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ABSTRACTS

CHEMICAL EDUCATION (ED)

ED-01 Initiation in Research in Science Education for Chemistry Students. *Lectícia Tarquinio de Souza Parente* (Centro de Ciências do Estado do Rio de Janeiro).

Improvement in behaviour of students that receive a grant of FAPERJ in the first level of initiation in research are commented as the first results of an investigation. (CNPq, FAPERJ).

ED-02 Verification of Conceptual Levels of Elementary Concepts in Study of Matter in High School Pupils of Rio de Janeiro. *Lectícia Tarquinio de Souza Parente* (Centro de Ciências do Estado do Rio de Janeiro)

Making an usual form of ink used in the elementary class-rooms the teachers are conduced to observe the conceptions present in the mind of pupils about: phases, mixture, substance and colour. (CNPq, FAPERJ)

ED-03 The Integration of Science, Technology and Society: an Innovative Programme to Teach Chemistry at Agricultural Schools of the State of Santa Catarina (Brazil). *Reni Scaranto* and *André Valdir Zunino*. (Universidade Federal de Santa Catarina. Centro de Educação. Mestrado em Educação).

A diagnostic of agricultural schools (N = 7) has showed the application of collection type curriculum of BERNSTEIN (1971). Based on this a program was developed supported by the integration of Chemistry, Technology and Society and evaluated by Iluminative Methodology. A prelimary results have demonstrated that the chemistry teachers weakened the hierarchy with their students who have had plus participation on their own knowledge; a minor gap between theorical chemistry knowledge and its application or usefulness in the agricultural context and plus social implications with the consencioness of the rural realities.

ED-04 The Teaching of Chemistry in Habilitação Específica para o Magistério (Hem): Why a Proposal? *Maria Cristina de Zensi Zancul* (Rede Estadual de Ensino), *Maria Lúcia Ribeiro* and *Dilene Cristina Alarcon Martinez* (Instituto de Química-UNESP).

The present work has the purpose of presenting the justification for the development of a proposal of Chemistry contents for Habilitação Específica para o Magistério (HEM), taking into account that the present stage of Chemistry teaching presents imperfect and discussible points. The work has been developed based upon researches accomplished within public-net school teachers and students of the cities of Araraquara and Américo Brasiliense. From the analysis of the results it has been possible to draw a profile of HEM Chemistry teaching. The necessity of a appropriate and specific proposal for the future formation of teachers has been reinforced.

ED-05 A Sociological Analysis of Chemistry Teaching at a Public School of The State of Santa Catarina (Brasil) Based on Bernstein's Theories. *Albertino Della Giustina* and *André Valdir Zunino*. (Universidade Federal de Santa Catarina. Centro de Educação. Mestrado em Educação).

This research looks for a diagnostic about the transmission of chemistry knowledge at a public school of the State of Santa Catarina. It is supported by the ideas about the educational codes such as collection and classification types which give form to curriculum, pedagogy and assessment of chemistry teaching. Results from a Department of chemistry (N = 17), through an iluminative/etnographic evaluation, have showed that the pedagogic works of the teachers are greatly influenced by hierarchical social class of the students, that there is a gap in the acquisition of the knowledge unfavorably to workers class.

ED-06 The Teaching of Chemistry Based on Principles of Pedagogic Action. *Luiz Carlos Naujorks* (Universidade de Passo Fundo/RS) and *André Valdir Zunino* (Universidade Federal de Santa Catarina).

The present research project is based on Stenhouse's (1978) theories wich attempts to operationalize Principles of Pedagogic Action (PPADs). Secondary chemistry teachers (N = 18) have chosen 3 or 4 Principles, for example, "to give to the students the oportunity to design and execute their own experiments" and applied one year. The Iluminative Evaluation (Parlett and Hamilton, 1978) was used to accompany the research. Results have demonstrated that the teachers are plus able to reelaborate, to utilize and to apply the chemistry knowledge, as well as, to be less dogmatic and plus critical for their own work.

ED-09 A "Dialogic" Conception to Prepare Undergraduate Students to Teach Chemistry. *Maria Celina Aydos* (Departamento de Química da Universidade de Mato Grosso do Sul), *André Valdir Zunino* (Centro de Educação da Universidade Federal de Santa Catarina).

An educational experience called "dialogical conception", based on Paulo Freire's ideas and applied at the university level with chemistry students when preparing them to teaching at secondary level. The identification of "generators theme" from the students has given the possibility to choose their own significative contents. This has contributed for the formation of future chemistry teachers, when they have operationalized at secondary schools, because they were able to bear with the conflicts and contraditions of the contents and schoolar system. Conclusions were made that they were better prepared for their professional life.

ED-10 A Methodological Approach to the Betterment of Chemistry Teaching Trough Modular Programme to Adults Workers. *Sônia Maria Chaves Haracemiv.* (Universidade Federal do Paraná, Centro de Estudo Supletivo).

The following problems were identified, at the fase III, chemical education at distance (CES): emphans on contents without taking into account the previous knowledge of the students and assessment based mainly on memory. At the CES the teaching is modular, individual and the assessment is through a bank of questions. Innovations such as team work (debates, discussions in small groups, experimentation and workshop), which enfocus three themas: chemistry at lunch time, home economy and chemistry and art. Through etnographic research the following results were gathered: the development of manipulative skills' students interests of chemistry contents, student's active participation on their own knowledge and the application of chemistry in day-to-day life. This essay has evidenced that the students have become more critical and consciouness of their reality, especially in their work environment, as well as, the necessity to study deeply theories of Paulo Freire and Bertein which could be applied in this essay.

ED-11 Using "Projeto de Rede" to Show How a Brazilian University Can Help in Enhancing Chemistry Teaching at Secondary Level, *Arnaldo R. Carvalho*, (Dept^{o} de Química Fundamental) and *M. Oscarina Godoy*, (Dept^{o} Energia Nuclear, UFPE, Brazil).

In order to enhance chemistry teaching at public schools, a training course was devised for secondary level teachers and students. Classes were improved with introduction of conceptual discussions, demonstrations and building of experimental set ups. Project evaluation stressed the importance of university – school collaboration, in up date chemistry teaching knowledge and in assist the science clubs at secondary schools.

ED-12 Formation in Chemistry: a Controversial Question. *Maria Alves de Toledo Bruns* (Department of Psychology and Education - University of São Paulo - Ribeirão Preto, SP) and *Maria Ines Fini* (Faculty of Education, Campinas State University - Campinas, SP).

The low productivity of the chemistry course of the F.F.C.L. of the University of São Paulo, evidenced by a decreasing number of graduates since its formation in 1961, is investigated in an attempt to determine why enrollment is always decreasing and the number of drop-outs is always increasing. An important factor is inherent to the chemistry profession in Brazil, that is lack of interaction of the university with either high school or industry resulting in distorted perspectives of the chemical profession and employment opportunities. The increasing number of drop-outs is also caused by factors inherent in the chemistry course at the F.F.C.L.

ED-13 Who Our Students Are and What They Think. *Lenalda Dias dos Santos, Djalma Andrade Heliane Freire Cabral* (Departamento de Química da Universidade Federal de Sergipe) and *Daisy Fonseca Ximenes* (Escola Técnica Federal de Sergipe).

As a follow up of our project "The Search for Professional Efficiency", the present work gives a break down of "Who our students are and what they think". The research method was a questionnaire given to students enrolled on the final three semesters of the course. The evaluation of the data collected by this research will permit us to take positive actions towards our goal which is to turn out professional teachers who understand and be more sensitive to our educational problems.

ED-14 The Utilization of Low Cost Materials for Teaching Chemistry. *Geronimo Wisnewski* (Secretaria da Educação do Paraná), *André Valdir Zunino* (Universidade Federal de Santa Catarina, Centro de Educação).

The lack of didatic materials, mainly for practical work, can be substituted by low cost locally available (LCla). Some critics have been said about the vulgarization of chemistry and non quantative aspects. Experiments have been developed with LCLA materials, not isolated in themselves, but conjugated with local or regional resources and Principles of Pedagogic Action (PPADs). Using the Iluminative Evaluation, the results have showed that the teaching of chemistry at secondary level became less dogmatic and the pupils had the opportunity to participate on their own knowledge, as well as, to reelaborate the chemistry contents.

ED-15 The Use of a Microcomputer as Chemistry Tutor, Part V: Principles of Quantum Chemistry – Wave Functions of Hydrogen Atom. *Flávio Romero Alves de Souza, Newton César Viana Costa, José Regis Botelho* e *Wolfango Eloy Sanchez Lemus*. (Chemistry Department, Universidade Federal da Paraíba).

This communicaton is about a interactive software for study hidrogen wave funtions. The program have five independent parts and the interface with the user has the access by menu. The program, written in

Applesoft BASIC, has being executed on any microcomputer Apple II Plus compatible. This software should be useful to acquaint students with the role of computer graphics in explaining the wave functions and orbitals shape.

ED-16 Lasers in Chemical Dinamics, a Summer Course for Undergraduates, *Isaac M. Xavier Jr.*, (Departamento de Química Fundamental, UFPE).

Chemical dynamics is one the most active field in chemistry nowadays. With the advent of lasers, major progress has been achieved in experimental chemical dynamics. A summer course on laser applications towards mode seletive chemistry was offered to engineering and science undergraduate students at UFPE, justo to share the excitement of doing research on this challenging field.

ED-17 Application of Multiple Regression Method to the Water/Phenol Phase Diagram: an Experiment for Undegraduate Physical-Chemistry Laboratory. *Edvaldo Sabadini, Marcelo Ganzarolli de Oliveira, Pedro Antonio Muniz Vazquez*, (Deptº Físico-Química, Instituto de Química, UNICAMP, C. Postal 6154, 13081, Campinas, SP).

To ilustrate the calculation procedure used to fit the best polynomium to experimental data we developed an experiment based on the partial water/phenol miscibility. The miscibility temperatures are determined by the students for several water/phenol compositions and the resulting diagram is plotted. Students are requested to hand calculate, by matrix methods, the 2nd degree regression polynomium which fits the experimental data and analyse the goodness of fit. Computer made calculations are carried out for 3rd and, 4th degree polynomials. Results shows a better fitting for the 4th degree polynomium.

ED-18 Gibbs-Rozeboom Diagrams. *N. M. Rodrigo Leygue-Alba, Vania Slaviero, Cláudio A. Perottoni* (Dept. de Física e Química, Universidade de Caxias do Sul - RS).

Suggested application of three-dimensional graphics by Gibbs-Rozeboom's charts to represent three fractions of components and density of a liquid system. For example was studied six systems with benzene, methyl ethyl ketone (MEK) and cyclohexane as the organic solvents, ethanol and acetone as consolutes and water as basic solvent.

PHYSICAL-CHEMISTRY (FQ)

FQ-001 Determination of the Activation Energy for the Transport of Water in Polyethylene Films, Employing Different Drying Agents. *Maria do Socorro Medeiros Cabral, Djalma Ribeiro da Silva* and *Francisco Gurgel de Azevedo* (Grupo de Polímeros - Departamento de Química e Cursode Mestrado em Engenharia Química da UFRN).

Our objective in this work is the understanding of the morphological structure of low-density polyethylene films. We have been studying the adsorption and water transfer properties through thermal drying and humid Payne cup technique. We also study, the influence of the drying agents and temperature in the water transfer in low-density polyethylene films. With the flux values we establish the activation energy from Arrhenius representation and propose a possible mechanism to the water transport in these films.

FQ-002 Equilibria of Positively Charged Indicators in Water-In-Oil Microemulsions (W/O uEs). *Ana M. Chinelatto, Maria T. M. Fonseca, Nilo Z. Kiyan,* and *Omar A. El Seoud* (Instituto de Química, USP, C.P. 20.780, 01498 São Paulo, S.P., Brazil).

Acid-base equilibria of two positively-charged phenol derivatives was studied spectrophotometrically in the presence of W/O uEs of the surfactants benzylcetyldimethylammonium chloride (cationic) in benzene and Brij-30 (non-ionic) in heptane. Buffer independent micellar pKa values could be obtained, provided that the starting pH of the buffer solutions were corrected for ion exchange with the counter-ion of the cationic surfactant, and for the effect of the lower polarity of the micelle-solubilized water. The electrostatic potential of the W/O interface, at the average solubilization site of the indicators (i.e., at the center of the water "pool") was calculated to be between 28 to 67 mV (CNPq, FAPESP, FINEP).

FQ-003 Poly (Dimethyl-Co-Diphenyl) Silane - Synthesis and Properties. *Patrícia P. C. Sartoratto* and *Inez Valéria P. Yoshida* (Institute of Chemistry - UNICAMP - P.O. BOX 6154 - Campinas - SP).

Copolymers of polysilanes having $SiPh_2$ and $SiMe_2$ units were synthetised by a Wurtz type reaction with different molar ratios of the appropriated dichlorodiorganosilanes. Soluble and insoluble materials were obtained and characterized by IR and ^1H NMR spectroscopy. The thermogravimetric analysis showed that the copolymers having larger $SiPh_2/SiMe_2$ ratios were thermically more stable. All soluble copolymers were amorphous solids as observed by powder X-Ray diffractometry and displayed a strong absorption in the U.V. region, corresponding to a $\sigma \rightarrow \sigma^*$ transition, and λ_{max} moved to longer wavelenghts as the $SiPh_2/SiMe_2$ ratio increased.

FQ-004 Interfacing a Tensile Testing Machine With an IBM PC/XT Compatible. *Valmir Fascio Juliano* and *Marco-A. De Paoli* (Instituto de Química, UNICAMP) and *Claudimir L. do Lago* (Instituto de Química, USP).

We developed an interface for conecting a Tensile Testing Machine to an IBM PC/XT compatible microcomputer. It reads the crosshead shift and the tensile strength. The interface also controls the temperature of an oven used to heat the samples. By using the appropriate software and furnishing the cross-section dimensions of the specimen it is possible to obtain the stress/strain curve and to calculate: elongation at yield, yield stress, elongation at break, stress at failure, modulus and the area under the curve. These values can be calculated for each specimen or as an average of a series of measurements.

FQ-005 Stabilization of Poly(Vinylchloride) With Organometallic Polymers. *Roni F. F. Mori* (Departamento de Química, UFSM) and *Marco-A. De Paoli* (Instituto de Química, UNICAMP).

Nickel organometallic polymers have been prepared by the reaction of bis (cyclo-octadiene) Nickel (0) with poly(vinylchloride), PVC, in the presence of the following ligands: 2,2'-bipyridine, 4,4'-bipyridine, triphenylphosphine, pyridine and chinoline. These were characterized by elemental analysis, infrared and ^1H-NMR, as well as vyscosimetric measurements. By comparing the photo and thermo-oxidation of these polymers with pure PVC we conclude that the nickel organometallic anchored to the polymer chain has a strong stabilizing effect.

FQ-006 Transition-Metal Complexation in Polymer Blends. *Alfredo T. Nunes Pires* (Federal University of Santa Catarina) and *Laurence A. Belfiore* (Departament of Chemical Engineering, Colorado State University, CO, USA.)

The transition-metal-containing model compounds are zinc salts of acetic acid, lauric acid, stearic acid and 2,4-pentanedionate. Binary mixtures of these small molecule with poly(vinyl pyridine) were prepared. Thermal analysis via Differential Scanning Calorimetry was used to probe the phase behavior of these blends at the macroscopic level. High-resolution carbon-13 solid state NMR was used to probe intermolecular complexation at the sitespecific level. Strong interactions are operative between the zinc cation and the imide group in this model system. The carbon-13 NMR spectral perturbations are only observed when the ionic copolymer is neutralized with zinc, and when the pyridine nitrogen is in the four position with all small molecule studied. However, only zinc acetate and 2,4-pentanedionate form complex with poly(2-vinyl pyridine).

FQ-007 Graft Copolymerization of Chitosan and Polyacrylonitrile Using Ceric Ammonium as a Redox Initiator. *Elisa H. S. Moecke* and *Mauro C. M. Laranjeira* (Departamento de Ciência e Tecnologia de Alimentos e de Química, UFSC).

The polymer chitosan that consists mainly of β-(1,4)-2-amino-2-desoxy-D-glucose units, is prepared from exoskeleton of shrimp *(Xyphopénaeus kroyeri)* by basic hydrolysis from chitin. Polyacrylonitrile was grafted onto chitosan using ceric ammonium nitrate (CAN) as redox initiator in an homogeneous aqueous medium. The percentages of grafting and efficiency are dependend on concentrations of CAN and monomer, and reaction time. The evidence of grafting was confirmed by infra red spectroscopy, differential scanning colorimetry (DSC) and microanalysis.

FQ-008 Kinetic Study of Extraction of Proteins Combinated With Chitin from Shrimp Shells. *Regina Célia Zimmermann* and *Mauro César Marghetti Laranjeira* (Departamento de Química - UFSC - Florianópolis - SC - 88.049).

The kinetic study of extraction of proteins combinaded with chitin in the matrix of the shrimp were made by basic hydrolysis using different concentration of alkali. The absorbance values of proteins were determinated by the biuret reaction after withdraw the samples from the mixture at different time intervals. When absorbance values are plotted against time, a curve is obtained which represents two consecutives reactions. The more rapid represents the reaction in the interface and the second reaction in the shell intersticis. The kinetics under pseudo-order condictions showed linear dependence on NaOH concentration.

FQ-009 Soluble Polyaniline. *Walter Mendes de Azevêdo* (Departamento de Química Fundamental, UFPE).

The reaction of aniline with $KMnO_4$ or MnO_2in well defined acid medium yields electroconductive polyaniline which is soluble in common polar organic solvents, especially in dimethylformamid and dimethylsulfoxide. The solution electronic spectrum of samples prepared by chemical reaction between aniline salt and $KMnO_4$ in aqueous medium, exhibits two strong band, one at 320nm and 370nm and two weak band at 440nm and 580nm; no band is observed at region of 800nm. The electrical conductivity of the examples prepared up to now is $0.31 Scm^{-1}$.

FQ-010 Water-Ethanol Pressure-Driven Pervaporation *D. Windmoller (UNICAMP-SP/UNIJUI-RS) and F. Galembeck* (UNICAMP/SP).

Pervaporation experiments were performed, keeping the feed current at pressures in the 1-15 atm range, above and below normal boiling temperatures. Flow rates increase up to 40-fold, as the temperature is raised in this range, but they have a small dependence on feed pressure. Pervaporation of 10% (w/w) aqueous ethanol feed at 2.5 atm through a 100 μm silicone membrane yields 9.22 x 10^{-6} ml/cm^2.sec of 40-50% ethanol at 100°C.

FQ-011 Study of $|Fe(OH)_3|_n$ Cristalization in Solid Medium by the Use of Diffuse Reflectance Spectroscopy. *Ana Maria Rocco* and *Inés Joekes* (Physical-Chemistry Department - UNICAMP).

This work aimed to evaluate the Diffuse Reflectance Spectroscopy (DRS) technique as a tool for the study of crystallization of amorphous Fe(III) hidroxoacetate. DRS and infrared spectroscopy were obtained for samples comprising amorphous material and material previously heated from 40 to 780°C. KBr pellets containing amorphous Fe (III) hidroxoacetate were heated to 150°C and 450°C and DRS and infrared spectre were obtained. These were different for all steps. The technique is sensible to changes in the shape and size of the particles.

FQ-012 An All-Glass Vacuum Line for Static Measurement of Specific Surface Area by the Bet Method. *Celso Ulysses Davanzo* and *Marcia C. Branciforti* (Instituto de Química - Universidade Estadual de Campinas - Caixa Postal 6154 - Campinas - S.P. - Brazil).

An all-glass apparatus was constructed for static measurement of specific surface area using absorption of nitrogen or argon at liquid nitrogen temperature and applying the BET method.

FQ-013 Thermal Isomerization of Dimethyl Yellow: Solvent and Polymer Matrix Effects. *Marcelo Ganzarolli de Oliveira* and *Teresa Dib Zambon Atvars* (Departamento de Físico-Química, Instituto de Química, UNICAMP).

A spectroscopic study of dimethyl yellow (DY) thermal isomerization was carried out by monitoring relaxation to equilibrium after trans-cis photoisomerization of the dye in several solvents and in polymer matrices. Results show that in low viscosity solvents relaxation follow a simple first order kinetic, while in polymer matrices the process is described by at least two rate constants suggesting two different enviroments. Relaxation rate increase sharply for polar and hydrogen-bonding solvents and matrices and is related to an stabilization of DY exited states in this cases. Kinetic studies show that DY can be used to probe physical aging in low-polar amorphous polymers.

FQ-014 Study of the Interaction Between Trypsin and Cetylpyridinium Chloride (CPC). *Eloi Alves S. Filho* and *Pedro L. O. Volpe* (Institute of Chemistry, UNICAMP, P.O. Box 6154, Campinas, SP, Brazil).

The factors which are responsible for the stabilization of the secondary and tertiary structure of bilogical macromolecules can be to some degree understood from studies of interactions which lead to the breakdown of native structure. Ionic surfactants are unique in this context in that they denature proteins at concentrations of the order of millimolar in marked contrast to denaturant such as urea which function only at 6-8 M. Equilibrium dialysis technique was used to study the interaction between trypsin and a cationic surfactant (CPC) below the CMC in a phosphate buffer (pH = 6.8). Kapp, the apparent binding constant of protein-surfactant complex was calculated using the Wyman binding potential concept (Wyman, J.J., Mol. Biol. *11* 631-644, 1965). (CNPq - FAP).

FQ-015 The Interaction Between Benzyltrimethylammonium Bromide and *Saccharomyces Cerevisiae*. *Carmem S. R. Bazzani, Tereza D. Z. Atvars* and *Pedro L. O. Volpe* (Institute of Chemistry - UNICAMP - P.O. BOX 6154 - Campinas - SP - BRAZIL).

A study has been made of the interaction between Benzyltrimethylammonium Bromide and Saccharomyces cerevisiae in isotonic medium. The binding data were obtained at 25 and 33°C using a suspension of Saccharomyces cerevisiae cells (10^7 cells/ml) the absorbance measurements were made at 261 nm after the cells centrifugation. The binding isotherms in both temperatures shows that in 40 minutes the equilibrium is achieved. (CNPq - FAP - HERGA).

FQ-016 Monitoring Heat Effects of the Glucose Fermentation by Flow Microcalorimetry. *Carmem S. R. Bazzani, Márcio A. Almeida, Pedro L. O. Volpe* (Institute of Chemistry - UNICAMP - P.O. BOX 6154 - Campinas - SP) and *Silvia Egushi* (Fundação Tropical de Pesquisas e Tecnologia - P.O. BOX 1889, Campinas).

The various metabolic events which occur within cells are all reactions which produce heat. Thus, by monitoring the heat effects with sufficiently sensitive calorimeters, we can study fermentation processes. An LKB 2277 Bioactivity Monitor was used to determine the thermograms curves for the fermentation in differents glucose concentrations. The medium was defined in terms of pH. The growing kinetic, exhaustion time, production of energy in function of time were obtained. (CNPq - FAP).

FQ-017 Immunoglobulin Fractionation by Zonal Centrifugation in Density Gradients, Osmocentrifugation and Centrifugue Ultrafiltration. *Gilza Maria Piedade Prazeres, Ilna Gomes da Silva, Maria Célia Pires Costa* (Depto. de Química, Universidade Federal do Maranhão) and *Fernando Galembeck* (Instituto de Química, Universidade Estadual de Campinas).

Zonal centrifugation in density gradients has been widely used in biological particle preparative and analytical fractionation. Osmocentrifugation and Centrifuge Ultrafiltration are new membrane-assisted concentration and fractionation techniques when macromolecular solutions are contained within dialysis cells. We have used these techniques in immunoglobulins concentration and fractionation of hiperimune human serum. After centrifuging for the desired times, the fractions obtained are analysed by gel filtration.

FQ-018 Dissipative Structures in Dialysis Cell: Looking for an equilibrium point. *Sirlane Aparecida Abreu Santana, Mariano Oscar Aníbal Ibañe Rojas, Antonio Benedito da Silva* and *Maria Célia Pires Costa* (Depto. de Química, Universidade Federal do Maranhão).

In experiments of zonal osmocentrifugation in density gradients (Ficoll-Sacarose) of concentrated eythrocruorin and human hemoglobin, many bands, are formed throughout the dialysis cells, producing a temporal and spatial organization. A model for the formation of these structures is proposed, based on formation of concentration gradients in the sedimenting zone, followed by convection. Recent experiments that objectify the equilibrium point of these structures show that as time goes by the bands will be widened and finaly organize a continous gradient of concentration.

FQ-023 Catalysis by Ion-Exchange Resins. V. the Activation Energy of Esterification and Self-Etherification Reactions. *Flávio B. dos Santos, Maria Edwiges da S. Barreira* (Institute of Chemistry/UFRJ) and *David Tabak* (Institute of Chemistry/UFRJ and Bayer of Brazil S.A.).

The tentative reaction between benzoic acid and benzyl alcohol catalyzed by the resin Lewatit SP-112 in the acidic form was carried out in the presence of toluene as the solvent. Even using equimolecular amounts or excess of any of the reagents, the products were benzyl ether and those from the alkylation of the solvent by the alcohol instead of the ester. In order to study the effect of substituting the phenyl group of the alcohol by an alkyl one (n-propyl), the reaction of benzoic acid with n-butanol in toluene was carried out. Th only product, except for water, was n-butyl benzoate. The activation energy of the process determined by the Arrhenius equation was 19 Kcal.mol^{-1}. The self-etherification of benzyl alcohol in the presence of cyclohexane as the solvent has been studied, with an activation energy of 22 Kcal.mol^{-1} being determined for the process.

FQ-024 Precipitation of Ferrous Sulfide. *Ana Maria Travalloni Louvisse, Sandra M. Saraiva, Gaspar González,* (PETROBRÁS Research Center, Ilha do Fundão, Quadra 7, CEP 21910, Rio de Janeiro, Brazil).

The precipitation of FeS produce a colloidal suspension containing negatively charged particles of less than 1μm in diameter. Electrolytes and surfactants increase the particle size and cationic surfactants produce positively charged particles.

FQ-025 Sentido de Torção em Liomesofases Colestéricas Induzidas por Carboidratos. *T. M. H. do Aido, M. R. Alcântara, O. Felippe Jr., A. M. G. Pereira* and *J. A. Vanin* (Inst. Química - Univ. de São Paulo).

The handedness for induced cholesteric lyotropic liquid crystals was determined by polarizing microscopy techniques. Carbohydrates were added to lyomesophases based on the amphiphiles potassium laurate, sodium decylsulfate and decylammonium chloride. The resulting cholesteric phases correspond to Ch_D systems. The analysis of the helical twist sense, its correlation with the chiral solute structure and its chemical properties can play important role to clarify the cholesterization process in lyotropic systems. (CNPq, FAPESP, FINEP).

FQ-026 Solubilization of Ionic Solutes in Micellar Solutions. *Angela Ramalho Diniz* and *Fred Yukio Fujiwara* (Instituto de Química, Universidade Estadual de Campinas).

In order to study the behavior of benezenesulfonate, toluenesulfonate, benzoate and anilinium ions in micellar solutions, the order parameter were determined in nematic mesophases and the C-13 NMR relaxation times were measures in isotropic micellar solutions in order to characterize the rotational diffusion anisotropy. The results indicate that these ionic solutes, due to their hydrophobic nature, tend to be incorporated into micelles with the same electrical charge. The interaction of these solutes with micelles of the opposite charge depends on the nature of the solute and of the surfactant.

FQ-027 Estudo de Liomesofases Colestéricas à Base de Decilsulfato de Sódio Induzidas por Anfifílicos Derivados de Amino-Ácidos. *A. M. G. Pereira, T. M. H. do Aido, M. R. Alcântara, O. Felippe Jr.* and *J. A. Vanin* (Inst. Química - Univ. de São Paulo).

Nematic lyomesophases of sodium decylsulfate were converted to a choloesteric phase by doping with N-dodecanoyl amino acid surfactantants. The resulting systems were studied by polarizing microscopy technics and refractometry. The cholesteric phases correspond to Ch_D systems. The pitches, helical twist senses and birrefringences were determined. The data suggested that the structure of the amino acid part of the surfactant, the ionic strength and pH play a significant role on the formation of lyomesofases. (CNPq, FAPESP, FINEP).

FQ-028 Aplicação de Dicroismo Linear ao Estudo de Ordenamento de Liomesofases Nemáticas. *O. Felippe Jr., T. M. H. do Aido, M. R. Alcântara, A. M. G. Pereira* & *J. A. Vanin* (Instituto de Química da Universidade de São Paulo).

The present work reports the use of linear dichroism techniques in order to determine the location of solutes molecules in liquid crystal micelles. It was prepared phases based on sodium decilsulfate (SDS), sodium sulfate and water using bromothymol blue as a solute. Absorbance measurements for lyomesophases previously oriented in a 14 kG magnetic field was obtained in an usual spectrophotometer. The resulting order parameters show that bromothymol blue molecule is located into the amphiphile bilayer instead of the micellar surface.

FQ-029 Synthesis and Characterization of Liquid Crystals Containing the Heterocyclic Rings Furane and Thiophene. *Delcio Hartmann* and *Hugo Gallardo* (Department of Physics – UFSC).

Mesomorphic systems containing structural units of 2,5–substituted thiophene and furane were studied. The 2–substituents were: $4–NO_2–C_6H_4$ or $4–EtO–C_6H_4$ and the 5–substituents: $4–C_nH_{2n+1}O–C_6H_4–N{=}CH–$. The homologous serie Ia shows nematic properties and as a function of aliphatic chain length the temperature interval of existence of the nematic phases varies from 24°C to 50°C, For n = 9 the phase sequence Sc–N–I is observed. Serie Ib is similar, but the interval of the nematic phase is less varying from 16°C to 38°C. Transition heats of all phase transition were measured using a DSC. Compounds containing the furane ring do not show mesomorphic properties.

FQ-030 Mesomorphic Properties of Phenyl-Thiophene Derivates. *Ida Eunice Favarin, Veroni Valenti* and *Hugo Gallardo* (Departamento de Física, UFSC).

We have synthesized the homologus serie 2–octanoil–5–(4–alcoxiphenyl)thiophene (alcoxi = $–OC_nH_{2n+1}$). The textures of the phases were observed in the polarizing microscope and the transition heats and temperatures measured using a DSC. Compounds with n = 1 to 12 were synthesized. For n = 2 to 10 only a smectic A phase was observed. For n = 12, two smectic phases S_I and S_{II} observed. The thermodynamic data are discussed and compared with the analogous biphenyl serie.

FQ-031 Phase Separation of Low Molecular Weight PEG/PPG Blends Under Shearing. *E. C. Muniz* (Univ. Est. Maringá), *S. P. Nunes* (UNICAMP, Institute of Chemistry, C. P. 6154, 13081, Campinas, Brazil), *B. A. Wolf* (Universitat Mainz).

UCST phase diagrams were obtained for low molecular weight PEG/PPG blends by turbidimetric and viscometric methods. Phase separation is affected by shearing. For instance, the demixing temperature of 50% PEG and 50% PPG 1000 blends increase under shearing and has a maximum value at ca. 500 s^{-1}. Curves of viscosity as a function of composition showed a non-linear behaviour, specially at a low temperature and a high shearing.

FQ-032 On the Influence of the Presence of Ir (III) in a Solution of H_2IrCl_6, When Ir/Al_2O_3 Catalysts Were Prepared by Impregnation. *Carlos Oscar Brandão Falcão* (IME), *David dos Santos Cunha* (INPE) and *Gilberto Marques da Cruz* (Depto. de Química da PUC/RJ).

Ir/Al_2O_3 catalysts, with 4% in weight in metal, were prepared by impregnation with solutions of H_2IrCl_6. The catalysts contained either a mixture of Ir (III) and Ir (IV) or Ir (IV) only. The initial presence of Ir (III) undermined the complete dechlorination, even under H_2 at 1073°K. It led to an increase of metalic particle sizes, when the reduction was carried out at moderate temperatures. The catalysts prepared with the Ir (III) and Ir(IV) mixture have shown turnover numbers three times greater than those with Ir (III) for benzene hydrogenation. This result was attributed to a greater hydrogen interstitial adsorption.

FQ-033 Study of Solubilization in Micelles Through the Measurement of Diffusion Coefficients. *Cláudio A. Tonegutti, Watson Loh* and *Pedro L. O. Volpe* (Institute of Chemistry - UNICAMP - P. O. BOX 6154 - Campinas, SP - Brazil).

This communication reports measurements of diffusion coefficients for the homologous series of m-alkoxyphenols. These coefficients were obtained using a hollow Teflow tubing (8 m length and 0.7 mm i.d.) connected to the HPLC equipment Varian 8500 (with UV detector) by the Taylor's dispersion technique. Diffusion coefficients, both in water and CTAB 2mM solutions, decreased with the increase of the side chain. The decrease of the solute diffusion coefficient with the micellar solubilization allowed the determination of the partition coefficients. The linear decrease of the Gibbs function of transfer from water to micelles observed along the homologous series confirmed the consistence of the method. (CNPq - FAP).

FQ-034 Fluorokinetic Reagents: a Study of the Reaction Between Bichromophoric Substrates, Derived from Haloacetamides, and N-Heptanthiol in Micellar Solution. *Pedro Berci Filho* (Instituto de Física e Química de São Carlos, IFQSC/USP); *F. H. Quina* (Instituto de Química-USP/São Paulo) and *Miguel Guilhermo Neumann* (Instituto de Química e Física de São Carlos, IFQSC/USP).

The reaction of haloacetamide, containing bichromophores, with n-heptanthiol was taked as a model to study the effects of cationic micelles on the nucleophilic substitution reactions. The fluorescence reborn emission allowed to follow the reaction course and to calculate the $\underline{k}$ values, in bulk and micellar media. The data analysis shown that the rate constants increase were owed only to the increase of the local concentraction of the substrates in micellar phase.

FQ-035 Electrochemical Reduction Study of 2-Chloro, 4-Dimethylaminebenzylidenepyruvic acid. *João Olímpio Tognolli, Maria Valnice Boldrin Zanoni* and *Cristo Bladimiros Melios* (Inst. de Química, Depto. de Química Analítica, UNESP, Araraquara, SP).

The electrochemical reduction of 2-cloro, 4-dimethylaminebenzylidenepyruvic acid was investigated in buffered aqueous solution over the pH range 2.0 - 12.0 using D. C. and square-wave polarography and cyclic voltammetry. The redox behavior of the system is dependent of pH. In neutral medium the process occurs in a sole two-electron step and complete reduction of the olefinic unsaturation. Different protonations are responsible for the two successive reduction step observed in acid medium. A further reduction wave was observed at pH > 10.

FQ-036 Polarographic Reduction of Phenyl-p-Nitrobenzenesulphonate. *Luciana Vanni Gatti* and *Nelson Ramos Stradiotto* (Departamento de Química da FFCLRP - USP - Ribeirão Preto, SP).

The polarographic reduction of phenyl-p-nitrobenzenesulphonate (FNBS) in dimethylformamide was studied using the tecnique of alternating (AC) and direct current (DC) polarography. The FNBS shows three reduction steps. The first step is a transfer of one electron, the second and third reduction steps are processes involving an one electron transfer coupled to a chemical reaction.

FQ-037 Voltammetric Reduction of Phenyl 3-Nitrobenzoate in N, N-Dimethylformamide. *Sônia Maria Alves Jorge* and *Nelson Ramos Stradiotto* (Faculdade de Filosofia, Ciências e Letras de Ribeirão Preto - Universidade de São Paulo - Departamento de Química - Av. Bandeirantes, 3900 - 14049 - Ribeirão Preto, SP, Brasil).

The electrochemical behaviour of the phenyl 3-nitrobenzoate in 0.1 M Bu_4NBF_4/DMF was investigated by cyclic voltammetry at a vitreous carbon disc electrode. The results show that the ester is reduced in two cathodic steps. The first one at - 0,97 V vs SCE is an EC mechanism. The second reduction step at 1,91 V vs SCE seems to be an EC mechanism, in which the dianion produced undergoes a chemical reaction.

FQ-038 Electrochemical Characteristics of Complexes Type $RuCl_2(CO)_2(L)_2$, Were L = N-Heterocyclic. *Lis Regina V. Olmo; Sandra Aparecida Onofre* and *Alzir Azevedo Batista* (DQ/UFSCar).

We have investigated the electrochemical behavior of the isomers (tcc), (cct) and (ttt) $[RuCl_2(CO)_2(M - Im)_2]$, where M-Im=1-methylimidazole. The Ru(II)/Ru(III) potentials of the half waves for these compounds are 1,75;1,45 and 0,85V vs $Ag/AgI/CH_2Cl_2$/0,1 M-Bu_4NClO_4/25°C reference electrode for (tcc), (cct) and (ttt) isomers, respectively, at which ferrocene is oxidized at 0.60V. In the same conditions the (cct) isomer of the complex where L = pyrrolidine present their $E_{1/2}$ = 0,80V. The complex with pyridine is electrochemically irreversible and shows the oxidation potential at 2,1V.

FQ-039 Potentiometric Studies of the Corrosion Inhibition of Unsaturated Alcohols on Copper in H_2SO_4 Aqueous Solution. *Claudio Regis da Luz Silva* and *Reinaldo Simões Gonçalves* (Departamento de Química da UFSM - CEP 97119 - Santa Maria - RS).

The protection efficiency of three unsaturated alcohols was determined by potentiometry in 0,1N H_2SO_4 aqueous solutions at 25°C and 65°C for copper electrode. The corrosion processes related with the copper electrodes in these conditions were well blocked in the presence of Allyl Alcohol at 25°C and in the presence of Propargyl Alcohol at 65°C. The protection efficiency depends on the alcohol concentration.

FQ-040 Potentiometric Studies of the Corrosion Inhibition of Unsaturated Alcohols on Low-Carbon Steel in H_2SO_4 Aqueous Solution. *Zilda Manfio Baratto; Izabel Cristina Riegel* and *Reinaldo Simões Goncalves* (Departamento de Química da Universidade Federal de Santa Maria, CEP 97119 - Santa Maria - RS).

The protection efficiency of three unsaturated alcohols was determined by potentiometry in 0,1N H_2SO_4 aqueous solutions at 25°C for low-carbon steel. The corrosion processes related with the low-carbon steel in this medium were well blocked in the presence of propargyl alcohol. 1 - propanol and allyl alcohol were not as efficient as the other one. The protection efficiency depends on alcohol concentration.

FQ-041 Construction and Performance of a Cyclic Voltammetry Instrument. *José Luiz Bruçó, Carlos Alberto Pelá, Antonio Carlos Dias Ângelo* and *Nelson Ramos Stradiotto* (Faculdade de Filosofia, Ciências e Letras de Ribeirão Preto - Universidade de São Paulo - Av. Bandeirantes, 3900 - 14049 - Ribeirão Preto, SP, Brasil).

The cyclic voltammetry is perhaps the most versatile electroanalytical technique for the study of the electrode reactions. This work describes the construction of a equipment for cyclic voltammetric studies. The tests realized using a known electrochemical system show that the equipment presents a good performance in respect of commercial instruments.

FQ-042 The Effect of the Addition of 2,2 - Bipyridine (BIPY) on the Deposition of Lead on Hg, Ag/Hg and Ag Electrodes. *C. A. Moritz* and *J. C. Rubim* (Instituto de Química da USP, C.P. 20780, 01498, São Paulo).

The effect of the addition of bipy on the electrodeposition of lead has been investigated by chronopotentiometry and cyclic voltammetry. For the Hg electrode the electrochemical processes are reversible in the absence and presence of bipy, while for the Ag/Hg electrode the reversibility is observed only in the presence of bipy. The behaviour of the Ag electrode is quite different due to the under deposition (UPD) of lead. The bipy shifts and broadens the oxidation-reduction waves of UPD.

FQ-043 Measurement of the Self-Diffusion Coefficient of Na^+ in Aqueous Solution of Sodium Chloride - Open-Ended Capillary Method. *José Schifino* and *Raquel Maria Olegário* (Departamento de Física-Química - UFRGS).

The capillary method was used to measure the self-diffusion coefficient of Na^+ in aqueos solution of NaCl 0.1 M using ^{22}Na. The capillary containing the $^{22}Na^+$ was placed in contact with a large amount of NaCl 0.1 M solution and the decreasing activity was continuously monitored as a function of time. An automatic data acquisition system was employed and the calculation of the self-diffusion coefficient was performed by an iterative procedure. A diffusion coefficient of 1.272 x 10^{-5} cm^2/s was obtained at 25°C which compares well with those reported in the literature.

FQ-044 Electrochemical Behavior of Jatropholone in N,N-Dimethylformamide. *Marília O. F. Goulart, Antônio Euzébio Goulart Sant'Ana* (Deptº de Química/CCEN/Universidade Federal de Alagoas, Maceió, Al), *Josealdo Tonholo* and *Julien F. Boodts* (Deptº Química/FFCLRP/Universidade de São Paulo, Av. Bandeirantes, 3900, 14049 - Ribeirão Preto, SP, Brazil).

The electrochemical behavior of jatrofolone a natural product isolated from *Jatropha elliptica* was studied in DMF (TBAP 0.100 M) by d.c. polarography and controlled potential coulometry techniques. The coulometric technique showed the transference of two electrons. The ill-defined single wave, close to the supporting electrolite discharge, was solved by the use of Gauss-Newton algorithm method. This wave was characterized as being from a quasi-reversible process or a reversible process with a coupled chemical reaction. A difusional limiting current was found.

FQ-045 The Corrosion Behavior of Amorphous and Crystalline Ni-P Alloys in NaCl Solutions at Different pH's. *F. J. B. Rabelo, A. M. M. M. Adam* e *E. R. Gonzalez* (Instituto de Física e Química de São Carlos/USP - C.P. 369 - 13560 - São Carlos, SP–Brazil).

Co-P alloys were prepared by an electrodeposition process under different conditions. Two of the alloys were selected for testing. The amorphous structure was confirmed by X-ray diffraction. Potentiodynamic anodic polarization experiments in 0.1N NaCl (pH 2.8, 3.5 and 6.0) were carried out for two different compositions. All the amorphous Co-P alloys examined were spontaneously passive in NaCl solutions, but anodic polarization at high potentials results in passivity breakdown. The passivation behavior was better observed at low pH values. (CNPq).

FQ-046 Anodic Behavior of Ni-P Alloys in HCl Solutions at Different Temperatures. *P. de Lima Neto, F. C. Nart, A. M. M. M. Adam* e *L. A. Avaca* (Instituto de Física e Química de São Carlos/USP - C.P. 369 - 13560 - São Carlos, SP - Brazil).

Ni-P alloys have been prepared by electrodeposition at different current densities. The amorphous and crystalline states were examined by Xray diffraction. The potentiodynamic behavior of two crystalline and two amorphous Ni-P alloys, with 18 and 23 weight percent of P, in 0.1N HCl at different temperatures showed: i) the anodic behavior is not affect by the P content, ii) corrosion is more accentuated at higher temperatures, and iii) the amorphous alloys are more resistent to corrosion processes. (CNPq).

FQ-047 Effect of the Purity of the Water on Corrosion Studies With ABNT 347 Stainless Steel in Sulfuric Acid Medium. *Hernani Aranha, Silvia Maria Leite Agostinho* (Departamento de Química Fundamental do Instituto de Química da USP) and *Idalina Vieira Aoki* (Departamento de Engenharia Química da Escola Politécnica da USP).

This work presents a study of the effect of the purity of the water on corrosion studies for the system ABNT 347 stainless steel in naturally aerated 2.0 M H_2SO_4 solutions at room temperature. The study comprises weight loss experiments in the presence and absence of the benzotriazole (BTAH) inhibitor, and potentiostatic anodic polarization curves. Water of three differents purity degrees, destilated, bidestilated and destilated-deionized, was employed. The weight loss experiments results show that the surface coverage degree (Θ) by BTAH depends on the nature of the water employed: Θ increases as the purity of the solvent decreases. These results were interpreted as a synergetic effect of metallic cations and organic molecules. Potentiostatic studies, on the other hand, show no dependence of the anodic behavior of the stainless steel on the purity of the water, in this medium without BTAH.

FQ-048 Ionic Exchange Studies of Zirconium (IV) Phosphosilicate by Specific Conductivity (k) of the Solutions. *Edegar Ozorio da Silva, Reinaldo Simões Gonçalves* (Departº de Quím. da Univ. Fed. de Santa Maria - CEP 97119 - Santa Maria, RS), and *Yoshitaka Gushikem* (Inst. de Quím. da UNICAMP - CEP 13081 - Campinas, SP).

The kinetic process of ionic exchange of zirconium (IV) phosphosilicate (HZPS) with Cu (II) was accompained by the specific conductivity variation (Δk) of the solutions with time (t). In this process, it was determined that the relation $\Delta k/\Delta t$, depends on the initial concentration of Cu (II) and that the number of active sites present in 1,0 g of (HZPS) were saturated when the copper concentration in solution was 10^{-3}M.

FQ-049 Development of the Indirect Iterative Method for the Hydrogen Evolution Reaction Mechanistic Study. *E. L. de Sá, A. A. Tanaka* and *E. R. González* (Instituto de Física e Química de São Carlos/USP - C. P. 369 - CEP 13560 - São Carlos, SP - Brazil).

This work presents a new method, the Indirect Iterative Method (I-IM), for the determination of surface coverage by adsorbed Hydrogen atoms in the HER. This method is based on pthe simple calculation of isotherm parameters with data from steady-state polarization curves. The IMN has been applied to the HER on iron electrodes in alkaline media and showed an excellent agreement between calculated and experimental data for a Volmer-Heyrovsky mechaninsm.

FQ-050 Construction of an Infrared Cell to Study Solid-Gas Interface. *Celso Ulysses Davanzo, Heloise O. Pastore, Hermes A. de Freitas* and *Arlindo Furquim* (glass blower) (Instituto de Química - Universidade Estadual de Campinas - C.P. 6154 - Campinas, SP - Brazil).

The cell is constructed borosilicate glass and has two bodies joined by a TS 65/40 plane joint. One side of the cell is used as a furnace and the other one to measure the i. r. spectra. The sample holder moves hanged in a kind of glass made rail.

FQ-052 Raman Spectroscopy of Pyrimidine Derivatives. Part 1. Sers of Barbituric Acid Adsorbed on a Silver Electrode. *L. F. C. de Oliveira, P. S. Santos* and *J. C. Rubim* (Instituto de Química da Universidade de São Paulo - C.P. 20780 - 01498 - São Paulo - Brazil).

The surface enhanced Raman scattering (SERS) of barbituric acid (BA) on a silver electrode at pH 8.4 is reported for the first time. The intense fluorescence in the visible region precludes the obtention of the ordinary Raman spectrum of BA, the available normal coordinate analysis being based on the ir data. The more substantially enhanced Raman featuares are those at 641 and 675 cm^{-1}, assigned to ring breathing and C = O, respectively. The voltammograms show that BA is not involved in oxidation-reduction processes in the 0.0 to -1.0V potential region.

FQ-053 Raman Spectroscopy of Pyrimidine Derivatives. Part 2. Raman Spectroscopy of Alloxan [2,4,5,6(1H,3H)-Pyrimidinetetrone]. *L. F. C. de Oliveira* and *P. S. Santos* (Instituto de Química da Universidade de São Paulo, C.P. 20780 - São Paulo - Brazil).

The present work reports for the first time the Raman spectrum of alloxan with laser excitation. The normal coordinate analysis available in the literature was based in the ir data only. Raman spectra were obtained for the solid and saturated aqueous solution using the 647.1 nm line of a Kr^+ laser. A tentative assignment was made by comparison with the ir data. The strongest lines are assigned to ring breathing (648 cm^{-1}) and C=O stretching (1749 cm^{-1}), both of A_1 symmetry.

FQ-054 Effect of the Agglomeration of Silver Colloidal Particles in the Surface Enhanced Raman Spectra (SERS) of Pyridine. *Paola Corio* and *Joel C. Rubim* (Instituto de Química da USP - CP. 20780 - 01498 - São Paulo - Brasil).

In this work, the behaviour of Raman and absortion spectra of pyridine in aqueous and silver colloidal solutions has been presented. The Raman spectra of colloidal solutions prepared in the presence of pyridine (50mM) do not present SERS effect. Upon the addition of Cl^- an enhancement of the Raman signals is observed. The effect of Cl^- is to cause an aggregation of the colloidal silver particles, which shifts the absortion spectra of the colloid to the visible.

FQ-055 Vibrational Spectra of Some Inorganic Polymers of Squarate and Croconate Ions. *L. F. C. de Oliveira, J. H. do Amaral and P. S. Santos* (Instituto de Química da Universidade de São Paulo, C.P. 20780 - 01498 - São Paulo - Brazil).

Polymeric complexes of squarate ion [$MC_4O_4 \cdot 2H_2O$ (M=Zn^{2+}, Fe^{2+}, Cu^{2+}, Co^{2+} and Ni^{2+})] and of croconate ion [$MC_5O_5 \cdot 3H_2O$ (M= Fe^{2+}, Cu^{2+}, Co^{2+} and Ni^{2+})] were prepared and characterized by chemical analysis and ir data. Here the Raman spectra of such compounds are reported for the first time. Such spectra strongly suggest that the high symmetries of the anions are conserved in the polymers. The unusual large intensities of some non-totally symmetric bands of the free ions, formerly assigned to Jahn-Teller distorsion, are surprisingly observed in the complexes as well.

FQ-056 Raman Spectra of Chloroquine Adsorbed on a Silver Electrode. *D. L. A. de Faria* and *P. S. Santos* (Instituto de Química da USP - C.P. 20780 - São Paulo, SP).

Chloroquine diphosphate (DCQ) is one of the most employed antimalarial drug, obviating the motivation for a detailed study of its properties. In the present work the Surface Enhanced Raman Spectra (SERS) of DCQ were investigated, in particular the dependence on pH, applied potential support electrolyte and drug concentration. The obtained results strongly suggest that depending on the applied potential the molecule can adsorb at the electrode through the quinolinic nitrogen or paralel to the surface.

FQ-057 Determination of Primary Molecular Structure and Average Molecular Weight on the Natural Rubber From *Manihot caerulescens* Pohl. *André Luiz H. Cardoso, Nágila M. P. S. Ricardo* and *Judith F. Rodrigues* (Departamento de Química Orgânica e Inorgânica da Universidade Federal do Ceará).

Studies of primary molecular structure and average molecular weight from latex of *Manihot caerulescens* Pohl have been investigated in toluene solutions by viscometry and infrared spectroscopy. The values of intrinsic viscosity, $[\eta]$, show that NR from *Manihot caerulescens* Pohl have a visible decrease on the molecular weight ($\bar{M}$v) when compared with N. R. from *Manihot glaziovii*. This $\bar{M}$v value folls within the typical range for *Heves brasiliensis* NR. It was not observed bands characteristic of 3,4 and 1,2 structures, as well as those due to the 1,4 - trans linkage.

FQ-060 Optical Analogs of Pulsed NMR Spectroscopy in Chemical Dynamics. *Isaac M. Xavier Jr.* (Departamento de Química Fundamental, UFPE).

Coherent transient phenomena have long been utilized in nuclear magnetic resonance. Only recently, through the use of acousto-optic modulation, have phase-coherent optical pulse sequences become available. With this technique, the execution of a sequence of phase-coherent laser pulses is reduced to generating the corresponding sequence in the radiofrequency domain. Results are shown for photon echo and photon locking pulse sequences applied to iodine molecule.

FQ-061 Ionic Fragmentation of Molecules Induced by High Energy Electron Impact. I. A Time of Flight Mass Spectrometer. *G. G. B. de Souza* (Instituto de Química da UFRJ - Cidade Universitária - RJ - CEP 21910) and *Aldo Soares Cardoso* (Centro Brasileiro de Pesquisas Físicas).

A Time of Flight Mass Spectrometer has been added to a previously built electron energy-loss spectrometer, in order to study double and multiple ionization process resulting from high energy (0.2 - 2.0 KeV) electron impact with gas - phase compouds. The ionic trajetories have been studied using the SIMION program. Examples of spectra obtained with the methane and sulphur hexafluoride molecules will be discussed.

FQ-062 Ionic Fragmentation of Molecules Induced by High Energy Electron Impact. II - Interfacing A Quadrupole Mass Spectrometer to an 8-Bit Microcomputer). *G. G. B. de Souza, F. C. Pontes, J. B. Maciel, A. I. da Silva Jr.* (Dep. de Físico-Química - IQ-UFRJ, Laboratório de Impacto de Elétrons).

A commercial quadrupole mass spectrometer has been attached to a previously built electron energy loss spectrometer in order to study the formation of positive and negative ions through high energy (200 – 2000 eV) electron impact. The long range purpose consist in correlating molecular fragmentation with accessible channels for excitation and ionization. The mass spectrometer has been interfaced to an inexpensive microcomputer. In the present work we present the corresponding hardware and software.

FQ-063 Electronic Excitation of the $TiCl_4$ Molecule in the VUV Region (0 – 500 eV). *Rosana Janot Martins; Gerardo Gerson Bezerra de Souza* and *Carlos Alberto Lucas*. (Instituto de Química, Departamento de Físico-Química, Universidade Federal do Rio de Janeiro).

Angle-resolved electron energy-loss spectra of gas phase $TiCl_4$ have been measured in the 0 to 500 eV energy range. The incident electron energy was 1 keV and the energy resolution 0.6 eV. Two new bands have been observed at 12 eV and 15 eV and the GOS's for the bands located at 6.8 and 10 eV have been determined, showing that they can be associated with dipole-allowed electronic transitions. Excitation of Ti 2p and 3p, as well as of Cl 2p electrons has been observed, centered respectively at 465, 43 and 213 eV.

FQ-064 Ionic Molecular Fragmentation With High Energy Electrons. III - Simulation of Ionic Trajectories. *G. Gerson B. de Souza* (Instituto de Química da UFRJ, Cidade Universitária, RJ, Brazil, CEP 21290), *Aldo S. Cardoso* (Centro Bras. Pesq. Físicas, Av. Dr. Xavier Sigaud 150, RJ, Brazil 20000) and *M. Suely Pedrosa* (Instituto de Física da UFRJ, Cidade Universitária, RJ, Brazil 21910).

Positive ion trajectories in a Time-of-Flight mass spectrometer have been simulated using the SIMION program. In our experimental set-up ions are formed through the interaction of high energy electrons (0.2 – 2.0 keV) with gas-phase compounds. The analysis has shown that unit mass resolution is obtained only for light ions (up to 18 a.m.u.) with the present TOF unit.

FQ-065 Generalized Oscillator Strengths of the $\tilde{A} \leftarrow \tilde{X}$ Transition of NH_3. *Heloisa M. Boechat Roberty* (Observatório do Valongo - UFRJ, Ladeira Pedro Antônio 43 - CEP 20080) and *G. Gerson B. de Souza* (Instituto de Química da UFRJ, Cidade Universitária, RJ, Brazil, CEP 21910).

The behavior of the Generalized Oscillator Strength (GOS) as a function of momentum transfer K^2, for the $\tilde{A} \leftarrow \tilde{X}$ transition in NH_3 has been determined by electron impact spectroscopy at 1 keV incident electron energy. The measurements were made in the angular range of $1.5° - 15.0°$ ($K^2 = 0.05 - 5.0$ ua) and an energy resolution of 0.6 eV. The extrapolated optical oscillator strength agrees with previously determined results. A minimum at $K^2 \cong 0.9$ u.a. in the GOS curve has been observed.

FQ-066 Electron Impact Excitation of Valence and Inner-Valence Electrons of the Sodium Atom. *J. C. Nogueira* (Departamento de Química da UFSC, São Carlos, SP, Brazil, CEP 13560), *G. G. B. de Souza, C. A. Lucas* and *C. E. Bielschowsky* (Instituto de Química da UFRJ, Cidade Universitária, RJ, Brazil, CEP 21910).

Differential cross section and generalized oscilator strengths has been determined both theoretically and experimentally for the following excitation process in Na atom: $1s^2 2s^2 2p^6 3s^1 (^2S_{1/2}) \rightarrow 1s^2 2s^2 2p^6 3p^1 (^2P_{1/2,3/2})$ and $1s^2 2s^2 2p^6 3s^1 (^2S_{1/2}) \rightarrow 1s^2 2s^2 2p^5 3s^2 (^2P_{1/2,3/2})$. Elastic differential cross section has been also determined. The experimental results have been obtained at the impact energy of 1 keV, and energy resolution of 0.6 eV. The theoretical results were determined with the Glauber and Born-Ochkur approximation.

FQ-067 Theoretical Calculations of the Electronic Structures of Mesoionic Compounds. *S. E. Galembeck* (Universidade de São Paulo, Faculdade Filosofia, Ciências e Letras - RP, 14.049 - Ribeirão Preto, SP); *A. M. Simas, J. Miller, M. B. de Oliveira* (Universidade Federal de Pernambuco, Departamento de Química Fundamental, 50.739 - Recife, PE) and *A. Echevarria* (Universidade Federal Rural do Rio de Janeiro, Departamento de Química, 23.851 - Itaguai, RJ - Brasil).

The geometries and electronic structures of five-membered ring 1,3,4-triazol-1-ium -5-thiolate and of 2-amino-1,3-dithiol-1-ium-4-thiolate mesoionic systems have been calculated by means of semi-empirical (MNDO, AMl and MNDO-PM3) and ab-initio techniques (STO-3G/6-311+G** and GVB-PP). The charge distributions predicted by all techniques used show that there is separation of charge within the ring as well as over the entire molecule. Contrary to the electronic structures generally accepted for mesoionic systems based on pi-electron considerations, results show that there is separation of charge within the ring as well as over the entire molecule; the hydrogen atoms of the substituents becoming strongly positively charged. Comparison is made with the geometries determined by x-ray diffraction of members of the 2-amino-1,3-dithiolium-4-thiolate system. (CNPq, FINEP, FAPESP).

FQ-068 An MNDO Study of the Tautomeric Equilibrium of Pyrrole-2-Carboxaldehyde. *N. B. da Costa Jr., A.M. Simas, M. N. Ramos* (Depto. de Química Fundamental, Univ. Federal de Pernambuco, 50739, Recife, PE, Brasil) and *J. T. Lopez-Navarrete* (Depto. de Quimica Física, Facultad de Ciencias, 29071, Malaga, Espanha).

MNDO calculations have been performed to study the tautomeric equilibrium of pyrrole-2-carboxaldehyde ($\underline{1}$) and its hydroxide form ($\underline{2}$). Analysis have been made considering thermodynamic, electronic and vibrational properties. The results reveal that: (i) $\Delta G^{\neq}$ ($\underline{1} \rightarrow \underline{T}$) and ΔG_r ($\underline{1} \rightarrow \underline{2}$) are 243KJ/mol and 31.3KJ/mol at 308K, respectively, (ii) CH stretching frequencies and intensities have been useful to characterize intramolecular interactions and stability differences. As a result, the CH infrared intensities were parametrized in terms of atomic charges and charge-fluxes.

FQ-069 A QSAR-MO Study of N-Phenyl-Phtaliide Derivatives. *M. D. Coutinho Neto, B. B. Neto, F. W. J. Demnitz* and *M. N. Ramos* (Departamento de Química Fundamental - Universidade Federal de Pernambuco, 50739 - Recife - PE).

N-Phenyl-phtalimide derivatives, which are hypolipidemic agents in mice, have been subjected to a QSAR study based on several empirical and theoretical parameters, the latter being obtained from AM1 molecular orbital calculations at optimized geometries. A regression analysis shows that the substituent's lipophilicity and the atomic charge on the carbon atom bound to the nitrogen in the five-membered ring are the parameters that best correlate with the hypolipidemic activity. The derivatives in which the substituent is o-NO, p-NO, p-$SOCH_3$ or p-$CONH_2$ are predicted to have stronger hypolipidemic activities than the ones whose activites have been experimentally determined.

FQ-070 Effect of an External Uniform and Strong Electric Field on the Structure and Reactivity Indices of Molecules as Revealed by Semi-Empirical Molecular Orbital Techniques. *Sérgio Emanuel Galembeck* (Depto. Química, FFCLRP / USP - Ribeirão Preto, Brazil) and *Alfredo M. Simas* (Depto. Química Fundamental - UFPE, Recife, Brazil).

Calculations of the molecule of nitrobenzene in presence of an external electric field are performed with the AMl method. An electric field in the directon of the dipole moment lowers the total energy of the molecule. A field contrary to the dipole moment first raises the total energy, and as the field becomes stronger, lowers that. Results indicate that the reactivity indices are nor to sensitive to an external electric field until magnitude becomes of the order of 1 V/Å. After that, the molecular charge density and the molecular orbitals changes dramatically. This indicates that there are orientation effecs due to the electric fields in reactions of the nitrobenzene.

FQ-071 Comparison of Vibrational Frequencies Calculated by Semi-Empirical Molecular Orbital Techniques. *Ivana Aparecida Borin* and *Sérgio Emanuel Galembeck* (Fac. de Filosofia, Ciências e Letras de Ribeirão Preto – Deptº Química - Univ. de São Paulo).

We have analysed the quality of the vibrational frequencies calculated by semi-empirical methods, MINDO/3, MNDO, AMI and MNDO-PM3. We have studied fifteen molecules of C,H,O,N. The geometry were fully optimized and the derivatives of the energy are calculated by numerical diferences. The results indicate that the use of analytical derivatives do not alter the results, not the use of greater precision in the convergence criteria. The absolute error are sometimes too big ($\sim$500 cm^{-1}). All the methods present inversion of frequencies or artificial degenerecencies. The MNDO-PM3 presents the best results, followed by AMI, MINDO/3 and MNDO.

FQ-072 3-D Views of Molecular Normal Vibrations: an Aid to Normal Coordinate and Molecular Mechanics Calculations. *Pedro Antonio Muniz Vazquez* (Instituto de Química, UNICAMP, Campinas, SP).

A set of subroutines of the PLUTO package were modified and adapted to run in the FORCES molecular mechanics program producing a 3-D screen view of normal vibrational modes. The atomic displacements are displayed as perspective cilynders and the output can be piped to a plot file. The program is running in VAX/VMS and MSDOS environments. (INTEL Co.)

FQ-073 Computational Baseline Correction of Spectral Data: an Interactive Method Using a Generating Polinomial. *Pedro Antonio Muniz Vazquez* (Instituto de Química, UNICAMP, Campinas, SP).

An interactive procedure for baseline correction of spectral data was developed for IBM/PC and compatible microcomputers. A polinomial regression is done over a set of data points arbitrarily choosed by the user. Then a line is generated over the entire data and, if satisfactory, the baseline removed through subtraction, otherwise a new set of points is required. (INTEL Co.)

FQ-074 S_N2 Reaction Between CN^- and CH_3F: an AB Initio Study. *Paolo Roberto Livotto* and *Yuji Takahata* (Instituto de Química, Universidade Estadual de Campinas).

The reaction coordinate of the S_N2 reaction between the CN^- and CH_3F was studied using an ab initio method. A basis set of DZ plus polarization with difuse functions on heavy atoms was employed. MP2 correlation energy was also included. The structures of ion-dipole complexes (IDC) was calculated to have C_{3v} symmetry in direct reaction, while a C_S symmetry IDC was calculated in the reverse reaction. An activation energy of 14.8 kJ/mol was obtained for the reaction.

FQ-075 Modification of Computer Program MM2 (1977), Version PC, to Use the 1987 Force Field. *Yoshiyuki Hase* (Instituto de Química, Universidade Estadual de Campinas).

The PC version of the Allinger's computer program MM2 was modified to carry out the 1987 force field calculations. First, the MM2 parameters were separated from the main program as databank to facilitate the changes in force field and also to expand the program capacity up to 99 kind of atoms. Next, the electronegativity correction was incorporated to obtain the better bond distances. Finally, the program was changed to allow the mechanical treatment of aromatic molecules as new option; this is at first time in this program.

FQ-076 Molecular Mechanics Calculations and MM2 Force Field of Fluorophosphines and fluorophosphine Oxides. *Yoshiyuki Hase* (Instituto de Química, Universidade Estadual de Campinas).

A new parameter set of MM2 theory was determined for fluorophosphines and fluorophosphine oxides. The parameters suggested were obtained mainly from the normal coordinate analysis carried out using the vibrational spectral data. By the use of these parameters, molecular geometries, dipole moments and rotational barriers of titled molecules were well reproduced comparing with the known experimental data. The parameters are totally compatible with the Allinger's 1977, 1985 and 1987 force fields.

FQ-077 Quasiclassical Dynamical Study of the Reaction H + ClO = OH / HCl /OH + Cl / O(3P) Chemical Reaction on a Singlet Potential Surface. *C. Fenelon & H. H. R. Schor* (Departamento de Química, ICEx, UFMG).

The dynamics of the reaction H + OCl has been studied by the quasiclassical trajectory approach employing a singlet potential surface that presents minima along the reaction paths. The calculation were performed for reactants in the ground vibrational and rotational state at collisionenergies 1.0, 2.5, 5.0, 10.0 and 20.0. Cross sections for both reactions and for intermediate OHCl formation was calculated. The energy effects were investigated and compared with the experimental results of Wategaonkar and Setser (J. C. P. 90,6223,1989). Rotational energy plays an important role in these reactions. Calculations with J>0 are being performed. (CNPq/FINEP).

FQ-078 Laser Control of Electronic Branching Ratios in Unimolecular Dissociation. *H. H. R. Schor* (Departamento de Química, ICEx, UFMG).

The present study center attention on laser control of electronic diabatic processes. As our model system we choose the laser induced dissociation on BrBr following resonance transitions from the ground state (X) to two interfering electronically excited states (B and II). We suggested a sequence of two femtoseconds laser pulser for exciting the system and subsequently for inducing the desired transition of the dissociative wave packet towards the desired product channel. (CNPq, FINEP).

FQ-080 Statistical Mechanics of Short Peptide Chains. *Carlos A. P. almeida* (Universidade Federal de Minas Gerais, Instituto de Ciências Exatas, Depto. de Química) and *Saul G. Jacchieri* (Universidade Federal de Minas Gerais, Instituto de Ciências Biológicas, Depto. de Bioquímica e Imunologia, Belo Horizonte, 31270 MG).

By selecting values of dihedral angles that lie in the minima of rotational barriers we choose a finite number of rotational isomeric states from the infinite number of conformations that the chain may adopt. Conformations generated in this way that closely match crystalographic structures are not the most stable nor the most entropically favored. We conclude that our data is correct in the absence of intermolecular and solvent interaction and that inclusion of these factors will change the order of probabilities in our table of conformations without changing the structures of these conformations.

FQ-081 A Semi-Empirical Study on the Conformation of Cholic Acid. *Maurício L. Operti, Joaquim D. da Motta Neto* and *Ricardo Bicca de Alencastro* (Instituto de Química da UFRJ - Rio de Janeiro - Brazil).

We report the results of a MM2 calculation of the geometry of cholic acid which conforms to experimental crystallographic data (1). Our calculations were carried out in a VAXstation II/GPX. We found that the MM2 method does not reach the description of any intramolecular hidrogen bond. (1) S. Candeloro de Sanctis e E. Giglio, *Acta Cryst.* **B35**, 2650-2655 (1979).

FQ-082 Fundamental Vibrational Frequency Correlation. *J. M. Trevas dos Santos, I. M. Brinn, O. M. Herrera* (Instituto de Química, Universidade Federal do Rio de Janeiro, Ilha do Fundão - Rio de Janeiro, RJ - 21941 - Brasil).

D_{3h}-D_{3d} conformer pairs have been studied using a program which automatically classifies each vibrational mode according to its representation in its point group. In the case of ethane-d_0, ethane-d_6, 2-butyne-d_0 and 2-butyne-d_6 it was found that the frequencies were identical for the corresponding modes in each D_{3h}-D_{3d} pair. The use of non-diagonal terms is indicated to differentiate and correlate vibrational frequecies in these point groups.

FQ-084 A Theoretical Study of the Electronic Transitions $A^2\pi$–$X^2\Sigma^+$ and $C^2\Sigma^+$ - $X^2\Sigma^+$ of the BeH and BeD Molecules. *Francisco Bolivar Correto Machado* (Instituto de Estudos Avançados - IEAv - CTA e Intituto de Química - USP) and *Fernando Rei Ornellas* (Instituto de Química - USP).

A theoretical description of the $X^2\Sigma^+$, $A^2\pi$ and $C^2\Sigma^+$ electronic states of the BeH molecule has been carried out within the multi-reference singles and doubles configuration interaction (MR-SDCI) approach using a very large atomic basis set of (14s9p4d1f) and (8s2p2d) of primitive gaussians contracted to [9s7p4d1f] and [5s2p2d] on beryllium and hydrogen, respectively. The vibrational levels spacings calculated with the theoretical potential energy curves differ from the experimental ones by no more than 20 cm^{-1} on the average. The dipole moment function shows a clear variation of molecular polarity as the internuclear distance changes and the Einstein emission coefficients predict strong transitions between the states $X^2\Sigma^+(v'' = 0-3) - A^2\pi(v' = 0)$ and $X^2\Sigma^+(v'' = 5-7) - C^2\Sigma^+(v' = 0)$.

FQ-085 A Theoretical Study of the SO^+ Ion Using the Configuration Interaction Method. *Antonio Carlos Borin, Fernando Rei Ornellas* (Instituto de Química, Universidade de São Paulo - C.P. 20780 - São Paulo - 01498 - SP - Brazil) and *Francisco Bolivar Correto Machado* (Instituto de Estudos Avançados, Centro Técnico Aeroespacial - C.P. 6044 - Sao José dos Campos - 12200 - SP - Brazil).

Potential energy curves, dipole moment functions and vibrational levels are computed for the two lowest $^2\pi$ states of the SO^+ ion. It is shown that the inclusion of a relatively high d expoent (8.35) on sulfur is important to describe the behavior of the system near equilibriuminternuclear distance region. To properly describe the dissociation it is shown that an f function has to be also included in the basis set of the system.

FQ-086 A Characterization of the Ground State and the First Excited States of the BeN Molecule by the Configuration Interaction Method. *Orlando Roberto Neto* (Inst. de Estudos Avançados, Divisão de Lasers – CTA e Inst. de Química – USP), *Francisco Bolivar Correto Machado* (Inst. de Estudos Avançados, Divisão de Física Teórica – CTA) and *Fernando Rei Ornellas* (Inst. de Química – USP).

The non–existence of any experimental study on the BeN molecule has raised the question whether this species is a stable one or not. In this study, using the multireference singles and doubles configuration interaction method (MR–SDCI) and the gaussian basis set (13s5p1d)/[7s3p1d] on beryllium and the set (13s5p1d)/[7s3p1d] on nitrogen, we have calculated potential energy curves and found the states $^4\Sigma^-$, $^2\pi$ and $^2\Sigma^+$ to be stable with the quartet lower in energy followed by the $^2\pi$ state. Dipole moment function, vibrational levels spacings and spectroscopic constants are also presented for the first time.

FQ-087 A Molecular Analogue for Phase Diagram. *João Cardoso Pereira Netto* (Universidade de Mogi das Cruzes) and *Newton Bernardes* (Universidade de São Paulo).

Substituing the pressure by one molecular variable as interaction force–fi between particle, is possible to construct a phase diagram which reproduces the information contained in the classical diagram P x T and provides other consequences as molecular definitions for critical and triple points and the inexistence of the critical point solid liquid.

FQ-088 X–Ray Line Broadening in Hematite Derived Goethite by Heating. *Milton F. de Jesus Filho, Geraldo M. da Costa* (Departamento de Química) and *Eustáquio Galvão da Silva* (Departamento de Física – Universidade Federal de Minas Gerais – 31270 – Belo Horizonte – Brasil).

The differential X–ray line broadening in hematite derived goethite by heating was studied in several samples obtained from synthetic goethites of varying crystallinity. Mossbauer results discard the hypothesis of iron atoms off their equilibrium positions in the hematite samples. The integrated intensities of the H(110)/H(104) planes are nearly constant. No distortion component was observed and the broadening could be explained by small particle size in acicular samples.

FQ-089 The Crystal and Molecular Structure of a Complex Formed by Picric Acid and Nitropyridine N–Oxide. *Anna Paula Bechepeche, Regina Helena de Almeida Santos* (IFQSC – USP).

The ACP.NPNO crystallizes in the triclinic system, Pl; a = 3.683(2), b = 12.956(2), c = 13.662(2) Å, α = 94.35(2), β = 94.91(2), γ = 91.30(2)°; V = 647.3(8); Z = 2; D_C = 1.663 g/cm^3; μ = 0.97 mm^{-1}; F(000) = 332; 1844 unic reflections with 1172 observed ($I > 3\sigma(I)$). The two independent complex presents very strong hydrogen bonds (2.429(7) and 2.531(4) Å) between the O from NPNO and OH from ACP. (CNPq, FINEP).

FQ-090 Crystal and Molecular Structure of Bis (Diethanoldithiocarbamate) Zinc(II). *M. T. P. Gambardella, R. H. de Almeida Santos, G. O. Chierice, A. L. B. Marques* (Instituto de Física e Química de São Carlos – USP).

The solid complex Zn(DEDC)$_2$ were prepared by precipitation of aqueous solutions of Zn^{2+} with NH$_4$(DEDC) in ca. 1:2 molar ratios. Crystal Data: orthorhombic; Pbcn, a = 29.570(1), b = 8.456(1), c = 13.833(1) Å^3, Z = 8, F(000) = 1775.92, μ = 18.24 mm^{-1}. Structure solved by Patterson and difference Fourier methods and refined by least–squares with R = 6.38 and R_W = 4.84%. The crystal consists of dimeric units. In each molecule, one ligand acts as a simple chelate and the other as a highly asymmetric chelate. The sulphur atom which participates of the weakest interaction binds itself more strongly to the metal related by inversion. (CNPq, FINEP, CAPES, FAPESP).

FQ-091 Crystal and Molecular Structure of the Crotonepetin. *M. S. de Lima Marques, R. H. de Almeida Santos* (IFQSC–USP), *E. Rocha Silveira, S. Maia de Morais* (Departamento de Química Orgânica, Universidade Federal do Ceará).

The acetyl derivative of VJ–1 obtained from the hexane extract of the leaves of **Croton nepetifolius** was recrystallized from ethanol. Crystal Data: $C_{22}H_{33}O_3$, orthorhombic, $P2_12_12_1$, a = 7.363(3), b = 14.597(3), c = 19.538(3) Å, Z = 4, V = 2099.6(5) Å^3, F(000) = 756, μ = 0.75 mm^{-1}, d_C = 1.093 g.cm^{-3}. Structure solved by direct methods and refined by least–squares with R = 0.0582 and R_W = 0.0668 for 1183 observed reflections. (CNPq, FINEP).

FQ-092 The Crystal and Molecular Structure of the Crotomicasin. *M. S. de Lima Marques, R. H. de Almeida Santos (IFQSC–USP), E. Rocha Silveira, M. A. Sousa Lima* (Departamento de Química Orgânica, Universidade Federal do Ceará).

The structure of CMRH–1, obtained from the **Croton micans** root, was determined. The ethanolic and hexanolic extract of this plant present biological activities. Crystallographic Data: $C_{20}H_{22}O_3$; orthorrombic, $P2_12_12_1$; a = 9.810(2), b = 12.738(2), c = 13.570(2) Å; Z = 4; V = 1695.8(6) Å3; F(000) = 664.00; μ = 0.751 mm^{-1}; D_C = 1.216 g/cm^3. Structure solved by direct methods and refined by least–squares with R = 0.0510 and R_W = 0.0534 for 1086 observed reflections. (CNPq, FINEP).

FQ-093 The Crystal and Molecular Structure of The 1:1 Complex Formed by 4–Nitropyridine N–Oxide + 2–Aminobenzoic Acid. *R. Moreno Fuquen* (Univ. del Valle, Colombia), *J. R. Lechat, R. H. de Almeida Santos* (IFQSC/USP).

The crystal structure of the 1:1 molecular complex of 4–Nitropyridine N–oxide and 2–Aminobenzoic Acid obtained by solid state reaction or by slow evaporation of equimolecular solution of the starting compounds in acetonitrile has been determined. *Crystallographic Data:* $C_{12}H_{11}N_3O_5$, monoclinic, Cc, a = 9.522(3), b = 10.637(4), c = 12.611(3) Å, β = 104.31(2)°, Z = 4, V = 1237.7(4) Å^3, F(000) = 576.00, μ = 0.111 mm^{-1}, Dc = 1.488 g.cm^{-3}, mp = 133–137.5°C. The complex is formed by charge transfer and hydrogen bonding of the N–oxide oxygen 0(3) to the hydroxyl 0(4) of the carboxylic group with an 0(3)•••0(4) distance equal to 2.630(4) Å. (CAPES, FINEP).

FQ-094 Crystal and Molecular Structure of 6,6–Dimethyl–9, 10–Dimethoxy–2–(3,4–Dimethoxyphenyl)–4H,6H–Benzo[1,2–b:3,4–b']Dipyran–4–One. *Ana Maria G. D. Rodrigues, Johannes R. Lechat, Regina H. P. Francisco* (DQFM – IFQSC – USP).

The crystal data for the title compound are: $C_{24}H_{24}O_7$, triclinic, P$\bar{1}$, a = 8.199(1), b = 16.170(3), c = 16.568(3) Å, α = 104.78(1), β = 90.10(1), γ = 100.61(1)°, Z = 4, V = 2088 Å^3, F(000) = 896, d_C = 1.35 g.cm^{-3}, μ = 0.6 mm^{-1}. Crystal dimensions 0.45 x 0.35 x 0.15 mm^3, Mo Kα, 2976 reflections with $F > 6\sigma(F)$. Direct methods, full–matrix least–squares refinement with R = 0.086 and R_W = 0.093. Both independent molecules are aproximately plane and present similar structural features. (CNPq, CAPES, FINEP, FAPESP).

FQ-095 Structures of: (I) [4R,9R,10S]–9–Carbomethoxy–7–Isopropyl–4–Methyl–Tricyclo [8.3.0.0^{4,9}]–1,6–Tridecadienedione–5,8 and (II) [4R,9R,10R]–9–Carbomethoxy–7–Isopropyl–4–Methyl–2–Phenylthio–Tricyclo [8.3.0.0^{4,9}]–1,6–Tridecadienedione–5,8. *J. Zukerman-Schpector, E. E. Castellano, C. A. De Simone, G. Oliva* (Instituto de Física e Química de São Carlos – USP – C.P. 369 – 13560 – São Carlos – SP – Brazil), *T. J. Brocksom* and *V. Catani* (Departamento de Química - Universidade Federal de São Carlos).

(I): $C_{19}H_{24}O_4$, F_W=316.40, orthorhombic, Pbca, a=12.444(1), b=14.615(3), c=18.797(5) Å, V=3419(2) Å^3, Z=8, Dx=1.229 gcm^{-3}, μ(MoKα)=0.49 cm^{-1}, F(000)=1360, T=298K, R=0.050 for 1060 observed reflections. (II): $C_{25}H_{28}O_4S$, Fw=424.56, monoclinic, $P2_1/n$, a=10.338(2), b=14.538(3), c=15.829(2) Å, β=106.54(1)°, V = 2281(1) Å^3, Z=4, Dx=1.236 gcm^{-3}, μ(MoKα) = 1.30cm^{-1}, F(000)=904, T=298K, R=0.050 for 2032 observed reflections. In both structures the five membered ring is in an envelope conformation, the cyclohexene ring in a essentially halfchair conformation and the cyclohexene–dione in a sofa conformation. The basic tricyclic system adopts in both structures an overall hemispherical configuration.

FQ-096 Crystal Structures of Two Methyl–Mercury Compounds: (I) $CH_3Hg(C_{12}H_{10}PS_2)$ and (II) $CH_3Hg(C_6H_{10}N_3S)$. *J. Zukerman-Schpector* (Instituto de Física e Química de São Carlos – USP – C.P. 369 – 13560 – São Carlos – SP – Brazil), *E. M. Vazquez, M. C. Rodrigues-Arguelles, M. I. Suarez, A. Sanchez, J. S. Casas* and *J. Sordo* (Depto. de Química Inorgánica, Universidad de Santiago de Compostela, España).

Crystal data for (I): $C_{13}H_{13}HgPS_2$, Fw=464.94, triclinic, P$\bar{1}$, a=9.189(1), b=11.945(3), c=6.9930(9) Å, α=105.79(1), β=96.03(1), τ=78.92(2)°, V=723.7(3) Å^3, Z=2, Dx = 2.133 gcm^{-3}, μ(MoKα)=10.63mm^{-1}, F(000)=436, T=298K, R=0.030 for 1414 independent reflections. *Crystal data for* (II): $C_7H_{13}HgN_3S$, Fw=371.86, monoclinic, $P2_1/c$, a=8.793(2), b=15.247(3), c = 7.511 (2) Å, β=101.90(2)°, V=1005.5(8) Å^3, Z=4, Dx = 2.456 gcm^{-3}, μ(MoKα)=14.99mm^{-1}, F(000)=688, T=298K, R=0.040 for 1519 independent reflections. Distances (Å) and angles (°) around the Hg(II). (I); Hg–S(2)=2.379(3), Hg–C(13)=2.08(1), Hg–S(1)=3.869(3), Hg–S(1)$^{\rm I}$ = 3.152(3); S(2)–Hg–C(13)=175.0(3), S(2)–Hg–S(1)$^{\rm I}$ = 83.54(8), S(1)$^{\rm I}$–Hg–C(13)=98.2(3). $^{\rm I}$symmetry code: 1–x,1–y, –z (II): Hg–S=2.380(3), Hg–N=2.537(8), Hg–C=2.09(1); S–Hg–N = 74.8(2), S–Hg–C=167.7(4), N–Hg–C=116.8(4).

FQ-097 Crystal Structure of Ni Tetrakis (2–pyridylmethyl) Ethylenediamine $(Cl0_4)_2$ 2/3 H_2O. *Ivo Vencato* (Depto. Física – UFSC – Florianópolis – SC – Brazil) and *Yvonne Primerano Mascarenhas* (IFQSP – USP – São Carlos – SP).

The complex $C_{26}H_{28}N_6Ni(Cl0_4)_2$.2/3 H_2O is monoclinic, space group C2/c, with a = 41.205(5), b = 9.492(2), c = 23.930(3) Å, β = 108.02(3)°, V = 8900(6) Å^3, Z = 12, d_{calc} = 1.55 g cm^{-3}, λ(MoKα) = 0.71073 Å, μ = 8.31 cm^{-1}. The structure was solved with MULTAN–80 and was refined by least – squares to an R value of 7.1 % for 3258 observed reflections with $I > 3\sigma(I)$. The structure consists of one distorted Ni^{2+} ion in special position and one Ni^{2+} ion in the general position, both octahedrally coordinated by nitrogen atoms.

FQ-098 Crystal Structure of (1SR, 3RS)3–Chlorine Aniline–1, 3–Diphenyl–1–Propanol. *Ivo Vencato* (Depto. de Física – UFSC), *Yvonne P. Mascarenhas* (Inst. de Física e Quím. de São Carlos – USP), *Ronaldo A. Pilli* and *L. C. Dias* (Inst. de Quím. – UNICAMP, Brazil).

$C_{21}H_{20}$O N Cl, M_r = 337.89, triclinic, space group P1, a = 6.147(2), b = 11.438(2), c = 12.926(2) Å, α = 79.91(1), β = 86.86(2), γ = 80.48(2)°, V = 882.1(5) Å^3, Z = 2, D_{calc} = 1.27 Mg.m^{-3}, λ(MoKα) = 0.71073 Å, with 1312 observed reflections and R = 5.6%. The observed relative configuration syn is consistent with the quelate intermediate participation involving the Zn^{2+} cation, during the corresponding β–aminocetone reduction with zinc borohydrate.

FQ-099 Studies on Crystal Modification of Linear Trans–Quinacridone Pigments.

D. S. Filho, C. M. F. Oliveira (Instituto de Macromoléculas, Universidade Federal do Rio de Janeiro).

Linear trans–quinacridone are pigments of outstanding light, heat and chemical fastness. It is said to exist as six different crystallographic modifications α, β, γ, γ', δ and ϵ), although the forms β and γ are of commercial importance. We have examined these modifications by a combination of infrared spectroscopy, spectrophotometric and differential thermal analysis, optic and scanning electron microscopy, X–ray diffraction and specific surfaces.

FQ-100 Nucleation and Polymorphic Structure is Isotactic Polypropylene.

D. S. Filho, C. M. F. Oliveira (Instituto de Macromoléculas, Universidade Federal do Rio de Janeiro).

The addition of the six differents crystallographic modifications of the linear trans–quinacridone to isotactic polypropylene (iso–PP) have been examined. The dyestuff can exist in α, β, γ, γ', δ, and ϵ modifications. The modification α do not nucleate the β form crystalline of iso–PP but the modification δ have showed to be more effective than others forms. Perhaps the shape of crystal and its space–group symmetry is one of the reasons why the β form of iso–PP is formed. We examine the relationship between the crystal structure of linear transquinacridone and the efficiency of formation of the beta iso–PP phase.

FQ-101 Study of Fluorescence Spectra of Complexes Obtained Between Uranyl Nitrate or Uranyl Acetate With Disulfoxides.

João Aldésio Pinheiro Holanda, Maria Mozarina Beserra Almeida, Emelvira Bravo de Paiva e Sá, Maria Isaura Diógenes Holanda and *Sérgio Maia Melo* (Centro de Ciências da Universidade Federal do Ceará).

The fluorescence spectra of the uranyl nitrate hexahydrate, uranyl acetate dihydrate and some derivatives complexes of disulfoxides, were recorded at room and liquid N_2 temperatures, in the range of 450 to 610nm. At room temperature, the spectra exhibited five bands at approximately 490, 510, 533, 555 and 586nm while at liquid N_2 temperature these bands are split in some sharp lines. These spectra seem to show different crystal field effects.

FQ-102 Positron Annihilation Studies in Tetrakis (α–Hydroxicarboxylate)Hafnium(IV) Complexes.

A. Marques Netto, M. E. S. R. Silva, V. J. P. Gouveia, J. C. Machado, W. F. Magalhães (Dep. de Química – ICEx – UFMG – Belo Horizonte – MG) and *Roberto S. Barbiéri* (Dep. de Química Analítica – IQ – UNESP – Araraquara – SP).

Positron annihilation studies were performed on a series of complexes of the type [Hf(glycol$_4$] (glycol = lactate, I; isobutirate, II; mandelate, III; 2–hydroxyhexanoate, IV and 2–hydroxidodecanoate, V). The formation probability, I_3, of *orto*–positronium in this series is supposed to be dependent on the charge delocalization processes. It was also observed that within a same homologous series, for example, compounds I and II or compounds IV and V, I_3 decreases, significantly, with the number of carbon atoms of the ligands. (CNPq, FINEP).

FQ-103 Positron Annihilation (e+) Studies in Apatites [$Ca_5(PO_4)_3X$; X=F, Cl...] Using Lifetime Spectroscopy (LS).

Jane Magalhães Alves, Sheyla Maria de Castro Máximo Bicalho, Viviane Viana Silva (Departamento de Química, Instituto de Ciências Exatas, Universidade Federal de Minas Gerais).

Preliminary measurements were carried out on LS of annealed and non–annealed samples of Araxá apatites and the lifetime spectra were resolved for three lifetime (ζ_i) and intensity (I_i) components. A peculiar oscillation of I_2 values was observed with maxima for annealing temperatures of 300 and 700°C. When statistically significant measurements are made it is expected that is possible to correlate LS data to lattice defects and thermal reordering of apatites through supporting information by X–rays difraction, thermoluminescence, fission–tracks counting and other physical chemical investigation methods.

FQ-104 Chemiluminescence of C_2 and CH in the Ethanol/Cl_2 System.

Harrald Victor Linnert and *José Manuel Riveros* (Instituto de Química – Universidade de São Paulo – Caixa Postal 20780, 01498 – São Paulo – SP).

The time dependent behavior of transient chemical species, CH and C_2, has been observed by the infrared laser induced multiphoton decomposition of ethanol in experiments with Cl_2 as a radical scavenger. The time evolution of the emission signal has been analyzed by a kinetic model which includes: a) an ill–defined pseudo–first order process as a consequence of collisionally modulated multiphoton vibrational excitation and b) decay through spontaneous emission, chemical reaction or radiative quenching. The resulting kinetics were fitted to the experimental curves by computer simulation.

FQ-105 Picosecond Emission of o–Hydroxyphenyl Oxadiazoles.

Carlos E. M. Carvalho, Ira M. Brinn (Instituto de Química – UFRJ – Rio de Janeiro – Brasil) *Francine Heisel* and *Joseph A. Miehe* (Centre de Recherches Nucléaires, Cronenbourg, France).

The kinetics of intramolecular proton transfer in 5–(2–hydroxyphenyl)–3–phenyl–1,2,4–oxadiazole has been investigated using steady state and time dependent picosecond LASER spectroscopy. On the basis of these experimental determinations the structures of the two fluorescing species observed have been assigned to two different conformations of the same molecular structure, both being present in the ground state. Rapid proton transfer in the excited state is observed here as the predominat quenching process of one of these conformations.

FQ-106 Self–Absorption of the Luminescence of Solid Uranyl Nitrate Hexahydrate Investigated by Time Resolved Spectroscopy.

João B. Marques Novo, Francisco B. T. Pessine (Depto. Físico-Química) and *Oswaldo L. Alves* (Depto. Química Inorgânica – Instituto de Química – UNICAMP – Brazil).

A systematic investigation of the self–absorption in solid uranyl nitrate hexahydrate on the relative intensities of the time resolved emission vibronic spectra and the excited state lifetime was performed. We found that the sample's granulometry and the irradiation angle, keeping the ortogonal geometry for the excitation/detection, were very critical parameters to be adjusted and they can be responsible for severe errors on the spectroscopic and kinectics measurements.

FQ-107 Time Resolved Spectroscopy: Interfacing of a Microcomputer to a Luminescence Spectrometer.

João B. Marques Novo, Francisco B. T. Pessine (Depto. Físico-Química) and *Oswaldo L. Alves* (Depto. Química Inorgânica – Instituto de Química – UNICAMP – Brazil).

The interfacing of a microcomputer to a luminescence spectrometer through the SR245 A/D interface (Stanford Research) is described. BASIC programs for acquisition and treatment of 3D–surfaces of time resolved spectra (TRS) were developed. The latter included normalization, subtraction, addition and baseline adjustment of TRS as well as excited state lifetime calculations.

FQ-108 Prealbumin Tryptophans Environmental Changes Induced by Thyroxide.

Gustavo González and Guilhermina Tapia (Instituto de Química, Universidade Católica de Valparaiso, Valparaiso, Chile).

Three–dimensional structure of prealbumin shows different environments for its two tryptophans. Fluorescence techniques as quenching by thyroxine and acrylamide, as well as the excitation spectra analysis of tryptophans show a burying of the exposed Trp–41 and polarity increase in the environment of the hydrophobic Trp–79 upon binding of the ligand thyroxine.

FQ-109 Resolution of the Lowest Energy of the Electronic Absorption Spectra of 1–Anilinonaphtalene–8–Sulfonate.

Juan E. Brunet (Instituto de Química, Universidad Católica de Valparaiso, Casilla 4059, Valparaiso, Chile) and *Victor Vargas* (Departamento de Química, Facultad de Ciencias, Universidad de Chile, Casilla 653, Santiago, Chile).

The lowest energy absorption band of 1–anilinonaphtalene–8–sulfonate (1, 8–ANS) have been investigated. Two gaussian bands were fitted to the observed band profile. From the absorption spectra and the fluorescence excitation spectra of 1,8–ANS in glycerol at 5°C, two anisotropy resolved bands were also obtained. These findings confirms the presence of two electronic transitions under the first observed absorption band of 1,8–ANS with maxima at 380 nM and 345 nM.

FQ-110 O Processo de Colesterização Estudado Através dos Parâmetros de Ordem em Sistemas Liomesomórficos.

O. Felippe Jr., T. M. H. do Aido, M. R. Alcântara, A. M. G. Pereira & J. A. Vanin (Inst. Química – Univ. de São Paulo).

Linear dichroism was applied to the study of the cholesterization process of a potassium laurate / diacetone-sorbosde (DAS) system, using congo red as a probe. The inducer solute, DAS, was added in several concentrations. The order parameters of the resulting cholesteric phases calculated from absorbance measurements at 495 nm, lead to an exponential dependence on the inducer concentration. This fact sugests the use of the order parameters, obtained from linear dichroism, as a measurement of the micellar interaction strenght in cholesteric systems.

FQ-111 Solvent Effects on the Formation of Triplet Exciplexes of Acetone and Mesitylene or Durene.

G. L. Indig, L. H. Catalani and *T. Wilson* (The Biological Laboratories, Harvard University, Cambridge, USA and Instituto de Química da Universidade de São Paulo, São Paulo, Brazil.

Triplet exciplexes formation between excited acetone and ground state mesitylene or durene can be monitored by energy transfer to 9,10–dibromoanthracene followed by fluorescence from its S_1 state, which was analysed by time resolved single photon counting. All kinetic parameters can be recovered from the analysis of the triple exponential decay curves, which show build up and decay of the exciplex. Solvent effects showed dependence of the rate of complex formation with solvent polarizability within the series acetonitrile, n–hexane, isooctane, dodecane and c–hexane.

NATURAL PRODUCTS (PN)

PN-01 Flavonol Glycosides of *Ocotea Vellosiana* (Meissn.) Mez. (Lauraceae). *Walmir S. Garcez* (DQI, CCET, UFMS), *Massayoshi Yoshida* and *Otto R. Gottlieb* (IQ-USP).

The new acylated flavonol glycoside kaempferol–3–0–(4''–*trans*–p–coumarocyl)–α–L–rhamnopyranoside and the known compounds kaempferol–3–0–β–D–glucopyranoside, kaempferol–3–0–α–L–rhamnopyranoside, quercetin–3–0–β–D–glucopyranoside, and quercetin–3–0–α–L–rhamnopyranoside, were obtained from leaves of *Ocotea vellosiana*. The structure of the isolated compounds were determinated by spectroscopic methods: UV, ^{1}H (2D HOMO COSY ^{1}H–^{1}H) and ^{13}C NMR. Previously we reported isolation and identification of six aporphine alkaloids from leaves and fruits of this specimen. (CNPq, CAPES, FINEP, FAPESP).

PN-02 Some Flavonoids From *Iryanthera* Species (Myristicaceae). *Lúcia Maria Conserva, Massayoshi Yoshida* and *Otto R. Gottlieb* (Instituto de Química, USP).

Ethanol extract of trunck wood of *I. ulei* Warb. and *I. paraensis* Huber afforded two flavonoid types. From *I. ulei* were isolated five dyarylpropanoids and one dihydrochalcone and from *I. paraensis*, one diarylpropanoid and dihydrochalcone. Among the diarylpropanoids, 1–(2',4'–dihydroxy–2'–methoxy–3',5'–dimethylphenyl)–3–(2''–hydroxy–4'',5''–methylenedioxyphenyl)–propane is unknown. Structure elucidation of this new compound was based on 2D NMR technique (Long Range ^{1}H–^{13}C Hetero COSY).

PN-03 3–Methylsulphonylallyl Cinnamate From *Cinnamomum Australe* (Lauracene). *Frida M. Pagliosa* (DQ, UFSM), *Vanderlan da S. Bolzani* (IQ, UNESP), *Massayoshi Yoshida* and *Otto R. Gottlieb* (IQ, USP).

Phytochemical analysis shows that main constituent in hexane extract of barks of *Cinnamomum australe* is *trans*–3–methylsulphonylallyl *trans*–cinnamate. Sulphonyl compounds occur frequently in species of Alliaceae, but in Lauraceae, occur only in species of *Phoebe* and *Cinnamomum* genera.

PN-05 Neolignans From *Aniba Ferrea* (Lauraceae). *Dionéa C. Rodrigues* (FCFRP-USP), *Massayoshi Yoshida, Otto R. Gottlieb* (IQ-USP) and *Lúcia M. X. Lopes* (IQ-UNESP).

From barks and trunk wood of *Aniba ferrea* were isolated, besides (7S,8S,1'R)–3'–methoxy 3,4–methylenedioxy–4'–oxo– $\Delta^{1,3,5,3',5',8'}$–8.1',7.0.6'– neolignan (burchellin); (7S,8S,1'S,3'S) –3',5'–dimethoxy–3,4–methylenedioxy–4'–oxo–$\Delta^{1,3,5,5',8'}$–8.1',7.0.6'– neolignan (canellin B) and *rel*–(7R,8S,1'R,2'S)–2'–hydroxy–5–methoxy –3,4–methylendioxy–3'–oxo–$\Delta^{1,3,5,4',8'}$–8.1',7.0.2'–neolignan (ferrearin A). This communication reports for first time the occurrence of burchellin and canellin B in *Aniba ferrea*.

PN-06 Cyanogenetic Glucoside in Annona Crassiflora,Mart. Seeds Can Explain the Folk Medicinal Use of the Roasted Seeds. *Alaíde Braga de Oliveira, Lúcia Pinheiro Santos* and *Maria Amélia Diamantino Boaventura* (Departamento de Química – ICEX – UFMG)

As part of a program to study folk medicinal plants with activity against snakes bites, an ethanol extract of seeds of *Annona crassiflora*,Mart., was investigated. This extract showed a dose–dependent inhibitory effect on agonist(acetylcholine, histamine and bradykinin) induced contractions of isoled guinea pig ileum. Chemical analysis of the ethanol extract have led to the isolation of several alkaloids, of a cyanogenetic glucoside,(S)–proteacin, and a linear acetogenin. The presence of a cyanogenetic glucoside can explain, in part, the folk use of roasted seeds.

PN-09 A Monoterpenic Furocumarin From *Dorstenia Cayapiaa* Vell. *Wagner Vilegas, Gilberto L. Pozetti* (IQAr. – UNESP), *Janete H. Y. Vilegas* (Rhodia S/A – Centro de Pesquisas de Paulínia), *Gabriel Llabres* and *Marcel Baiwir* (Inst. de Physique – B5 – Liege Belgica).

In order to know the chemical composition of the *Dorstenia* genus (Moraceae), we have begun the study of *D.cayapiaa* vell. The phytochemical investigation of the rhyzomes of this plant afforded Bergapten (1) and the furocumarin (2). The isolation of both was performed by means of the conventional chromatographyc techniques. The structural elucidation of (2) was obtained by means of 1D and 2D NMR, IR, UV and MS. (FUNDUNESP, FAPESP, Fonds national de la Recherche Scientifique of Belgium).

PN-10 Quercetin From Vismia Species. *Tanus Jorge Nagem* and *Vera Lúcia Alves*, (Departamento de Química, Universidade Federal de Minas Gerais, Belo Horizonte, Brazil).

Chemical investigation of *Vismia magnoliaefolia* gave besides aliphatic compounds as fiedelin, dammaradienol, sitosterol, betulinic acid, six aromatic compounds known as vismiaquinone, madagascin, bianthrone, vismiaquinone B, 1,7–dihydroxyxanthone and quercetin a flavonol isolated before from several plants but never found before in the genus *VISMIA*.

PN-11 Flavan Phenylpropanoids From *Ocotea Porosa* (Lauraceae). *Jorge M. David, Massayoshi Yoshida* and *Otto R. Gottlieb* (Instituto de Química, USP).

South American genus *Ocotea* is well known, through detailed studies, as source of neolignans and alkaloids. However, in contradistriction, few chemical examination has been made with polar constituents. Ethanol extract of barks of *Ocotea porosa* afforded, besides epicatechin, two new flavan phenylpropanoids similar to cinchonains, previously isolated from *Cinchona succimbra* (Rubiaceae) and *Phyllocladus trichomanoides* (Podocarpaceae).

PN-12 Recents Progress in the Phytochemical Studies of *Ocotea Porosa*. *Jorge Maurício David, Massayoshi Yoshida* and *Otto Richard Gottlieb* (Instituto de Química – USP).

Nine neolignans were isolated from barks of *Ocotea porosa*, collected around vicinity of Santa Maria (RS). Among them, six neolignans are new, and belong to benzofuran and *seco*–benzofuran types.

PN-13 Biflavonoid Isolated From *Ouratea Hexasperma*. *Mário G. de Carvalho, R. Braz Filho, Victor Rumjanek* and *D.C. Sobrinho* (Departamento de Química, Universidade Federal Rural do Rio de Janeiro).

The structure proposed for a biflavonoid isolated from *Ouratea hexasperma*, family Ocnaceae, was revised on the basis of two dimensional NMR experiments.

PN-14 Flavonoid Glycoside Isolated From *Stygmaphyllon Tomentosum*. *Joaquim B. de Toledo* (Departamento de Química, Universidade Federal de Ouro Preto), *Paulo Cesar de Abreu Aragão, Victor M. Rumjanek, Anselmo A. Morais* and *R. Braz Filho* (Departamento de Química, Universidade Federal Rural do Rio de Janeiro).

The flavonoid glycoside isolated from the ethanolic extract of the leaves of *Stygmaphyllon tomentosum*, family Malpighiaceae, was characterized as 3,7–di–O–α–L–rhamnopyranosyl–4',5–dihydroxyflavonol through spectral data analysis, mainly ^{1}H and ^{13}C NMR.

PN-15 Alkaloids From *Rollinia Exsucca*: *Joaquim de Carvalho Bayma, Heriberto Rodrigues Bitencourt* (Departamento de Química – Universidade Federal do Pará) and *Alaíde Braga de Oliveira* (Departamento de Química – Instituto de Ciências Exatas – Universidade Federal de Minas Gerais).

One oxoaporphine alkaloid, liriodenine and two tetrahydroprotoberberine alkaloids, scoulerine and corytenchine, were isolated from the bark of *Rollinia exsucca*.The structure elucidation of alkaloids was achieved through interpretation of ^{1}H–NMR, ultra-violet, infrared and mass spectra.

PN-16 Isoquinoline Alkaloids in *Duguetia Pycnastera*. *Ana Júlia de Aquino Silveira, Joaquim de Carvalho Bayma* (Department of Chemistry, Universidade Federal do Pará) & *Alaíde Braga de Oliveira* (Department of Chemistry, Universidade Federal de Minas Gerais).

Phytochemical studies of the *D. pycnastera* has led to the isolation of the O-methylmoschatoline, lysicamine, and stephadiolamine β–N–oxide. The identity of the compounds was inferred on the basis of spectral data.

PN-17 Chemical Constituents of *Vanyllosmopsis Arborea*. Baker. *Maria Elisa Oliveira Matos, Mirian Pinheiro de Souza, Maria Iracema Lacerda Machado* and *Edilberto Rocha Silveira*. (Departamento de Química Orgânica e Inorgânica da Universidade Federal do Ceará).

The wood of *V. arborea* furnishes an essential oil with high content of α–bisabolol. From the hexanic and ethanolic extract were also isolated αbisabolol and a hydroxylated derivative of this sesquiterpene respectivelly. In continuation of the study of the mentioned specie were obtained lupeol from the cloroformic extract of the bark and the lignan syringaresinol as an acetylated derivative in the ethanolic extract of the wood. The structural propositions for these compounds were supported by spectral analysis of iv, ^{1}H–nmr and ^{13}C including APT and DEPT. (FINEP, CNPq).

PN-18 Chemical Composition ofr *Vernonia Scaber*Pers. *Maria Iracema Lacerda Machado, Maria Elisa Oliveira Matos, Kerusa Gurgel Tamiana*, (Departamento de Química Orgânica e Inorgânica da Universidade Federal do Ceará) and *Raimundo Braz Filho* (Departamento de Química da Universidade Federal Rural do Rio de Janeiro).

In this work we report the preliminary examination of *Vernonia* *scaber*. In the genus is common the presence of lactones sesquiterpenes which show antitumoral properties and flavonoids and triterpenes. The present study led to the isolation of lupeol and 3',4',5,7 – tetrahydroxy–3– methoxyflavone. The structural assignments were based on physical and spectral data on acetylated derivatives. (FINEP, CNPq).

PN-19 Tephrosia Candida Rotenoids. *César Cornélio Andrei, Rosana Aparecida Giacomini* (Chem. Deptº/FUEL) and *Raimundo Braz Filho* (Chem. Deptº/UFRRJ).

From the roots of *Teprhosia candida* (Fabaceae), eight rotenoids were isolated: 6a, 12a–dehydrotoxicarol in mixture with vilosol, 6a, 12a–dehydrorotenone in mixture with 6a, 12a–dehydrodeguelin, a 6a, 12a–dehydrotoxicarol isomer with dimethylchromene ring on linear position with the D ring, toxicarol, degueline and tephrosin. In vew of the rotenoid presences this plant showed inseticide potential against the beatle *Diabrotica speciosa* in biological tests.

PN-20 Neolignan Tetrahydrofuran-Type From *Aniba Puchury-Minor* (Lauraceae). *M. de F. da Silva* (CCEN–UFPa), J.G.S. Maia, (DEL-MPEG), *M. Yoshida* and *O. R. Gottlieb* (IQ–USP).

From the hexanic extract of trunk wood of *Aniba puchury–minor*, called "casca–preciosa–arana", colected on Serra de Carajás (PA), in mining area, were, isolated isoelemicin, *cis*– and *trans*– asarone (as a mixture), methyleugenol, methylisoeugenol, 2,4,5–trimethoxibenzaldehyde, sitosterol and a tetrahydrofuran type neolignan identified as magnosalicin, previously found in *Magnolia salicifolia* (Magnoliaceae). Chemosystematics interactions occurs in this two families. (CNPq).

PN-21 New Bicyclo–Octanoid Neolignans From *Aniba Squarensis* (Lauraceae). *G. M. S. P. Guilhon, J. G. S. Maia,* (CCEN-UFPA), *M. Yoshida* and *O. R. Gottlieb* (IQ-USP).

In previous communication [Guilhon et al.Cien.Cult. 41 (Supl.),522(1989)], it was reported the isolation of two bicyclo–octanoid neolignans from the hexanic extract of the trunk wood of *A. squarensis*. Now, from the same extract were identified six others bicyclo–octanoid neolignans. Four of them were isolated previously, while the other two are new compounds but with the same structural–type of the neolignans detected in *Ocotea veraguensis* [Khan et al. Phyt.: *26*, 1155 (1987)] and *Aniba* sp [Fernandes et al.: Pyt. *15*, 1033 (1976)], except on oxigenation pattern of the aromatic ring.

PN-22 Tephrosia Tunicata Flavonoids. *César Cornélio Andrei, Mary Leiva de Faria* (Chem. Dept./FUEL) and *Raimundo Braz Filho* (Chem. Dept./UFRRJ).

The hexane extract of the roots of *Tephrosia tunicata* showed the presence of four flavonoid compounds. Two flavones, candidine and 5–hydroxy–3–methoxy–2",2"–dimethylpyrano–(5",6":7,8) flavone, the flavonone obovatine and 5–methoxy–2",2"–dimethylpyrano–(5",6":7,8) flavonol. The structure of the compounds were determined by NMR, IR and MS spectral data.

PN-23 Isolation and Characterization of Tetracyclic Diterpenoids From Xylopia Frutescens. *B. H. de Oliveira* (Dep. Química, Universidade Estadual de Maringá, PR, Brazil), *A. E. G. Sant'Anna* (Dep. Química, Universidade Federal de Alagoas, AL, Brazil) and *J. R. Hanson* (School of Chemistry, University of Sussex, UK).

Two tetracyclic diterpenoids have been isolated from the bark of Xylopia frutescens hitherto not described for that species. The first, less polar compound, was identified as kaur–16–en–19–oic acid. It was compared with authentic sample and gave consistent spectrometric data (IR, NMR). The second and more polar compound was identified as ent–16–hydroxy–kaur–19–oic acid by comparison with NMR data from the first compound. The stereochemistry at C16 was determined by NMR experiments (Nuclear Overhauser Effect Difference Spectra).

PN-24 Selective Enolization of Methyl Populnonate. *José Rego de Souza* and *Grácia Divina de Fátima Silva* (Departamento de Química, ICEx – Universidade Federal de Minas Gerais; 31.270 – Belo Horizonte (MG), Brazil).

To an ice-cold suspension of methyl populnonate (3–oxofriedelan–20–α–methylcarboxylate) from *Austroplenckia populnea* Reiss in acetic anhydride, were added drops of percloric acid. After stirring for 20 min., the reaction mixture was neutralized (pH 6) with 5% NaOH and filtered. Recristalization of the residue in $CHCl_3$/MeOH gave only 3–acetoxy–friedelan–3–en–20α–methylcarboxylate, m.p. 283–284°.

PN-25 X-Ray Crystal Structure of Epikatonic Acid. *José Rego de Souza, Grácia Divina de Fátima Silva* (Departamento de Química, ICEx – Universidade Federal de Minas Gerais; 31.270 Belo Horizonte (MG), Brazil), *Yvonne Primerano Mascarenhas* and *André Barros Cota* (Instituto de Física e Química de São Carlos – USP; 13560 – São Carlos (SP), Brazil).

The structure of epikatonic acid (3β–hydroxy–olean–12–en–20α–carboxylic acid) from *Austroplenkia populnea* Reiss. has been established from spectral data. The single–crystal X–ray analysis showed the presence of a dimer with hydrogen bonds from the hydroxyl group at C–3 and the carbonyl group at C–20.

PN-26 Composition of the Roots of *Baccharis dracunculifolia*. *Cleusa Conceição da Silva* and *Anita J. Marsaioli* (Instituto de Química, UNICAMP, CP 6154, 13081 Campinas, SP, Brasil).

Baccharis dracunculifolia is responsible for the essential oil of Vassoura, which is used in perfumery industries. We have recently studied this oil and identified 43 components, many of them are sesquiterpene alcohols possessing the cadinane and aromadendrane skeletons. Motivated by this plant industrial application we began to study the composition of its roots. Biological tests will be realized at SANOFI (France). Preliminary results reveal the presence of dihydromatricaria ester and of several other polyacetylenes as well as of triterpenes.

PN-28 Terpenoids from *Ocotea Corymbosa* (Meissn.) Mez. (Lauraceae). *Juceni P. Chávez* (FFAR-UFBA); *Walmir S. Garcez* (DQI, CCET, UFMS); *Massayoshi Yoshida; Otto R. Gottlieb* (IQ–USP).

Hexane and dichloromethane extracts of barks of *O. corymbosa* yielded by chromatographic fractionation, besides 7–hydroxy–calamenene, two new compounds: a simmetric dimer formed by linkage between two 7–hydroxy–calamenene unities and assimetric dimer obtained by linkage between 7– and 8–hydroxy–calamenene.

PN-29 Identification of Keto–Triterpenes From *Scoparia Dulcis* (Scrophulariaceae). *Roberto S. Gallegos Olea* and *Nidia F. Roque* (Institut of Chemistry, USP) *Sonia M. Freire* and *Luce M. B. Torres* (Departament of Chemistry, UFMa).

Glutinol proved to be the active substance of the aqueous and ethanolic extracts of *Scoparia dulcis* L., known as vassourinha (S. M. Freire et all, Brazilian-Sino Symposium on Chemistry and Pharmacology of Natural Products, Rio de Janeiro, december 1989). Besides this triterpene; betulinic acid, dulcioic acid, ifflaionic acid, friedelin and αamyrin were previously described to occur in the plant (Phytochemistry *1981*, 20 (1), 171–3). A mixture of ca. seven constituents of keto–triterpenes was separated from a less polar fraction of the ethanolic extract. Lupenone, boehmerone, α–amyrenone e β–amyrenone were identified in this mixture by ^{13}C NMR techniques. A reassignment of the ^{13}C chemical shifts of glutinol is proposed. (CNPq, FINEP).

PN-30 Aliphatic Compounds From *Cuphea Aperta* Koehne. *Dirceu Martins* and *Nidia Franca Roque* (Instituto de Química, USP, São Paulo, Brazil).

The species *Cuphea aperta* Koehne (Lythraceae), known as sete sangrias, have been used in the brazilian folk medicine, against hypertension. In a previous communication we have related the presence of Kaempferol, quercetin, gallic acid, protocatechuic acid and methyl gallate in the aqueous extract of the whole plant. The hexane extract afforded mixtures of hydrocarbons ($C_{24}H_{50}$, $C_{25}H_{52}$ and $C_{27}H_{56}$), steroids (β–sitosterol, stigmasterol and stigmast–4–en–3–one, stigmasta–4,22–dien–3–one and 24–methyl–cholest–4–en–3–one) and triterpenoids (α–amyrin, β–amyrin and lupeol). NMR ^{1}H, NMR ^{13}C and MS were used in the identification of the mixture constituents. (CNPq).

PN-31 Triterpenoids of *Lafoensia Densiflora* Pohl (Lythraceae). *Fernanda Rodrigues Garcez* and *Walmir Silva Garcez* (Depto. de Química, CCET, Universidade Federal de Mato Grosso do Sul).

The triterpenoids betulin, betulinic acid, oleanolic acid acetate, 3β–acetoxi–11α,12α–epoxioleanan–28,13β–olide and 3β–acetoxi–12α–hidroxioleanan–28,13β–olide, were isolated from the bark of *Lafoensia densiflora*, in addition to β–sitosterol, hexacosanoic acid and manitol hexacetate (the last one after acetylation). The structural elucidation of these compounds was based on spectroscopic data. The triterpenoids isolated so far have not been reported in other species of the Lythraceae family. (CPQ/PROPP–UFMS).

PN-32 The Essencial Oil of *Spilanthes Acmella* Murr. *Telma Leda Gomes Lemos, Otília Deusdênia Loiola Pessoa, Francisco José Abreu Matos, José Wilson de Alencar, Afrânio Aragão Craveiro* (Laboratório de Produtos Naturais – Depto. de Química Orgânica e Inorgânica – Universidade Federal do Ceará).

Spilanthes acmella populary known as "Agrião" it's a herb cultivated in humid tropical areas. It's utilization as folk medicine is very diversified and includes use against: toothache, gingivits, expectorant, and mouth disease. Aereal parts of the plant were submitted to steam distillation to obtain the oil. GC–MS of the oil allowed to identify the major constituints: β–caryophyllene, thymol, γ– muurolene, β–phelandrene, cis–ocimene, β–pinene, α–humulene, myrcene, γ–cadinene, α–copaene, sabinene, δelemene and estragole. (FINEP, CNPq, CAPES).

PN-33 Antimicrobial Activity of Essential Oils From Brazilian Plants. *Telma Leda G. Lemos, Francisco José A. Matos, Afrânio A. Craveiro* (Laboratório de Produtos Naturais – Depto. Química Orgânica e Inorgânica – Universidade Federal do Ceará) *Rita de Cássia S. B. Barbosa* and *Edeltrudes de O. Lima* (Universidade Federal da Paraíba – Laboratório de Tecnologia Farmacêutica).

Essential oils from Brazilian Northeastern plants *(Croton mucronifolius, Lippia gracillis, Croton Zehntneri, Xilopia sericea, Cimbopogon naruds, Cedrela odorata, Lippia microphylla, Cinamomun zeilanicum, Croton triangulares)*, were screened against several microorganisms such as: *E. coli, S. aureus, S. epidermis, B. cereus, C. albicans, C. tropicalis*. GC–MS analysis of the oils allowed the identification of individual components and in several examples the pharmacological activity is in agreement with the action of the major substances presents in the oils.

Among these oils several are already used by rural population in folk medicine. (FINEP - CNPq).

PN-34 Phyllacanthin, a New Bis-Nor-Diterpene From *Cnidoscolus Phyllacanthus*. *Telma Leda G. Lemos, Edilberto R. Silveira, Mailcar F. de Oliveira* (Depto. de Química Orgânica e Inorgânica – Universidade Federal do Ceará), *Raimundo Braz Filho* (Depto. de Química Orgânica da UFRJ).

Cnidoscolus phyllacantus (Euphorbiaceae), popularly known as "favela preta", is a shrub sometimes a small tree well dispersed through the norteastern "caatinga". Chromatografic analysis of the hexane extratives from the trunk bark yielded a bis–norditerpene I, methyl–7–oxo–(16,17)–bis–nor–isopisiferin, (Phytochemistry 1985, *24,* 1545–5). We are now reporting the isolation of a similar diterpene II, (16,17)–bis–nor–isopisiferin. Structure determination has been accomplished by two dimensional NMR techniques as COSY and HETCOR. As far as we known I and II are the first examples of a new class of terpenoides we named "phyllacanthane". (Supported by: CNPq, CAPES).

PN-35 Copaifera Cearensis Oil Constituents. *Waldenir F. Braga, Octávio A. C. Antunes* and *Angelo C. Pinto* (IQ/UFRJ).

Copaifera cearensis oil is a mixture of organic compounds having anti-inflamatory and analgesic properties. HRGC–MS analysis of this oil showed the presence of sesquiterpene hydrocarbons and alcohols, and diterpenes. The two major diterpenes present in this oil showed an MW of m/z 316 ($C_{20}H_{28}O_3$) and m/z ($C_{20}H_{30}O_3$). HRGC–IR showed the presence of C=O and –OH groups. These two compounds, upon CH_2N_2 esterification, yielded two compounds with MW of m/z 330 and 332. Based on the similarities between the mass spectra of these acids with the labdane fragmentation pattern (m/z 221 and 223), and the presence of a furan ring (m/z 81 and 96), it was possible to propose 15,16–epoxy–13(16), 14–labdadien–19–carboxilic and 15,16–epoxy–8(17), 13(16),14–labdatrien–19–carboxilic acids as the structures for these compounds.

PN-36 Relative Configuration of a New *Nor*–Triterpene From *Lophanthera Lactescens*. *Heber dos S. Abreu, R. Braz-Filho* (Departamento de Química, Universidade Federal Rural do Rio de Janeiro), *Hugo E. Gottlieb* (Department of Chemistry, Bar–Illan University) and *James N. Shoolery* (NMR Applications Laboratory, Varian Associates).

The relative configuration of a new *seco-nor*–triterpene isolated from *Lophantera lactescens,* family Malpighiaceae, was decided on the basis of results obtained with the aid of NOE experiments.

PN-37 Essencial Oils of the Family Leguminosae and Compositae of the Sergipe State. *Wellington Oliveira dos Santos, Péricles Barreto Alves* (Departamento de Química da Universidade Federal de Sergipe, Campus Universitário, 49100 – Sergipe – Brasil) *Afrânio Aragão Craveiro* and *José Wilson de Alencar* (Departamento de Química Orgânica e Inorgânica da Universidade Federal do Ceará, Campus Pici – 60.000 – Fortaleza – Brasil).

In this work, will be continued the project "Essencial Oils of Plants of the Sergipe State". The primary objective of this study is to identify the chemical components in each oil. Two species of Compositae family *(Vernonia sp)* and Leguminosae *(Rhynchosia mínima)* were analised, using G.L.C/ME and computer. In *Vernonia sp* was detected α – pinene, β – pinene, p – cimene, 1,8 – cineol, Terpinene – 4 – ol, α – copaene, β – cariofilene, α – humulene, α – muurolene and Δ – cadinene, and in the *Rhyncosia mínima*, was detected, limonene, β – cariofilene, α – humulene, β – cubebene and Δ – cadinene.

PN-38 Two New Essential Oils From Verbenaceae Cultivated in Brazilian Northeastern. *Afrânio Aragão Craveiro, José Wilson de Alencar, Francisco José de Abreu Matos* and *Maria Iracema Lacerda Machado* (Universidade Federal do Ceará – Laboratório de Produtos Naturais).

The essential oils composition of two Verbenaceae cultivated in Brazilian Northeastern is described: *Lippia aff. geminata* contains: limonene, alpha–terpinolene, citronellal, geranial and geranil acetate as major constituents besides 58 minor constituents and *Vitex agnus-castus* contains: estragol, delta–3–carene methyl–eugenol and beta–cariophyllene and other 44 terpenes in minor quantities. The oils were analised by GLC/MS and identified with aid of Kovats, indices confirmed by visual inspection of mass spectra. (FINEP, CNPq, CEME).

PN-39 New Essential Oils From Brazilian Northeastern Labiatae *(Aeolanthus suavis* and *Mentha* X *villosa)*. *Afrânio Aragão Craveiro, José Wilson de Alencar, Francisco José de Abreu Matos, Maria Iracema Lacerda Machado* and *Francisco José Queiroz Monte* (Laboratório de Produtos Naturais, Universidade Federal do Ceará).

The Essential Oil Composition of two Brazilian Northeastern Labiatae is described: *Aeolanthus suavis* (I) and *Mentha* X *villosa* (II). The specie *Mentha* X *villosa* shows amebiase and giardiase activities. The principals constituents of essential oils of two species are the following: I contains linalool and 14 minor components and II contains limonene, diosfenoleno?, beta–cariophyllene, gama–muurolene and others 31 minor constituents. The identifications were done by GLC/MS with the aid of kovats indices and confirmed by visual inspection of the MS Spectra. Work is in progress to identify active substance (s). (FINEP, CNPq, CEME).

PN-40 Triterpenes From *Palicourea Rigida*. *Vanderlan da S. Bolzani, Ligia M. V. Trevisan* (Inst. de Química, UNESP – Araraquara) and *Maria Claudia M. Young* (Inst. de Botânica – São Paulo).

Continuing our studies on Rubiaceae from "cerrado" regions, we have examined the species *Palicourea rígida.* In this communication we report the isolation and identification from the leaves of the 3β–Gl–sitosterol, stigmasterol, lupeol, α–amirin, β–amirin, friedelin and of two triterpenes whose structures are being determined.

PN-41 Chemical Study of *Vellozia Candida Fruits*. *Rosângela de Almeida Epifanio* and *Angelo C. Pinto* (Instituto de Química – Universidade Federal do Rio de Janeiro).

The hexanic extracts of the seeds and pericarp of *V. candida* were submited to an HRGC-MS-C analysis. This preliminary study shows that the seeds extract contains fatty acids (15:0, 16:0 and 18:0), alkanes (C_{23}, C_{25}, C_{27}, C_{29} and C_{31}) and the steroid β–sitosterol. The pericarp extract contains palmitic acid, β–sitosterol and the n–alkanes C_{23}, C_{25}, C_{26}, C_{27}, C_{28}, C_{29}, C_{31} and C_{33}. This latter extract still has an isomeric mixture of sesquiterpenes. Probably the caracteristic fragance of the fruits is due the presence of these compounds.

PN-42 *Vellozia Declinans Hydrocarbons*. *Rosângela de Almeida Epifanio* and *Angelo C. Pinto* (Instituto de Química – Universidade Federal do Rio de Janeiro).

The more apolar fraction of the hexanic extract of roots, stems and leaf sheaths of *Vellozia declinans* were analysed by HRGC-MS-C. It showed that this fraction contains n–alkanes (C_{21} to C_{34}), isoprenoids (C_{14} to C_{16}), n–alkylcyclohexanes(C_{13} to C_{15}) and three aromatic cleistanthanes (6,8,11,13–cleistanthatetraene; 8,11,13–cleistanthatriene and 6,8,11,13,15–cleistanthapentaene). This is the first report on these diterpenes in *V. declinans.*

PN-43 Characterization of Triterpenoids From Species of Polypodiaceae. *M. L. Patitucci* (Departamento de Química – Universidade Federal do Paraná), *A. C. Pinto* and *J. N. Cardoso* (Instituto de Química – Universidade Federal do Rio de Janeiro).

From dry rhyzomes of six Brazilian species of Polypodiaceae were characterized the following pentacyclic triterpenoids: olean–12–ene, fern–7–ene, fern–8–ene, fern–9(11)–ene, hopene and a fernene acetate. These triterpenoids were characterized by HRGC, HRGC–MS–C, Mass Fragmentography and application of the principles of the organic geochemist's approach.

PN-44 Crotonepetin, a New Casbane Diterpene From *Croton nepetaefolius*. *Edilberto R. Silveira, Selene M. Morais* (Lab. de Produtos Naturais, Deptº de Química Orgânica e Inorgânica – UFC), *Regina Helena A. Santos, Maria Sivânia L. Marques* (Instituto de Física e Química – USP – São Carlos).

The *Croton* genus (Euphorbiaceae) is well represented in the characteristic vegetation of brasilian northeast flora named "caatinga". Several *Croton* species have folk medicinal uses and the literature documents a series of physiological activities related to either *Croton* plants or substances isolated from them. From *Croton nepetaefolius* collected in Jacobina-BA, we have isolated a new casbane diterpene named *crotonepetin,* 4–hidroxy–5–oxo–casbene (J. Chem. Soc. Perkin Trans. I 1981, 2666-69). The structural proposition based on spectroscopic analysis of the acetyl derivative has been confirmed by X–ray crystallography. (CNPq/FINEP).

PN-45 Crotomicansin, a New Crotofolane From *Croton micans*. *Edilberto R. Silveira, Mary Anne S. Lima* (Lab. de Produtos Naturais, Deptº de Química Orgânica e Inorgânica – UFC), *Raimundo Braz Filho* (Deptº de Química – UFRRJ), *Charles D. Hufford* (Deptº of Pharmacognosy University of Mississippi – U.S.A.), *Regina Helena A. Santos, Maria Silvania L. Marques* (Instituto de Física e Química – USP São Carlos).

Due to its occurence and abundance the *Croton* genus is one of the most important of the Euphorbiaceae family represented at the northeastern "caatinga". Chromatographic analysis of the hexane extractives from roots of *Croton micans* collected in Jacobina - BA yielded cuparene and a unstable diterpene named crotomicansin, 6,10–7–oxo–crotoxide A(J. Org. Chem. 1989; *54*, 1654–7). Structure determination utilizing modern NMR spectroscopic techniques such as HECTOR, COSY, DEPT and two dimensional INADEQUATE has been unambiguosly confirmed through X–ray crystallography analysis. (FINEP/CNPq).

PN-46 Chemical Constituints of *Pilocarpus Spicatus* Saint Hillaire (Rutaceae). *Edilberto Rocha Silveira, Manoel Andrade Neto,* (Deptº de Química Orgânica e Inorgânica da Universidade Federal do Ceará), *Raimundo Braz Filho,* (Deptº de Química da Universidade Federal Rural do Rio de Janeiro).

In order to find new substances of therapeutical interesting properties, we have collected leaves of *Pilocarpus spicatus* and proceeded the extraction with ethanol and hexane in Soxlet. Two triterpenes were isolated from the hexane extract. Both belonging to the damarane type, bearing unknown carbon skeletons in the side chain and containing thirty-three carbon atoms. The structural determination was done with I.R.E.M. and specially ^{1}H and ^{13}C NMR spectroscopical analyses, including the most recent techniques such as APT, DEPT and HETCOR. (CAPES, CNPq and FINEP).

PN-47 Essential Oils From Lemongrass, Palmarosa, Citronella and Mentha Cultivated in The Semi-Arid Region of Brazil. *Afrânio Aragão Craveiro, José Wilson de Alencar, Francisco José de Abreu Matos, Maria Iracema Lacerda Machado* and *Francisco José Queiroz Monte*. (Universidade Federal do Ceará – Laboratório de Produtos Naturais).

The essential oils from *Cymbopogon citratus* (I), *Cymbopogon winterianus* (III), *Cymbopogon nardus* (II) and *Mentha arvensis* (IV) cultivated in the garden of Medicinal Plants in the Laboratory of Natural Products of UFC were compared with the commercial samples of the same oils. The oils presented almost the same compositions, showing as major products the following compounds: I – myrcene and citral, II – geranial, III – citronellal, however in the case of IV *(Mentha arvensis)* the major compound Menthol is present in the yield of 71%, whith high content in this valuable monoterpene, making atractive study of the Mentha production in Brazilian Northeast. (CNPq/FINEP/MCT).

PN-48 A New Source of Nerolidol – *Zantoxylon gardneri*. *Afrânio Aragão Craveiro, José Wilson de Alencar, Francisco José de Abreu Matos* and *Maria Iracema Lacerda Machado* (Laboratório de Produtos Naturais – Departamento de Química Orgânica e Inorgânica – Universidade Federal do Ceará).

Nerolidol is found in low concentrations in essential oil of belonging to the plants genus Citrus (Rutaceae) as well in volatiles of *Myroloxilon balsamun, Melaleuca* spp and *Cananga odorata*. The essential oil of *Zantoxylon gardneri* (Rutaceae), (F1073) analysed by GC/MS showed nerolidol in high concent.as the chief constituent (90%).

PN-49 New Essential Oils From Brazilian Northeastern Compositae *(Eupatorium ballotaefolium* and *Trichogonia* sp). *Afrânio Aragão Craveiro, José Wilson de Alencar, Francisco José de Abreu Matos, Mª Iracema Lacerda Machado* and *Edilberto Rocha Silveira* (Laboratório de Produtos Naturais, Universidade Federal do Ceará).

The essential oil composition of two Brazilian Northeastern is described: *Eupatorium ballotaefolium* (I) and *Trichogonia* sp (II). (I) contains: metil carvacrol, p–cimene, limonene, beta-cariophyllene and others 17 minor components. II contains: alpha-pinene, p-cimene, limonene, beta-elemene, beta-cariophyllene, alpha-humulene and others 36 minor components. The identifications were done by GLC/MS with the aid of Kovats indices and confirmed by visual inspection of the MS Spectra. (FINEP, CNPq e CEME).

PN-50 New Essential Oil From Brazilian Northeast Plant – *Croton micans*. *Afrânio Aragão Craveiro, José Wilson de Alencar, Francisco José de Abreu Matos, Mª Iracema Lacerda Machado* and *Edilberto Rocha Silveira* (Univ. Fed. do Ceará, Laboratório de Prod. Naturais).

The essential oil composition of *Croton micans* leaves contains canfene hydrate as major constituent besides minor quantities of sabinene, beta-caryophyllene, and gama-elemene and eleven other terpenes detected by GLC/MS analysis. The identification were done with aid of Kovats indices and confirmed by visual inspection of the mass spectra. (FINEP, CNPq & CEME).

PN-51 Isolation of the Triacontanoic Acid From *Eucalyptus Grandis* Milled Wood Lignin. *S. A. L. Morais* and *D. Piló-Veloso* (Chemistry Department – UFMG – Belo Horizonte – Brazil) and *E. A. Nascimento* (Chemistry Department – UFU – Uberlândia – MG – Brazil).

Eucalyptus grandis milled wood lignin (MWL) obtained according to Björkmann and adaptations by Bland and Menshun presents signaes due to high shielded protons in the NMR spectrum which are not characteristic of lignins. Carbon 13 NMR spectrum presents signals due to high shielded carbons which are not caracteristic of lignins too. Other types of *Eucalyptus grandis* lignin (hydrolytic, Klason, Brauns) present the same resonance signals. After exhaustive extraction of the MWL with chloroform, a compound was isolated whose proton and carbon 13 NMR, IR and mass spectra, and fusion point correspond closely with those of triacontanoic acid ($C_{29}H_{59}COOH$).

PN-52 Chemical Study of Oils Extracted of Fruits Pataua Native Plants of Amazon Region. *Maria Helena da Silva Bentes, Harry Serruya, Márcia Gorette Lima da Silva* (Chemical of Natural Products/CCEN/UFPA).

The authors in another before communication (SBPC, 1981) notice the great likeness of pataua *(Jessenia bataua,* Mart.) oil pulp with oliva oil. This led us to carry out an investigation of seventy (70) samples of this fruits for to selete seends for his rational plantation. It were determined humidity, the compositions of fruits, oil yield and his fatty acid compositions. The results of analyses of the kernel, 59% to 72% and the shell+pulp, 28% to 41% of the fruits was the folowings: humidity 31,1% to 40,4% and 36.1% to 50.5%; oil yield 0.01% to 0.47% and 2,3% to 15,0% and the fatty acid in majority proportion was oleic, 52.5 to 79,6 and palmiltoleic, 10.8% to 38.9%.

PN-53 Chemical Study of Green Vegetables From Para State. *Maria Helena da Silva Bentes, Harry Serruya,* (Chemical of Natural Products/CCEN/UFPA), *Maria Dulcimar de Brito Silva* (QPN/UFPA) and *Davi de Jesus Oliveira* (UFPA).

The results of chemical analysis of thirteen (13) samples of green vegetables cultivated near Belem city (Para) and used like foods are presented. The standard methods were changed and improved for to work with samples "in natura" and with small quantities. It is mentioned, also, its medicinal use.

PN-54 Bitter Glicosides of the Vatairea Macrocarpa Ducke(Amargoso). *Maria Goretti de Vasconcelos Silva, Francisco José de Abreu Matos* and *José Paz Parente* (Laboratório de Produtos Naturais – Universidade Federal do Ceará).

The plant *Vatairea macrocarpa* Ducke, family leguminosae, known popularly "Amargoso" has a bitter wood from which was isolated three anthraquinones (M.G.V. Silva, F.J.A. Matos e J.P. Parente. Ciência e Cultura, 38(7) Suplem. 597, 1986) and two anthrones glicosides. The bitter constituent of the plant were isolated of the aqueous extract partitioned with ethyl acetate. The structural assigments were based on physical and spectral data. The compounds are no report in literature. (MCT/CNPq/FINEP).

PN-55 Utilization of Silicotungstic Acid as Alkaloid Detection Reagent. *S. R. Sargenti; J. H. Y. Vilegas; M. Garcia; V. L. B. Campos; A. M. Favare* (Centro de Pesquisas de Paulínia – Rhodia S.A.).

We have developed an alkaloid screening procedure using silicotungstic acid, which forms a reversible complex with alkaloids, that can be destroyed and used for TLC analysis. This reagent was evaluated against Dragendorff's, Reinecke's and Mayer's reagent for the detection of some secondary, tertiary and quarternary alkaloid standards. The silicotungstic acid appeared to be useful for the detection of all types of above alkaloids and can substitute the traditional detection reagents, especially the modified preparation silicotungstic acid/sulphuric acid.

PN-56 Fatty Analysis of the Sex Pheromone Glands of *Spodoptera latifascia* Walker (Lepidoptera: Noctuidae). *Agostinho Lelis Teixeira* and *João Sabino de Oliveira*. (Deptº Química, Univ. Fed. Viçosa).

The moth *Spodoptera latifascia* occurs on some economic plants such as soybeans, cotton, potato and gramineous species. Previously we have identified, from a the crude extracts of virgin female glands, two components of the sex pheromone of *S. latifascia* the (Z)–9,(E)–12–tetradecadienyl acetate and (Z)–9–tetradecenyl acetate. Female pheromone gland extracts of *S. latifascia* were analyzed for pheromone precursors. The acids were identified on the basis of capillary gas chromatography-mass spectrometry. The gland extracts contained large amounts of hexadecanoic acid, hexadecenoic acid, octadecanoic acid and octadecenoic acid.

PN-57 Xylopic Acid From *Xylopia Frutescens* Aubl. *Maria Amélia Diamantino Boaventura, Jacqueline Aparecida Takahashi* and *Alaíde Braga de Oliveira* (Departamento de Química – ICEX – UFMG).

Several species of the genus *Xylopia* (Annonaceae) are used in popular medicine as antidote against snake venons. In the present communication we related the study of the chemical composition of the green fruits of *Xylopia frutescens*. In the hexane and ethanolic extracts we observed the spontaneous precipitation of a solid that was recrystallised and identificated as 15– acetoxi–(–)–kaur–16–en–19–oic acid, or xylopic acid, isolated previously of *Xylopia aethiopica*. Antimicrobial activity of xylopic acid were related. Another substances with kaurane skeleton are already isolated of *Xylopia aromatica*.

ANALYTICAL CHEMISTRY (QA)

QA-01 Studies on the Absorption of Acids and Bases in Polymeric Containers. *Marcelo Sartoratto, Isabel Cristina Fontes Sales Jardim* and *Kenneth E. Collins* (Instituto de Química, Universidade Estadual de Campinas).

Small containers of PTFE, Delrin and polypropylene were subjected to tests of the absorption of organic and inorganic acids or bases using a semimicro titration technique. Organic acids and bases are significantly less absorbed than the commun inorganic ones. These results have implications on the use of acid cleaned or base cleaned containers for chemical reactions requiring careful control of acidity or basicity during the reaction.

QA-02 Ion Retention in Membrane Ultrafiltration: Preliminary Results. *I. L. Küchler, N. Miekeley* (Chemistry Department; Pontifical Catholic University; 22453 Rio de Janeiro; Brazil).

Experiments were performed in a AMICON 8400 stirred cell with several membranes, in particular AMICON YM2, to assess the effect of ion retention in ultrafiltration. This membrane, which has a nominal molecular weight (MW) cut-off of 1,000 dalton, showed a measurable retention for all the studied inorganic cations. In the presence of multivalent anions, such as sulphate or phosphate, cation retention was much higher than in the presence of monovalent anions (e.g. nitrate, chloride). The charge of the cation had also some influence on ion retention. For membranes with higher MW cut-off (⩾ 10,000 dalton) ion retention was negligible.

QA-04 Determination of Fluoride Ions in Toothpastes. *R. Colombara* and *J. Lichtig* - (Instituto de Química da Universidade de São Paulo, C.P. 20780, 01498, São Paulo, Brazil).

The determination of fluoride ions in toothpastes can be made by any methods, only if fluoride is free of interferents. This can be done by the previous solution distillation, and we miniaturized a simple apparatus of quantitative distillation of 1 ppm fluoride in 15 minutes. The ionic fluoride content of the toothpastes determined by us using eletrode and a spectrofotometric method is ca. 1000 ppm. Comparisons between these data and aqueous dispersion of toothpastes, measured without distillation, are made.

QA-05 A New Method for Xenotime Decomposition – *Gilson Zapparolli* and *Osvaldo Antonio Serra* (Departamento de Química da Faculdade de Filosofia, Ciências e Letras de Ribeirão Preto - SP.)

Xenotime is a mineral of heavy rare earth. It is a phosphate and Yttrium is the main element. With the development of new materials as superconductors, heavy rare earths became very important. Xenotime is decompose a by two methods: by fusion with carbonates at ~1000°C or by sulphuric acid at 200–250°C. (Dodezol, J.; Povondra, P. and Sulcek, Z. in "Decomposition Techniques in Inorganic Analysis" – London Iliffe Books Ldd 1968). We describe a simple method using a mixture of $NaOH/Na_2CO_3$ and heating at ~400°C, with total extraction of the rare earths. (CNPq–FINEP/PADCT/UMBRAL).

QA-06 Heavy Metals Preconcentration from Brines by Coprecipitation With Ammonium Pyrrolidenedithiocarbamate(APDC). *Haroldo Silveira Dórea; Antonio Celso Spinola Costa* (Instituto de Química. Universidade Federal da Bahia. Salvador. Bahia) *Rui Jesus Lorenzo Garcia* (GELAB/CEPED. Camaçarí. Bahia).

Traces of Cd, Co, Cu, Fe, Mo, Ni, V, and Zn in brines can be determined by coprecipitation with APDC, using cellulose powder (Cellulose PEI TLC Baker) or zinc pyrrolidenedithiocarbamate as carriers, at pH 4,6±0,1. After filtration with a membrane filter, the precipitate is digested with HNO_3 and the solution is diluted to 25,0 mL. Measurements are made simultaneously with an "Atom Comp 800 Jarrel Ash" ICP-AES. Recuperations better than 90% were obtained for all metals, in a concentration range of 25-250 ppb.

QA-07 Cation Exchange Chromatography of Cadmium and Nickel by Using Organic Solvent and Hydrocloric Acid. *Martha B. Penteado, Mauro Ramos* and *Miuaco K. Kuya* (Instituto de Química, Universidade de São Paulo, SP).

The separation of cadmium from nickel in a mixture of their hydroxides, originating in the processing of Ni/Cd battery wastes, is described. The mixture is dissolved in excess HC1 0,5M and percolated through a strong cationic resin equilibrated in HC1 0,5M. Nickel is selectively retained in this medium while cadmium freely passes through because of its tendency to form anionic chloro complexes. The difference in distribution coefficients of the metals is improved by using ethanol (or acetone)–hydrocloric acid 0,5M (50%, v:v) mixture. Succesfull separation is thus achieved.

QA-08 Further Studies on Extraction, Separation and Identification of Annato Pigments *(Bixa orellana L.)*. *Silvia M. B. de Souza, Asae S. Endo & Keiko Takashima* (Departamento de Química - Universidade Estadual de Londrina).

The most effective mixture for the extraction was arrived at 60% ethanol, sodium hydroxide 0.7%, vitamin E 0.525g%, pH 5 and two hours of shaking. By silica gel 60 columm chromatography five pigments of different polarities were obtained with sequential application of solvents of increasing polarity. The red pigment thus obtained were examined by TLC. The spot with Rf (0.03cm) was shown to have aborption bands at 427,449 and 476nm near to the Ones found by TAN and SEDERSTROM for beta-caroten.

QA-09 Phoshate Adsorption in Soils of A_1 Horizon of Piauí State. *Afonso Sena Gonçalves, José Carvalho Cordeiro, Maria do Socorro Lages Veras* and *Rosa Lídia Morais da Silva* (Universidade Federal).

A_1 horizons of twelve soils samples of Piauí State were submited to phosphate adsorption in aqueous solutions with concentrations between 1 to 6 g P/ g soil. The results showed that in general this adsorption is one half that adsorptions in B_{22} horizons.

QA-10 General Equation for Calculation of the Titration Curve of a Metal With a Ligand: Reaction With Coefficient Diferent of One. *Antonio Celso Spinola Costa; Adelaide Maria Mendonça Amorim* and *André Alay Esteves* (Instituto de Química, Universidade Federal da Bahia, Salvador, Bahia).

For the titration of a metal M with a ligand L, with a reaction $M + nL = ML_n$ the calculation of the titration curves $pL' = f(V)$ e $pM' = f(V)$ can be made with the general expressions:

$$V = V_0 \frac{|L'|}{C-|L'|} \times \left(1+\frac{C_0(\beta'_1+2\beta'_2|L'|+3\beta'_3|L'|^2+\ldots+n\beta'_n|L'|^{n-1})}{1+\beta'|L'|+\beta'_2|L'|^2+\ldots+\beta'_n|L'|^n}\right)$$

$$|M'| = \frac{C_0V_0}{V+V_0} \times \frac{1}{1+\beta'_1|L'|+\beta'_2|L'|^2+\ldots+\beta'_n|L'|^n}$$

where C_0 and V_0 are the initial concentration and volume of the metal solution; Ç is the concentration of Titrant and V are the additioned volumes of the titrant. The others symbols have the usual signification.

QA-11 Simulation On Precipitation Reactions. *Daniel Rodrigues de Moura* (Departamento de Química - ICEx - UFMG - Caixa Postal 702 - 30.161 - Belo Horizonte, MG, Brazil).

Mathematics equations were developed in order to simulate the precipitation's reactions which are based in the equilibrium:

$$wA + yB \rightleftharpoons AwBy\downarrow,\ Ksp$$

The method, without mathematics approximations, allows the complete monitoring of the process, acquainting the operator how to fix, as wished, the ideal conditions of work.

QA-12 Simple Graphical Data Input Tablet. *Paulo Sérgio de Souza* and *Célio Pasquini* (Instituto de Química - Universidade Estadual de Campinas - Campinas - São Paulo - Brasil).

A simple graphical data input tablet was constructed based on the change in the electrical resistance of two linear resistors (38 and 27 cm longer) placed perpendicularly on the vertical and horizontal directions. A dynamic ADC interface (11 bits resolution) was used to read the analogic voltage from a pair of operational amplifiers used to translate the change in the electrical resistance to a voltage. The interface also selects the channel (horizontal or vertical) to be read. Data are transferred to an Zenith IBM-AT clone microcomputer. The estimated resolution was 0.3 mm in the horizontal direction and 0. 2 mm in the vertical one.

QA-13 Automatic Device for Sample Introduction in Flow Injection Analysis. *Celio Pasquini* and *Lourival C. de Faria*. (Instituto de Química - Universidade Estadual de Campinas - Campinas - São Paulo - Brasil).

The device was assembled employing three 3-way sub-miniature Teflon valves (NResearch, 12 V, 80 mA) and a "T" junction. The module manifold is such that when the valves are off the carrier liquid is pumped directly to the detector while the sample is being collected. When the valves are simultaneously turned on the sample is carried towards the detector. A timer circuit based on the 555 IC and a transistor switch (TIP 121) has been used to drive the module. Sample volumes over 15 1 can be introduced with rsd equal or lower than +1%.

QA-14 Conductometric and Colorimetric Determination of Volatile Acidity of Wines by Flow-Injection Analysis. *Matthieu Tubino* and *Flávio G. Barros* (Instituto de Química-UNICAMP).

Conductometric and colorimetric flow-injection systems were developed for the determination of volatile acidity of wines. The two systems consist of the injection of the sample in a deionized water stream that then flows past a PTFE membrane separator. In the first system, the diffused acetic acid changes the conductivity of another deionized water stream that passes through a conductivity cell. In the second system, it changes the colour of a Bromocresol Purple indicator solution that passes through a flow cell in a spectrophotometer set at 560 nm.

QA-15 Toxicity Tests Using *Escherichia Coli*: Monitoring of Co_2 by Fia. *José Roberto Guimarães* and *Wilson de Figueiredo Jardim* (Instituto de Química, Departamento de Química Analítica, Universidade Estadual de Campinas, CP 6154, 13081 Campinas).

Short-term toxicity tests using the bacteria *E. coli* as a test orgamism were developed. The inhibition of the microbial respiration for different agents such as Cd(II), Ni(II) and the cetyltrimethyammonium chloride was investigated. The total concentration of carbon dioxide in the aqueous culture medium was determined by using Flow Injection Analysis (FIA) with a conductometric detector. Toxicity tests results were presented as EC_{50}. For Ni(II), Cd(II) and the cetyltrimethylammonium chloride, the EC_{50} (60 minutes) were 76.67, 14,62 and 7.88 mg/1, respectively.

QA-16 Physical Immobilization of Enzymes On Amperometric Sensors With Polymeric Films. *Lúcio Angnes, Sílvia H. P. Serrano, Ivano G. R. Gutz* (Instituto de Química da USP) and *Joseph Wang* (Chem. Department, New Mexico State University).

Physical immobilization is obtained applying a thin layer of a solution containing NAD and formaldehyde dehydrogenase (EC 1.2.1.46) on a glassy carbon electrode and, after drying, covering it with a cellulose acetate solution layer. This modified electrode compares favorably with chemical immobilization and allows the amperometric determination of formaldehyde, in a FIA system at a 30 samples/hour throughput and 10^{-6}M detection limit. The NADH formed during the enzymatic oxidation of formaldehyde is electrochemically reoxidated to NAD at the electrode.

QA-17 Malic Acid Assay in Orange Juice Using an Enzyme from Microbial Source. *Hideko Yamanaka, Maristela de Freitas Sanches Peres* e * *Cecília Laluce*. (Instituto de Química, Departamento de Química Analítica, UNESP, Araraquara (SP), * Departamento de Bioquímica).

The use of intracellular malate dehydrogenase from *Streptomyces aureofaciens* in analytical chemistry was evaluated. Some kinetic parameters were determined for the malic acid assay using the crude enzymatic extract. The assay condictions were also defined for the malic acid determination in orange juice.

QA-18 Glucose Determination in Instant Coffee With Enzyme Electrode. *Roberto Matsukura, Luiz M. Aleixo, Oswaldo E. S. Godinho, Graciliano de Oliveira Neto* (Instituto de Química da Universidade Estadual de Campinas) and *Kenji Hata* (Johnson e Johnson de Jaguariuna).

An amperometric method utilizing a enzyme electrode has been developed for the determination of the glucose content of instant coffee. The electrode was constructed by combination of a oxygen electrode with the immobilized B-D-glucose oxidase collagem membrane, the electrode response is linear in the concentration range of $1.0x10^{-2}$ to $1.0x10^{-4}$ M. The sensor was stable for more than 2 weeks and 80 assays.

QA-19 Determination of Water in Ethanol by Conductometric Measurements. *Kenneth E. Collins, Sérgio H. Pezzin, Fábio Augusto* (IQ-UNICAMP).

Ethanol solutions containing water, in the range from zero to 25% (v/v), were saturated with various electrolytes and the conductances of these solutions were measured. A strong dependence of electric conductance on the concentration of water was observed. A simple and sensitive method, using NaCl as the dissolved electrolyte, was developed based on this dependence. The procedure, which envolves the agitation of 50ml of an alcohol-water solution with 1g of NaC1, prior to the measurement of conductance, shows a $1.1x10^{-4}$S change in conductance for a 1% difference in concentration of H_2O, in the range 0–10% H_2O. The procedure was applied to comercial samples sold for household, beverage and automotive fuel purposes.

QA-20 Potentiometric Study of Cooper (II)-Azide Complexes in Aqueous Medium - *Orliney Maciel Guimarães* and *José Fernando de Andrade* - Chemistry Department, F.F.C.L.R.P. - USP, Ribeirão Preto, 14.049, SP, Brazil.

The stepwise formation of copper(II)-azide complexes in aqueous sodim perchlorate medium has been investigated at 28,0 ± 0,1°C and ionic strength 1,0M. By the indirect potentiometric method employed, using a glass electrode and under the conditions studied, four mononuclear complexes were detected. No evidence of polynuclear species was found. The final analysis of the data, by solution of simultaneous equations (matrix method) leads to the following overall formation constants: $\beta_1 = 2{,}44.10^2\ M^{-1}$, $\beta_2 = 1{,}20.10^4\ M^{-2}$, $\beta_3 = 1{,}45.10^5\ M^{-3}$ and $\beta_4 = 3{,}18.10^7\ M^{-4}$. The graphical method of Leden, with some refinements, was also utilized initially.

QA-21 Standard Addition Method for Trace Ions Determination by Biamperometry: A New and Advantageous Application - *Maria Olímpia de Oliveira Rezende* (Instituto de Física e Química de São Carlos - Departamento de Química e Física Molecular - Universidade de São Paulo - Cx.P. 369 - 13.560 - São Carlos - SP - Brasil), *Jaim Lichtig* (Instituto de Química - Universidade de São Paulo - Cx.P. 20780 - 01.498 - São Paulo - SP - Brasil).

The two indicator electrodes analysis becomes identical to that with only one indicator electrode if ΔE imposed between electrodes is large enough to produce current depending to difusion process, but not so great to avoid another indesirable reactions, e.g., 100 mV, like Kolthof demonstrated. We also verified when one substance of the reversible pair is present in a large excess, the linearity response is better.

QA-22 Determination of Sb(III) at Trace Level in Electronic Grade Reagents. *Mauro Aquiles La Scalea, Lúcio Angnes, Ivano G. R. Gutz* (Instituto de Química - Universidade de São Paulo).

The purity requirements for electronic grade reagents are high, and the presence of As and Sb is tolerated only at ppb level because they are dopants of silicon semiconductors. Conditions have been established for the determination of antimony in electronic grade acids at the ppb level by anodic stripping voltammetry. The determination was made by standard addition method and the calibration curves are linear.

QA-23 Use of Stainless Steel as pH Electrode in Flow Injection Analysis. *Ivo M. Raimundo Jr.* and *Gentil C. Paixão* (Instituto de Química - Universidade Estadual de Campinas - São Paulo - Brasil).

A tube of 316 stainless steel 0. 8 mm longer with i. d. 1. 0 mm was employed as a pH sensor in a single line, limited dispersion potentiometric FIA. The tube was previously immersed for 20 min in a hot (70–80 °C) solution 2. 5 M of CrO_3 in H_2SO_4 5 M. A Ag/AgC1 electrode was employed as reference. The electrode response was linear from pH 2 to 13 (r=0. 999). The sensitivity found was 53 mV per pH unit and samples can be introduced at the rate of 50 per hour, and the precision expressed as the rsd for six replicates measurement of ten buffers solutions was 1. 8% or lower.

QA-24 A Modified Method for the Determination of Mercury by Atomic Absorption Spectrometry. *Gastón East Ponce, Antônio C. Barbosa* (Departamento de Química, Universidade de Brasília, Brasília-DF), and *Romino B. Ornelas* (Companhia de Água e Esgotos de Brasília, Brasília-DF).

A modified method for the determination of traces of Hg by Cold Vapor Atomic Absorption Spectrometry by direct transfer of the vapor to a cell by means of a disposable syringe is described. The cell was made out of Pyrex glass tubing, 11.5 cm long 15 mm i.d., with silica end windows attached with epoxy resin and injection and outlet ports. Approximate volume 20 mL. A calibration curve from 4.4 to 43.5 ng mL^{-1} was linear with r - 0.9990. Hair samples were analysed for Hg in this manner and the results were not significantly different from those obtained by ASV with rotating gold disk electrode. Determination of Hg in sediments of Paranoá Lake, Brasília, is currently in progress.

QA-25 Study of the Experimental Conditions for Hydride Generation of As and Sb Coupled to ICP-AES. *Ary A. B. Buzatto* (University Mackenzie), *Elisabeth de Oliveira* (Chemistry Department, University of São Paulo).

Experimental studies about hydride generation of As and Sb coupled to ICP-AES were developed to determine the best operational conditions (low back-ground signal, reprodutibility and feasability). It was achieved that polypropilene filter TFP-501 plus teflon are the more suitable support for the gas-liquid separator. In the adequate conditions, the detection limits obtained were 1.2 ppb for As and 0.70 ppb for Sb. 99.5% of acuracy was obtained for the NRC - Canada Standards BCSS-1 and MESS-1, measuring AsIII and SbIII.

QA-26 Simultaneous Determination of Rare Earths by ICP Emission Spectrometry: A Sistematic Study of Interferences. *Sidney Lopes da Silva, Carlos Henrique Piloto* and *Luiz Augusto Morino* (Solvay do Brasil S.A., R&D Laboratory, CP 2801 - 09211 - Sto. André - São Paulo).

Interference among rare earths is a shared problem to many analytical technics and ICP spectrometry is no exception, but its advantages in detection limits and speed justify their study. In multi-component sample analysis, instrumental (SPECTROFLAME ICP) parameters were optimized to minimal interference conditions. Seven analytical wavelengths were choosen for each rare earth. The best wavelength in nm, as determined by the least sum of interferences were: La, 398.85; Ce, 413.77; Pr, 418.95; Nd, 415.61; Sm, 446.73; Eu, 412.97; Gd, 310.05; Tb, 356.17; Dy, 353.17; Ho, 345.60; Er, 369.27; Tm, 346.22; Yb, 328.94; Lu, 261.54; Y, 371.03. Solvay do Brasil SA.

QA-27 Determination of Rare-Earths in High Purity Ittrium Oxide and Xenotime by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES) - *Rosa Ana Conti, Luiz Rinaldo Bizaio* (Fundação de Tecnologia Industrial - Centro de Materiais Refratários - Lorena - SP), *Richard C. Floyd* (Applied Research Laboratories - EUA).

Rare-Earths materials are applied in several branches of high technology. ICP/AES was applied to determine RE trace impurities in high purity Y_2O_3 and RE components in xenotime (RE ore). Analyses were made in the presence of other. RE elements, without any separation or preconcentration. The following detection limits were obtained: Yb = 1.4, Er = 0.3, Tb = 0.5, Dy = 0.4, and Ce = 2.4 μ g/g. High purity Y_2O_3 doped with known amounts of these RE showed relative erros < 10%. Typical values for xenotime concentrates were Y = 39%, Er = 13%, Dy = 9%, Yb = 18%, Tb = 1%, Ce < 0.2%.

QA-28 Determination of Trace Metals in Brines By ICP-AES With Preconcentration by Precipitation With Diethyldithiocarbamate. *Haroldo Silveira Dórea; Antonio Celso Spinola Costa* (Instituto de Química. Universidade Federal da Bahia. Salvador. Bahia) *Rui Jesus Lourenzo Garcia* (GELAB-CEPED. Camaçarí. Bahia).

Several metals in brines can be separated from the matrix and concentrated by precipitation with sodium diethyldithiocarbamate (DDTC). Working with 200,0±0,1 g of brine (200–300g/L NaC1), we added 10,0 mL do 1% DDTC, after pH adjusting to pH 5,6±0,1. Zn (II) or cellulose powder, (Cellulose PEI TLC Baker) are used as carriers. After filtration in a 0,45 μm membrane filter, the precipitate is dissolved with HNO_3 and diluted to 25,0 mL. Measurements are made by ICP-AES using a multichannel instrument and recuperations better than 90% were obtained for Cd, Co, Cu, Mn, Pb, V in a range of 25-500 ppb.

QA-30 Determination of Trace Metallic Impurities in Pure Titanium and Titanium Alloys by Inductively Coupled Plasma Atomic Emissin Spectrometry (ICP/AES) - *Rosa Ana Conti, Luiz Rinaldo Bizio* (Fundação de Tecnologia Industrial - Centro de Materiais Refratários - Lorena - SP.)

The new applications of pure metallic titanium and its alloys demand trace and alloying elements characterization in these materials. A 3410 Minitorch ARL ICP sequencial spectrometer has been used for determination of trace and major components (e.g., Aℓ, Fe, V) in EB melted titanium metal and Ti6%Aℓ4%V alloy. The determinations were carried out without any further sample preparation than dissolution and subsequent dilution. The following detection limits have been obtained: Aℓ=1,8; V=3,8; Fe=0,7 μg/g. The typical values of Ti metal are: Aℓ=20; V=30; Fe=450 μg/g and for TiAℓV alloy are: Aℓ=5,8; V=3,9; Fe=0,5 wt%, ICP/AES has proved to be a fast and reliable technique for these analyses.

QA-31 Comparision of the Filters Digestion Methods for the Determinationof Metals With ICP-AES. *Roseli F. Gennari, Elisabeth de Oliveira,* (Department of Chemistry - University of São Paulo).

Four methods of filters digestion were studied. The samples were taken in the period from 24 to 25 July 1989, and analyzed for aluminium, arsenic, calcium, cadmium, cobalt, cromium, copper, iron, magnesium, manganese, nickel, lead, vanadium and zinc. The method with HNO_3 and $HC1O_4$ in the ultrasonic bath was specific for the determination of cromium and copper.

QA-32 Refractory Metals Determination by Flame Atomic Absorption Spectrophotometry. *Gilberto Luís Jardim Pinto da Silva, Francisco Ribeiro, Angelo Capri Neto* (Fundação de Tecnologia Industrial - Centro de Materiais Refratários - Lorena - SP.)

A method is described for direct determination of aluminium, vanadium and iron in Ti-6%Aℓ-4%V alloy by flame atomic absorption spectrophotometry. Analyses in synthetic samples showed that titanium intereferes only in vanadium determination. This interference was compensated by matrix matching the calibration standard solutions of vanadium or by using the standard addition technique. This methodology has been used for analitycal characterization of Ti-Aℓ-V alloys refined by electron beam.

QA-33 Analitycal Characterization of Potassium Fluortantalate by Absorption and Emission Atomic Spectrophotometry - *Gilberto Luís Jardim Pinto da Silva, Francisco Ribeiro* (Fundação de Tecnologia Industrial - Centro de Materiais Refratários - Lorena - SP).

A method is described for determination of tantalum, potassium, sodium, iron, silicon and magnesium in potassium fluortantalate (K_2TaF_7) by atomic emission and absorption spectrophotometry. Potassium was determined by flame emission and no interferences were detected. Potassium interferes in tantalum détermination by flame atomic absorption, and tantalum interferes in all other determinations. These interferences were satisfactorily compensated by matrix matching the callibration standard solutions.

QA-34 Comparative Study Between Atomic Absorption and Emission Flame Spectrophotometry for Determination of Low Contents of Niobium - *Angelo Capri Neto, João Flávio Pellegrini* (Fundação de Tecnologia Industrial - Centro de Materiais Refratários - Lorena - SP).

The determination of low contents (up to 200 μg/ml) of Nb by atomic absorption and emission spectrophotometry with acetylene nitrous oxide flame was studied. The results showed that: 1) The addition of aluminum (0.05 to 0.2% w/v) increase the linearity and sensitivity of calibration curves of niobium both for absorption and emission techniques. 2) Above 200 μg/ml of Nb, both techniques showed similar results. 3) The sensitivity, under the conditions herein considered, is of 3.2 μg/ml of niobium for emission and 12.2 μg/ml for atomic absorption.

QA-35 Determination of Cu, Fe, Mn and Zn in Fruits by Flame Atomic Absorption Spectroscopy. *D. S. Vaitsman, I. S. Azevedo, V. L. R. Matos, Z. T. C. Leite* and *E. P. Vaitsman.* (Departamento de Química Analítica - Instituto de Química - Universidade Federal do Rio de Janeiro).

The amounts of Cu, Fe, Mn and Zn were determined in fruits usualy consumed by the population of Rio de Janeiro in Brazil, using a varian AA-175B atomic absorption Spctrograph. 24 different kinds of fruits, making up a whole of 66 samples were weighed and macerated before the treatment of elimination of the organic matter by a 450°C furnace incineration followed by an adition of diluted mineral acids so as to obtain a mineral residue. (CEPG/UFRJ; DQI-IQ/UFRJ; CNPq)

QA-36 Comparison of Digestion Methods for the Determination of Heavy Metals in Sediments by AAS. *Romino B. Ornelas,* (Companhia de Água e Esgotos de Brasília); *Antonio C. Barbosa* and *Gastón A. E. Ponce* (Departamento de Química, UnB, 70910, Brasília, DF).

Two digestion methods for the determination of Cu, Pb, Zn and Cd in sediments of the Paranoá Lake of Brasília by flame AAS, were compared. In method A, HC1 and HNO_3 were used, and HNO_3, H_2SO_4 and 30% H_2O_2 was employed in method B. The mean value for the analysis of Cuby method A was 17% higher than that obtained by method B. On the other hand, no significant difference for Pb and Zn by using either method was found. The content of Cd lies below the detection limit of the technique. The relative standard deviations were 9,52 and 8,52% for Zn; 8,41 and 8,96% for Cu and 7,50 and 7,17% for Pb, by method A and B, respectively. Recovery using method A was 96,8; 100,0 and 99,9% for Cu, Pb and Cd, respectively.

QA-37 Chemical Weathering of Scheelite Tailings. *Sylvia Cristina lacerda da Costa Pereira, Lúcia de Fátima Lacerda da Costa Pereira,* (Departamento de Química Aplicada, Universidade Federal de Pernambuco).

All the chemical phenomena which produce a chemical modification of the rocks are known as chemical intemperism. To work with conditions similar to ground water under the action of microorganisms, a cytric N/10 solution was prepared. Spectography and atomic absorption showed a substancial improvement of the corrective action of the pH with the reduction of the acidity and the continuous liberation on of the lower elements essential to growth of plants.

QA-38 Spectrophotometric Determination of Copper (II) With Zinc Diethanoldithiocarbamate. *Aldaléa Lopes Brandes Marques* (Dept. Téc. Química/UFMA); *Gilberto Orivaldo Chierice* (Ins. Fís. Química - S. Carlos - Caixa Postal 369 - CEP 13560 - São Carlos/SP).

A simple and direct spectrophotometric method for the determination of copper (II) at the ppb level is proposed. The method is based in the reaction of desplacement of the zinc by copper (II) in the zincdiethanoldithiocarbamate complex. The new reagent has shown be most suitable than others dithiocarbamates salts. (CAPES-PICD).

QA-39 A New Reaction of $CS_2N_3^-$ Ions and its Analytical Utilization (II). *Edmar P. Marques,* (Universidade Federal do Maranhão); *Tito de Abreu Cassoni,* (Instituto de Química, UNESP, Araraquara); *Douglas Wagner Franco,* (Instituto de Física e Química de S. Carlos, USP).

The oxidation reaction of sulfite ions by oxygen is catalized by 1,2,3,4-thiatriazol-5-tiolate ions according to:

$$2HSO_3^- + 0_2 \xrightarrow{CS_2N_3^-} 2HSO_4^-$$

The rate constant of sulfite formation and the rate constant of oxygen uptake are equal to 0.7 ± 1 s^{-1} ($C_{CS_2N_3^-} = 7 \times 10^{-5}$M). Both rate constant are in agreement with that previously obtained by spectrophotometric measurements of $CS_2N_3^-$ absorbance decrease at 313 nm in the same conditions. (CAPES, CNPq, FAPESP).

QA-40 Colorimetric Determination of TiO_2 With H_2O_2 in Chromite Samples. *Jacinta Enzweiler,* (Instituto de Geociências, Universidade Estadual de Campinas - Caixa Postal 6152 - 13081 - Campinas, SP).

The TiO_2 content (beside other elements) in chromites rich samples its important to characterize geochemically the deposit. TiO_2 was determined by the colorimetric method with H_2O_2 after the sample sintering with Na_2O_2. The Cr(VI) interference was avoided by precipitation of Fe and Ti with NaOH. The collected precipitate was redissolved in hot dilute H_2SO_4 and the TiO_2 was determined spectrometrically at 410 nm after complexation with H_2O_2. For 7 different sinterings, the average content of TiO_2 was 0,34% with a standard deviation of 0,0195.

QA-41 Complexation of Trivalent Lanthanides by 4-methoxybenzylidenepyruvate in Aqueous Solution. *Helena Redigolo, Cristo B. Melios, Massao Ionashiro* and *Marco A. Gonçalves.* (Instituto de Química, Departamento de Química Analítica - UNESP, Araraquara - SP.).

The complex formation equilibria in M-4-MeO-BP systems, where M = Ce(III), Nd(III), Eu(III), Gd(III), Tb(III), Dy(III), Ho(III), Er(III), Tm(III), Yb(III), Y(III) and 4-MeO-BP = 4-methoxybenzylidenepyruvate (pka=1.473), were investigated spectrophotometrically at 25.0 ± 0.1°C and ionic strength 0.500 M, held with sodium perchlorate. The study is mostly concerned with the determination of formation constants and molar absorptivites. The termodynamic stability order found for 4-MeO-BP binary complexes is: Nd(III) > Eu(III) > Gd(III) > Yb(III) > Ce(III) > Tm(III) > Tb(III) > Er(III) > Dy(III) > Ho (III) > Y(III).

QA-42 Spectrophotometric Determination of Silicon Traces in Metallic Niobium. *Maria Lúcia Caetano Pinto da Silva, Maria José Ayres, Luiz Rinaldo Bizaio.* (Fundação de Tecnologia Industrial - Centro de Materiais Refratários - Lorena - SP).

A method is described for separation of silicon traces from metallic niobium for spectrophotometric determination. The silicon is distilled as SiF_4 and collected in an absorbing solution consisting of boric acid, ammonium molybdate and sulfamic acid. By addition of ascorbic acid, silicomolybdic acid is formed and spectrophotometrically determined at 815 nm. The method was applicable to samples of metallic niobium refined by electron beam containing less than 20 μg/g of silicon. A relative standard deviation of 7% was obtained for samples containing between 3.3 and 12.3 μg/g.

QA-43 A New Method for Total Protein Determination in Bean. *Dimas A. M. Zaia, Nilson J. Santos, Silvana R. Rockenbach, and Wagner J. Barreto.* (Depto. de Química, CCE, FUEL, Londrina - PR, Brazil).

A new method for total protein determination was developed and applied to samples of bean. The method is based on the products of the reaction between p-benzoquinone and protein, which show a charge-transfer band at 350 nm. The effects of concentration, reaction time, and pH were studied. The best results with the p-benzoquinone method were obtained by addition of standard at pH 6.0 (phosphate buffer 0.2 M): casein standard (24.2 + 0.4, n = 11) and protomax 90 standard (24.7 + 0.7, n = 15). These results agree very well with Kjedahl's method (23.0 + 0.4). (CONCITEC).

QA-44 The Potentiality of Emission Detectors for Empirical Formulae Determination in Gas Chromatography. *Antonio Luiz Pires Valente,* (Instituto de Química, UNICAMP, Brasil) and *Peter C. Uden,* (Chemistry Department, University of Massachusetts, USA).

A microwave induced plasma (MIP) and multichannel spectrometer system coupled to a gas chromatograph was used for empirical formulas determination of organohalogenated compounds. The use of data reduction methods and polynomial regression analysis has permitted considerable reduction of the errors usually found for hydrogen coefficients. These errors seem to be due to a dependence of the MIP signal with the hydrogen concentration.

QA-45 Alocation of Retention Times of Gaussian Chromatographic Peaks by Polynomials: Polynomial Grade and Regression Window Effect. *Antonio Luiz Pires Valente* and *Fabio Augusto* (Dept. of Analytical Chemistry, IQ – UNICAMP: C. P. 6154, 13081 Campinas – SP, Brazil).

The effect of the polynomial degree and of the number of points (regression window) in polynomial allocation of retention times of simulated gaussian chromatographic peaks was studied. Polynomials from 2nd to 5th degree and 7 to 25 point windows were used for peaks with base-width from 2.0 to 8.0 s. Retention time errors ranged from – 81 to + 8 ms. Narrow peaks need higher polynomial and smaller regression windows; the opposite is true for larger peaks. The choice of parameters is critical for narrow peaks.

QA-46 Software for Van Deemter Equation Determination. *Antonio Luiz Pires Valente* and *Fabio Augusto* (Dept. of Analytical Chemistry, IQ – UNICAMP; C. P. 6154. 13081 Campinas, SP – Brazil).

A program that calculates the A, B and C parameters of the van Deemter equation was developed. A least squares regression algorithm is used. Input data are sets of height equivalent to a theoretical plate and linear mobile phase velocity. The program also calculates the A, B and C confidence limits and the curve equation fitness. It has been successfully used for GC and HPLC data. The knowledge of A, B and C may permit studies of column characteristics, such as solute diffusibility in the stationary phase. These studies are progress in our laboratory.

QA-47 Application of Digital Filters for Noise Reduction in Digitized Chromatograms. *Antonio Luiz Pires Valente* and *Fabio Augusto* (Dept. of Analytical Chemistry, IQ – UNICAMP; C. P. 6154, 13081 Campinas – SP, Brazil).

The efficiency of moving average, MA, and partial least squares, SG (Savitzky-Golay), noise reduction filters was studied. The best number of points (convolution window) used in each filtration step was evaluated, as well as the polynomial degree (2nd or 4th) for SG filter. Simulated chromatograms with noise levels of 10, 50 and 100 mV were used. The MA filter is better then SG for noise reduction, but it causes more peak deformation than 4th degree SG. Deformation is proportional to the size of the convolution window; narrow peaks are more easily deformed.

QA-48 The Effects of Mobile Phase Flow Rate on Quantitative HPLC. *Carol H. Collins, M. Fátima S. Trindade* (IQ – UNICAMP) and *Paulo Roberto N. Carvalho* (ITAL, Campinas).

It is well known that changes in mobile phase flow rate modify the retention times and peak heights of the separated peaks. Thus, use of peak areas has been suggested for species quantitation under the supposition that this value is independent of flow rate. Although this supposition is probably valid for mass–sensitive detectors such as are used in gas chromatography, we have found significant differences in area determinations using concentration sensitive spectrophotometric detectors at both high and low flow rates. However, the ratios of peak areas (or heights) remain constant, suggesting that the internal standard method is a better choice than the external standard method for quantitative work.

QA-49 Thermal Stability a Non–Polar Liquid Stationary Phase Immobilized Onto Chromosorb W–HP by Gamma Irradiation. *Maria José J. F. dos Santos, Kenneth E. Collins* and *Carol H. Collins* (Instituto de Química, Universidade Estadual de Campinas, Campinas – SP).

Although the effects of gama radiation on non–polar liquid stationary phases used in capilary columns are well described in the literature, little is know about gamma immobilization of phases used in packed columns. This study compares the thermal stability, extractability and chromatographic properties of SE–30 on Chromosorb W–HP without irradiation, after gamma irradiation to six different doses and after post–irradiation extraction. The latter phases showed the highest chromatographic efficiencies as well as the greatest thermal stabilities. (CNPq).

QA-50 Relation Between the Retention Indices in Non–Polar Stationary Phases and Connectivity Indices of DDT and Compounds Structurally Related. *Vilma E. F. Heinzen* and *Rosendo A. Yunes,* (Departamento de Química – UFSC, Florianópolis – SC – Brasil, 88049).

Retention indices (I) of DDT and correlated compounds in non–polar stationary phase (Apiezon–L at 215°C) were correlated by one and two variable equations with molecular connectivity indices ^{1}X, $^{1}X^{V}$, $^{3}X_{pc}$, $^{4}X_{pc}$ and $^{4}X^{V}_{pc}$. The best one variable equation was obtained with $^{1}X^{V}$ (R = 0,8905; F = 68,99) and the best two variable equations with $^{1}X^{V}$, $^{4}X^{V}_{pc}$ (R = 0,8997; F = 36,09) and $^{1}X^{V}$, $^{3}X_{pc}$ (R = 0,9008; F = 36,58). The $^{1}X^{V}$ that is proportional to the number of the bonds, the insaturations and the heteroatoms existing in the molecule is the principal factor that governs the magnitude of the $I^{215}_{\text{Apiezon–L}}$ values.

QA–51 Analysis of Organochlorine Pesticide Residues in Blood Serum I. *Elizabeth Victória Minelli* and *Maria Lúcia Ribeiro* (Instituto de Química – UNESP).

This paper proposes a analytical procedure for organochlorine pesticide residues in blood serum. Serum is treated with a special silica gel containing three different water contents and the pesticides are extracted from the suspension by hexane: acetone mixture (9:1). After separation and concentration the organic phase is transferred to the aluminium oxide column. Elution is carried out with n–hexane, the eluate is concentrade and analyzed by gas chromatography.

QA-52 Thermal Analysis of Hydrido–Complexes of Platinum (II). *Roberto Santos Barbiéri, Júlio César Rocha, Carlos Roberto Bellato* (Dep. de Química Analítica, Instituto de Química – UNESP – Araraquara, SP) and *Eustáquio Galvão de Silva* (Dep. de Física, Instituto de Ciências Exatas – UFMG – Belo Horizonte, MG).

The compounds $[Pt(H)(X)(PPh_3)_2]$ (X = Cl, Br, I, SCN, NO_2, PPh_3 = triphenylphosphine) were prepared and characterized by infrared espectroscopy and by carbon and hydrogen elemental analysis. Simultaneos thermogravimetric (TG) and derivative thermogravimetric (DTG) curves of these complexes under air are studied. These compounds show relative resistance to thermal decomposition. Mass loss considerations indicate conversion of the complexes into metallic platinum. The thermal decomposition of the halogen complexes ocurred in various steps and the thiocyanate complex decomposes in only one step. It was possible to formulate thermal decomposition mechanisms for all the compounds under study except for the nitro complex. (CNPq).

QA-53 Development of a Combustion Calorimeter Combined With a Graph–Plotter. *Francisco Gurgel de Azevedo* and *Glícia Maria Galvão Damasceno* (Departamento de Química e Curso de Mestrado em Engenharia Química, Universidade Federal do Rio Grande do Norte).

Description of a combustion calorimeter with a graph–plotter, which system consists of a conventional calorimetric bomb, a temperature sensor, a Wheatstone bridge and a graph–plotter. One of the arms of the Wheatstone brigde is a thermometer of platinum resistance, which is immersed in a bath where the calorimetric bomb is present; this arm is the temperature sensor. The sensitivity of the circuit is of 0.98 V K^{-1}, and this when the potential feeding the bridge is 1.35 V. The calorimeter has been used to determine the combustion energy of turfs, marine algas and animal ration. The precision of the measurements is given in terms of standard deviation, calculated on six values. Using a common thermometer the standard deviation is around 0.4% and in the case of the temperature sensor and the graph–plotter it falls to 0.1%.

QA-54 Thermal Stability of the Double Selenates of Lanthanides (Ceric Earth) and Lithium in the Molar Ratio 1:1 and 1:2. *Paulo Marcio Cruz; Clóvis Augusto Ribeiro; Miriam Hisami Miyano* (Instituto de Química de Araraquara – UNESP, SP).

Thermogravimetry, differential thermal analysis and other methods of analysis have been used in the characterization and study of the decomposition of the Double Selenates of Lanthanides (Ceric Earth) and Lithium in the molar ratio 1:1 and 1:2. The results permit suggestions concerning the compositions and mechanisms of thermal decomposition of these compounds.

QA-55 Preparation, Characterization and Thermal Stability of Complexes of Zn, Co and EDTA. *Marcia Keiko Oshiro, Marisa Spirandeli Crespi, Clóvis Augusto Ribeiro* e *Massao Ionashiro.* (Instituto de Química de Araraquara, Departamento de Química Analítica, UNESP).

Complexes of Zn, Co and EDTA were prepared by mining equimolars quantities of correspondants carbonates of metals and EDTA, evaporating in a desiccator over anydrous calcium chloride. These compleplexes were determined by conventional methods of analysis, differential thermal analysis (DTA) and Termogravimetry (TG). Analytical and thermal analytical permitted to study the stability, the thermal decomposition, the stoichiometric and suggest the mechanism of the thermal decomposition of this compounds.

QA-56 Thermoanalytical Identification of Some Sulfa Drugs. *Elcio Rogerio Barrak* (Departamento de Física e Química da Escola Federal de Engenharia de Itajubá) and *Ivo Giolito* (Instituto de Química da Universidade de São Paulo).

Sulfacetamide, sulfadiazine, sulfadoxine, sulfaguanidine, sulfamerazine, sulfametoxazol, sulfamonometoxine, sulfanilamida and sulfisoxazol were separately identified in two different ways: a) Using the onset temperature of the first weight loss in a thermogravimetric curve and b) Using the maximum of the endothermic melting peak in a differential scanning calorimetric curve.

QA-57 Thermal Analytical Study of Hexamethyl Fosforamide of Lanthanide Trifluoromethanesulfonate. *A. Oliveira da Silva, D. M. Araújo Melo* and *J. D. G. Fernandes* (Universidade Federal do Rio Grande do Norte - Departamento de Química). *G. Vicentini* and *L. B. Zinner* (Universidade de São Paulo - Instituto de Química).

Rare earth trifluoromethanesulfonate complexes with hexamethylfosforamide (HMPA), $Ln(CF_3SO_3)_3{\cdot}nHMPA$, (n = 6 to La-Eu and n = 4 to Gd-Lu), have been prepared and the thermal behaviour studied by TG and DTA techniques. The ligands are lost in two steps. The anhydrous lanthanoid trifluoromethanesulfonates formed around 350°C rapidly decompose with evolution of COF_2 and SO_2, giving LnF_3 as the final product.

QA-58 Thermal Analytical Investigation of Hexamathylfosfamide Complexes of Tervalent Lanthanoid Perchlorates. *J. D. G. Fernandes, D. M. Araújo Melo* and *O. A. de Oliveira* (Universidade Federal do Rio Grande do Norte - Departamento de Química). *G. Vicentini* (Universidade de São Paulo - Instituto de Química).

Tg-DTA analyses of $Ln\ (ClO_4)_3{\cdot}nHMPA$ (n = 6 to La-Eu and n = 4 to Gd-Lu) were carried out in a nitrogen and air atmosphere. The observed values of the characteristic temperatures show systematic changes along the series, owing to the effects of the "lanthanoid contraction".

QA-59 Thermogravimetric Behaviour of Complexes of Di-N-Butyldithiocarbamate with Phosphorus (III), Arsenic (III), Zinc (II), Cadmium (II) and Mercury (II). *Antonio Gouveia de Souza, Gilson Francisco de Oliveira* and *Marco Aurélio Rodrigues Melo* (Departamento de Química - CCEN - Universidade Federal da Paraíba).

The thermogravimetric curves were obtained on a DU PONT model 951 thermobalance using oxigen or nitrogen furnace atmospheres. Sample sizes ranged in mass from 5 to llmg; a furnace heating rate of 5°C per min was employed. The metal complexes were all studied in nitrogen atmosphere: $P(S_2CNBu_2^n)_3$, $As\ (S_2CNBu_2^n)_3$, $Zn(S_2CNBu_2^n)_2$ $Cd(S_2CNBu_2^n)_2$ and $Hg(S_2CNBu_2^n)_2$ and in oxigen atmosphere: $P(S_2CNBu_2^n)_3$ and $As(S_2CNBu_2^n)_3$ - CNPq.

QA-60 Thermogravimetric Study of the Oxidation Kinetics of Copper. *José Schifino* and *Mateus A. G. Andrade* (Departamento de Físico-Química - Instituto de Química - UFRGS).

The oxidation kinetics of copper was studied by thermogravimetry at varying temperatures in the range 500°C to 950°C. The weight increase of a sample was recorded as a function of time employing a thermogravimetric system Perkin Elmer DSC-4 TGS2. The balance sensitivity allows samples of about 1 mg to be studied in experimentas lasting less than 3 hours. The reaction rate is inicially fast and as the oxide layer grows the kinetic becomes diffusion controlled according to the parabolic law $x^2 = 2kt$. Rate constants follow an equation of the type $k = 2{,}93 \times 10^{-2} \exp(-34{,}4/RT)\ cm^2/s$.

QA-61 Studies on the Kinetics of Chemical Etching of Polycarbonates as Fission Track Detectors. *Maria Lúcia C. P. Carvalho* (Dep. Química Analítica, UFRJ), *Heliane Zylberberg and João A. Medeiros* (Dep. Química, PUC/RJ e Laboratório de Análise Mineral/CNEN, Rio de Janeiro).

Polycarbonates are important fission track detectors for uranium and thorium determination, mainly at very low levels, as in water and biological samples. Etching kinetics of aromatic linear ("Lexan") and cross linked, alifatic, ("CR-39") polycarbonates were studied before and after reactor irradiation up to 3.10^{17} n/cm². Mass variation at 10^{-5} g constant weight, diameters of 90 degrees- ^{252}Cf fission tracks were used to determine bulk etching rate (v_b). A new method was developped to determine v_b through the etch products in NaOH or KOH solutions, which proved to be more sensitive and accurate than those.

QA-62 Thin Layer Chromatography for Separations of Gamma Radiolysis Fragments of Bis-Phenylalaninate Copper (II). *Elias Mansur Netto* (Faculdade de Farmácia - UFMG), *Hyedda Nancy Sander Mansur, Ana Maria Tonani Pereira, Luiza de Marilac Pereira Dolabella* and *Simone de Fátima Barbosa Tófani* (Instituto de Ciências Exatas - Departamento de Química - UFMG).

The study of radiolysis of water solutions of bis-phenylalaninate copper (II) complex was made from fractions separations fo complex and ionic copper in 0,25 mm thickness celulose and silica thin layers, using some elluents solutions. The agent detection used was a water solution of sodium diethylditiocarbamate. For silica thin layers, the best elluents was ethanol-water-acetone-acetic acid (45:35:18:2) solution, giving a Rf of 0,69 for Cu^{2+} and 0,22 for the complexed copper. For cellulose thin layers, the best elluent was water-methanol-acetic acid (49:50:1) solution, giving Rf values of 0,81 for Cu^{2+} and 0,18 for the complexed form.

QA-63 Gamma-Radiolysis of Indoline in Alcoholic Solutions. *Jose Talamoni, Eny Maria Vieira* and *Maria Olímpia de Oliveira Rezende* (Instituto de Física e Química de São Carlos - Departamento de Química e Física Molecular - USP - Cx. P. 369 - 13.560 - São Carlos - SP - Brasil).

In this work we studied the chemical and radiolytic stability of products by radiation from Indoline in an alcoholic solutions. Doses were from $2x10^4$ to $3x10^5$ Gy and the samples concentration were 1:1 by volume. We have observed significant effects for high radiation doses with formation of Quinoline and 1, 2, 3, 4, tetrahydroquinoline as main products. Lower doses affected the solvent with the production of long polymeric hydrocarbons (C_9 to C_{29}). Products formed were characterized by capillary gas chromatography coupled to mass spectrometry (CG/MS). We have used a fused capillary column 25 m long, 0.2 mm internal diametre, covered by a film of cross-linked 5% phenylmethyl silicone 0.33 μ m tickness. (FAPESP, CNPq, CAPES).

QA-64 Self-Radiolysis of $Ca^{14}CO_3$ at High Dose. *Guadalupe Albarrán*, (Instituto de Ciências Nucleares, UNAM, México - D. F.), *Kenneth E. Collins, Carol H. Collins* (Instituto de Química, UNICAMP - Campinas, SP).

Ionizing radiation of solid calcite ($CaCo_3$) produces the radicals CO_3^-, CO_3^{3-} and CO_2^-. These radicals can interact in the solid state or react upon dissolution to produce various organic compounds. High specific activity (290 mCi/g) $Ca^{14}CO_3$ was prepared and stored for 10, 690, 840 and 2400 days. The organic products obtained on dissolution were separated and identified by ion moderated partition chromatography and quantified by liquid scintillation counting of the carbon-14. Significant changes in the product distribution of five organic acids and two non-polar organic compounds were observed to accompany the increase in dose from the self-radiolysis of $Ca^{14}CO_3$. (CONACYT, CNPq).

QA-65 Ethanol-Fueled Light-Duty Vehicles Equipped With Catalytic Converters: Formaldehyde and Acetaldehyde Emissions. *Jailson B. de Andrade*, (Chemistry Institute, U.F.Ba, 40.210 Salvador, Bahia, Brazil) and *Antonio H. Miguel* (Chemistry Institute, USP, C. P. 20.780, São Paulo, SP, 01498, Brazil).

Aldehydes and other carbonyls emitted to the atmosphere can participate in a complex chain of reactions that lead to the formation of important smog components such as peroxyacetyl nitrate (PAN), nitric acid and several others. We determined the profile of aldehydes emitted by two Brazilian ethanol-fueled vehicles –equipped with three-way catalytic converters– operated under the urban driving cycle. Without catalysts, the (weighted) average emissions of formaldehyde and acetaldehyde were in the range of 0.019 and 0,246 g/km, respectively; with catalysts they were lower by 72.6% to 83%, respectively.

QA-66 Formaldehyde Collection Methods Comparison and Ambient Concentrations During the São Paulo Atmospheric Characterization Experiment (SPACEX-I). *Denise R. M. Pomaleski, M. Cecília G. de Mendonça,* and *Antonio H. Miguel* (Chemistry Institute, USP, C. P. 20.780, São Paulo, SP, 01498, Brazil), *Michael Schilling* and *Dieter Klockow* (Institute of Spectrochemistry and Applied Spectroscopy - ISAS, Dortmund, FRG), *Ciomara R. de Carvalho* (Technology Center of Minas Gerais and Chemistry Department UFMG, BH, Minas Gerais) and *Jailson B. de Andrade* (Chemistry Institute, UFBa, Salvador, Bahia, Brazil).

Formaldehyde (HCHO) and other carbonyls play a major role in tropospheric smog processes. During SPACEX-I, carried out in the Austral winter of 1989, two groups made independent measurements of HCHO, each sampling with a different technique: C18 Sep-Pak cartridges (Waters) and denuder tubes, all coated with 2,4-DNPH /H_3PO_4. Concentrations ranged between 1.18 ppbv and 17.8 ppbv. During sunny periods, good agreement was observed between the two methods. However, during some rainy periods, denuder results were up to a factor of two higher, indicating that aqueous phase HCHO may have been cocollected with gas phase HCHO, and/or "micro scale" differences between the location of the samplers.

QA-67 Aldehydes and Carboxylic Acids Concentrations During the First São Paulo Atmospheric Characterization Experiment (SPACEX-I). *Antonio H. Miguel, Denise R. M. Pomaleski* (Chemistry Institute, USP, C. P. 20.780, São Paulo, SP, 01498, Brazil), *Michael Schilling* and *Dieter Klockow* (Institute of Spectrochemistry and Applied Spectroscopy (ISAS) Dortmund, FRG), *Ciomara R. de Carvalho* (Technology Center of Minas Gerais and Chemistry Department UFMG, BH, Minas Gerais).

Formaldehyde (HCHO) and acetaldehyde (CH_3CHO) are toxic, while formic (HCOOH) and acetic acid (CH_3COOH) are corrosive. They are emitted directly to the atmosphere or formed *in situ*. We have obtained the first data base of the profiles of these species in the atmosphere of São Paulo, a city heavily impacted by alcohol and diesel emissions. Day time (8 am to 8 pm) HCOOH/HCHO and CH_3COOH/ CH_3CHO ratios varied between 0.33 and 0.64 and between 0.23 and 0.36, respectively, suggesting a common source (vehicles) for these aldehydes and their respective carboxylic acids. Day time concentrations (ppbv) ranged from 0.75 to 7.67 for the acids and between 0.70 and 29.3 for the aldehydes.

QA-68 Aspects of Transportation and Deposition of Atmospheric Sulfate Nitrate and Chloride in the Recôncavo, Bahia, Brazil. *T. M. Tavares, V. C. Rocha, E.M. Nogueira* (Analytical Chemistry Dept. Federal University of Bahia), *P. Jacob, D. Klockow* (ISAS, Institut für Spektrochemie und Angew. Spektroscopie, Dortmund, Germany), *R. Van Grieken* (Chemistry Dept., University of Antwerp, Belgium).

NO_3^-, $SO_4^=$ and $C1^-$ have been analysed by IC in the following atmospheric samples: gaseous phase components, (HNO_3, $HC1$ and NH_3); continuous fractions of rain events; size fractionated particulated matter. Individual particle analysis for NO_3^- and $SO_4^=$ at different depths were performed in these samples by LAMMA. Most NO_3^- and $SO_4^=$ sits on the surface of the seaspray. NO_3^- enrichment is predominant in particle size between 0.55 and 5.00 μm ($\geqslant 50\%$ of size 1.70 μm). This enrichment is found in all sites of the Recôncavo, including "remote" coastal sites. The origin of this NO_3^- has not been all identified. Seaspray has great influence in humid deposition. (GTZ, Stiftung Volkswagenwerk, COMCITEC, CNPq).

QA-69 Comparative Study of Atmospheric H_2O_2 in São Paulo and Bahia (Brazil). *P. Jacob, M. Schilling* (ISAS); *D. Klockow* (ISAS, Universidade de Dortmund, Alemanha); *U. QuaB* (Universidade de Dortmund, Alemanha) and *T. M. Tavares* and *V. Rocha* (Instituto de Química, UFBa).

Concentrations of H_2O_2 in atmospheric gas and liquid phases were determined in São Paulo city and in three different environments in the State of Bahia. Gaseous H_2O_2 were collected by cryogenic and rain by wet only sampling. Analysis were performed with the peroxyoxalate chemiluminescence method *in situ*. The measured gas phase concentrations in São Paulo city ranged from 0.009-0.38 ppbv, whereas in Bahia ranged from 0.2-3.9 ppbv. The corresponding concentration ranges in rainwater were 0.03-1,9 ppmw for São Paulo compared to 0.9-6,8 in Bahia. (GTZ, Stiftung Volkswagenwerk, CONCITEC, CNPq).

QA-70 Determination of $HNO_{3(v)}$ by Ion Chromatography After Sampling Onto Carbonate-Coated Cellulose Filters. *Nadia M. Aragão, Fátima Regina A. Justo* and *Jailson B. de Andrade*, (Instituto de Química - UFBA, Campus de Ondina 40.210 - Salvador, Bahia, Brasil).

A new method is described for the determination of $HNO_3(v)$ collected on carbonate-coated cellulose filters. Following extraction in deionized water and previous filtration the extract was directely injected in the ion chromatography system. A comparison study between this method and collection on NaC1-coated filters and quantification by spectrophotometry did not show significant differences.

QA-71 A Comparison Study Among the Methods of Direct Addition, U Tube and Diffusion Cell in the Determination of Benzene, Toluene and Xylene Adsorption/Desorption Efficiency Over Activated Charcoal. *Albertinho B. de Carvalho*[1,2], *Mina Kato*[1] e *Jailson B. de Andrade*[2]. [1]Fundacentro-Bahia e [2]Instituto de Química-UFBA, Campus de Ondina, 40.210 - Salvador, Bahia, Brasil.

We compared the methods of direct addition, U tube and diffusion cell to evaluate the adsorption/desorption efficiency of benzene, toluene and xylene over activated charcoal. The recovery efficiency for these substances were higher than 94%, for the three methods, while the temperature was 25±1 and the relative humidity ranged between 60% to 80%.

QA-72 Determination of Formaldehyde by HPLC as 2,4-DNPH Derivate After Sampling Onto Bisulfite-Coated Cellulose Filters. *Jailson B. de Andrade* (Instituto de Química-UFBA, Campus de Ondina, 40.210 - Salvador, Bahia, Brasil); and *Roger L. Tanner* (Brookhaven National Laboratory, Upton, New York, 11973).

A new method is described for determination of formaldehyde collected at high-volume flow rates as hydroxymethanesulfonate on bisulfite-coated cellulose filters. Following extraction in aqueous acid (HCl 1.0 M) and removal of unreacted bisulfite by heating, the hydroxymethanesulfonate is decomposed by base (NaOH 0.1M) and formaldehyde determined by 2,4-DNPH derivatization and HPLC. A limit of detection of 0.2 ppbV is achieved for 30 min sampling times.

QA-73 Development of a Fractionation Scheme for Characterization of Polar Particulate Organic Compounds Present in Ambient Air. *Lilian R. F. de Carvalho* (Instituto de Química - Universidade de São Paulo - Brazil); *Lara Gundel* and *Joan M. Daisey* (Lawrence Berkeley Laboratory - California - U.S.A.).

The acid-base partitioning was achieved for polar extract and HPLC results show that acid/base extraction does not separate into clean groups of acid/base/neutral fractions, in spite of the recovery of mass in the fractions has been significantly high. Another fractionation scheme based on polarity differences was developed. Ionic cromatography analysis show that a significant portion of the original polar extract is comprised of inorganics. The HPLC results from this late procedure suggest that this preliminary fractionated scheme is suitable for the characterization study of the polar organic compounds.

QA-74 Variation of Emission Rates of Reduced Sulfur Gases in Air from São Paulo City. *Arnaldo Alves Cardoso* (IQ. Araraquara, Dep. Quím. Analítica - UNESP) and *Luís Roberto M. Pitombo* (IQ. USP, São Paulo).

The diurnal variations in reduced sulfur compounds emission, particularly dimethyl sulfide (DMS) were observed in São Paulo city. Analysis of reduced sulfur gases, present in atmosphere, were done by using a chloropalladate(II) impregnated solid adsobent. The solid absorbs the reduced sulfur gases from the air flow, leading to the formation of a complex extractable into toluene. The UV absorbance of the dissolved complex is proportional to the amount of reduced sulfur gases. The result showed a regular diurnal cycle in the production of DMS, with highest emission rates in daylight and low rates over night.

QA-75 Determination of Copper in the Atmosphere of Belo Horizonte Metropolitan Region by Atomic Absorption Spectrometry. *Simone de Fátima Barbosa Tófani, Hyedda Nancy Sander Mansur* and *Silvânia Vaz de Melo Mattos* (Instituto de Ciências Exatas - Departamento de Química - UFMG); *Elias Mansur Netto* (Faculdade de Farmácia - UFMG).

Samples for Copper analysis were collected by sucction of a known volume of air, in Belo Horizonte atmosphere. It was used a glass fiber filter in collecting them, and we choose seven differents regions of the town, in the months of january and july. After dissolution of the samples in HNO_3 3M and HCI 3M, they were pre-concentrated in a cationic exchanger resin. Samples and standards were analysed by Atomic Absorption Spectrometry, in a air/acetylene flame. The mean concentration of Copper obtained has been $0.03 \mu g/m^3$, which fall below the maximum allowable content in comparison with results from other more industrialized countries.

QA-76 Peat: An Adsorbent for Anionic Detergents. *Célia Mª A. Matos* (Departamento de Química da Universidade Federal de Sergipe), *João S. d'Ávila* (Departamento de Engenharia Química da Universidade Federal de Sergipe), *Regivânia L. de Menezes* and *Patrícia L. de Carvalho* (Bolsistas da Nitrofértil).

The principal aim of the Peat Project, is the developpment of alternatives methods directed to environment protection, by using dried peat as an adsorbent material. The analytical methodology used to determine the adsorption capacity of peat related to anionic detergent was based in methilene blue calorimetric method. Preliminary results show, in the optimized analytical conditions of the process that: a) the average adsorption efficiency was 75%; b) the granulometry is about 10 – 30 #, the pH is about 5 and humidity is about 5%; c) all these results were obtained in a residence time equal to 15 minutes, in such a way that by increasing the detergent concentration, the quantity adsorbed also increases. (ITPS/FIPEC/UFS).

QA-77 Chemical Oxygen Demand (COD) Using Microwave Digestion. *Jarbas J. R. Rohwedder and Wilson F. Jardim* (Instituto de Química - Dep. de Química Analítica - UNICAMP - C.P. 6154 - Campinas - SP).

A new approach to determine the Chemical Oxygen Demand (COD) using a microwave digestion to replace the conventional time consuming reflux method is proposed. The procedure uses a microwave oven for the digestion of small volumes of sample (from 500 up to 2,000 ul), in a completely closed 60 ml Teflon vessel. For samples showing COD values up to 1,000 mg $0_2 1^{-1}$, the digestion step is completed in 7 min, compared to the 2 h needed for the conventional reflux method. An average precision of 1,0% was obtained using membrane filtered natural samples.

QA-78 Degradation of Phenol and Urea Under Solar Light Using TiO_2 as Catalyst. *Márcia M. Kondo, Wilson de F. Jardim* and *Ricardo J. Ferro* (Instituto de Química - Dep. Química Analítica - UNICAMP - Caixa Postal 6154 - 13081 - Campinas).

Photodegradation of urea and of phenol in aqueous solution were investigated under near-UV and visible radiation, using pure and Ag-loaded TiO_2 (anatase) as catalyst. In 20 min, a solution containing 100 $mg.1^{-1}$ of urea was almost completely degraded (83%) using Ag-loaded oxide, while for the pure form of TiO_2, only 16% of urea in solution was degraded. Similar results were observed when a $1.0 x 10^{-3}$ M solution of phenol was irradiated in presence of solar light. The degradation reached 39% and 85% when using pure and Ag-loaded oxide, respectively.

QA-79 The Behavior of Dissolved Inorganic Nutrients in the Real River Estuary. *J. P. H. Alves; C. A. Xavier; L. P. C. Romão* and *G. S. Sales* (Departamento de Química, Universidade Federal de Sergipe, 49000 Aracaju-SE, Brasil).

The Real River Estuary is part of the complex estuarine Piauí - Fundo Real, situated in the State of Sergipe, northeast of Brazil (LAT. 11º, 09-30'S; LOG 37º, 15-30'W). A first insight of the behavior of the nutrients is presented. The salinity distribution in the estuary, is governed by the local regime of rainfall. During the wet period (high rainfall) the colum of water is stratified, and during the dry period it is closed to homogenity. Phosphate levels are practically invariant over the whole salinity range of the estuary. Silicate exhibited, in general, a conservative behavior. The behavior of dissolved nitrate is non-conservative, except during periods of very high fresh water flow, when the supply increase and the utilization decrease.

QA-80 Determination of Heavy Metals in Water and Sediment of Paranoá Lake, Brasília. *Antonio C. Barbosa, Gastón A. E. Ponce* (Departamento de Química, UnB, 70910 - Brasília, DF); and *Romino B. Ornelas* (Companhia de Água e Esgotos de Brasília).

Zn, Cu, Pb and Cd were determined by ASV and flame AAS in water and sediment in six sites of Paranoá Lake of Brasília. Samples were digested with HNO_3–H_2O_2 for water and HNO_3–HC1 for sediment. The results showed higher levels of Cu, Zn and Pb near to the points of the lake having a high quantity of biomass. The maximum level of Cu, Zn and Pb found in water remained below the "minimum risk concentration" defined by CONAMA. However, the level of Cu in sediment in all the sites was higher than accepted by international conventions. The same is true for the concentrations of Pb and Zn in a few sites. The content of Cd remains below the limit of detection of the AAS method.

QA-81 Estudo da Densidade e do Número de Moléculas por Mililitro em Misturas Binárias Aquosas. *N. M. Rodrigo Leygue-Alba, Cláudio A. Perottoni* (Dept. de Física e Química - Universidade de Caxias do Sul - RS) and *Walter Martins* (UNICAMP - SP).

Studies of density and molecules number for mililitro (NMPm) are performed with lineals functions, in methanol, ethanol, 1-propanol, 2-propanol and glycerin - water systems. The diference between ideal functions and experimental data provide measures of the solvents interactions. Scale of interaction grade and indication by stechiometric relations in solvents association are proposed.

QA-82 Determination of Cd and Pb in Sediments and in the Bivalve Myte LLA Falcata of the Mundau Lagoon, Maceió Al, By GFAAS. *R. C. Campos*[1,2] and O. L. C. Amaral[1] · [1]Dep. de Química, PUC-RJ, [2]Dep. de Química, UFRRJ.

The determination of Cd and Pb in sediments and organisms of the Mundau lagoon by GFAAS was performed, after acid digeston, STPF conditions and two different background correctors were used. Zeeman GFAAS showed good agreement between the certified and found values of both elements in reference materials. D_2 compensation showed overcorrection due to fast background during the Cd determinations. The overcorrection can be avoided if the zero ramp is not used. An intermediate step between pyrolysis and atomization was also necessary.

APPLIED CHEMISTRY (AP)

AP-01 Fermentation of Sucrose Solutions by "S. cerevesiae" Immobilized on Activated Chrysotile. *Paulo José Samento Moran, Inês Joekes, José Augusto R. Rodrigues, Oswaldo Parizotto Junior, Beatriz Helena Lameiro de Noronha Sales and Maria Mary Kawakubo* (Instituto de Química, Universidade Estadual de Campinas).

The activities of immobilized and free cells in the fermentation of 12%, 20% and 30% sucrose solutions at 30 and 40°C in reactors solutions without shaking were compared. In all experiments, the rate of CO_2 evolution was higher and the reductor sugar concentration lower with immobilized cells. The ethanol concentration was always higher for the free cells. The immobilized cells showed a better performance than the free cells in the reuse experiments. The 12% sucrose solution was eluted in a column filled with immobilized cells during 18 days without losing its activity.

AP-03 Mutual Coagulation of Colloidal Gold and Iron Oxides. *Jacinta Enzweiler* (Instituto de Geociências, C. P. 6152) and *Inês Joekes* (Instituto de Química, Cx. P. 6154 - Universidade Estadual de Campinas - 13081 - Campinas, SP).

The interaction between colloidal gold and iron oxides may have some importance in secondary enrichment of lateritic gold occurrences. The mutual coagulation of colloidal gold with synthetic goethite and hematite was measured at 25°C in slightly acidic medium. It was observed that the presence of inert electrolyte until 10^{-2} M $NaNO_3$ enhances the mututal coagulation effect. On the other hand, the presence of ions such as citrate, which adsorbs specifically on the oxides, affects the interaction by lowering the p.z.c. of the oxides, inhibiting the gold adsorption.

AP-04 The Recovery of Gallium From Industrial Waste. *Mattieu Tubino* and *Maria Elizabeth Afonso de Magalhães* (Institute de Química, UNICAMP).

The gallium demand, on industrial market, have been increasing in the last few years, because of the application of this element in the electronic industry. This element is always found in nature at low concentration. Carbonation, one of the most used methods to concentrate gallium, brings the gallium concentration to 0.4-0.5% as Ga_2O_3. On the present work, we studied the gallium extraction and re-extraction from residual liquor of an aluminium plant, using bauxite as source. The obtained final gallium concentration in this work is significatively higher then that obtained by the carbonation process.

AP-05 Use of the Violet Crystal to Detect Surface Alteration of the Human Hair. *Graziella Ciaramella Moita* (Departamento de Química da Universidade Federal do Piauí).

The hair surface is a protective shell of the fiber. It's in contact with the environment, and it's directly attacked for it. Thus the adsorption capacity of dye from aqueous solution in hair surface may be used for detection of surface alteration due a environment agression and action of cosmetic products. The adsorption capacity of normal hair, bleached hair and smooth hair, in contact with solution of Violet Crystal 9.10^{-5}M during 8 hours, at room temperature is $1{,}55.10^{-5}$; $2{,}27.10^{-5}$ and $2{,}20.10^{-5}$ mols of violet crystal/gram of hair, respectively.

AP-06 Alterations of Viscosity of Comestibles Oils When Submitted to Thermic Treatment. *Iolanda da Cruz Vieira, José Arimatéia Dantas Lopes and José Machado Moita Neto* (Departamento de Química da Universidade Federal do Piauí).

Prolonged heating of comestibles oils in presence of air provoke the formation of oxipolimers. The extension of this reaction may be accompanied for viscosimetries measures. Comercial samples of maize oils were submitted to heating in water-bath by periods of 10, 20, 30 and 60 minutes. After this treatment were measured the viscosity and density of each sample, at temperature of 30°C. The results indicate sensibles alterations on viscosity of oils in the first 20 minutes of heating (increasing between 7 and 16%), and, flutuation around this values after 30 and 60 minutes of heating.

AP-07 Caracterization of Brazilian Coal Oils Obtained by Supercritical Liquefation. *José Luiz Ribeiro* and *Tania Maria Hubert Ribeiro* (Grupo de Carboquímica, Instituto de Química, Universidade Federal do Rio Grande do Sul).

Coal liquids are obtained by utilization of an apparatus for supercritical liquefation. Water is used as supercritical fluid. The liquid product thus produced is split into three fractions according to their solubility in different solvents. The oil fractions, are analysed using Infrared Spectroscopy, Gas Chromatography and Mass Spectrometry. Several hydrocarbons (saturated and aromatics) and phenols are isolated and identified.

AP-08 Coal Liquids Obtained by Sonic Treatment. *Tania M. H. Ribeiro* and *José Luiz Ribeiro* (Grupo de Carboquímica, Instituto de Química, Universidade Federal do Rio Grande do Sul).

The influence of an ultrasonic field on coal was studied by experiments on coal in liquid suspensions. The extraction yield depends on the time of sonic treatment and mineral content of coal.

AP-09 "Magnetic Susceptibility" and it's Relation Shits With Logic Lithology and Quantities of Fe, Mn, Zn, Cu, Ti and Co. *S. A. D. Ferreira, M. E. Fontes, D. R. Santanta, J. D. Fabris* (EMBRAPA - CNPMS).

The experiment was realized with eleven soils samples in order to determine the magnetic susceptibility and the quantities of Fe, Mn, Zn, Cu, Co e Ti. The characterization showed the basic evidences that the measurement of magnetic susceptibility (Faraday's Method) has relation with the folowing itens. (1) The origin material: susceptibility is more intensive in soils derived of mafic rocks. (2) The quantity of iron that is elevated in red soil richerd in iron. (3) The presence of ferrimagnetic minerals, particularly magnetite and maghemite. (4) The quantity of Zn, Cu, Mn, Co and Ti in the various fractions soils.

AP-10 Identification of psicoative substances in thin-layer chromatography using urine. *Teresa Maria de Jesus Ponte Carvalho, José Wilson de Alencar* and *Maria Iracema Lacerda Machado* (Departamento de Química Orgânica e Inorgânica da Universidade Federal do Ceará), *Helena Lutéscia Luna Coelho* (Departamento de Farmácia da Universidade Federal do Ceará).

A simple metodology for clinical diagnosis involving psicoative substances, chromatographic and spectrometric data is proposed to be implemented an program for microcomputer. The present comunication deals with the padronization of the chromatographic steps of the method to be pplied to samples of urine enriched with the standards to be tested. (FINEP, CNPq).

AP-11 Micronutrients in Sugar-Cane. *Zenice Chaves Pinto* and *João Augusto Caminha Barbosa Júnior* (Departamento de Química Aplicada – Universidade Federal de Pernambuco).

The aim of this research was to suggest proper techniques to analyse sugar-cane samples and defining analytical parameters to determine Zn, Cu, Mn and Fe by atomic absorption. The technique was to reduce the sample to ashes by evaporation and by acid treatment. The analyses were realized in two parts: immediately after the sample collection and after 6(six) months of storeage in a freezer. It was concluded that the acid treatment is the best indicated method for this type of analyses and that the samples can be storeaged during the proposed time without any alteration.

AP-12 Vegetable Tar Corrosion Power Reduction Over Iron–Steel. *Fernando Carazza, Marília Otoni da Silva Pereira, Flávio Marcelo Correia* (Depto. de Química, ICEx, UFMG).

The corrosion power of vegetable tar samples treated with H_2O and CaO, Na_2CO_3, and NaOH solutions over Iron–Steel, were compared. The best results were observed with samples treated with NaOH solution.

AP-13 NP Partialy Soluble Fertilizer: One Alternative for the Production From Natural Phosphate and Ammonium Sulphate(AS). *Arão Horowitz, Maria Olímpia Medeiros Carneiro* and *Graciliane Nobre da Cruz* (Departamento de Química Aplicada – Centro de Tecnologia – Universidade Federal de Pernambuco).

In recent years the partialy soluble phosphates had been shown to be more efficient for tropical soils than the soluble phosphates. They are generally obtained by treatment with sulphuric acid. As this country does not have appreciable sulphur deposits and this element is becoming more expensive the possibility to produce it by reaction of phosphorite with AS, obtained by the recovery of carbon dioxide wasted in alcohol production, is becoming increasingly interesting. The best fertilizer so far obtained was by the wet griding of phosphorite with equimolar proportion of AS and sulphuric acid.

AP-14 The Processing of Molasses Distilery Waste: From Polluter to Valuable Fertilizer. *Arão Horowitz, Maria Olímpia Medeiros Carneiro* and *Graciliane Nobre da Cruz* (Departamento de Química Aplicada – Centro de Tecnologia – Universidade Federal de Pernambuco).

It was shown that by adding fertilizer ammonium bicarbonate (AB) obtained by the recovery of carbon dioxide wasted in alcohol production, thereby greatly reducing the cost of ethanol, the molasses distilery waste could be transformed into a valuable fertilizer. On the other hand 30% of the potassium in this waste is containd in the organic matter and so is not easily leachable. From the results it was possible to conclude that 40 m^3/ha of the waste treated with adequate amount of AB so as to give the appropriate amount of nitrogen are to be used for optimum yield of sugar cane crops.

AP-15 Catalitic Converstion of Oil From Para Rubber Seeds *(Hevea brasiliensis)*. *Maria Helena da Silva Bentes* (Chemical of Natural Products/CCEN/UFPA), *Gerard Diegá–Mariadassou* (Laboratoire de Réactité de Surface et Structure/U. Paris VI) and *Harry Serruya* (Chemical of Natural Products/CCEN/UFPA).

The data obteined by hydrocracking should be used to compare the mechanism reaction of refined and crude vegetal oil. The oil of Para rubber seeds *(Hevea brasillensis)* was analysed before the reaction and submited to hydrocracking process later at discontinuous reactor.The products were analysed by CG in capillary column. Despite the reactor's limitations, it has been possible to observe difference in composition of resultant products of soy–bean refined and Para rubber crude oils. It was observed, also, that the time of contact of the reagent, inicial pressure and the time of reaction change the results. In same conditions, the crude oil from Para rubber was superior to the refined soy–bean oil and it would be apply to producing alternative fuel.

AP-16 Essential Oils and Fine Chemicals(III). Myrcene Derivatives. *Afrânio Aragão Craveiro, Ícaro Gusmão Pinto Vieira, Francisca Noélia Pereira Mendes* and *João Capistrano Nobre de Abreu.* (Fundação Centro de Pesquisa e Desenvolvimento da Indústria Química do Estado do Ceará – Laboratório de Produtos Naturais – Departamento de Química Orgânica e Inorgânica – Universidade Federal do Ceará).

Myrcene a major by-product from Lemongrass essential oil destillation is being used as starting material for the production of fine chemicals used in perfumery and cosmectics. Thus reaction of hidroclorination gave a mixture of geranyl and linalyl chlorides in 79,5% yield. These chlorides were transformed in the acetates using sodium acetate in 80% yield. The late reaction was studied in the presence of several catalysts and solvents. (CNPq/FINEP/MCT/RHAE).

AP-17 Essential Oils and Fine Chemicals (II). Eucaliptus Oils and Their Derivatives. *Afrânio Aragão Craveiro, Ícaro Gusmão Pinto Vieira, Francisca Noélia Pereira Mendes, Sandro Maia Sobreira.* (Fundação Centro de Pesquisa e Desenvolvimento da Indústria Química do Estado do Ceará – Laboratório de Produtos Naturais – Departamento de Química Orgânica e Inorgânica – Universidade Federal do Ceará).

Destillation of Eucaliptus oil gave pure citronellal. Citronellal was transformed in Isopulegol using silica in hexame with yield of 83%. Hidration of Isopulegol with H_2SO_4 68% produce the p–menthane–3,8–diol with 50% yield. Transformation of citronellal to hydroxicitronellal was done with H_2SO_4 58 to 78% in 36.4% yield.

AP-18 Castor Oil in Thin Chemistry (II) Esterification of Undecanoic Acid. *Afrânio Aragão Craveiro, M. G. Vasconcelos Silva, C. R. B. da Silva* and *P. Q. da Costa* (Fundação Centro de Pesquisas e Desenvolvimento da Indústria Química do Estado do Ceará – Laboratório de Produtos Naturais – Universidade Federal do Ceará).

Castor oil is largely used as feedstock in the industry. In contimation to the studies of transformation of this oil in more valuable materials, it is described the hydrogenation of undecilenic acid and the esterification of undecanoic acid with glycerol giving the dihydroxi, ester (IV) which has aplication in cosmetic. MCT/RHAE/FINEP.

AP-19 Kinetic of Fluorescent Whitening Agent Absorption in Cotton Fibers. *Judith Feitosa Rodrigues* and *Cláudia C. B. S. Carneiro.* (Departamento de Química Orgânica e Inorgânica da Universidade Federal do Ceará).

Fluorescence emission at 434 nm, with excitation at 350 nm, was employed to study the kinetic of fluorescent whitening agent (FWA) absorption in cotton fibers. When 0,64 g of textile contacted with aqueous solution of detergent OMO (0,3%), at 25°C, 85% of whitening agent was absorbed, independent of operation process. Time to reach the saturation point was 10 and 60 hours, for the system with and without agitation, respectively. To absorbe 50% of FWA the textile, in agitation process, needs 10 minutes, while, without agitation, was necessary 8 hours of contact.

AP-20 Decolorization of Bleached Kraft Effluent Lignins by Photochemical and Biological Systems. *Nelson Durán, Jaime Rodriguez* and *Márcia Dezotti* (Inst. Química – UNICAMP) *Irene Ferrer* (Pont. Universidad Catolica de Chile) and *Lucia Innocentini-Mei* (Eng. Química – UNICAMP).

Chrysonilia sitophila, an Ascomycete, showed high ligninolytic ability in numerous woody materials. This report defines the requirement for the decolorization of Kraft bleachery caustic extraction stage effluent by this fungs in free pellets and immobilized conditions. The best results with mycelial pellets was 86% of decolorization in 16 h at 27°C. Better results with the immobilized fungi were obtained. Kraft effluent was oxidatively decomposed by a semiconductor–photocatalyzed reaction containing oxygen and the semiconductor ZnO. Under continued illumination (1 h) the effluent became colorless and the chemical oxygen demand value decreased. The combined bio–and photochemical decoloration showed a good potentiality in the effluent treatment for pulp and paper industry.

AP-21 Polyvinyl Alcohol Degradation by Microorganisms. *Leda Coltro, Nelson Durán* (Inst. Química – UNICAMP), *Pamela Cañas, Monica Riffo, Victoriano Campos* (Fac. Cienc. Univer. Catolica Valparaiso, Chile).

Polyvinyl alcohol (PVA) degradation has been principally investigated with bacteria. In this work we have studied 27 strains of different aerobic bacteria and 3 fungi, using the PVA as carbon sources in the nutrient. The most efficient bacteria were the folowing: *B. pumillus, B. subtilis, Ps. aeruginosa, E. aerogenes, S. aureaus, K. pneumoniae, A. calcoaceticus* and the fungi *C. sitophila* and *C. clodosporoides* and one unknown strain. The parameters studied in this work with fungi were the following: pH variation, growth (dry mycelium) viscosity(hydrolase activity), phenoloxidase activity and secondary alcohol oxidase activity. The results have shown that fungi are relatively efficient in PVA degradation when compared with those reported by bacteria.

AP-22 Formic Acid Pulping of *Eucaliptus grandis* Associated With a Biological Pre-Treatment. *Norma de Magalhães Erismann, Marco Alarcón, Nelson Durán* (Instituto de Química – UNICAMP), *Juanita Freer* and *Jaime Baeza* (Department of Chemistry, Faculdad de Ciencias, Universidad de Concepcion, Chile).

Organosolv pulping mediated by formic acid of untreated and pre–treated *Eucaliptus grandis* chips with *Chrysonilia sitophila* was evaluated. The best condition for untreated chips were found with formic acid (99%) at 95°C, 0,22%(w/v) catalyst (HC1), charge 1/10. The pulp yield was 43%, kappa number 31, reject content 0,1% and 8% of Klason lignin. Preliminary results show that a microbiological pre–treatment of chips improves the delignification of the final pulp. In this case, the kappa number were 46 and 39±3, Klason lignin 15% and 11±1% for the organosolv pulp obtained with chips of the steril control and pre–treated with *C.sitophila*, respectively.

AP-23 The Good Use of Vegetable Tar From Wood Pyrolysis. Bromination With NBS. *Fernando Carazza, Ana Maria de Resende, Nelson de Souza Pereira* (Depto. de Química, ICEx, UFMG).

During the studies to improve vegetable tar constituents separation, the NBS bromination of several phenols derivatives were performed. The conditions for aromatic bromination of anisole, veratrole, 1,2,3–trimethoxy–benzene, phenyl acetate, guaiacyl acetate, and 2,6–dimethoxyphenyl acetate were stablished. The phenyl and guaiacyl didn't react under the conditions used. The benzilic bromination of 4–propylguaiacyl acetate, and 4–proyl–veratrole with NBS were tried, without succes for the last compound.

AP-25 Chemical Analysis in Chilean Native Hardwood Lignins. *Héctor Mansilla, Marcela Haun*, Nelson Durán*** (Departamento de Ciencias Biológicas, Universidad de Talca, Casilla 747, Talca, Chile.(*) - Instituto de Biologia e(**) Instituto de Química, Universidade Estadual de Campinas, CEP 13081, Campinas, SP.

Dioxane lignins extracted from five Chilean native hardwoods *(Nothofagus oblicua, Nothofagus glauca, Lithraea caustica, Quillaja saponaria* and *Cryptocarya alba)* were chemically characterized. Relative content of functional groups (∅OH, C=O, OCH_3) were correlated with the light emission in the HRP/H_2O_2 enzymatic system. Free phenolic hydroxyl content is closely conected with the chemiluminescence intensity.

AP-26 Pyrolisis of *Euphorbia Copiapina* Dichloromethane Extract With Zeolitic Catalysts. *Sara Gnecco, Marcela Montecinos, Ruby Cida, Rosa Catalán* (Departamento de Química, Faculdade de Ciencias, Universidad de Concepción. Casilla 3–C. Concepción – Chile).

Euphorbia copiapina, a chilean species growing in arid areas, is a promising source of botanochemicals compounds. CH_2C1_2 extract from stems and leaves was pyrolyzed with various zeolitic catalysts, under different experimental conditions. Conversion degree and quality of pyrolytic products were estimated by means of thermogravimetry, differential scanning calorimetry and combustion heat. Results show that, besides ZSM–5 Mobil, the synthetized catalyts: Co(1) NaY, Mo(3) NaY and Co(1) Mo (0.5) NaY were the most promising.

AP-27 Bark as Formulating Agent for Controlled Release Herbicides. *Graciela Palma* (Universidad de la Frontera, Tenuco (CHILE), *Juanita Freer* and *Jaime Baeza* (Depto. Química, Universidad de Concepción (CHILE)).

Bark of *Pinus radiata* D. Don, modified bark (CH_2O/HNO_2), phlobaphenes and catechin (as model) were used as formulating agents for controlled release pesticides either by chemical and physical methods. The pesticides incorporated were trichloric acetic acid (TCA) and 2,4–dichlorophenoxiacetic acid (2,4–D). The modified bark incorporated about 25% of 2,4–D by chemical method and 15% by the physical one. Release rates have been determined under "in vitro" conditions by inmersion of the formulation in water at pH 5.5.

AP-28 Biomimetic Reaction in the Delignification of *Pinus Radiata* D. Don and *Eucalyptus Globulus*. *Ana M. Fernández* (Depto. Ingenieria en Maderas, Universidad del Bio-Bio (CHILE); *Juanita Freer, Jaime Baeza* (Depto. de Química, Universidad de Concepción (CHILE); and *Nelson Durán* (Instituto de Química, Universidad Estadual de Campinas, S.P. (BRASIL).

Chips and organosolv (formic acid) from *Pinus radiata* D. Don and *Eucalyptus globulus* and pine Kraft pulp were treated with hemin (as a biomimetic model) and hydrogen peroxide in aqueous solution at reflux temperature. Analyses of treated pulps and chips indicate selective delignification mainly in *E. globulus*. Excellent bleaching of pine Kraft pulp was obtained.

AP-29 Separation of Unsaturated Fatty Acids From Fish Oil. *A. A. Craveiro* and *M. F. G. Lopes*. (Laboratório de Produtos Naturais – Universidade Federal do Ceará).

Saturated and unsaturated methyl ester of fatty acids from fish oil were separated using oclusion complex with urea. The saturated methyl esters are trapped inside the urea complex whereas the unsaturated esters are excluded by solvent extraction. This process was used with sucess in the separation of acids from fish oils from Brazilian Northeast and commercial preparation. (MCT/RHAE/FINEP/CNPq)

AP-30 Organosolv Pulping of Wood of *Pinus Radiata* D. Don. *Silvio Urizar, Ana Pedreros, Juanita Freer, Eckhard Schmidt* and *Jaime Baeza* (Laboratorio de Recursos Renovables, Depto. de Quimica, Universidad de Concepción, Concepción-CHILE).

Formic acid was found to be an efficient delignify for hardwood *(Eucalyptus globulus)* at milder conditions. For softwood *(Pinus radiata)* stronger conditions were needed. The conditions for pulping the pinus wood were evaluated. The following range of conditions has been tested for pinus: Formic acid concentrations: 85–98%; HC1 concentrations: 0.025–0.05 %p/v; Pretreatment: wet chips were autoclaved at 120°C for 1 h, and soaked in pulping licor for 1 h; Heating time: 120 min; Cooking: 90°C, 180 min. The yields obtained are in the range from 58.1 to 71.3% and the Kappa number range from 84 to 101.

AP-31 Chrysonilia Sitophila (TFB–27441 Strain): Production, Partial Purification and Characterization. *S. O'Reilly*, E. Curotto*, R. Torres*, V. Campos*** and *N. Duran**** (Instituto de Química*, Instituto de Biologia**, Universidad Católica de Valparaíso, Casilla 4059, Valparaíso, Chile e Instituto de Química, Universidad Estadual de Campinas, Brazil***).

Chrysonilia sitophila, a fungus from Tribolium ferrugineum a brazilian insect found in rice, is used to study its production of extracellular β–glucosidase. Cellobiose, pretreated Pinus radiata and Eucalyptus globulus sawdust where used as carbon source. Crude extract β–glucosidase induced by cellobiose was purified and kinetically characterized.

AP-32 Formic Acid–Lignins From *Pinus Radiata* D. Don. *Susana Flores, Juanita Freer, Carlos Aguilera* and *Jaime Baeza* (Laboratorio de Recursos Renovables, Depto. de Química, Universidad de Concepción, Concepción–Chile).

Chemical and spectroscopic characterization of formic acid lignins from *Pinus radiata* D. Don is presented. The chips were pulped with formic acid–HCl(98%–0.02%). The samples (charge 20:200 g wood/ml solvent) were first preheated slowly (1.5 h between 20–50°C and 1 h between 50–90°C) and then cooked at 90° for periods of 0, 45, 90, 135 and 180 min. The content of metoxyl groups obtained are in the range from 12 to 8%, while the content of phenolic hydroxyl groups range from 9.11 to 1.98%.

BIOLOGICAL CHEMISTRY (QB)

QB-01 Quantitative Structure Activity Relationship for the Lethal Effect of Hydrochlorides of N,N-[(Dimethyl amino)Ethyl] 4-Substituted Benzoates: *A. T. do Amaral, Y. Miyazaki, L. Caprara* and *A. C. Oliveira* (Chem. Dept., I. Q.; Dept. of Pharmacol., I.C.B., USP, C.P. 20780, CEP-01498 SP, Brasil).

A classic QSAR analysis has been carried out on the set of the title compds. Application of stepwise regression analysis has shown that both electronic and lipophilic effects, approached, at first, in terms of σ and parameters, contribute to the lethal effect of the compounds, evaluated, experimentally, by means of the LD_{50}.

QB-02 SB-73: An Immunomodulating Drug, its Synthesis, Characterization and Biological Activities. *Nelson Duran* (Instituto de Química, UNICAMP), *Marcela Haun, Fawcy Dawood, Elisabeth C. Ficher, Alba R. M. Souza-Brito, Lucia da Silva* (Instituto de Biologia, UNICAMP), *Regina Pisani, Francisco Pisani* (Esc. Medicina, UNICAMP), *Denise B. Ciampi* (Centro Corsini), and *Odilon D. S. Nunes* (CEDECAB).

We have developed a new biosynthetic compound (SB-73) which has immunomodulatory properties and atoxic SB-73 exhibited antiviral activities in vitro against Herpes Type 1 virus, Stomatitis vesicular virus and Adenovirus Type 5. Acts efficiently on Canine Parvovirus and Distemper virus infections, Lethal dose (LD[50]) was 3 g/Kg in mice intraperitonealy inoculated. It is non toxic, cytotoxic or genotoxic to limphocytes. SB-73 exhibited antineoplastic activities on lymphosarcoma-180 and Ehrlich sarcoma. Due to the wide range of biological activities, probably through its immunomodulating capacity, and no toxicity, SB-73 appears as potential drug in treatment of immunodepressive deseases. (Supported by CEDECAB).

QB-03 Structural Parameters and Analgesic Activity of 5-Arylamino-3-Methyl-4-Nitro-N-Phenylpyrazoles. *C. J. C. Correa* (IQ, UFRJ), *E. J. Barreiro, A. C. C. Freitas* e *A. L. Pereira* (DTF, Fac. Farm., UFRJ).

In a project aiming at bioactive pyrazoles, a series of 5-arylamino-3-methyl-4-nitro-N-phenylpyrazoles has been prepared by reaction of the corresponding 5-chloropyrazole with substituted anilines. Several of these compounds showed analgesic activity. With the goal of establishing a QSAR profile for these substances, as related to the influence of the substituent in the arylamino group, equation 1 was generated: $\text{Log } 1/ED_{50} = 1.97\pi^2 - 0.98\pi - 1.96\sigma_m - 0.19\sigma_p + 0.28E_s + 1.15$ (1) (n=9; R = 0.94; Standard Mean Deviation = 0.18). Obtained after eq. 1, the p–CF_3 analog was quite active.

QB-04 Synthesis and Antibiotic Activity of Mesoionic Compounds 1,3,4-Thiadiazolium-2-Aminide. *Astréa M. Giesbrecht, Cristina Z. Nassis* (Instituto de Ciências Biomédicas, USP), *Carlos A. Montanari* (Instituto de Ciências Exatas, UFPO), *Yukino Miyata* and *Joseph Miller* (Instituto de Química, USP).

Previous studies of antibiotic activity of 1,3,4-oxadiazolium-3-aminide mesoionic compounds, showed that they possessed strong antibacterial and antifungal properties. Seven new mesoionic compounds from the system 1,3,4-thiadiazolium-2-aminide and the corresponding chlorides, were now synthetized and tested against several bacteria, yeasts and dermatophytes, by the cupplate agar diffusion method. Five pairs of compounds (chlorides and free bases) showed strong activity against the Gram-positive bacteria, being the chlorides more potent than the bases. (CNPq, CAPES).

QB-05 Determination of Lipophilicity of Substituted N-Benzilideen-3,3-Diphenylpropilamines in Octanol/Water System. *Marta Albernaz, Aurea Echevarria* (Dep. Química, Universidade Federal Rural do Rio de Janeiro) and *M. Graça Nascimento* (Dept. Química, Universidade Federal de Santa Catarina).

The study of drug potency in biological systems requires an understanding of chemical structures in terms of physical-chemical properties: transport and distribution of a drug. The largest progress has been made in describing the lipophilicity of drugs and in understanding the dependence of drug activity on lipophilicity. The diphenylpropilamines Schiff bases showed reasonable biological activity against *Candida albicans*. The lipophilicity values were obtained by partition coefficients in octanol/water system. The measurements of partition coefficients were carried out by determining concentrations in both organic and aqueous phases by U.V. spectroscopy. No good linear relationship was obtained between π and σ, indicating that factors other than electronic are involved.

QB-06 Schistosomicidal Activity of 2-(Alkylamino)-1-octanethiosulfuric Acids II. *Marcus L. O. Penido, David L. Nelson* and *Dorila Piló-Veloso* (Departamento de Bioquímica-Imunologia, ICB; Departamento de Química, ICEx, UFMG, Belo Horizonte, MG, Brasil).

Utilizing the sequence of reactions presented previously (Penido, Nelson & Veloso, Ciência e Cultura *40* (7): 480 (1988), two new zwitterionic Bunte salts have been synthesized: 2-[(methylpropyl)amino]-1-octanethiosulfuric acid mp = 219-22°C (d) and 2-(propylamino)-1-octanethiosulfuric acid mp = 207-9°C (d) in 90% and 84% yields, respectively. Spectral analyses were in accord with the structures proposed for the products. Contrary to the acids tested previously, no schistosomicidal activity was observed for these products with the doses utilized (300 mg/kg/day x 5 days) in experimentally infected mice.

QB-07 Oxidation of Cis-1,2-Bis(Hydroximethyl) Cyclohexane (BHMC) Catalyzed by Horse Liver Alcohol Dehydrogenase. *Lucia M. C. Paiva* - (Instituto Nacional de Tecnologia); *Gerson F. Pinto* and *Enrique G. Oestreicher* - (Depto. de Bioquímica do Instituto de Química da UFRJ).

A kinetic study of the oxidation of BHMC by horse liver alcohol dehydrogenase has been carried out in order to identify kinetic factors that can be significant in preparative-scale asymetric synthetic applications of the enzyme. Initial rate studies performed with variable concentrations of each substrate (BHMC and NAD) at different fixed concentrations of the other, showed that when large concentration of BHMC where used, activation by the substrate was present whereas, inhibition by excess of NAD is observed at low concentrations of BHMC. This complex behaviour is explained by a mechanism involving alternative pathways for formation of the reactive ternary complex and the formation of an abortive complex E-NADH-BHMC from which NADH dissociates more rapidly than from the normal E-NADH complex.

QB-08 Evaluation of the Isozymes L_1 and L_3 of Soybean by the Carotene Bleaching Method. *Vera Maria Klajn* and *Maurílio Alves Moreira* (Departamento de Química, Universidade Federal de Viçosa. 36570 - Viçosa-MG-Brasil).

Lipoxygenase isozymes from soybean seeds catalyses the hydroperoxidation of poliunsatured fat acids. These reactions are considered to be the main cause of the beany and bitter flavor normally associated with soybean products. Lipoxygenase secondary reactions also promotes pigment bleaching such as the carotene bleaching. Based upon this principle it has been possible to adapt a very sensitive and non-destructive methodology to detect the presence/absence of lipoxygenases L_1 and L_3 in F_2 seeds from a breeding program oriented to eliminate these isozymes in soybean seeds.

QB-09 DNA Transformations by Carbon-Centered Radical. *Giselle Z. Justo, Paolo R. Livotto, Nelson Durán* (Instituto de Química, UNICAMP) and *Adelaide Faljoni-Alário* (Instituto de Química, USP).

In this communication we report the specificity of the carbon-centered radical, 2-phenylethyl, to the DNA attack. Experiments with Tb(III) and DAPI fluorescence demonstrated that these radicals are able to attack Calf Thymus DNA, leading to steric effects on the double helice. The same was observed by reaction with formaldehyde. In separate experiments, we also establishment the induction of DNA strand breaks, when the plasmid pBR 322 was submitted to the radical reaction. Theoretical correlations evaluated the possibility of a free radical mechanism and pointed out the guanine base as the preferencial site to the attack. Supported by FAPESP, CNPq, FINEP, OAS, CT/INTRA (BID) and UNESCO programms.

QB-10 Swelling Studies of Connective Tissues in DMSO:Water Mixtures - *Cassia Maria Eiko Yasuoka Moriwaki* and *Gilberto Goissis* (Instituto de Física e Química de São Carlos - Departamento de Química e Física Molecular - USP - Cx. P. 369 - 13.560 - São Carlos - SP - Brasil).

A study on the swelling of connective tissue at variable DMSO concentration and ionic strength was undertaken. The main observed effect was an increase on the swelling capacity by factors up to 14 times, on comparative basis. The molecular intergrity of the tissue was shown to be dependent on DMSO, salt concentration as well as the order of cations addition. The observed overall effect may depend on the disruption of native hydrogen bonds present associated with a decrease of hydrophobic interactions, responsible for the stability of the microfibrils. (CNPq).

QB-11 Purification and Kinetic Characterization of Chymopapain B and Papaya Proteinase from Latex of *Carica papaya*. *Carmelita A. Basilio* and *Pedro L. O. Volpe* (Instituto de Química, UNICAMP), *Lúcia Pereira-da-Silva* (Instituto de Biologia, UNICAMP).

A purification method for papaya proteinase latex was developed which permited almost complete purification of these enzymes in a single step. This method uses column chromatography on CM-celulose of the latex extract previously dialysed against acetate buffer, pH 5.0, and elute with the same buffer at increasing ionic strenght. Each enzyme, chymopapain B and papaya proteinase, purified by this method, showed a single protein component when submitted to 15% polyacrylamide gel electrophoresis at pH 4.5, in a nondenaturing system. The kinetic parameters, K_M and V_{max}, were determined using casein as substrate. (CNPq - FAP).

QB-12 Galactomannan from the Seeds of *S. Barbatiman* (Barbatimão). *Sirlei C. S. Leitner, João B. C. Corrêa, Maria R. Sierakowski, Joana L. M. S. Ganter* and *Fany Reicher* (Departament of Biochemistry and of Chemistry, UFPR .. P. O. Box 19046 (81504), Curitiba, Paraná, Brasil).

Powdered, defatted seeds of the native leguminosae *S. barbatiman*, were extracted with water, producing a viscous solution. Ethanol precipitates were obtained (yield 30%) and g.l.c.analyses of derived alditol acetates and C^{13}-n.m.r.showed the presence of mannose and galactose in the ratio of 1.5:1.0. The galactomannan was freed from protein and analyzed by gel filtration on Sepharose CL 4B(mol.wt.-1.5 10^6). Other galactomannan fraction treated with Fehling or TCA 2.5% showed Mol.wt.2.5 10^5, suggesting molecular dissociation and/or degradation. The anomeric configuration of mannose is β and the galactose is α. (Finep, CNPq and UFPR).

QB-13 Polysaccharides from the Seeds of *Senna Multijuga*. *Carem Gledes Vargas, Fany Reicher, Maria Rita Sierakowski, Joana Léa Meira Silveira Ganter, João Batista Corrêa* (Departament of Biochemistry and Departament of Chemistry of UFPR., P.O. Box 19046 (81504), Curitiba, Paraná, Brasil).

The seeds of S. multijuga were powdered, deffated, extracted with water (room temp.), and treated with ethanol, resulting in 2 insoluble forms. The major one, a galactomannan (man:gal 1.8:1) was purified by Fehling precipitation. The second one was treated with Cetavlon and precipitate (pH 7) contains $\underline{O}$-acetyl groups (confirmed by ^{13}C n.m.r. δ 173.23 $\underline{C} = O$ and δ25.75 $\underline{C}H_3$). Their location was analysed by the phenylcarbamate mathylation procedure of Bouveng (FB) and the modified method of Corrêa et al (FC). Resulting partially $\underline{O}$-methylated products were hydrolised and converted into a mixture of partially $\underline{O}$-methylated alditol acetates. G. l. c. analyses showed 2,3-di-$\underline{O}$-methyl xylose, monomethyl xylose, arabinose and xylose (19:12:4:65 FB and 16:15:3:64 FC). (Finep, CNPq and UFPR.)

QB-14 Studies of Lignin Biodegradation During Wood Decay by *Chrysonilia Sitophila*. *André Ferraz and Nelson Durán*, (Instituto de Química, Biological Chemistry Laboratory), UNICAMP C.P. 6154 Campinas S.P. Brazil.

We have studied the lignin biodegradation during decay of *Pinus radiata by C. sitophila*. This strain showed previously ability to degrade lignin and lignocellulosic materials. Milled wood (MWL), neutral di-oxane (NDL) and methanol (ML) lignins of undecayed and decayed (3 months) *P. radiata* were isolated and chemically characterized. MWL degraded and undegraded gave MW > 28,000 no change in phenolic and $\gamma - C = O$ groups were observed. However $\propto - C = O$ was increased in the latter. Very low phenolic groups in NWL was found. Spectral characterization (UV, IR, and NMR) of these lignin indicated the same type of changes. (FAPESP, CNPq and FINEP.)

QB-15 Cellulose, Lignin, Hemicellulose and Extractives Transformation from *Eucalyptus Grandis* Wood by *Thermoascus aurantiacus*. *Angela Machuca* and *Nelson Durán* (Instituto de Química, Laboratório de Química Biológica, UNICAMP, Campinas, Brasil).

Thermoascus aurantiacus, a brazilian strain, isolated from piles of Eucalyptus chips produced cellulases and ligninases in the culture fluid. The cultivation of *T. aurantiacus* at 50° C with E. grandis sawdust (45-60 mesh) and 0.3% glucose resulted in weight loss of 5.4% after 21 days. *T. aurantiacus* degrades only 11.3% of lignin and the levels of cellulose and hemicellulose didn' t show changes after the treatment. The fungal attack was more efficient on extractives than the other components of the wood above mentioned. The extractive loss was 53.7% in the 21 days period. Supported by CNPq, FAPESP, FINEP.

QB-16 Cellular Targets for the Toxic Action of a Riboflavin-Tryptophan Photoadduct. *Hoffmann, M.E., Soraggi, C.L.* (Universidade Estadual de Campinas, Departamento de Bioquímica, Campinas, S.P. Brazil) and *Silva, E.. (Pontificia Universidad Catolica de Chile, Facultad de Quimica, Santiago, Chile).*

It has been previously shown that one photoadduct of riboflavin and L- tryptophan induces hepatic dysfunction in rats (Donoso et al., Nutr. Rep. Internat., 37:599, 1988). In the current work we report the induction of cytostatic and cytotoxic effects of this adduct, following exposition of V79 hamster fibroblasts to the irradiated solution. Attempting to evidenciate the involvement of the DNA as the target for these toxic effects we analysed the DNA synthesis rate in adduct exposed cells. We have found that toxic concentrations of the adduct were not able to inhibit the DNA replication in V79 cells. This suggests that other key-components are the targets for the photoadduct toxic action.

QB-17 Amminoacid Sequence Analysis Computer Program Prototype. *R. A. Müller* (Departamento de Química – Universidade Federal de Viçosa).

Accuracy, velocity and fitness will be achieved with the aid of a fortran computer program for nucleic acid sequence analysis. The program can perform the ending code detection, codon setting, aminoacid matching, base inclusion and exclusion, cistron localization with frequency numbering and aminoacid comparation.

QB-18 Heterogeneity Study in Phospholipid Bilayers. *Margarita Baganara* and *Carlos P. Sootmayor* (Instituto de Química, Universidad Católica de Valparaíso, Chile).

Fluorescence decay of certain compounds that are monoexponential in normal solutions are multiexponential when they are included in lipid bilayers. This fact can be related with membrane heterogeneity and has been used to study changes in dimyristoylphosphatidylcholine bilayers caused by different agents. DPH fluorescence decay was studied using multifrequency phase and modulation fluorometry. The data obtained was analyzed using a continuos distribution in order to consider bilayer heterogeneity.

QB-19 Comparative Studies of the Production of Carotenes in four Species of Dunaliella. *Sonia Erazo*; Andrés Markovitcs**; Gustavo Parada*** and *Pia Gianelli***. (*Instituto de Quimica and **Escuela de Ingenieria Bioquimica, Universidad Católica de Valparaíso, Casilla 4059, Valparaíso, Chile).

Four halotolerant microalgaes were cultivated and the growth, production of carotene and the relative amounts of their isomers were compared. The isomers were separated by HPLC. In the two chilean algae, D. utex and D. bardawil the contens of α-carotene were higher than in β-carotene were detected, with different relativa amounts. In the chilean specie RMB18 the mayor constituent of β-carotene was the isomer 9-cis-β-carotene.

INORGANIC CHEMISTRY (QI)

QI-001 Electron Transport in Proteins. *A.A.S. da Gama* (DQF/UFPE 50739 - Recife-PE).

We have developed a Green's function technique to solve the Dyson's equation for a model systems, donor-intermediate-acceptor which represents the electron transfer path way in proteins. Simple Huckel parameters are used for calculation in real systems.

QI-002 Ternary Complexes in Copper(II) – Histidine – Hydroxamic Acid Systems: Spectrophotometric Titrations in The Visible. *Eucler B. Paniago* and *Sandra Carvalho*, (Dep. Química – ICEx/UFMG).

Spectrophotometric titrations of solutions containing copper(II) ions, histidine (his) and acetohydroxamic (aha) or glycinehydroxamic (gha) acid wer made in the visible region at 25 °C, I=0,1 M $(NaC10_4)$. With the Cu(II)-his-aha system, as the pH is raised, there is a shift to lower values of the wavelenght corresponding to the absorption maximum, which can be attributed to the appearance of one neutral (610 nm) and one negative ternary species (590 nm), which are the predominant species at higher pHs. These findings are in good agreement with the calculated species distribution. Similar results were obtained with the Cu(II)-his-gha system, for which a protonated species was also proposed.

QI-003 Equilibrium in The Fe(III)-Acetohydroxamic Acid System. *Eucler B. Paniago*, (Dep. de Química); *Emílio Suyama*, (Dep. de Estatística) and *Liliam V. Leonel*, (Dep. de Química) (ICEx-UFMG).

The interaction between Fe(III) and acetohydroxamic acid was followed spectrophotometrically in the pH range 2.2-5.4. Results of factorial analysis gave 4 as the number of light absorving species in this system. Using the program SQUAD (Legett et al, 1975), formation constants and molar absortivities in the range 300-600 nm for the following species were determined : FeL ($\log \beta = 11.06$), FeL_2 ($\log \beta = 20.79$), FeL_3 ($\log \beta = 28.45$).

QI-004 Alkane Hydroxylation Catalyzed by Iron Porphyrins. I-Atropisomers of Porphyrins 2-N-Alkyl-Pyridyl. *Ynara Marina Idemori* (Instituto de Ciências Exatas - Deptº de Química - Universidade Federal de Minas Gerais), *Osvaldo Antonio Serra, Soraya Saadeh Albuquerque, Yassuko Iamamoto* (Faculdade de Filosofia, Ciências e Letras de Ribeirão Preto - USP).

The hydroxylation of unactivated alkanes has been observed in a catalytic systems containing iodosylbenzene and various iron prophyrins. The two fractions $\alpha\beta\alpha\beta + \alpha\alpha\beta\beta$ (A) and $\alpha\alpha\alpha\alpha + \alpha\alpha\alpha\beta$ (B) atropisomers of Chloro (5,10,15,20-Tetrakis-tetradecyl-2-pyridylporphyrinato) iron (III)Fe(TMy2PyP)C1] were isolated. These fractions have been used as catalyst in the hidroxylation reaction of cyclohexane in acetonitrile at O°C in aerobic and anaerobic system and iodosylbenzene as oxidant. For the fraction B, the low concentration enhance the catalytic activity with higher yield.

QI-005 Study of Intermediate Species in the Reactions of Iron-porphyrin $FeTM_4$–PyP $(C10_4)_5$ and Iodosylbenzene at Low Temperature. *Shirley Nakagaki, Yassuko Iamamoto* (Departamento de Química - Faculdade de Filosofia, Ciências e Letras de Ribeirão Preto - USP) and *Otaciro R. Nascimento* (Instituto de Física e Química de São Carlos - USP).

The ironporphyrin tetra(4-N-methylpyridil)porphyrinato-iron (III) perchlorate has been studied as catalyst and iodosylbenzene as oxygem donor for cyclohexane hydroxylation in methanol and methanol-dichloromethane with yields of 3 and 5% of cyclohexanol respectively. In acetonitrile the yield was increased to 20%. The reaction in acetonitrile system was studied by UV-VIS at –40°C and EPR Spectroscopy at –150°C. The following intermediates species are evident: (i) iron III rhombic species g=6.64 and g=5.44; (ii) ferryl species (FeIV), with desappearing of iron III high-spin EPR signal and 550 nm band; (iii) iron N-oxides species (Fe–o–N) g=4.3. The active specie Iron IV porphyrin π–cation radical may be present, because the highest yield was observed in acetonitrile but it is not confirmed in this experimental condition.

QI-006 Spectroelectrochemistry and Resonance Raman Spectra of a Polynuclear Iron Porphyrin. *Koiti Araki, Henrique E. Toma* and *Paulo S. Santos* (Instituto de Química – Universidade de São Paulo – Caixa Postal 20780 – São Paulo - Brazil).

Spectroelectrochemical and resonance Raman data of the polynuclear $Na_4[Fe–4–TPyP(Ruedta)_4C1]$ complex are reported. The reduction of the complex occurs in two steps, shifting the Soret band, from 400 to 428 nm, and leading the appearance of a broad absorption around 460 nm. This band, in the spectrum of the totally reduced complex, can be inequivocally assigned to a metal-to-ligand charge-transfer transition at the peripheral $[Ru^{II}edta(pyP)]$ complexes, based on resonance Raman spectroscopy.

QI-007 Uma Rota Alternativa para a Síntese de $Cp_3Co_3(\mu_3–NO)_2$. *Jörn Müller* (Institut für Anorganische und Analytische Chemie – Technische Universität Berlin) and *Gelson Manzoni de Oliveira* (UFSM - Departamento de Química).

Treatment of $CpCo(C_2H_4)_2$ with NO in hexane yields $Cp_3Co_3–(\mu_3–NO)_2$together with the dinitrosoethane complex $CpCo(NO)_2C_2H_4$; the mixture of these known compounds can easily be separated.

QI-008 Determination of Equilibrium and Rate Constants for Simple Ferrous Porphyrins. *Carmita Freitas Portela* (Departamento de Química Fundamental – UFPE – Brazil) e *Teddy G. Traylor* (Department of Chemistry, University of California, San Diego – USA).

Until recently the use of simple ferrous porphyrins as model for reversible oxygen carrying hemoproteins was frustated because addition of unhindered, strong field ligands, such 1–methylimidazole, to four–coordinated hemes in solution do not give stable five–coordinated species but leads predominantly to six–coordinated complexes. The use of high ligand concentration and nanosecond laser photolysis greatly simplified the kinetic investigation of heme–base– CO interactions. This method avoids dissociation to the four-coordinated heme, the principal problem in the study of heme–base mixtures.

QI-009 Effect of the Presence of Copper Sulphate in the Development of Microorganisms in Soil. *Silvana A.M. Critter* and *Claudio Airoldi* (Instituto de Química, UNICAMP, Caixa Postal 6154, 13081 – Campinas – SP).

The development of microorganisms in ampules of soil sample (1.5g) containing glicose plus $(NH_4)_2SO_4$ (12.0mg) and variable amount of $CuSO_4$ (0; 0.75; 1.5; 3.0; 4.0mg), and water (0,8ml) was followed by means of a LKB 2277 microcalorimetry. For each sample the thermic effect was recorded at 298.15 K and the results were adjusted to a kinetic equation $T_p(i) = T_p(o) + 1/c\ i \exp(n)$, showing a linear behaviour of

T_p (i) vs time, which depended on the amount of $CuSO_4$ in soil. These results indicated that the development of microorganisms is inhibided by the presence of this salt.

QI-010 Crystal Structure and electrochemistry of a New Vanadium (III) Species With Bioinorganic Relevance.

A. Neves; A. S. Ceccato (Departamento de Química – Universidade Federal de Santa Catarina – Brasil) and *B. Nuber; J. Weiss* (Anorganisch–Chemisches Institut der Universität Heildelberg – F. R. G.).

The potentially hexadentate Ligand N, N',N,N' –bis[(2–hydroxybenzyl)(2–methylpyridyl)etilenediamine($C_{28}H_{32}N_4O_2$,H_2BBPEN) has been synthesized and the corresponding green vanadium (III) complex[(V(BBPEN)]PF_6(1) has been isolated. The crystal structure of 1 has been determinied by X-ray crystallography: Triclinic, Space group $P_{\bar{1}}$;a=11,036(6); b=11.102(7), c=12.017(7)A°, α = 90.78(4), β = 91.46(4), γ = 108.14(4), V = 1398,35A$^{\circ 3}$, z = 2, Rw = 5.0. Cyclic voltamograms of 1 in acetonitrile show three quasi–reversible one–electron process ($V^{V/IV}$ = + 1.11, $V^{IV/III}$ = + 0.27,$V^{III/II}$ = –1.79V vs Fc^+/Fc respectively. The results are discussed in terms of coordination ability of the ligand H_2BBPEN.

QI-011 Spectrometric Study of the Metal Complexes of Rubrofusarin.

Elene Cristina Pereira and *Lucia Tosi*, (Departamento de Química, UFMG).

The interactions of rubrofusarin (Rfg, 2–methyl–5,6–di–hydroxy–8–methoxynafto–γ–pyrone) with Ca(II), Mg(II), Al(III) and Fe(III) in aqueous solution were studied by means of UV–visible spectroscopy. The formation constant β = [ML] / [M^{n+}] [L^-] in which [ML] stands for the concentration of the complex, [M^{n+}] and [L^-] for those of the free metal ion and the ligand, respectively, were calculated. The metal complexes of Rfg may function as structural models for those of anhydrotetracycline.

QI-012 Study of the Fe(III) Complexes of Anhydrotetracycline and Rubrofusarin.

Elene Cristina Pereira, Lucia Tosi, and *Heloisa Beraldo,* (Depto. de Química); *Anuar Abras,* (Depto. de Física, UFMG).

The Fe(III) complexes of anhydrotetracycline (AHTC) and rubrofusarin (Rfg, 2-methyl–5,6–dihydroxy–8–methoxynafto–γ–pyrone) were prepared. The possible structures were suggested. Mössbauer spectra indicate the presence of paramagnetic relaxation phenomena. The estimated isomer shifts (δ) and quadrupole splittings (Δ) at T = 85 K, namely δ_{Fe} = (0.44 ± 0.04) mm/s and Δ = (1.56 ± 0.07) mm/s for the Rfg complex and δ_{Fe} = (0.42 ± 0.04) mm/s and Δ = (0.70 ± 0.08) mm/s for the AHTC complex, are indicative of high spin Fe(III). From the δ values one may infer that the coordination spheres are essentially the same. The Δ values indicate lower symmetry for the Rfg complex.

QI-013 Coordination of VO^{2+} and Cu^{2+} With Alginic Acid Isolated From the Brown Seaweed *Laminaria brasiliensis*.

Antonio Salvio Mangrich, Stela Maris de Moraes Romanowski*, José Hazencleve Duarte**, Maria Eugênia Rabello Duarte*** and *Cesar Cusatis**** (Departamentos de Química*, Bioquímica** e Física*** da Universidade Federal do Paraná) and *Ney Vernon Vugman* (Instituto de Física da Universidade Federal do Rio de Janeiro).

Complexes of alginic acid and of its fractions rich in β –$\underline{\underline{D}}$–manuronic acid and α –$\underline{\underline{L}}$–guluronic acid (isolated from the brown seaweed *L. brasiliensis*), were prepared with VO^{2+} and Cu^{2+} ions. They were studied by EPR spectroscopy and X–ray diffraction. The results of EPR showed that, in both VO^{2+} and Cu^{2+} complexes, carboxylates groups of the polysaccharide structure act as ligands. The others positions of the metal ion coordinatioon spheres are occupied by water molecules. The metal complexes studied didn't give sharp x–ray patterns for unequivocal conclusions.

QI-014 Reductive Degradation of Humic Acid from the Jucu River Estuarine Region, Brazil.

Armando Pereira do Nascimento Filho (Instituto de Química da Universidade Federal Fluminense) and *Antonio Salvio Mangrich* (Laboratório de Química de Húmus e Fertilizantes, Departamento de Química da Universidade Federal do Paraná).

Humic acid from a sediment of the Jucu River estuarine region, Espírito Santo State, Brazil, was studied by Na-amalgam reduction and the products obtained were analised by GC-MS methods. Salycilic acid and vanillin were identified in the products and were supposed to result mainly from lignin. The existence of the lignin structure in the humic acid suggest the participation of terrestrial plants on the formation of the estuarine region. Salycilates are the principal coordination sites for metal ions in this humic acid (A. S. Mangrich and N. V. Vugman, *Sci. Tot. Environ., 75: 235, (1990)).*

QI-015 Mossbauer and Electronic Spectroscopy of Macrocyclic Iron (III) Complex, With Formation of μ–Peroxo–Bridged Species.

Noel M. Levy, Eduardo Stadler (Depto. de Química) *Maurício A. C. Melo* and *Valderes Drago* (Depto. de Física) (Univ. Fed. de Santa Catarina – Cx. Postal 476 – Florianópolis-SC).

In this work we have studied the properties of iron macrocyclic complex [Fe^{II}(TIM)$(L)_2$], where the abbreviated TIM is the ligand 2,3,9,10–tetramethyl–1,4,8,11– tetraazacyclotetradeca–1,3,8,10 –tetraene, with emphasis, in electronic and Mossbauer spectroscopy of dimeric process in iron macrocyclic complex with a formation of μ–peroxo bridged, Eq 1, 2[Fe^{III}(TIM)$(H_2O)_2$] + $2O_2$ $\leftrightarrows$ [$Fe_2{}^{III}$$(TIM)_2(H_2O)_2(O)_2$]+$H_2O$ (1) we have investigated their possible relevance to the understanding of the biological processes.

QI-016 Electronic and Resonance Raman Spectra of Iron(II) Tetraazamacrocyclic Complexes With N-Heterocyclic Ligands.

Fábio S. Nunes, Henrique E. Toma, Eduardo Stadler, Luiz A. Morino, and *Paulo S. Santos* (Instituto de Química – Universidade de São Paulo – Cx. Postal 20.780 – S. Paulo – Brazil).

Iron(II) complexes of synthetic macrocyclic ligands meso-5,5,7,12,12,14–hexamethyl–1,4,8,11–tetraazacyclotetradeca 1,3,8,10–tetraene (ENO), bis(dimethylglyoximato) (DMGH) and bis(diacetylmonoxime-imino) 1,3–propane (DOHpn) with N–heterocyclic ligands exhibit intense bands in the visible region, $\in > 1000$ $M^{-1}cm^{-1}$, characteristic of metal-to-ligand charge transfer transitions. The resonance Raman spectra of these complexes show a selective enhancement of the vibrational modes of the macrocyclic ligands, when the excitation wavelenghts concide with the low energy band, supporting the assignment of a metal–to–macrocyclic charge tranfer transition. The ligand DOHpn show an intermediate structure between bis–α–diimine and bis–oxime, exhibiting enhancement of vibrational modes common to both functional groups.

QI-017 Kinetic and Thermodynamic Aspects of the Interaction of Cis-Platinum With Papain.

Yeda P. Dick, Viviane Fassina Clenia Saldanha and *Andréia P. S. Daudt.* (Departamento de Físico-Química, Instituto de Química, Universidade Federal do Rio Grande do Sul).

The interaction of cis–platinum (cis $Pt(NH_3)_2Cl_2$), the well known anti-tumour drug, with papain, a thiol-proteinase, has been investigated. The enzyme is inactivated by relatively high concentrations of the complex, being the inhibition of the competitive type, in the temperature range of 15° to 40°C. The thermodynamic parameters ($\Delta G^{\circ\prime}$, $\Delta H^{\circ\prime}$, $\Delta S^{\circ\prime}$, and $\Delta C_p^{\circ\prime}$) of the binding process have been determined. The spontaneity of the coordination derives only from the enthalpic contribution since the entropic factor is unfavourable. A Scatchard's plot suggests that the binding is anticooperative. (Supported by CNPq, FAPERGS).

QI-018 Nickel (II) and Iron (III) Complexes With Sulfa Derivatives of 5–Azo–8–Hydroxyquinoline as Ligands.

Astréa Souza Silva, Edward Ralph Dockal (Universidade Federal de São Carlos, Departamento de Química), *Maria Inês Gonçalves* (Universidade de São Paulo, Faculdade de Ciências Farmacêuticas).

As part of the study of compounds of sulfa derivatives of 5–azo–8–hydroxyquinoline which are possible anti-malarial drugs, Ni(II) and Fe(III) complexes of these ligands were synthesized. The infrared spectra, 4000 to 200 cm^{-1}, obtained on the Perkin Elmer 283, are evidence for the preferencial mode of coordenation of the ligands in the solid state in KBr. These ligands can act as neutral bidentates or monobasic bidentates, forming chelate rings of 6, 5 or 4 members respectively. The streching frequencies of OH (fenolic), C=N, S=O, N–H and metal-ligand appear to indicate the preferencial coordination either of the 8–hydroxyquinoline moeity or the sulfa moeity.

QI-019 Synthesis and Characterizaton of Adducts Between Rhodium (II) Carboxylates and Sulphadiazine.

Rubén Dario Sinisterra Millán and *Renato Najjar.* (Inst. de Química, Depto. de Quím. Fundamental, Universidade de S. Paulo, São Paulo, SP - Brazil).

Rhodium (II) carboxylates are substances with antitumour activity. However, that compounds are very toxic when injected in animals and we are looking for complex with carrier molecules that have selectivity for malignant tissue. The literature has shown that sulphadiazine preferentially concentrates in tumours when compared with liver and bone marrow, (bone marrow and liver toxicity often limits the amounts of antitumor agents given clinically). We have synthesized compounds of general formula: $Rh_2(R\text{-}COO)_4.2SD$ (R = alkyl or aryl radical and SD = sulphadiazine). The I.R., UV–visible, magnetic susceptibility besides the toxicity toward mice were studied up to now.

QI-020 EPR Study of Cu(II) Adsorbed on 1,4 Diazabicycle |2.2.2| Octane Grafted on Silica Surface.

W. C. Moreira, Y. Gushikem (IQ-UNICAMP-SP) and *O. R. Nascimento* (IFQ – USP – SP).

The EPR spectra of $CuC1_2$ and $Cu(C10_4)$ adsorbed on a silica gel surface, modified with 1,4 diazabicycle |2.2.2| octane, showed that the structure of surface complex depends of the anion in the surface. When chloride anion was present in the surface the complex has a tetrahedral symmetry, alternatively if the anion is perchlorate the complex formed has square planar symmetry. Adsorption of NH_3 on the surface with chloride anion and $CuC1_2$ adsorbed, the complex presents octahedral symmetry with tetragonal distortion. If $Cu(C10_4)_2$ was adsorbed on the same surface and when perchlorate anion was the contraion, the amincomplex formed has square planar symmetry.

QI-021 Silica Gel Modified With Metal Oxide Catalysts. *E. V. Benvenutti; Y. Gushikem* (Instituto de Química - UNICAMP, C.P. 6154 - 13081 - Campinas, SP - Brazil).

Oxides of Sb (V), Fe (III) and Mn (IV) supported on silica gel surface were obtained. The surface area of the pure silica gel was 340 m^2 g^{-1} and after treatment it was about 500 m^2 g^{-1}. The thermal stability (determined by DSC and TGA) and chemical stability in acidic medium were studied. The amount of the metals and the surface area suggested a monolayer of the metal oxides on the silica surface.

QI-022 Physical Characterization of Monolayer Dispersed TiO_2 on Silica Gel Surface. *L. T. Kubota, Y. Gushikem* (Instituto de Química - UNICAMP), and *S. Castro* (Instituto de Física - UNICAMP).

The XPS studies of Silica-Titanium (ST) samples submitted to thermal treatment at 200, 400 and 700°C were carried out using a spectrometer ESCA-36 of McPherson Co., at a pressure of 2.10^{-7} Torr. In all the observed cases the binding energies were characteristic of the Ti atom in TiO_2 form. The atomic concentration of Ti on silica surface was 8.2, 7.7 and 6.1% for the 200, 400 and 700°C treated samples. The decrease on the Ti/Si ratio could be due to an increase of the particle size of Ti or a migration of Ti into the silica. The sharpenning of the line width of Ti 2p from 2.4 to 2.1 eV when heated from 200 to 700°C, suggests a decrease in the number of chemical species of the metal on the surface.

QI-023 Monolayer of TiO_2 Immobilized on Silica Gel Surface and Its Utilization in the Cr(VI) Extraction. *L. T. Kubota* (Depto. de Química da FC da UNESP - Campus de Bauru), *J. C. Moreira* (Inst. de Química da UNESP - Campus Araraquara), *Y. Gushikem* (Inst. de Química - UNICAMP).

Ti (IV) oxide bonded to silica surface was obtained reacting activated silica gel with Ti (IV) in an organic solvent under inert atmosphere. The amount of the metal oxide on the solid was 1.8 $mmol.g^{-1}$ and the surface area 308 m^2 g^{-1}. This material was used to adsorb dichromate ion from acid and neutral solutions. The affinity increased with the acidity of the medium and decreased with the thermal treatment. Preconcentration and recovery of small amounts of Cr (VI) (about 0.5 ppm) studies were carried out using the column technique.

QI-024 Adsorption Proprieties of Monolayer Dispersed Nb(V) on Silica Gel Surface. *S Denofre* and *Y. Gushikem* (Instituto de Química - UNICAMP - C.P. 6154 - 13081 - Campinas, SP - Brasil).

Silica gel having a particle size between 0.063-0.200 mm and specific surface area of 242 m^2 g^{-1}, was chemically modified with Nb(V). The amount of the attached metal on the surface was 0.38 x 10^{-3} $mol\text{-}g^{-1}$. The adsorption isotherms were determined for various metal ions and the adsorption capacity determined for each one was (in μmol g^{-1}): Cu(II) = 12, Co(II) = 17, Ni(II) = 11, Zn(II) = 8, Cd(II) = 7 and Hg(II) = 30.

QI-025 Cianide Ion Coordination on Ruthenium Complex Anchored on Modified Silica Gel. *Douglas Wagner Franco* (Instituto de Física e Química de São Carlos - USP), *Sonia Maria Carvalho Neiva* (Universidade Federal do Piauí - FUFPI) e *Yoshitaka Gushikem* (Instituto de Química - UNICAMP).

Immobilization of ruthenium complexes on silica gel modified with 3(1-imidazolyl) propil groups has been previously reported (40ª SBPC Annual Meeting). In this work, an adsorption study of a cianide derivative complex is presented. The coordination of the cianide derivative on the modified ruthenium complex has been characterized by electronic (λ = 444 nm) and infrared (υ_{CN}= 2040 cm^{-1}) of spectroscopies. The synthesis for the coordination of the cianide ion in mon-aqueous media have been stablished and according to the following reaction: $[\equiv Si(CH_2)_3imN\text{-}\ Ru(NH_3)_4(H_2O)]C\ell_2 + CN^- \xrightarrow{ethanol} [\equiv Si(CH_2)_3imN\text{-}\ Ru(NH_3)_4CN]\ C\ell$, where imN represents the imidazol group. (CAPES, CNPq, FAPESP).

QI-026 Imobilization of Ruthenium Complex on Silica Gel. *Douglas Wagner Franco* and *Maria Regina Martins* (Instituto de Física e Química de São Carlos - USP) and *Yoshitaka Gushikem* (Instituto de Química - UNICAMP).

Ruthenium complex $[\equiv Si(CH_2)_3NHP\varnothing_2\text{–}Ru(NH)_3)_4SO_2(H_2O)]^{2+}$ was supported onto silica gel modified with iminopropyldifenilfosfine groups. The imobilization capacity was determined as higher than 95%. (CAPES, CNPq, FAPESP).

QI-027 Silanes Incorporation to Sicosil Surface and Its Use With Epoxy Resin. *Mário F. Góes* (Fac. de Odontologia de Piracicaba, Univ. de Campinas), *Angélica A. Isawa, Eduardo J. Nassar, M., Elisabete D. Zaniquelli* (Fac. Filosof. Ciênc. Letras Rib. Preto, USP, Depto. Química).

Silica surface can be modified by incorporation of organofunctional silanes. Surface modified silica has been used in a number of applications, e.g., as coupling agent in polymeric systems. In this work we use sicosil 63 M (colloidal silica from Rhodia) as substrate, gamma-aminopropyltriethoxysilano and gamma-glycidoxypropyltrimethoxysilano from Union Carbide, as coupling agents; both were analized by NMR and FTIR. Silanized sicosil particles were incorporated to epoxy resin (XD 4704 - Ciba Geigy). Three different methodologies were used for the silane incorporation: i) sonication (10 min.), ii) shaking (5 h.) and iii) reflux (18 h.), using toluene as the solvent. The sicosil surface was submited to acid or basic pre-tratments and was vacuum dried before the silane reaction. The particles were analysed by FTIR before and after silane treatment. Two bands at 2938 and 2882 cm^{-1} were observed only for the modified sicosil samples and attributed to assymmetrical and symmetrical C-H streching. The best result was obtained with the particles submited to acid pre-tratment and shaking for 5 h. Silanized particles can be incorporated to epoxy resin in 50% mass percent while the percentage for non-silanized particles is 20%. Besides this we observed different dispersion characteristics in CCl_4 for the samples. FAPESP/FUNCAMP.

QI-028 Sorption of lanthanides on Silica Gel Chemically Modified by Sodium P-Aminobenzoate. *Regina Célia Retamero, Elizabeth Berwerth Stucchi* and *José Celso Moreira* (Instituto de Química, UNESP - Araraquara, SP).

Sodium p-aminobenzoate on silica gel was used for preconcentration of $\mu g.ml^{-1}$ levels of Pr, Nd, Eu, Dy e Ho from aqueous solution at different pH values in batch experiments. The concentrations of metal ions were determined spectrophotometrically with I-(2-pyridylazo)-2-naphthol (PAN).

QI-029 Adsorption of Metal Ions From on an Sodium 3-Aminobenzoate Modified Silica Gel. *Manoel L. Menezes* and *José Celso Moreira* (Instituto de Química, UNESP - Araraquara, SP).

Sodium 3-aminobenzoate group attached to a silica gel surface have been used for the adsorption of metal ions from ethanol. This organofunctionalized silica adsorbs copper, cobalt, cadmium, iron, manganese, nickel and zinc from ethanol by method bach. The maxima adsorption capacity determined for each metal ion was as follows (mol x g^{-1}): Cu^{+2} 2,7 x 10^{-4}, Co^{+2} 1,3 x 10^{-4}, Cd^{+2} 1,0 x 10^{-4}, Fe^{+3} 2,6 x 10^{-4}, Mn^{+2} 2,2 x 10^{-4}, Ni^{+2} 7,5 x 10^{-5} and Zn^{+2} 4,8 x 10^{-4}.

QI-030 Siliceous Supports for Enzyme Immobilization. *H. C. Trevisan, L. H I. Mei*, (Faculdade de Engenharia Química, DCTM, Universidade Estadual de Campinas, SP - Brazil).

Macroporous silica gel was obtained by hidrothermal treatment of a silica prepared from sodium silicate and HCl. The silica (pore diameter = 440 Å) and one grade of diatomaceous earth, celite 545 (pore diameter = 1,96 x 10^6 Å) were examined for their suitability as supports for glucose oxidase immobilization. The silica was loaded with up to 400 U/g of enzyme and the celite with up to 110 U/g. Despite the high enzimatic load to the supports, the maximum measured activity was 27 U/g for the silica and 50 U/g for the celite and it was dependent on the particle size, due to internal mass transfer resistances.

QI-031 Utilization of Precipitated Silica as Adsorvent in Column Chromatography. *Rui R. Bosshard, Eduardo Cruvinel, Janete H. Y. Vilegas e Marcos Garcia* (Centro de Pesquisas de Paulínia - Rhodia S. A.).

We have studied the viability of substitution of silicagel as chromatographic adsorvent by the precipitated silica Sicosil (Trade mark of Rhodia S. A.). The precipitated silica was tested' in vacuum filtrations and in mplc separations of plant compounds (terpenes, steroids, flavonoids, etc) and purifications of synthetic compounds (bisphenol derivatives). This material, cheaper than silicagel, can substitute common silicagel in these separations without loss of quality of the chromatographic separations.

QI-032 Preparation of Immobilized Inorganic and Biological Catalysts Using Surface Activated Chrysotile. *Roberto Alcântara M. Zucchetti, Osvaldo Parizzotto Junior, José Augusto R. Rodrigues, Paulo José Samenho Moran* and *Ines Joekes* (Instituto de Química, UNICAMP - Campinas - Brasil).

Brazilian Chrysotile asbestos (5RL - SAMA) was activat (Pat. BR. - 8.903.849) and used to immobilize a) *Saccharomyces cerevisiae*, b) Manganese dioxide (MnO_2), c) Cupper oxide (CuO) and d) Antimony trioxide (Sb_2O_3). We found maximun adsorption (aqueous media, 25°C and magnetic stirring) of 0.9 g of *Saccharomyces cerevisiae*, 0,7 g of MnO_2, 0.3 g of CuO and 0.6 g of Sb_2O_3 per gram of chrysotile, respectively. Adsorption values show that this material has a supporting efficiency comparable with the bests in the market. Qualitative results show that the supported biocatalyst has activity comparable to the free yeast and much longer life-time.

QI-034 Adsorption of Metallic Chlorides on Chemically Modified Silica Gel With 3-Chloropropyldiacetamide in Ethanol. *Claudio Airoldi* and *Edésio F. C. Alcântara* (Instituto de Química, UNICAMP - C. P. 6154 - 13081 - Campinas, SP).

The elemental analyses of nitrogen of the immobilized diacetamide on silica surface indicated the presence of 3.37 x 10^{-4} mole of this molecule per gram of silica. This surface extracts divalent cations from ethanol solution under stirring during 3 h at 298 K. The isotherms are fitted to a modified Langmuir equation, showing an amount of mole of solute adsorbed per gram of adsorbent (mol g^{-1}), and the equilibrium constant in the interface (1 mol^{-1}), as following: Cu(4.16×10^{-5}; 4.10×10^3), Co(1.02×10^{-4}; 1.21×10^3), Zn(1.13×10^{-4}; 2.77×10^3). This surface shows a small adsorption capacity in comparison with the acetonic medium.

QI-035 Cationic Adsorption on Immobilized Urea Silica Gel Surface. *Maria Rita M. C. Santos* (Departamento de Química, UFPI) and *Claudio Airoldi* (Instituto de Química, UNICAMP – C.P. 6154 - 13081 - Campinas, SP).

The reaction of 3-chloropropyltrimetoxysilane on silica gel produced surface I, which reacted with urea in N, N-dimethylformamide at 130°C during 8 h to give surface II. Analysis of the degree of chloride (5.43×10^{-4} moles/g) and nitrogen (3.80×10^{-4} moles/g), respectively, were obtained. The last surface extracts divalent cations from thermostatized non-aqueous solution at 298 K during 3 h, where the maximum of the isotherms (10^{-4} moles/g) were obtained: 1.40; 1.10; 0.998; 0.860; 0.750; 0.550 for Hg, Cu, Co, Ni, Cd, Zn in ethanol, and 3.59; 1.67; 1.19 for Cu, Co, Zn in acetone. The less interactive effect of acetone favours the cations adsorption on the surface.

QI-036 "Adsorptive Index of Cu(II) Aco on Freshly Prepared Silica Gel Surface". *José Geraldo de Paiva Espínola, José Marcedônio Porto de Freitas, HellenRose Ouriques Catão* (Departamento de Química - CCEN - Universidade Federal da Paraíba - JoãoPessoa, PB).

The adsorptive parameters of Cu(II) Aco on silica gel were reasured by complexometry using the EDTA-Murexide method. The adsorptive index obtained experimentally doesn't fit mathematically with the models already published, searching for another alternative equations for a better interpretation of the surface saturation of the gel with the complex. Observed values were in the range of 0,68 mmoles of the complex relative to lg of the support.

QI-037 The Reactions of $HIr_4(CO)_{10}$ (μ–PPh_2) With $PPh_2C{\equiv}CPh$. *M. H. A. Benvenutti* and *M. D. Vargas* (Instituto de Química, UNICAMP - Brazil). *D. Braga* and *F. Grepioni* (Dipartimento di Chimica "G. Ciamician", Universitá Degli Studi di Bologna, Italy).

The reaction of $HIr_4(CO)_{10}(\mu\text{-}PPh_2)$ (1) with $PPh_2C{\equiv}CPh$ in the presence of DBU, followed by protonation, yields the novel compound $HIr_4(CO)_9$ (μ–PPh_2) (μ_4–η^3–PPh_2C–CPh) (2) (up to 50%), that possesses a spiked triangular metal framework supporting the 6-electron donor $PPh_2C{\equiv}CPh$ ligand. Attempts at cleaving the P–C bond by reaction with Me_3NO, photolysis and pyrolysis resulted in decomposition. In contrast, compound (1) reacts with $PPh_2C{\equiv}CPh$ to give the CO substituted product $HIr_4(CO)_9(PPh_2C{\equiv}CPh)$ (μ–PPh_2) (4), which rearranges to $Ir_4(CO)_8(\mu\text{-}PPh_2)_2(\mu_3\text{–}\eta^2HC{\equiv}CPh)$ (5) via cleavage of the P-C bond and hydride transfer to the acetylide fragment.

QI-038 Reactions of $Ir_4(CO)_{11}PPh_2H$ With $[MPPh_3]^+$ Fragments, (M = Au, Ag). *F. S. Livotto, M. A. Rosa* and *M. D. Vargas* (Instituto de Química, UNICAMP – Brazil) and *D. Braga* and *F. Grepioni* (Dipartimento Di Chimica "G. Ciamician", Universitá Degli Studi Di Bologna, Italy).

The reactions of $Ir_4(CO)_{11}(PPh_2H)$(1) with $[AuPPh_3]^+$ in the presence of a base depend on the order of addition of the reagents and yield the products $Ir_4(CO)_{11}\{PPh_2Au(PPh_3)\}$ (2), upon deprotonation in the presence of the cation and $(Ph_3PAu)Ir_4(CO)_{10}(\mu\text{-}PPh_2)$ (3). Upon heating (2) in toluene above 70°C quantitative conversion to (3) is observed and kinetic studies suggest a rate law $k_{obs} = k_1$, with a [CO] dependent preequilibrium. Only (2) undergoes CO substitution with phosphines; reaction of (3) with $P(p\text{-}MeOC_6H_4)_3$ leads to PPh_3 substitution. The reaction of (1) with $[AgPPh_3]^+$, at room temperature, produces $(Ph_3PAg)Ir_4(CO)_{10}(\mu\text{-}PPh_2)$ (3a), with formation of the intermediate $Ir_4(CO)_{11}\{PPh_2(AgPPh_3)\}$ (2a) detected by IR (υ_{CO} region).

QI-039 Obtention of Organotin Sulphides and Their Reaction With Mercurated Derivatives of Ferrocene. *Geraldo Magela de Lima* and *Carlos A. L. Filgueiras* (Depto. de Química, UFMG).

Several mercurated derivatives of ferrocene were prepared, namely cpFecp-Hg-cpFecp, cpFecp-HgCp, ClHgcpFecpHgCl, and $[cpFecp\text{-}Hg]_n$. Reaction of these compounds with sulphides of the type $(Ph_3Sn)_2S$ and $[R_2SnS]_3$, where R = Me or Ph, produced heterobimetalic species in high yield, which occur as crystalline solids. By meane of modifications in the ferrocene derivative or the sulphide one can vary the structure and properties of the product, which have several possible applications, such as catalytic ones.

QI-040 Decarbonylation Reactions of $[Fe(CO)_5]$ Induced by Trimethylamine-Oxide in the Presence of Lewis Bases. *O. L. Casagrande Jr.* and *A. E. Mauro* (Instituto de Química de Araraquara - UNESP).

There is a great interest in the use of trimethylamine N-Oxide in organometallic chemistry, and this reagent is particularly useful in the decarbonilation reactions of metal carbonyls. This communication is concerned with the reactions of $[Fe(CO)_5]$ with ligands like phenantroline; 2,2'-bipyridine and 2-quinolinethiol induced by TMNO. The products obtained, $[Fe_2(CO)_7(phen)_2]$, $[Fe_2(CO)_7(bipy)_2]$ and $[Fe(CO)_3(qn\text{-}SH)_2]$ were studied by vibrational spectroscopy.

QI-041 Synthesis and Characterization of Compounds of the Type $[Fe(CO)_2(\eta^2$ –$CS_2)(EPh_3)_2]$ (E=P, $P(O)_3$; As, Sb). *Teresa K. Muraoka; Vânia Martins Nogueira; Antonio E. Mauro* (Instituto de Química - UNESP - Araraquara.

The reaction between $Fe(CO)_5$, CS_2 and EPh_3(E=P;$P(O)_3$; As, Sb), in ethanol, in the presence of TMNO (trimethylamine N-Oxide) gives compounds of the type $[Fe(CO)_2(\eta^2 - CS_2)\ (EPh_3)_2]$. The I. R. Spectra of these compounds show two absorption bands in the CO and one in the CS stretching regions. The eletronic spectra of these compounds were mesured in dichloromethane solution. The oxidative electrochemistry of the compounds has been studied in N, N-dimethylformamide at Pt electrodes by cyclic voltammetry.

QI-042 Reactions of Chromiumhexacarbonyl Compounds Suported in γ-Alumina. *Ione M. Baibich* and *João H. Z. dos Santos* (Instituto de Química - UFRGS).

The present work deals with the comparative study of the photochemical substitution reactions ($\lambda >$ 280 nm) of $Cr(CO)_6$ with ligands from Group V A (PPh_3, $P(OPh)_3$, $AsPh_3$ and $SbPh_3$) in presence of γ-Alumina. For L = PPh_3 and $P(OPh)_3$, the species $Cr(CO)_5L$, in solution, and *trans* – $Cr(CO)_4L_2$, impregnated in γ-Al_2O_3 were obtained. It was observed a reduction of the reaction stereoespecifity as L was changed from PPh_3 to $SbPh_3$. This effect was atributed to the cone angle decrease. In this way, a mixture of mono - and disubstituted *cis* and *trans* species were obtained.

QI-043 Catalytic Activity of Heteropolianions on the Olefines Epoxidation. *Maria Eliza Moreira Dai de Carvalho, Maganir Alaíde de Almeida* and *Gennaro Junho Gama* (Departamento de Química, Instituto de Ciências Exatas, Universidade Federal de Minas Gerais).

This work presents the catalytic activity of heteropoly-anions (HPAs) containing tungsten and silicon atoms on the epoxidation of olefines. The results of several systems allow the conclusion that the HPAs employed, when mixed with NaOCl, can mimic the behaviour of the metalloporphirine/NaOCl/olefine system, leading to the convertion of the unsaturated compounds into heavier derivatives. The yields shown by the systems above are comparable leading to a preference for the use of the HPAs, due to its greater simplicity of synthesis. (CNPq, FAPEMIG, FINEP).

QI-044 Organotin Cuprates of High Order. *Mark V. M. Emonds, Petee Steingel* and *J. P. Marino* (University of Michigan - Ann Arbor - USA) and *F. Simonelli, A. R. M. de Oliveira and J. Tércio B. Ferreira* (Departamento de Química - UFSCar - C.P. 676 - São Carlos, SP - Brasil).

Organic synthesis based on organotin reagents is becoming an increasingly visible area of research. A new organotin cuprate $(nBu_3SnCH{=}CH)_2\ CuCNLi_2\ 2LiCl(1)$, was developed in our laboratories. We found that the cuprate 1 in the presence of two equivalents of lithium chloride added to α, β –unsaturated ketones in high yield. (CNPq, FAPESP and IFS).

QI-045 Characterization of Polynuclear Species of $RuCl_2$(Dimethylsulfoxide)$_2$(Pyrazine)$_2$ With Pentacyanoferrate (II) Ions. *Denise de Oliveira* and *Henrique E. Toma* (Instituto de Química - USP - São Paulo, SP).

Polynuclear species of $RuCl_2$(dimethylsulfoxide)$_2$ (pyrazine)$_2$ complex with the aquopentacyanoferrate(II) ion exhibit $E_{1/2} = 0.67$ V (vs EPH; KCl 0.1M) and one Fe(d π) → pz (π^*) charge transfer band at 530 nm. The dissociation kinetics of these species are typically of first order with $K_d = 1.7 \times 10^{-4}\ s^{-1}$ (28°C). The kinetic and electrochemical results are consistent with a weak coupling between the metallic centers as a consequence of the decreasing of the Ru(II) π energy level, in comparison with that of Fe(III), caused by the strong Ru(II) → S (dmso) π - backdonation.

QI-046 Electrochemical Study of Fe(II) (Poly)Imines Complexes. *Izaura Hiroko Kuwabara* (Depto. de Química - UFPR - Paraná) and *Henrique Eisi Toma* (Instituto de Química - Universidade de São Paulo - C.P. 20.780 - São Paulo - Brasil).

A series of iron(II) complexes was synthesized by the condensation reaction of diacetylpyridine and primary amines (NH_2–R) in the presence of Fe(II) ion. All complexes have intense absorptions at 495 and 592 nm, assigned to $Fe^{II} \rightarrow$ py and $Fe^{II} \rightarrow$ imine charge-transfer transitions. The cyclic votammograms in CH_3CN and DMF exhibit two reversible waves ascribed to the FeL°/FeL^- (–1,40 V) and FeL^-/FeL^{-2} (–1,70 V) species. Based on spectroelectrochemical measurements the –1,70 V wave was associated with the ligand reduction.

QI-047 Nickel Pentacyanoferrates – Synthesis and Properties of Modified Electrodes.

Flávio M. Matsumoto e Henrique E. Toma (Instituto de Química, Univ. S. Paulo, SP).

Complexes of general formula $Ni_X[Fe(CN)_5L]_Y$, where L = isonicotinamide and benzotriazole, were synthesized and characterized based on electronic and resonance Raman spectra. The complexes were also generated electrochemically, leading to stable films displaying similar absorption spectra as the pure solids. The modified electrodes exhibit two broad waves around 0.2 and 0.5 V vs EPH, ascribed to the Fe(III)/(II) redox couple in two non-equivalent sites in the solid film. The redox waves increase in the sequence $K^+ > Na^+ > Li^+$, showing a selectivity with respect to the electrolyte size.

QI-048 Synthesis, Electronic Spectra and Electrochemistry of a Trinuclear Cluster of Ruthenium Acetate and 4-Vinyl Pyridine.

Anamaria D. P. Alexiou e Henrique E. Toma (Instituto de Química, Univ. São Paulo - C.P. 20780 - São Paulo, SP).

The μ-oxo trinuclear cluster $[Ru_3OAc_6(py)_2vpy]PF_6$ (Ac = acetate, py - pyridine e vpy = 4-vinylpyridine) was synthesized and characterized based on electronic spectra and cyclic voltammetry in acetonitrile solutions. Four redox waves were observed at $E_{1/2}$ = 1.62 V; 0.65 V; –0.37 V and –1.62 V vs $Ag/AgNO_3$ (0.01 M), ascribed to the sucessive redox couples $Ru^{IV}Ru^{IV}Ru^{III}/Ru^{IV}Ru^{III}Ru^{III}/\ldots Ru^{III}Ru^{II}Ru^{II}$, respectively. The enhancement of the peaks during the repetitive scanning of the potentials were indicative of film formation, presumably by a polymerization process.

QI-049 Spectroelectrochemical Behaviour of the Hexanuclear Cluster $[Ru_3O(Acetate)_6\{(aminopirazine)RuEDTA\}_3]^{n-}$.

Marco Antonio L. Olive and *Henrique E. Toma* (Instituto de Química, Universidade de São Paulo).

The spectroeletrochemistry of hexanuclear cluster $[Ru_3O(Ac)_6\{(aminopirazine)RuEDTA\}_3]$ prepared by the reaction of $[Ru(EDTA)(H_2O)]^-$ and $[Ru_3O(Ac)_6(ampz)_3]BF_4$ exhibit at 0.5 V vs Ag/AgCl (1M KCl) an absorption band around 720 nm, assigned to intracluster transitions. At 0.016 V this band shifts to wavelengths above 820 nm. At - 0.015 V an absorption band appeared at 480 nm, and was assigned to a RuEDTA-to-aminopirazine charge transfer transition.

QI-050 Raman Resonance Studies of the Heptanuclear Tris (Bipyrazine) Rutenium (II) Complexes Containing Peripheral (EDTA)Ru(II) Groups.

R. L. Sernaglia (Depto. de Química, UEM, PR). *H. E. Toma* and *P. S. Santos* (Instituto de Química, USP - C.P. 20.780 - São Paulo, SP).

Resonance Raman (RR) spectra are reported for the heptanuclear $[Ru(bpz)_3Ru(EDTA)_6]^{-10}$ complex in aqueous solution, with excitation at several wavelenghths through the metal-to-ligand charge-transfer (MLCT) bands in the visible region of the electronic spectrum. Three types of excitation profiles are observed, consistent with the existence of the MLCT bands involving the peripheral and central ruthenium(II) ions and two π* levels of 2-2'-bipyrazine. The data are consistent with the electronic assignments, demonstrating the common LUMO orbital involved in two of these transitions.

QI-051 Synthesis and Characterization of Phosphine Complexes of Ru(III) Containing N-Heterocyclic Ligands.

Edna Aparecida Polato; Alzir Azevedo Batista (DQ/UFSCar) and *Otaciro R. Nascimento* (USP/IFQSC).

It has been shown that ruthenium (III) complexes containing phosphines are especially effective catalysts in hydrogenation of olefin substrates. In this work we report the synthesis and characterization of the complexes $[RuCl_3(PPh_3)(M\text{-}Im)_2]$ and $[RuCl_2(PPh_3)_2bipy]Cl$, where M - Im = 1-methylimidazole and bipy = bipyridine. Special emphasis was given to electronic paramagnetic resonance and ciclyc voltammetry.

QI-052 Carbonyl - Complexes of Ru(IV) With N-Heterocyclic Ligands.

Lis Regina V. Olmo; Sandra Aparecida Onofre; Alzir Azevedo Batista (DQ/UFSCar) and *Otaciro R. Nascimento* (USP/IFQSC).

The compounds $[RuCl_4(CO)(M\text{-}Im)]$. M-Im and $[RuCl_4(CO)(py)]$ where M-Im = 1-methylimidazole and py = pyridine were isolated in the solid state and characterized by mean of elemental analyse and by their infrared spectra. The compounds were further investigated using spectroscopic and electrochemical techniques. The electronic paramagnectic resonance parameters are consistent wity C_{4V} symmetry for these compounds.

QI-053 The Desulphuration of N-Sulphinylarylamines Catalysed by Transition Metal Salts.

José Roberto da Silveira Maia and *Carlos A. L. Filgueiras* (Departamento de Química, UFMG).

Reactions involving N-sulphinylarylamines and Pd and Pt salts led to a desulphuration of the ligands and the formation of amine complexes. The metal salts catalyse the loss of SO_2 and the solvent used is also important in the process. A mechanism was proposed involving the initial complexation of the original ligand followed by a rearrangement which causes SO_2 to be given off.

QI-054 The Influence of Tin on the Deactivation of Ru-Sn/SiO_2 Bimetallic Catalysts.

Valéria Castro de Almeida, Pedro Paulo Nunes (Instituto Militar de Engenharia, Seção de Química, IME, RJ).

This paper reports a study about the deactivation of Ru-Sn bimetallic catalysts supported on silica under conditions of temperature and pressure which yield benzene by cyclohexane transformation. They were prepared by impregnation with H_2RuCl_6 and $SnCl_2.2H_2O$ solutions dried and then reduced under H_2. The catalytic activity was evaluated by cyclohexane transformation reactions in the temperature range of 433-533K. In order to explain the observed deactivation it is proposed a strong adsorption of chemical species that inhibit the catalyst surface, but are dessorved as the temperature increase, regenerating the active phase.

QI-055 On the Mechanism of the Acid Hydrolysis of Tris (Pyridine–2–Carboxaldehyde–N–Alkylimine)Iron II. Determination of the Activation Volumes of the Reactions.

Matthieu Tubino and *Iris Ney de Carvalho* (Instituto de Química, UNICAMP).

The water-assisted acid dissociation of the complex cations of iron II with α–diimines, have been extensively studied over the last forty years. Nevertheless, the mechanisms of this process is not yet unequivocally established. Two main mechanisms have been proposed. Gillard's proposition assumes nucleophilic attack on the ligand while Basolo's mechanism admits iron–water interaction. To contribute for the understanding of this kind of reaction, the activation volume of the acid hydrolysis of tris(pyridine–2–carboxaldehyde–N–alkylimine)iron II were studied (alkyl = methyl, ethyl, n–propyl, n–butyl). The volumes of activation obtained were respectively, 11.5 cm^3/mol, 14.2 cm^3/mol, 13.0 cm^3/mol and 13.1 cm^3/mol. The similarity of these results with those obtained for the acid hydrolysis of the complexes of iron II with 2,2'–bipyridine and 1,10–phenanthroline suggest that the mechanisms of the reactions are equal. Kinetic measurements were carried out in a spectrophotometer in a thermostatized high-pressure cell, between 0.1 and 100 MPa.

QI-056 The Mechanisms of the Reactions Between Zincon and Copper and Zinc.

Matthieu Tubino and Adriana Vitorino Rossi (Instituto de Química - UNICAMP).

The mechanisms of formation of the complexes of Zincon with copper II and zinc II, were studied in aqueous solutions. Copper II and zinc II react in different velocities, with this ligand in solutions where they are reciprocally absent. However they react as an only one specie when the two are present together. The experimental results indicate a two step mechanisms, in all cases, passing through a binuclear specie. This mechanism could be useful in the understanding of the biochemical reactions of copper and zinc.

QI-057 Synthesis, Characterization and Reactivity of the Potassium Pentacyano(4,4'–dithiodipyridine)Ruthenium(II) Complexes.

Ícaro S. Moreira (Departamento de Química Orgânica e Inorgânica da Universidade Federal do Ceará) and *Douglas W. Franco* (Instituto de Física e Química da Universidade de São Paulo de São Carlos).

The synthesis and characterization of the $K_3[Ru(CN)_5DTDP].3H_2O$ complex are described. The MLCT band was found at 348 nm. Oscilator strength calculations indicate the following order for back-bonding: $[Ru(NH_3)_5DTDP]^{3-} > [Ru(CN)_5DTDP]^{3-} > [Fe(CN)_5DTDP]^{3-}$. The $E_{1/2}$ value of 0.710 volts for the $[Ru(CN)_5DTDP]^{2-/3-}$ coupling was found to be most positive of the series. The rate constant of substitution reaction is $1.4 \times 10^{-4}s^{-1}$.

QI-058 A Study of the Unsymmetrical Binuclear Complexes $Na[(CN)_5FeLRu(NH_3)_5]. 6H_2O$ and $K[(CN)_5RuLRu(NH_3)_5].6H_2O$, L=4,4'Dithiodipyridine.

Ícaro S. Moreira (Departamento de Química Orgânica e Inorgânica da Universidade Federal do Ceará) and *Douglas W. Franco* (Instituto de Física e Química da USP de São Carlos).

The synthesis and characterizaton of the bridged unsymmetrical binuclear complexes $[(CN)_5FeDTDPRu(NH_3)_5]^-$ and $[(NH_3)_5RuDTDPRu(CN)_5]$ are described. The MLCT bands of the Fe(II)–Ru(II) and Ru(II)–Ru(II) complexes were found at 458 nm and 462 nm, respectively. The stepwise one eletron coupling determined by cyclic voltammetry indicates a very weak coupled system.

QI-059 Synthesis, Characterization and Reactivity of Complexes of Ru(edta) With Pyrazole Derivatives.

Luis Roberto Paschoal, Luiz Antonio Andrade de Oliveira, Maria Pedrina Dalponte Mattioli, Vânia Martins Nogueira. (Instituto de Química de Araraquara - UNESP).

The kinetics of formation of $[Ru(edta)L]^-$, where L = pyrazole and 3,5–dimethylpyrazole, by reaction of the aquo or the hydroxo ruthenium complex and the ligand L in aqueous solution, were studied by stopped–flow technique. The pyrazole and 3,5–dimethylpyrazole complexes showed absorptions in 350 and 360 nm, and Molar absorptivities of 1.2×10^3 and 1.1×10^3 and 1.1×10^3 M^{-1} cm^{-1}, respectively. Reversible cyclic voltammograms were obtained for both complexes, using saturated calomel electrode as reference and mercury plated platinum disc as working electrode.

QI-060 A ^{57}Fe Mössbauer Study of Mononuclear, Binuclear and Mixed Valence Pentacyanoferrate Complex With 4,4'–Dithiopyridine.

Ícaro S. Moreira, *Augusto L. Coelho* (Departamento de Química Orgânica e Inorgânica da Universidade Federal do Ceará), *Miguel A. B. Araújo* (Departamento de Física da Universidade Federal do Ceará) and *Douglas W. Franco* (Instituto de Física e Química da USP de São Carlos).

The Mössbauer spectra of the pentacyanoferrate complexes have been obtained. The spectrum of the binuclear, mixed valence complex showed only one dublet, wich is an indication of "trapped valence". It is also clear, from the isomer shift values, that the back-bonding is more effective in the mononuclear compound than in the binuclear compound.

QI-061 Synthesis and Characterization of the Sodium Pentacyano–4–Mercaptopyridineferrate(II) Complex.

Ícaro S. Moreira, *Augusto L. Coelho* (Departamento de Química Orgânica e Inorgânica da Universidade Federal do Ceará) and *Douglas W. Franco* (Instituto de Física e Química da USP de São Carlos).

The study of the $[Fe(CN)_5 4McPy]^{3-}$ complex is relevant due to the possibility of comparing it with the $[Fe(CN)_5 DTDP]^{3-}$ complex (DTDP= 4,4'–Dithiodipyridine). The $[Fe(CN)_5 4MPY]^{3-}$ MLCT band (398 nm) was found at higher energy, when compared with the similar band on the $[Fe(CN)_5 DTDP]^{3-}$ complex spectrum (412 nm). The values of the Mössbauer parameters, isomer shift (0.0521 mm/s) and quadrupolar shift (0.8033 mm/s) indicate a weaker π–acceptor character for 4McPY than for DTDP when coordinated to the same metal center. The $E_{1/2}$ value (0.200 v – SCE) found for the 4McPy complex strengthens this attribution.

QI-062 Study of the Reaction Between Triethylphosphite and Pentaamintristrifluoro–Methanesulfonate Osmiun III.

Luiz Constantino Grombone Vasconcellos and *Douglas Wagner Franco*. (Instituto de Física e Química de São Carlos - USP).

From the reaction between net $P(OEt)_3$ and $Os(NH_3)_5(TMFS)_3$ under argon atmosphere, a solid compound was isolated and caracterized by infrared and electronic spectroscopy, cyclic voltametry and Nuclear magnetic resonance ^1H. The infrared and NMR ^1H spectra suggest for this solid the following composition, trans-$Os(NH_3)_4(P(OEt)_3)_2(TMFS)_2$. Talking account NMR data, the increase order for the back-bonding is proposed: $Ru(CN)_5L^{-3} < Ru(NH_3)_4L_2^{+2} < Fe(CN)_5L^{-3} < Os(NH_3)_4L_2^{+2}$. The above order is also in agreement with the pKa data for $Ru(NH_3)_4H_2O\ P(OH)\ (OEt)_2^{+2}$ and $Fe(CN)_5P(OH)\ (OEt)_2$ complexes (3.7 and 5.1 respectively). (CNPq, FAPESP and PRONAQ-CAT).

QI-063 One–Step Synthesis of Cis–$[Ru(NH_3)_4$(2–Benzoylpyridine)$]^{2+}$ From $[RuCl(NH_3)_5]Cl_2$ and 2-Benzoylpyridine.

Adélia Silvia Angeli Teixeira de Paula, *Wagner Ferrarezi De Giovani* and *Elia Tfouni* (Departamento de Química da Faculdade de Filosofia, Ciências e Letras de Ribeirão Preto – USP).

The reaction of 2–benzoylpyridine with $[Ru(NH_3)_5(H_2O)]^{2+}$ (generated in situ from reduction of $[RuCl(NH_3)_5]^{2+}$) leads to cis–$[Ru(NH_3)_4$(2–benzoylpyridine)$]^{2+}$. The uv-visible region absorption spectrum, infrared spectrum and cyclic voltammogram in aqueous solution are identical to the ones taken from cis–$[Ru(NH_3)_4$(2–benzoylpyridine)$]^{2+}$ synthetized using cis–$[Ru(NH_3)_4\ (H_2O)_2]^{2+}$ as the starting complex. Results suggest that benzoylpyridine displaces a cis ammonea in the pentaammine leading to a chelate ring.

QI-064 A Bimetalic Catalyst $CuCr/SiO_2$ for Ethanol Desidrogenation.

Cesar Augusto Moraes de Abreu, *Celmy Maria Bezerra de Menezes Barbosa*, *Augusto Knoechelmann*, *Eleonora Maria Pereira de Luna Freire* (Departamento de Química Aplicada – Centro de Tecnologia – Universidade Federal de Pernambuco) e *Carlinda Farias* (Departamento de Engenharia de Minas – Centro de Tecnologia – Universidade Federal de Pernambuco).

A bimetalic catalyst, $Cucr/SiO_2$, by the impregnation method was prepared (19% Cu and 1% Cr). Cu and Cr were determined by AA and X-ray difraction. The activity tests were realized with a fixed bed reactor and the products were analized by gas cromatography. Acetic aldehyde was 48,35% and the conversion of the ethanol 72,83%. The yield was 35,58%.

QI-065 Oxidation of Isopropanol by Cobalt Complexes of Schiff Bases.

Neuza Costa dos Santos and *Edward Ralph Dockal* (Universidade Federal de São Carlos – Departamento de Química).

Cobalt complexes of Schiff bases show catalytic activity in organic reactions. In this study, the oxidation of isopropanol took place in the presence of cobalt(II) and cobalt(III) complexes with the Schiff bases (I) of the series ethylenediaminobis(salicylaldehyde) (Salen). The complexes are divided into three groups: $[Co^{III}(L)(H_2O)(OH)]$. $[Co^{III}(L)(OH)]$ and $[Co^{II}(L)]$. Of the 21 complexes tested 7 are capable of oxidizing isopropanol. The observed rat constants of the 2 or 3 consecutive reactions depend on the oxidation state of the cobalt and the structure of the Schiff bases. A mechanism for the oxidation of isopropanol involving all of the three series of complexes is proposed.

QI-066 Kinetic Studies on the Catalytic Activity of Manganese(II)–Gluconate Complexes in Reactions of Hydrogen Peroxide.

Cleonice Rocha and *Ana M. da Costa Ferreira* (Instituto de Química, Departamento de Química Fundamental, Universidade de São Paulo).

The oxidation of monoethyleneglycol by hydrogen peroxide, catalysed by manganese-gluconate complexes, has been investigated using spectrophotometric measurements, under nitrogen atmosphere. Kinetic data have shown a pseudo-first order dependence on both the concentration of the catalyst and of the peroxide, and a zero-order in relation to the pH, in the range 9.2 to 11.6. A second–order rate constant of $6.10 \times 10^{-1}\ M^{-1}s^{-1}$ was determined at 30°C, [gluconate] 0.10M and [glycol] 1.0M. Evidences for the formation of an aldehyde as a product of this oxidation was obtained.

QI-067 Solvatation of Trans–$[RuCl(CYCLAM)L]^+$.II. Substitution of 4–Acetylpyridine; Isonicotinamide and Pyridine by Dimethyl Sulfoxide.

Roberto Santana da Silva (UNESP/Instituto de Química (Araraquara) – Faculdade de Ciências Farmacêuticas de Ribeirão Preto/USP), *Elia Tfouni* (Faculdade de Filosofia, Ciências e Letras de Ribeirão Preto/Departamento de Química – USP) and *Luiz Antonio Andrade de Oliveira* – (UNESP/Instituto de Química (Araraquara)).

The macrocyclic complexes as trans–$[RuCl(cyclam)L]^+$, where L is 4–acetylpyridine, isonicotinamide or pyridine, show to be labile in relation to pyridinic ligands bonding to the ruthenium (II). The substitution these ligands have been studied in dimethilsufoxide solution. The kinetic of system have been studied in some temperatures at ionic strength $\mu = 0{,}1M\ (NaBF_4)$. Rates of reaction at 25°C to L = 4–acpy, isn and py are $5{,}8 \times 10^{-4} M^{-1}s^{-1}$; $6{,}74 \times 10^{-4} M^{-1}s^{-1}$ and $2{,}05 \times 10^{-3} M^{-1}s^{-1}$. The activation parameters to these species are: $\Delta H^{\neq} = 86{,}1\ KJ\ mol^{-1}$, $82{,}3\ KJ\ mol^{-1}$ and $76{,}3\ KJ\ mol^{-1}$.

QI-068 Ammonia Photoaquation on Trans-$[Ru(NH_3)_4 P(OEt)_3(H_2O)]^{2+}$.

Douglas Wagner Franco, *Selma Elaine Mazzetto*, *José Aroldo Viana dos Santos* (Instituto de Física e Química de São Carlos - USP), *Elia Tfouni* and *Maria Lúcia Bento* (Departamento de Química da Faculdade de Filosofia, Ciências e letras de Ribeirão Preto – USP).

The $P(OEt)_3$ exhibit a strong kinetic delabilizing effect over the ammonia ligands on the cis position on the *trans*–$[Ru(NH_3)_4\ P(OEt)_3\ (H_2O)]^{2+}$ complex ion. Contrasting with this thermical kinetic inertia, when irradiated at 313 nm, the mono phosphitge complex undergo photoaquation of the cis ammonia with a quantum yield of 0.1. (CNPq, FAPESP).

QI-069 Electron-Transfer Photosensitization Via $[Co(pic)_3]^{3+}$ Citrate Ion Pairs.

Maria Amélia de Almeida and *Neyde Yukie Murakami Iha* (Instituto de Química – USP – C.P. 20780 – Brazil). *Shunji Utsuno* (Shizuoka University – Japan).

$[Co(pic)_3]^{3+}$ – citrate ion pairs (pic = 2–aminomethylpyridine) exhibit a charge transfer absorption band around 310 nm. Light excitation at 313 nm of deoxygenated solutions cause an absorbance decrease as a consequence of the reduction of $[Co(pic)_3]^{3+}$ to $[Co(pic)_3]^{2+}$. In contrast with other labile cobalt(II) complexes, the electron-transfer process is reversible and the quantum yield is 0.09 at pH = 6.5, showing that this simple system can play the role of an electron–transfer photosensitizer.

QI-070 Synthesis, Purification and Photoinduced Electron Transfer Reaction of $[Co(sep)]^{3+}$ – Oxalate Ion Pairs.

Maria de Fátima Paredes de Oliveira and *Neyde Y. Murakami Iha* (Instituto de Química – USP – C.P. 20780 – Brazil.

The cobalt(III) cage complex, $[Co(sep)]Cl_3$, was prepared following a modified literature method and purified as a dithiocarbamate salt, without chromatographic procedure. The investigation of the photosensitized electron-transfer reaction of $[Co(sep)]^{3+}$/ oxalate ion pairs has been carried out in several conditions different from those that had already been studied. Excitation of the ion pairs in deoxygenated solutions (pH=5.5) results in a photoassisted reduction of the Co(III) complex with a quantum yield of 0.22 and 0.05 at 313 and 366 nm, respectively, and the oxidation of the oxalate ions.

QI-071 Light-Induced Ligand Substitution Reactions: Neighbouring Effect in the (2–Amine–5–Diethylaminepentane)Pentacyanoferrate(II) Complex.

Jailson Farias de Lima and *Neyde Yukie Murakami Iha* (Instituto de Química – USP – C.P. 20780, São Paulo, Brazil).

Excitation in the E(1) ligand field band of the title diamine complex in the presence of an excess of the ligand led to photolabilization of the cyanide ion with a quantum yield of (0.058 + 0.003). This value is markedly lower than those of other diamines studied, such as ethylenediamine, propylenediamine, 2–aminemethylpyridine, etc. These data show that the efficiency of the neighbouring effect depends on the length of the NN chain and/or the presense of bulk substituents.

QI-072 Synthesis and Photoreactivity of the Complex [Co(dinosar)]Cl_3. *Sílvia Naomi Motonaga* and *Neyde Yukie Murakami Iha* (Instituto de Química – USP – C.P. 20780 – Brazil).

The photochemical behaviour of a sarcophagine type complex, [Co(dinosar)]Cl_3, dinosar=1,8–dinitro–3,6,10,13,16,19–hexaazabicyclo–(6.6.6)eicosane, has been investigated and compared to that of the parent sepulchrate complex. The ligand field excitation of the complex was carried out in aqueous solution with an Oriel Hg(Xe) lamp, equipped with a filter. Light excitation of deoxygenated or air equilibrated solution of $[Co(dinosar)]^{3+}$, at a pH interval from 6.64 to 3.10, produced an absorbance increase at 370 nm. The apparent quantum yield based on these spectral changes is 0.02 at pH 6.64.

QI-073 Reactivity of: $[Ru(NH_3)_5(H_2O)]^{2+}$ Ion in Nitromethane. *Janete Jane Fernandes Alves, Ana Maria de Guzzi Plepis, Douglas Wagner Franco* (Instituto de Física e Química de São Carlos – USP) and *José Milton de Rezende* (Instituto de Ciências Exatas – UFMG).

A brown solid of composition $[Ru(NH_3)_5C_4H_5N_2](PF_6)_2$ was isolated from the dissolving of $[Ru(NH_3)_5(H_2O)](PF_6)_2$ in nitromethane. This solid was characterized by microanalysis, cyclic voltammetry, electronic spectrophotometry, infrared spectroscopy and nuclear magnetic resonance (^1H). The compound $[Ru(NH_3)_5C_4H_5N_2](PF_6)_2$ lose amonia with specific rate of 1.2 x $10^{-5}s^{-1}$ (pH = 3.0 CF_3COOH, μ ~0.1 $CF_3COOH/NaCF_3COOH$). (CNPq, FAPESP).

QI-074 Synthesis and Characterization of Trans-$[Ru(NH_3)_4(DMPP)_2][PF_6]_2$ Complex. *Joacy Batista de Lima, Benedito dos Santos Lima Neto, Douglas Wagner Franco.* (Instituto de Física e Química de São Carlos – Universidade de São Paulo).

The complex trans-$[Ru(NH_3)_4(DMPP)_2][PF_6]_2$ has been isolated and characterized by microanalysis, cyclic voltammetry, infrared and electronic spectroscopy. The aquation of trans–$[Ru(NH_3)_4(DMPP)_2]^{2+}$ to trans–$[Ru(NH_3)_4(DMPP)(H_2O)]^{2+}$ was studied spectrophotometrically employing isonicotinamide (isn) or pirazine (pz) as a scavenger. The specific rate constant for the aquation is (2.8 ± 0.1) x $10^{-5}s^{-1}$, at 25.0 ± 0.1°C, μ = 0,10 mol dm^{-3}, NaO_2CCF_3, $C_{H+} = 1.0 \times 10^{-3}$ mol dm^{-3}, CF_3CO_2H.

QI-075 Reactions in the System 2,2'–Bis(α–thioethanoic)propane Acid/Tetrachloropalladium(II) Acid – Synthesis of $[Cl_2PdC_7H_{14}O_4S_2](ClO_4)_2$. *Ilzemaro Schneider, Manfredo Hörner* and *Rodrigo Nogueira Olendski* (Departamento de Química – Universidade Federal de Santa Maria – Brasil).

$[Cl_2PdC_7H_{14}O_4S_2](C10_4)_2$ (1) is obtained from equimolar amounts of the 2,2'–bis(α–thioethanoic)propane acid. $C_7H_{12}O_4S_4$ and tetrachloropalladium(II) acid, H_2PdCl_4 in $HClO_4$(70%). The i.r. spectra of the free ligand $C_7H_{12}O_4S_2$ shows the deformation δCS_2 at 664 cm^{-1}. The absence of δCS_2 in the i.r. spectra of the complex (1), demonstrate the coordination of the S atoms to the Pd(II). X–ray photographs from single crystals of 1 (BUERGER METHOD), shows a triclinic unit cell. Cell parameters and the correct space group, will be defined by the solution of the crystal structure of (1).

QI-076 Topochemical Reaction of the Organic Groups in Layered Materials of Type γ–ZrP. *José Márcio Siqueira Jr.* and *Oswaldo Luiz Alves* (LQES – Lab. Quim. do Estado Sólido, IQ–UNICAMP).

Organic derivates of γ–ziconium phosphate (γ–ZrP) were prepared by the topochemical reactions with phenylphosphonic [A] and 2–carboxyethylphosphonic [B] acids. The characterization of the materials was carried out by XRD, FTIR, TGA,DSC and SEM techniques. The increase in basal spacing from 12.3 Å (γ–ZrP) to 15.1 Å [A] and 13.2 Å [B] and the IR spectra suggest that the incorporation of the groups O_3P–∅ and O_3P–$(CH_2)_2COOH$ takes place on the γ–structure.

QI-077 Study of Organic Layered Materials of Sn(IV). Part II: Thermal Behavior and Synthesis of Tin(IV) Bis–Dodecylphosphate Compounds. *M. E. Medeiros* and *O. L. Alves* (LQES – Lab. Quím. Estado Sólido, IQ/UNICAMP).

The layered material Tin(IV) bis-dodecylphosphate were caracterized by FTIR and XRD. The results indicate the formation of Sn(IV)–O–P–O–R bonds, similarly to the system Tin(IV) bis–phenylphosphonate and bis–carboxyethylphosphonate, the formation of a layered structure closed to α–ZrP. Was observed a value of 16,77 Å for the interplanar distance. The TGA curve for all materials exhibited a weigth loss of the organic moiety with formation of pyrophosphate at temperatures greater than 400°C. The thermal stability of these materials is large compared with the free phosphonic acids indicating the formation of new phases.

QI-078 Intercalation of Amines in $M(XHO_4)_2.1H_2O$ Layered Materials, Where M = Tetravalent Metals, X = P, As : Complementarity of XRD and FTIR Techniques. *M. E. Medeiros, J. M. Siqueira Jr., F. M. S. Garrido* and *O. L. Alves* (LQES – Lab. Quím. Estado Sólido, IQ, UNICAMP).

This work reports the results of the interaction of butylamine (BuAm), in vapour phase, with hydrogenphosphate of Zr, Ti, Sn, Ge and Pb, and hydrogenarsenate of Zr with α–layered structures. Was obtained intercalated single phases for the metals Zr, Ti and Sn, and more complex forms for Ge and Pb. The variation of d_{001} upon the intercalation is near 11 Å that suggests, as for the materials synthetized in aqueous medium, the formation of a bilayer of BuAm inside of the host structure. The FTIR spectra is very simillar for Zr and Ti compounds but present significative differences for Sn.

QI-079 The Effect of the Synthetic Conditions on the Crystallinity of Layered Materials of Zr(IV) With Large Organic Pendant Groups. *F. M. S. Garrido and O. L. Alves* (LQES - Lab. Quím. Estado Sólido/ IQ - UNICAMP).

This communication reports the synthesis and the improve of the degree of crystallinity of the layered compound $Zr(R\text{-}PO_3)_2.nH_2O$, where R = $-CH_2Ph(OH)(C(CH_3)_3)_2$. Two conditions were used: reaction of Zr^{4+} i) with the phosphonic acid obtained "in situ" by hydrolisis of diethylester (S-2) and ii) with the phosphonic acid in presence of HF (S-3). The X-ray patterns indicate that a semi-crystalline material was formed on S-2 and a well crystallized on S-3. The interlayer distance changed from 17,7 Å to 15,9 Å, as the degree of crystallinity increases. The FTIR spectra show several modifications that can be associated with the crystallinity.

QI-080 Synthesis and Characterization of Intercalation Compounds of FeOCl. *Ralpho Rinaldo dos Reis* and *Oswaldo Luiz Alves* (LQES - Lab. Quím. do Estado Sólido, IQ - UNICAMP).

Intercalation compounds were obtained by soaking FeOCl in some amines: n-butylamine and benzylamine. In these systems were studied the effects of temperature and reaction time. The materials were characterized by X-Ray diffraction, FTIR spectroscopy and thermal behavior (TGA). The expansion of b-axis of unit cell and IR data confirm the intercalation into FeOCl host.

QI-081 Solvothermal Synthesis of Iron Oxide: The Influence of NH_4Cl Mineralizer. *Eduardo Lyse Corrêa Netto Carvalho* and *Miguel Jafelicci Júnior* (São Paulo State University - Chemistry Institute - P O Box 355 - 14800 - Araraquara - SP - Brazil).

Iron oxide has been obtained from solvothermal treatment of isopropanolic iron hydrous oxide gel. The gel was prepared by addition of alcaline solution to isopropanolic iron nitrate solution under stirring. Ammonium chloride was used as mineralizer. Product was characterized by chemical analysis, x-ray powder diffraction, scanning electron microscopy and infrared spectroscopy. Uniform primary particles were formed but these ones agglomerate into large aggregates which the sintering process takes place at low temperature under solvothermal conditions. Remanescent ammonium chloride was found in the solid phase.

QI-082 Preparation Methods of Gadolinium Silicates Powders. *Jandira Aparecida Simoneti, Marian Rosaly Davolos e Miquel Jafelicci Junior* (Instituto de Química - Universidade Estadual Paulista - C.P. 355 - 14800 - Araraquara - SP - Brasil).

In order to improve different methods of luminecent rare earth silicates terbium activated powders it has been studied the $1Gd_2O_2$:$1SiO_2$ binary system. It was compared hydrothermal synthesis carried out at 510 and 680 atm, at 400°C for 1 h with usual solid state technique firing in an argon atm at 1.000 and 1.350°C. The products obtained were characterized by infrared vibrational spectroscopy and X-ray powder diffraction. It was found cristalline powders from both methods used, where the hydrothermal one is gadolinium oxyorthosilicate and the usual synthesis one probably is hidroxymetasilicate.

QI-083 Mecanismos de Desexcitação Cooperativa em Processos Up-Conversion no Par Tm^{3+} –Yb^{3+}. *G. F. de Sá, O. L. Malta, P. A. Santa-Cruz* (Universidade Federal de Pernambuco).

We have studied the infrared-to-blue up-conversion by a stepwise energy transfer process in the Yb^{3+} –Tm^{3+} pair, in glass ceramic hosts (PbF_2–GeO_2), the same matrix from which we have developed a very high sensitive 1.5μm quantum detector. The experimental curve involving the infrared-to-blue conversion versus sensityzer concentration (Yb^{3+}) was analyzed. A quenching of the Tm^{3+} ion blue emission at high Yb^{3+} concentration was observed, and a cooperative back-transfer mechanism was proposed.

QI-084 Interaction of Claylike Materials With Organic Bases-I-Preliminary Adsorption Study. *Aluisio S. Reis, Jr., José de A. Simoni* and *Aécio P. Chagas* (Instituto de Química, Universidade Estadual de Campinas).

The adsorption of N,N-dimethylacetamide, in n-heptane solution, on homoionic (H^+, Ca^{2+}, Zn^{2+}) claylike material, drying at 25 or

250°C, was studied by UV spectrophotometrie. The data was fitted to the equation N/X = 1/Xm(K–1) + N/Xm (N = solute molar fraction, X = adsorved solute amount, Xm = solute amount covering (monolayer) one gram of solid, K = equilibrium constant associated to adsorption process). From these six materials were obtained 10^4Xm = 1.3 to 2.3 and 10^{-4} K = 0.4 to 2.3 (correlation coeff. >0.99, for 6 or 7 points).

QI-085 Ionic Exchange of Ferric Antimoniate With Salts of Amines. *Daltamir J. Maia* and *Claudio Airoldi* (Instituto de Química, UNICAMP, Caixa Postal 6154, 13081 Campinas, SP).

The ferric antimoniate in acid form has been exchanged the protons with ammonium and other salts of amines in the chloride form, by means of the batch procedure. Experimentally, a series of samples of exchanger in bidistilled water containing variable amounts of salts, were shaked in a thermostat during 6 h at 298 K. Analysis of protons and excess of salts in the supernatant permitted the determination of the exchanger isotherms, which showed larger for NH_4^+. From a computer programm the selective coefficients have been calculated, showing the sequence: $NH_4^+ > CH_3(CH_2)_2NH_3^+ > CH_3NH_3^+ > (C_2H_5)_2NH_2^+$.

QI-086 Empirical Force Field Calculations on Mixed-Valence Ruthenium Complexes. *Cecília Cipriano* and *Juan Omar Machuca-Herrera* (Departamento de Química Inorgânica – IQ – UFRJ).

Mixed–Valence complexes continue to be an important kind of inorganic compounds for which neither experimental nor theoretical chemists can agree on whether the best description is a localized or a delocalized molecular structure. The well known Creutz–Taube ion (–pyrazine) decaamminodiruthenium (II, III) ions, is the prototype of binuclear mixed valence complexes. The aim of this work is to demonstrate the application of the empirical force field method (with convenient parametrization, along with Extended Huckel Theory) to studies of molecular and eletronicground state structures of the Creutz-Taube complex. Molecular geometries were fully optimized using FORCES, an empirical force field computer program.

QI-087 Spectroscopic Study of Cerium(IV)–Praseodimium(IV) Oxide Pigments in Gel Films. *Lisete Furtado* and *Henrique E. Toma*, (Instituto de Química, Univ. São Paulo).

The synthesis and spectroscopic properties of a series of cerium(IV)–praseodimium(IV) oxide pigments are reported. The pigments exhibit brick-red colours and are suitable for ceramic applications because of their high temperature stability. Electronic absorption spectra of the pigments suspended in a gel matrix of polyvinyl alcohol-sodium tetradecaborate mixture, consist of broad band with Gaussian components at 378 and 496 nm. These bands are ascribed to charge–transfer transitions from the occupied oxygen p-orbitals to the empty f levels of the lanthanides.

QI-088 Luminescent Vitreous Matriz Ceramics. *Lisete Furtado* (Instituto de Química – USP), *Rosemeiri Martins Campos* and *Osvaldo Antonio Serra* (Deptº de Química da FFCLRP – USP – Ribeirão Preto – SP).

Luminescent porcelains are one of the most important material for dental resto-rations. The manufacture of ceramics usually nivalves process where crystaline powders are compacted and then fired at temperatures that are high enough to develop useful properties. We prepared and characterized several materials with luminescent properties, by addition of Ce, Tb phosphates. (CNPq)

QI-089 Zeolites Containing Rare Earths. Luminescent Properties. *Ieda Lucia Viana Rosa* and *Osvaldo Antonio Serra* (Departamento de Química da FFCLRP – USP – Ribeirão Preto – SP).

A Sample of 360 HUA zeolite type HS–USY exchanged with Eu^{3+} has been studied by luminescence spectroscopy. The effect of differents thermal treatments on the location of Eu^{3+} ions in this zeolite was monitorated by luminescence emissions. On the basis of the observed $^5D_0 \rightarrow {}^7F_0, {}^7F_1$ e $^7F_2$ transitions, Eu^{3+} ons were found to be located in distinct environments. (CAPES, CNPq, FINEP.)

QI-090 Metaptiosphate Reactions in Solid State. *Rosemeiri Martins Campos, Ieda Lúcia Viana Rosa, Gilson Zapparolli* and *Oswaldo Antonio Serra* (Departamento de Química da FFCLRP – USP – Ribeirão Preto – SP).

Rare Earth trimetaphosphates heated at ~1000° decompose giving phosphates and loosing P_2O_5 (O. A. Serra and E. Giesbrecht, J. Inorg. Nucl. Chem. *30*, 793 (1968). We describe the reaction in solid state between a dopped trimetaphosphate and lanthanum oxide giving a new phosphate: $(Ce,Tb)P_3O_9 + La_2O_3$ —— $(Ce, Tb)PO_4 + 2LaPO_4$. (CNPq, FINEP)

QI-092 Synthesis of Cromosilicate Pentasil Zeolite of Highly Crystalline and Changes in The Infrared Spectra. *Heloise O. Pastore, José Silvio T. Mambrim, Celso U. Davanzo* and *Eduardo J. S. Vichi* (Instituto de Química – Universidade Estadual de Campinas – Caixa Postal 6154 – CEP 13081 – Campinas – S.P. – Brazil).

We synthetized a cronosilicate pentasil zeolite highly cristalline as revealed by x–ray diffraction and inspection a optical microscope. The observed ratio of the integrated intensities of the bands at 550 cm^{-1} and 430 cm^{-1} is 1,1. This is a unusual results as compared with the literature value of about 0.6 for pentasil zeolite considered of excellent cristanity.

QI-093 Structural Changes in Zeolitic Ferrisilicate Associated to Dehydrating Processes. *Eduardo J. S. Vichi, Heloise O. Pastore, Edison Stein* and *Celso U. Davanzo* (Instituto de Química, UNICAMP), *Helion Vargas, Edson C. da Silva, Flávio C. G. Granda* and *Ossamu Nakamura* (Instituto de Física, UNICAMP).

Structural modifications related to the processes of hydrating and dehydrating a ferrisilicate of the pentasil family have been studied by means of powder X–ray diffraction and DSC, and were related to ESR measurements. The results point to a structural phase transition occuring with symmetry changing from orthorhombic to monoclinic upon loosing water in the thermal process.

QI-094 Synthesis and Spectroscopic Characterization of a New Zeolite With Cr(III) in the Lattice. *Eduardo J. S. Vichi, Heloise O. Pastore, Edison Stein* and *Celso U. Davanzo* (Instituto de Química, UNICAMP) *Helion Vargas, Edson C. Silva, Mauro Bąesso* and *Ossamu Nakamura* (Instituto de Física, UNICAMP).

A hyghly cristalline zeolite was prepared with the following chemical composition: Al(0.61%, SiO_2/Al_2O_3 = 228); Fe(0.05%, SiO_2/Fe_2O_3 = 4650); Cr(0.41%, SiO_2/Cr_2O_3 = 540). The incorporation of Cr(III) in the framework of the zeolite is confirmed by the results of electron spin resonance and photoacustic spectroscopy measurements.

QI-095 Aluminum Polyphosphates or Is There an Alternative to Titanium Oxide? *E.C.O. Lima, F. Galembeck* (Unicamp, Caixa Postal 6154, 13081 Campinas, SP, Brazil).

Aluminum hexametaphosphates were prepared by admixture of aluminum nitrate, sodium hexametaphosphate and ammonium hydroxide aqueous solutions, following a three-factor two-level factorial plan. Eight different samples were obtained, and characterized by elemental analysis, IR, X–ray diffraction and transmission electron microscopy. Solids having Al/P ≧ 1 are refractory, non–coalescent. Al/P < 1 solids expand upon heating, generating hollow particles. This morphology is highly desirable in prospective white pigments, due to multiple scattering.

QI-096 Thermal Decomposition of Iron (III) Hydroxoacetate. *M. C. R. Varela, F. Galembeck* and *C. A. P. Leite* (UNICAMP, Caixa Postal 6154, 13081 Campinas SP, Brazil).

Magnetite can be obtained by heating iron (III) hydroxoacetate a reaction which has been studied by many techniques. DSC thermograms of the hydroxoacetate show two major features: a strong endothermic peak below 100°C and an inflexion, around 200°C. Electron diffractograms, taken in this temperature rangeon, show that this inflexion is due to the onset of magnetite cristallization, which confirms previous results obtained by ESR. The absence of other features in the thermogram can be explained by considering the enthalpies of the intervening reactions.

QI-097 Silicone Stabilization by Ferric Oxide. *W. Botter Jr.*, and *F. Galembeck* (Unicamp, Caixa Postal 6154, 13081 Campinas, SP, Brazil).

Iron (III) oxide can be added to cross–linked polydimethyl siloxane (PDMS) by sorption of $Fe(CO)_5$ followed by *in situ* oxidation. The modified PDMS has greater thermal stability than the original rubber, as observed by thermogravimetry. Temperature for 1% weight loss is 260°C in the original PDMS and increasses up to 340°C, depending on the actual modification procedure. However, iron oxide causes a decrease in the temperatures at wich weight loss is 5%.

QI-098 Exafs in Chemistry: Characterization of Superconductors Molecular Precursors. *M. Tsunoda* (Departamento de Química, Centro Técnico-Científico, Pontifícia Universidade Católica do Rio de Janeiro) and *L. Hubert–Pfalzgraf, R. Papiernik* (Laboratoire de Chimie Moléculaire/UA CNRS, Université de Nice).

EXAFS in K and L_3 absorption edges of Y and Ba respectively, were used to characterize the reaction products of $Y_5O(OiPr)_{13}$ with $Ba(OiPr)_2$ and $Ba_{(s)}$. Apparently the structure of the starting compound is maintained, and there is evidence for the formation of a heterometallic species.

QI-099 The Mean Enthalpy of Metal–Ligand Bond Determination of the Adducts Between MCl_2 (Zn, Cd) With Thiobenzamide. *Cláudio Airoldi* and *Edemir A. Digiampietri* (Instituto de Química, UNICAMP, Caixa Postal 6154, 13081 Campinas).

The adducts $ZnCl_2.2tb$ and $CdCl_2.tb$ (tb = thiobenzamide) were prepared from ethanol solutions and characterized by melting point, elemental analysis, thermogravimetry and infrared spectroscopy. The standard molar entalpy of the reaction $MCl_2(c) + n\ tb(c) = MCl_2.n\ tb(c)$, $\Delta_r H^\circ_m$ gave – 52.26 ± 0.69 (Zn) and –19.50 ± 0.20 (Cd) kJ mol^{-1}. From this value the standard molar enthalpy of formation –377.2 ± 2.2; –455.8 ± 1.5, the standard enthalpy of decomposition 259.1 ± 3.2; 122.9 ± 2.2, and the molar enthalpy of the lattice –408.1 ± 3.3; –303.7 ± 3.1 kJ mol^{-1}, respectively, were determined. Through a thermochemical the mean standard enthalpy of metal–sulphur bond 152.3 ± 4.0 and 200.3 ± 3 kJ mol^{-1}, respectively, were calculated.

QI-100 Calorimetry of Primary Amines With Alfa Titanium Interaction. *Cláudio Airoldi* (Instituto de Química, UNICAMP, Caixa Postal 6154, 13081, Campinas, S.P.) and *Severino F. de Oliveira* (Depto. de Química, UFPB, Campus Universitário, J. Pessoa, PB).

The α–crystalline titanium phosphate (αTiP) intercalates primary amines (C_1 to C_4) into the lamelae, which process was calorimetrilly followed through a precision LKB 8700 system. The standard enthalpy between αTiP and amines, $\Delta_R H^{\circ}_m$/kJmol^{-1}, –49.99 ± 0.21; –47.83 ± 0.27; –44.79 ± 0.11; –42.50 ± 0.15 in water, and –55.74 ± 0.06; –58.14 ± 0.41; –60.10 ± 0.14; –63.35 ± 0.11 in 1,2–dichloroethane (DCE) for methyl, ethyl, propyl and buthylamine, respectively, were obtained. The enthalpy of intercalation depends on the solvent and the number of the carbon of amine, which corresponds to –2.5 (DCE) and +2.6 kJmol^{-1} (H_2O) for each increment of CH_2 group in alkylamine chain. After intercalation the interlamellar distance (75.6 pm) changed to 129, 147, 167 and 190 pm (DCE), for the above amines, respectivelly.

QI-101 Thermochemical Properties of Chelates of Di-N-Propyldithiocarbamate With Phosphorus–Group Elements. *Lucildes Pita M. Neves, Mª de Lourdes M. Ribeiro Dias, Mª Helena P. Fernandes* and *Antonio Gouveia de Souza* (Departamento de Físico-Química – Instituto de Química – Universidade Federal da Bahia).

The standard molar enthalpy of reaction was obtain d by measuring the enthalpy of dissolution of reactants and products into acetone as calorimetric solvent, by means of the reaction: $ECl_3(1, cr) + 3[NH_2(C_3H_7)_2][S_2CN(C_3H_7)_2](cr) = E[S_2CN(C_3H_7)_2]_3 (cr) + 3[NH_2(C_3H_7)_2]C1 (cr)$, where E equivalve P, As, Sb e Bi. The values of ΔrH°_m were obtained by applying a convenient thermochemical cycle, which are: – (357,74 ± 9,38), – (108,97 ± 1,26), – (93,35 ± 0,46) e – (118,42 ± 0,45)kj.mol^{-1}. CAPES.

QI-102 Determination of Termodynamic Parameters of Chelate Di-N-Butyldithiocarbamate With Zinc(II) Chloride. *José Hilton de Souza, Antonio Gouveia de Souza* and *Carlos Davidson Pinheiro* (Departamento de Química – CCEN – Universidade Federal da Paraíba – João Pessoa – PB).

Through reaction–solution calorimetric the standard molar enthalpy of recation in condensaded phase were determined: $ZnCl_2(s) + 2Bu^n_2NH_2S_2CNBu^n_2(s) \rightarrow Zn(S_2CNBu^n_2)_2(s) + 2Bu^n_2NH_2Cl (s)$; $\Delta_r H^{\circ}_m$ = –(46,69 ± 2,22)kj.mol which enable in to obtain the enthalpy of formation of the chelate in the solid phase, $\Delta_f H^{\circ}_m(s)$ = –(473,26 ± 4,25)kj.mol . Through differential scanning calorimetry and estimative methods was determined the standard molar enthalpy of sublimation, $\Delta^g_s H^{\circ}_m$ = (84 ± 2)kj.mol . From these date the mean enthalpy of the M–S bond were obtained by considering the homolytic, <D>(Zn–S) = (197,29 ± 2,00) was well as the heterolytic breaking, <D>(Zn–S) = (761,36 ± 2,01)kj.mol^{-1}. (CNPq).

QI-103 Determination of the Lattice Energy of Di-Iso-Butilammonium Chloride. *Antônio Gouveia de Souza, Carlos Davidson Pinheiro* and *José Hilton de Souza* (Universidade Federal da Paraíba – Centro de Ciências Exatas e da Natureza – Departamento de Química – Campus I).

Through solution–reaction calorimetry and using equation: $Bu^i_2NH_2Cl_{(cr)} + OH^-_{(aq)} = Bu^i_2NH_{(1)} + H_2O_{(1)} + Cl^-_{(aq)}$ was measured the standard enthalpy of reaction and standard enthalpy of solution of the amine. To determine the standard enthalpy of formation was used the following expression: $\Delta_f H^{\circ}{}_m[Bu^i_2NH_2Cl_{,2}] = \Delta_f H^{\circ}{}_m(Bu^i_2NH:5.55H_2O] + \Delta_f H^{\circ}{}_m(H_2O_{,1}) + \Delta_f H^{\circ}{}_m(NaCl : 5{,}55H_2O) - \Delta_f H^{\circ}{}_m(NaOH : 5{,}55H_2O) - \Delta_r H^{\circ}{}_m$, however, using additional literature data to equation $U_t = \Delta_f H^{\circ}{}_m(H^+, g) + \Delta_f H^{\circ}m(Cl^-, g) + \Delta_f H^{\circ}{}_m(Bu^i_2NH, g) - \Delta_f H^{\circ}{}_m(Bu^i_2NH_2Cl,s) - P - 2.R.T$, was obtained the lattice energy U_t = 553 kJ.mol^{-1}. (CNPq).

QI-104 Differential Scanning Calorimetric Study of the Chelate Di-N-Propyldithiocarbamate With Zinc. *Antonio Gouveia de Souza* (Departamento de Química – CCEN – Universidade Federal da Paraíba – João Pessoa – PB) *Maria Aparecida Ruiz Piva de Carvalho* (EEPSG – Joaquim Abarca – Tupã – SP) e *Maria Lins de Medeiros Melo* (Departamento de Engenharia Química – Campus II – Universidade Federal da Paraíba – Campina Grande – PB).

Through DSC the standard molar enthalpy of fusion (30,35 ± 0,25)kJ.mol^{-1}, the heat capacities for the solid state (0,56 ± 0,09) and for the liquid phase (0,81 ± 0,01)kJ.$mol^{-1}K^{-1}$ were determined. The standard molar enthalpy of vaporization (60,92 ± 1,51)kJ.mol^{-1} and the gas–phase heat capacity (0,55 ± 0,14)kJ.$mol^{-1}K^{-1}$ were estimated from empirical equations. The application of all these auxiliary quantitles in methods of the literature enable the estimation of the standard, molar enthalpy of sublimation of the chelate, $\Delta^g_{cr} H^{\circ}_m$(298,15k) = (147 ± 2)kJ.$mol^{-1}$. (CAPES/CNPq).

QI-105 Thermal Decomposition of Some Thiocyanatos and Tetrahydroborate Copper(I) Complexes – *Carlos Cesar Porta* (Depto. de Química – Instituto de Biociências de Botucatu – UNESP) and *Antonio Eduardo Mauro* (Depto. de Química Geral e Inorgânica – Instituto de Química de Araraquara – UNESP).

The TG and DTG studies of $[Cu(SCN)(PPh_3)_2]_2$ and of it's derivatives with ligands like bipy, phen, py, tu, dmtu and tmen have been carried out. It was verified that the product of the decomposition is a mixture of $CuSO_4$ and CuO. The thermal decomposition of $[Cu(BH_4)_2(PPh_3)_2]$ gives a mixture of $Cu(BO_2)_2$ and CuO as a final product.

QI-106 Calorimetric Determination of Enthalpy of Vaporization for Chloroform–Tetrahydrofuran Mixture. *Geraldo L. Rolim, José de A. Simoni* and *Aécio P. Chagas* (Instituto de Química, Universidade Estadual de Campinas - UNICAMP).

By vaporization in a calorimetric cell, with N_2 flux, was determinated the molar enthalpy of vaporization, Δvap Hm, of chloroform (A)–tetrahydrofuran (B) system samples. The data was fitted to the equation: $\Delta vap\ Hm/kJ\ mol^{-1} = 31.33 + 3.822\ X + 6.123\ X^2 - 11.80\ X^3$, were X = A molar fraction, standard desviation = 0,45 kJ mol^{-1}. It was also obtained the excess molar enthalpy of vaporization of the mixture, $\Delta vap\ Hm^{exc} = \Delta vap\ Hm(mix) - X\ \Delta vap\ Hm\ (A) - (1-X)\ \Delta vap\ Hm\ (B)$.

QI-107 Comparative Study of Thermochemistry Adducts of Rare Earths With Amides Ligands (DMF, DMA and HMPA). *O. A. de Oliveira* and *D. M. Araújo Melo* (Universidade Federal do Rio Grande do Norte – Departamento de Química) and *L. B. Zinner* (Universidade de São Paulo – Instituto de Química).

Adducts of lanthanide trifluoromethanesulfonates and DMF, DMA, and HMPA have been prepared and the enthalpy variations were determined by solution colorimetry using suitable thermodinamic cycles. The results obtained show that the $\Delta\ H^Q_R$ values of HMPA > DMA > DMF. This fact is attributed to basicity of ligand HMPA. Ethanol presents a considerable donicity and competes with ligands, decreasing the metal–ligand interaction. The results indicate that even in ethanolic solution, the ligands remains coordinated to the lanthanides.

QI-108 Thermal Decomposition of Dimetilacetamide Complexes of Lanthanide Trifluoromethanesulfonates. *D. M. Araújo Melo, O. A. de Oliveira* and *A. Oliveira da Silva* (Universidade Federal do Rio Grande do Norte) and *L. B. Zinner* and *G. Vicentini* (Universidade de São Paulo – Instituto de Química).

In this paper we report on a thermoanalytical study of lanthanide trifluoromethanesulfonate complexes with dimethyl acetamide amploying the TG and DTA technique. Thermal studies were performed using a Perkin-Elmer TGA-DSC series 7 instrument set. The first step in the thermal degradation process in nitrogen and air is the loss of the water molecules. The starts at very low temperatures (~30°C). Dehydratation proceeds in on step; however, for the Cerium compound there is some evidence for the formation of $Ce(CF_3SO_3)_3.2DMA$. The anhydrous lanthanoid trifluoromethanesulfonates formed around 400°C rapidly decompose with evolution of COF_2 and SO_2, giving LnF_3 as the final product at ~470°C.

QI-109 Thermochemical Properties of Adducts of N-N-Dimethylacetamida and Tetramethylthiourea With Trihalides of Arsenic. *Luís Carlos Ramos dos Santos* (Depto. de Química – CCEN – Univ. Federal da Paraíba – João Pessoa – PB) and *Pedro Oliver Dunstan Lozano* (Instituto de Química – UNICAMP – Campinas – SP).

Adducts of AsX_3(X = Cl, Br e I) were synthesized and characterized with tetramethylthiourea (TMTU) and n,n–dimethylacetamida (DMA). The standard enthalpies determined calorimetrically at 298,15k by solution–reaction are, respectively: –(47,16 ± 0,51); –(36,76 ± 0,32); –(34,55 ± 0,38); –(100,98 ± 0,23), –(82,78 ± 0,10) e –(32,17 ± 0,14)kj.mol^{-1}. The energy average of arsenic–oxigen ($\bar{D}_{As-O}$) or arsenic–sulfur ($\bar{D}_{As-S}$) bonds were also estimated. The termochemical data lead to the conclusion of the acidity order $AsCl_3 > AsBr_3 > AsI_3$ and the general basicity order TMTU > DMA.

QI-110 A Thermochemical Study of the Adducts of Zinc, Cadmium and Mercury Bromides With 2–Pyrrolidone. *José C. de Queiróz, Francisco S. Dias* and *Eunice F. S. Vieira* (Depto. de Química Orgânica e Inorgânica da Universidade Federal do Ceará).

The complex compounds $MBr_2(Bul)n$, where $M=Zn^{2+}$, Cd^{2+} and Hg^{2+}, Bul = 2–pyrrolidone (or γ–Butyrolactam), and n= 1,2 or 3 have been obtained. Infrared assignments were discussed. The donor atom, in the 2–pyrrolidone molecule is the oxygen atom. The enthalpies of dissolution were measured calorimetrically at 25° in organic suitable solvents. Standard molar enthalpies of formation ($\Delta_f H^{\circ}$), reticular ($\Delta_L H^{\circ}$); and dissociation energies $\bar{D}$(M–O), have been calculated and compared with the similar thermodynamic quantities of complexes $MC1_2(Bul)_n$ and caprolactam complexes $MBr_2(CL)_n$.

QI-111 Application of Infrared Spectroscopy to Study Acid Zeolites HNaY Exchanged in Different Degrees III. Relation Between Acid Strength and Deshydration Velocity of Etanol. *Celso Ulisses Davanzo* and *Reginaldo Alberto Meloni* (Instituto de Química – Universidade Estadual de Campinas – Caixa Postal 6154 – Campinas – S.P. – Brazil).

We measured the number and strenght of Brönsted and Lewis acid sites following the intensity of bands at *ca.* 1540 cm^{-1} e 1460 cm^{-1} arising from the interaction of pyridine with ultrastable HNaY zeolites. The number and strength of the acid sites increase with the increase in the exchange degree. The same tendency is followed by the deshydration velocity of etanol.

QI-112 Study of the Simmetry of the Coordination Compounds of Cr(III) With Polyaminecarboxylic Acids, *Anna Cristina Pacheco Baptista* and *Judith Felcman,* (PUC/RJ, Department of Chemistry).

Coordination compounds of Cr(III) with polyaminecarboxylic acids can be considered to be of the type CrXnYm where X is O and Y is N. Experimental wavelenghts of the first absorption band in the visible spectra of 20 of these compounds were compared with the theoretical values. When m = 1, most of the compounds studied have C_{4v} simmetry indicating that the N is bound to Cr(III) and only two of them have O_h simmetry (N is not bound). When m = 2 some of them have D_{4h} simmetry (N in trans position) and others have C_{2v} simmetry (N in cis position); only in one case C_{4v} simmetry is observed indicating that one of the N is not bound.

QI-113 Determination of the Stequiometry of the Coordination Compounds of Cu(II) With Ethylenediamine–N,N'Diacetic Acid and Its Derivatives, *Maura Ferreira Mattos* and *Judith Felcman,* (PUC/RJ, Department of Chemistry).

Spectrometric studies of the coordination compounds of Cu(II) with ethylenediamine–N,N'–diacetic acid (EDDA), ethylene–diamine–N,N'–dimethylacetic acid (EDDMA); methylethylenediamine–N,N'dimethylacetic acid (MEDDMA), 1,4 dizaciclo-heptyl–N,N'–diacetic acid (DACHDA) and pyperazinediacetic acid (PIPDA) showed that only the CuL, compound is formed, except with PIPDA which forms both CuL and Cu_2L compounds.

QI-114 Study of d–d Transitions in Vanadyl Complexes of Tetradentatee Schiff Bases. *José Roberto Zamian* and *Edward Ralph Dockal* (Universidade Federal de São Carlos – Departamento de Química).

The electronic spectrum of many vanadyl complexes (VO^{2+}), principally those of low symmetry, are not well explained by the existing theoretical models. The present study, using Gaussian analysis the region of the spectra of the d–d transitions of vanadyl complexes of tetradentate Schiff bases (symmetry C_1 or C_s), has a its principal objective to add data concerning the behavior of the transitions by changing the ligand and thereby the local stereochemistry and the electronic density on the vanadyl ion. This Gaussian analysis of the eletronic spectra of various complexes suggest the following order of energies of the d–orbitals: $dxy < dxz < dyz < dz^2{-}y^2 << dz^2$.

QI-115 Effect of Formation of Ion Pair Over the Property Electrochemical and Espectroscopic of Complex Dicyanobis(1,10–Phenanthroline) Iron (II) in Nonaqueos Solvents. *Clóvis Antonio Rodrigues, Marcos Caroli Rezende* and *Eduardo Stadler* (Departamento de Química – Universidade Federal de Santa Catarina – C.P. 476 – Florianópolis – S.C).

Addiction of inorganic salt in the solutions induce the formation of ion pair between the complex and the metallic cations, change the values of redox potential $Fe^{(II/III)}$ and the MLCT band $Fe \rightarrow \pi^{\neq}$ phen. The ion pair cause a displacement in redox potential to values more positive and the position MLCT band to higher energy. A linear correlation was observed between the redox potential and the MLCT band displacement.

QI-116 Synthesis an Characterization of a New Mn (III) Complex Containing the Ligand N, N', N, N' – BIS [(2–Hidroxibenzil)(2–Methilpyridyl)]Ethylenediamine (H_2BBPEN). *A. Neves* (Depto. Química - Univ. Fed. SC); *S. M. D. Erthal* (Depto. Química - Univ. Fed. PR); *M. Horner* (Depto. Química - Univ. Fed. Santa Maria).

The hexadentate ligand H_2BBPEN reacts in methanolic solution with manganes (III) acetate yielding upon addition of NH_4PF_6 deep red crystals of $C_{28}H_{30}N_4O_2PF_6$(1). The compound crystallizes in the monoclinic space group $C_{2/c}$ with a = 13,465(7) Å; b = 24,071 (8) Å c = 18,639 (7) Å; β = 86,6°; V = 5998,9 (5) $Å^3$; Z = 8. CV of (1) in acetonitrile show two quasi-reversible one electron process $Mn^{IV/III} = +0{,}5$ V, $Mn^{III/II} = -0{,}4$ V vs Fc^+/Fc respectively. Spectroeletrochemical measurements for the oxidation process suggest the formation of a mixed-valence species with one intervalence band at 810 nm and ε = 2000 $1.mol^{-1}.cm^{-1}$.

QI-117 Synthesis and Spectroscopic Study of Some Copper (I) Perchlorate Complexes. *Vicente Alexandre de Lucca Neto; Antonio Eduardo Mauro* (Depto. de Química Geral e Inorgânica) e *Ademir dos Santos* (Depto. de Química Tecnológica e de Aplicação) - (Instituto de Química de Araraquara - UNESP).

Most of copper (I) complexes have this atom in a tetrahedral environment, although linear and trigonal-planar coordination have also been described. Some new compounds with the former geometry around the copper were synthesized by total or partial replacement of CH_3CN molecules, in $[Cu(CH_3CN)_4]ClO_4$, by ligands like dppe and phen. The compounds obtained were investigated by infrared spectroscopy.

QI-118 "Synthesis and Photophysics Studies of Nickel (O) Complexes". *Regina C. G. Frem; A. E. Mauro; A. C. Massabni* (Instituto de Química de Araraquara - UNESP).

Nickel (O) complexes of general formula NiL_4, where L = $AsPh_3$ or $SbPh_3$, were prepared from the reduction of Ni (II) compounds by $NaBH_4$. Nickel carbonyls derivatives has also been obtained. The compounds are yellow and show a strong orange emission when exposed to U. V. radiation. Excitation and emission spectra of the complexes are discussed.

QI-119 Carbon Determination in Coordination Compounds by Direct Combustion - Application to Ni, Pt and Sn Complexes. *Roberto Pellacani G. Monteiro e Oliêne dos Reis Fagundes* (Departamento de Tecnologia Química/CDTN-CNEN/BH - MG). and *Carlos Alberto L. Filgueiras, Gerimário F. de Souza e Bernadette de F. Trigo Passos* (Departamento de Química/ICEX/UFMG).

The direct combustion technique was applied recently to the elemental analysis of sulphur in coordination complexes. In this paper, the carbon determination by this technique in Ni, Pt and Sn complexes is described. The samples, weighed and mixed with an accelerator, were burned in a high frequency induction furnace (~1600°C) under an oxygen flux (~1*l*/min). The carbon was indirectly detected by thermal conductivity measurement and all the system was calibrated with NBS steel standards, 2-mercaptopyridine and S-trithiane. The analysis time was less than 1 min and the relative standard deviations were 5% or less for samples with mass up to 15 mg.

QI-120 Reactions Between Eu (III) Salts and 2,6-LNO. *Wanda de Oliveira, Hermi Felinto de Brito, Maria Cláudia França da Cunha* and *Ricardo Lourenço da Silva.* (Instituto de Química, Departamento de Química Fundamental, Universidade de São Paulo).

The addition compounds $EuCl_3$.3(2,6-LNO), $Eu(NO_3)_3$.4(2,6-LNO) and $Eu(ClO_4)_3$.8(2,5-LNO) were synthetized and characterized. Infrared spectra showed coordination of 2,6-LNO through the oxygen atom, and that nitrate ions were coordinated to Eu (III), but perchlorate ions not. The emission spectra at 77K suggested a D_3 symmetry around the central ion for the chloride and a D_{4d} for the nitrate and perchlorate adducts.

QI-121 Espectroscopic Behaviour for the EuX_3. 7.5 TSO ($X = CLO_4^-, PF_6^-, I^-$ and TSO - Tioxane Oxide), Complexes In D_{3d} Simmetry Site. *A. B. Nascimento* (Departamento de Química da Universidade Federal da Paraíba) and *G. Vicentini, H. F. Brito and M. C. F. Cunha* (Instituto de Química, USP).

Crystal-Field Parameters B_q^k of the title complexes were calculated from the $^5D_0 \rightarrow {}^7F_{0,1,2}$ transitions in the fluorescence spectra of the europun, by applying the tensorial operators method. The Scalar Crystal Field Strength parameter, Nv and Crystal Field Strength, S were also calculated and show a Crystal-Field splitting increasing in the order $PF_6^- < I^- < CLO_4^-$.

QI-122 $^3F_3$ and $^3P_2$ Energy Levels of Cubic Pr^{3+} Complexes Determination by the Equivalent Operators Method. *H. F. Brito and M. C. F. Cunha* (Instituto de Química, USP) and *A. B. Nascimento* (Departamento de Química da Universidade Federal da Paraíba).

Some considerations about the Pr^{3+} levels have been reported in the literature. This communication represents a contribution to the determination of crystal field parameters of Pr^{3+} complexes in cubic symmetry when J = 2 and 3 by the Stevens Method.

QI-123 Coordination Compounds Between 1,2-Bis (Alkylsulfinyl) Ethane and Uranyl Acetate. *João Aldésio Pinheiro Holanda e Maria Mozarina Beserra Almeida* (Departamento de Química Orgânica e Inorgânica, Universidade Federal do Ceará).

Solid coordination compounds formed by interaction between uranyl acetate dihydrate with disulfoxides: 1,2-bis (Methylsulfinyl) ethane, 1,2-bis (ethylsulfinyl)ethane and 1,2-bis (propylsulfinyl)ethane, all in the *meso* form, were investigated. The complexes were characterized by elemental analysis, melting ranges, molar conductance measurements, thermogravimetric data and infrared spectra. In all cases, the uranyl ion is bonded to the sulfoxide moieties via the oxygen atoms.

QI-124 Coordination Compounds Between Disulfoxides and Thorium (IV) Nitrate. *José Tarcísio Vasconcelos Chaves* and *João Aldésio Pinheiro Holanda* (Departamento de Química Orgânica e Inorgânica da Universidade Federal do Ceará).

The synthesis and characterization of complexes between thorium (IV) nitrate tetrahydrate and the *meso* form of the disulfoxides with the formula R-S(O)-CH_2-CH_2-S(O)-R, where R is methyl, ethyl or n-propyl group, are reported. Compounds of general formula $Th(NO_3)_4$. 2L were obtained. The infrared spectra shows the coordination through the oxygen of the sulfoxide in all the complexes. The espectroscopic and conductivity studies indicate the coordinate nature of the nitrate, probably as bidentate ligand.

QI-125 Compounds Formed Between i-Propyldiphenylphosphine Oxide and Rare Earth Perchlorates. *Airton Marques da Silva, Francisca Eliene Sobral* and *Marisleny Pereira Brito* (Departamento de Química Orgânica e Inorgânica da Universidade Federal do Ceará, Campus do Pici, C.P. 12.200).

The compounds were prepared from salts of rare-earth perchlorates and i-Propyldiphenylphosphine oxide. The analytical results indicated the following chemical formulas a) $Ln(CLO_4)_3.3L.xH_2O$ (Ln = La, Nd, Sm, Dy; x = 5 and l; L = i-PDPPO), b) $Yb(CLO_4)_3.4$(i-PDPPO). The vibrational studies at the infrared region showed that the interactions occured through the oxigen atom of the ligand. The molar electrolytic conductance showed that the compounds behave as 1:3 electrolytes in acetonitrile.

QI-126 Characterization of the Coordenates Compounds, Formed Between Some Salts of Lanthanides (III) and the Ligand 1,5-Diphenylcarbazide Through of Conductometric–Titrations, Emission Spectra and NMR^{1}H Spectra. *Edênia Guilherme Neto Brandão* and *Airton Marques da Silva* (Departamento de Química Orgânica e Inorgânica da Universidade Federal do Ceará).

This present research work is concerned with the characterization of the compounds that have the general composition: LnX_3yLzH_2O, where X = NO_3^-, Cl^-, Br^-; y = 3, 4, 5, 6, 8; z = 2, 4, 8; Ln = La, Pr, Nd, Eu, Dy, Ho, L = 1,5-DPCI. The conductometric titration obtained from solution of hydrated nitrates, chlorides and bromides of lanthanides (III) with solution of the 1,5-Diphenylcarbazide confirmed ascents curves behavior for all the compounds. Have been performed the emission spectra that suggest a microsymetry near to C_4v for the nitrate and chloride compounds and a microsymetry near to C_3v for the bromide compound, and have been performed the analyses of the NMR^{1}H spectra confirmed the formation of the addition compounds.

QI-127 Application of Nickel (II) Perchlorate to Purify TMSO. *Viktoria K. Lakatos Osorio* and *Mônica Ferreira* (Instituto de Química, USP - C.P. 20.780 - CEP 01498 - São Paulo, SP).

This paper deals with the purification of a commercial sample of tetramethylenesulfoxide (tmso) contaminated by the corresponding sulfone. The procedure proposed takes advantage of the preferencial coordinating ability of nickel (II) towards tmso. The impure dmso was used to prepare the compound $Ni(tmso)_6(CLO_4)_2$, from a methanol–diethyl ether solution. The solid obtained was dissolved in water and pure tmso was recovered by extraction with CH_2Cl_2, followed by evaporation of the solvent.

QI-128 Dithiocarbamate Complexes of Fe(III), Cu(II) and Ni(II) With Morpholine. *José Maria Barreto de Oliveira* and *Djalma Martins Rosa* (Departamento de Química Orgânica e Inorgânica da Universidade Federal do Ceará, C. P. 12.200 - Brasil).

Dithiocarbamates of Fe(III), Cu(II) and NI(II), with morpholine was prepared by directly addition metal salts with CS_2 and the corresponding amine. The complexes were characterized by elemental analysis, complexiometrie titration through EDTA and infrared absorption spectra.

QI-131 Ternary Complexes in Copper(II)-Histidine-Hydroxamic Acid Systems: Formation Constants Determination. *Eucler B. Paniago* and *Sandra Carvalho* (Dep. Química - ICEX/UFMG).

Formation constants have been determined by potentiometric titrations (glass electrode) at 25°C and I = 0,1 M ($NaCLO_4$) in aqueous solution, for the ternary systems copper(II)-histidine (A)-hydroxamic acid (B), where B refers to aceto-(aha) or glycinehydroxamic acid (gha). Ternary complexes of the type CuAB were detected in these systems and their stability has been compared with those of the corresponding binary complexes: they are more stable than would be expected from statistical considerations only. Other ternary complexes, CuABH and $CuAB(H_{-1})$, were found also in these conditions.

QI-132 Síntese da Cluster $Cp_3Co_2Fe(\mu_3{-}NH)(\mu_3{-}NO)$, Isoeletrônica A $Cp_3Co_3(\mu_3{-}NO)_2$. *Jörn Müller* (Institut für Anorganische und Analytische Chemie Technische Universität Berlin), *Gelson Manzoni de Oliveira* (UFSM - Departamento de Química).

The reaction of $(CpFeNO)_2$ with excess $CpCo(C_2H_4)_2$ in THF gives $Cp_3Co_2Fe(\mu_3\text{-}NH)(\mu_3{-}NO)$ as the main product, which is an isoelectronic congener of $Cp_3Co_3(\mu_3\text{-}NO)_2$. Another isoelectronic species of possible composition $Cp_3CoFe_2(\mu_3\text{-}NH)_2$ is found as a by-product.

ORGANIC CHEMISTRY (QO)

QO-001 Synthetic Methods III – Steric Hindrance in the Efficiency of the Reduction of α,β–Unsaturated Ketones by Hydrogen Transfer. *Maria Luiza Ambros von Holleben, Eduardo Rolim de Oliveira* and *Cláudia Alcaraz Zini* (Instituto de Química, Universidade Federal do Rio Grande do Sul - Porto Alegre, Brasil).

We have compared the development and the results of the hydrogen transfer reduction of 3–penten–2–one, 4–methyl–3–penten–2–one and 3,5,5–trimethylcyclohexen–2–one using limonene as hydrogen donor and Pd/C 10% as catalyst. In all the cases, the reduction was selective at the olefinic bond and we have verified that sterical hindrance effects both the yield of the reduction and the development of the parallel process, the disproportionation of limonene. The results are compatible with the mecanism of co–adsorption of acceptor and donor at the surface of catalyst.

QO-002 Blockage of Limonene 8,9 Double Bond. A New Reagent for Epoxidation. *Maria Luiza Ambros von Holleben, Francine Furtado Nachtigall.* (Instituto de Química – Departamento de Química Orgânica, Universidade Federal do Rio Grande do Sul, Porto Alegre, Brasil); *Joel Jones Jr.* (Instituto de Química, Departamento de Química – Universidade Federal do Rio de Janeiro).

The resulting peracid of the oxidation of anthracene/maleic anhydride Diels–Alder aduct was used as a bulky reagent for the epoxidation of 8,9 double bond of (+) limonene. The epoxidation, carried at room temperature, yielded 1,2–epoxi–limonene and 8,9–epoxi–limonene in a proportion of 1:1, approximately.

QO-003 New Improvement in the Sisstruc Expert System. *Jean Pierre Gastmans* (Faculdade de Engenharia de Guaratinguetá, Dep. de Física e Química – UNESP), *Denise Laura G. Fromanteau* (Instituto de Química de Araraquara, Dep. Q. Orgânica - UNESP) and *João Henrique G. Borges* (Departamento de Química – UFSCar).

In a former version of the SISSTRUC the computer generated the largest substructures as possible. In this version, we introduce a new algorithm which permits to decrease the number of combinations, avoiding combinative explosions.

QO-004 Correction of NMR Assignments by Computer the Cora Expert System (Correction of Assignments). *João Henrique G. Borges* (Dep. de Química – UFSCar), *Mário Cilense Jr.* and *Denise Laura G. Fromanteau* (Instituto de Química de Araraquara, Dep. de Q. Orgânica – UNESP), *Jean Pierre Gastmans* (Faculdade de Engenharia de Guaratinguetá, Dep. Física e Química UNESP).

We present a modified version of the CHECK-WHERE expert system (CORA). It is able to study molecules containing heteroatoms such as N,O,F,P,S,Se,Te etc... In this extended version, the computer is able to treat 39 different bonds and 40 extended nodules. With a complete and correct data set, CORA can help the chemist in pointing out and correcting erroneous assignments.

QO-005 Electronic Interactions Studies of Some ω–(Ethylsulfonyl)–*Para*–Substituted Acetophenones. *Giuseppe Distefano* and *Maurizio Dal Colle* (Dipartimento di Chimica dell'Università, Ferrara, 44100, Italy). *Paulo Roberto Olivato, Élida Bonfada* and *Mirta Gladis Mondino*. (Instituto de Química da Universidade de São Paulo, S.P., C.P. 20.780, Brazil).

This communication reports the IR, ^{13}C NMR and UPS studies of some ω(ethylsulfonyl)–acetophenones Y–$\phi C(O)CH_2SO_2Et$, being Y = electron–attracting, hydrogen and electron–donnating substituents. The υ_{CO} frequency shifts analysis together with the Non Additivity Effect of the α–methylenic carbon suggests in the title compounds occurrence of the $\pi^*_{CO}/\sigma_{C-SO_2}$ and $\pi_{CO}/\sigma^*_{C-SO_2}$ interactions. The comparison between the (I.E.)s of the SO_2 group orbitals for the $\phi C(O)CH_2SO_2Et$ and the corresponding orbitals for the Me_2SO_2 supports the $\pi_{CO}/\sigma^*_{SO_2}$ interactions. The slight stabilization of the uppermost π ring orbitals of $\phi C(O)CH_2SO_2Et$ in relation to the corresponding orbitals for the $\phi C(O)CH_2SEt$ agrees with the $\pi^*_{CO}/\sigma_{C-SO_2}$ interaction. (CNPq)

QO-006 Conformational and Electronic Interactions Studies of Some (Alkylthio)–Substituted Propanones and Their Mono– and Di–Oxidated Derivatives. *Paulo Roberto Olivato* and *Mirta Gladis Mondino* (Instituto de Química da Universidade de São Paulo, C.P. 20.780).

This communication deals with I.R. and ^{13}C NMR studies of (alkylthio)– (I), (alkylsulfinyl)– (II) and (alkylsulfonyl)– (III) propanones $H_2C(SO_nR)C(O)Me$, being R = Me, Et, ^{i}Bu, ^{i}Pr and ^{t}Bu; where n = 0, 1 and 2. The 1st overtone carbonyl stretching data together with the

Molecular Mechanics Calculations indicated for compounds I, II and III the existence of four, six and four conformers, respectively. The analysis of the carbonyl frequency shifts of the most stable *gauche* conformers G_1 for compounds (I–III) in comparison with the Non Additivity Effect of the α–methylenic carbons suggested the occurrence of the $\pi^*_{CO}/\sigma_{C-SO_n}$ and $\pi_{CO}/\sigma^*_{C-SO_n}$ interactions in the ground state of the title compounds. (CNPq).

QO-007 Anodic Oxidation of Ketene–S,S–Acetals. *Denise Curi, Hans Viertler* and *Vera Lúcia Pardini.* (Instituto de Química da Universidade de São Paulo, Caixa Postal 20.780, São Paulo - S.P. Brazil).

The eletrolyses of 1,1–bis(methylthio)–1–pentene (Ia) and 2–methyl–1,1–bis(ethylthio)–1–pentene (Ib) were performed at controlled potential in CH_3CN/H_2O (9:1^V/v)/0.2 M $NaC10_4$ employing a Pt anode, in order to study the influence of the substitution pattern of the carbon–carbon double bond. Oxidation of (Ia) at +1.70V x Ag/AgI afforded 4,5–bis(methylthiocarbonyl)octane as the major product while ketene–S,S–acetal monosulphoxide was predominately formed from (Ib), under similar conditions. Steric effects account for the observed results.

QO-010 Telurocyclofunctionalization of Alkenyl-Substituted 1,3-Dicarbonyl Compounds. *E. B. de Borba, J. V. Comasseto, H. M. C. Ferraz* (Instituto de Química, Universidade de São Paulo) and *C. A. Brandt* (Instituto Butantã - S.P.)

This work describes the use of aryltellurium trihalides as suitable cyclizing reagents for α and γ-alkenyl-substituted β-keto-esters and β-diketones. The reaction takes place via the enolic form of the dicarbonyl compounds, giving the corresponding cyclic teluroethers in excelent yields.

QO-011 α–Methylsulfinyl Benzylic Sulfones – Versatile Intermediates in the Ramberg–Backlund Reaction. *Blanka Wladislaw, Liliana Marzorati* and *Marcio Henrique Zaim.* (Instituto de Química, Universidade de São Paulo, C.P. 20.780 - Brazil).

The Ramberg–Backlund reaction of α–methylthiobenzil isopropil sulfones, not described in the literature, was investigated by Meyers method and under phase transfer conditions using trimethylcetyl ammonium chloride as phase transfer catalyst. In the first case a vinyl sulfoxide was the mainproduct, beside vinyl and dichlorocarbene insertion product. In the second case the vinyl chloride was the only product obtained. The mechanism of this reaction is under investigation.
(FAPESP, CNPq).

QO-012 New Synthesis of Some Ortho–Substituted 1–Deuterio-Benzaldehydes. *Blanka Wladislaw, Liliana Marzorati, Gunter Ebeling.* (Instituto de Química, Universidade de São Paulo, C.P. 20.780, Brazil).

This communication reports the study of deuteration for some, recently synthesized, *ortho*–substituted α–methylthio benzyl sulfones, followed by thermal decomposition of deuterated products, leading to the corresponding benzaldehydes 1–d. The latter compounds were isolated in ca. 80% yields, with isotopical purity of ca. 95%, as determined by RMN–^{1}H and mass spectrometry, and showed to be independent on the electronic character of the substituents. Therefore, this procedure may be considered as an alternative method of preparation of the *ortho*–substituted 1–deuteriobenzaldehydes.

QO-013 Isolation and Characterization of an Acyl–Thiosemicarbazide as an Intermediate in the Synthesis of Mesoionic Compounds. *M. Aparecida M. Maciel, A. Echevarria* and *Victor M. Rumjanek* (Departamento de Química – Universidade Federal Rural do Rio de Janeiro).

In the synthesis of some acyl–triazolic mesoionic compounds a mechanism was proposed which involves an acyl–thiosemicarbazide. The addition of base leads to the formatin of this intermediate which was characterized by I.R. spectroscopy. Treatment of this compound with dilute acid afforded the corresponding mesoionic species.

QO-014 Synthesis of the 4–Phenyl(3–,4–Substituted)4–Metoxi–1,1,1–Trichloromethyl–3–Buten–2–Ones. *Geonir M. Siqueira, Alex Claro Flores, Marcos A. P. Martins* (Departamento de Química, UFSM, RS).

–Alcoxivinyltrihalomethylketones are important syntons to obtain heterocyclic compound by the condensation with dinucleofiles. This work repports the synthesis of –alcoxivinyl–2–(3–,4–phenyl substituted) trichloromethylketones from the respective 3–,4– substituted acetophenones.

QO-015 Photochemistry of α–Chloro–2',4'–Dimethylacetophenone. *J. C. Netto Ferreira* (Departamento de Química, Universidade Federal Rural do Rio de Janeiro) and *J. C. Scaiano* (Division of Chemistry, National Research Council of Canada).

The transients involved in the photorearrangement of α–chloro–2',4'–dimethylacetophenone (I) were investigated by laser flash photolysis technique. Laser excitation of I in methanol leads to the formation of a short (λ_{max} = 340 nm, τ = 270 ns), **II**, and a long lived transient (λ_{max} = 380nm, τ 10 s), **III**, Quenching studies lead us to assing the short lived species to the triplet stat of the ketone I, whereas **III** was assigned to only one (the E-enol) of the photoenols derived from I. Two-laser, two-color experiments showed that enol **III** is readily photobleached by excitation of this transient at 420 nm., revealing a reasonable decrease in product formation under these conditions. These results are fully consistent with a mechanism involving two interconverting enols, only one of which (**IV**, the Z–enol) being responsible for product formation.

QO-016 The synthesis of Bottrospicatol. *Marco A. Ceschi* and *Timothy John Brocksom* (Departamento de Química da UFSCar, Caixa Postal 676, 13560 – São Carlos – SP, Brazil).

The bicyclic monoterpene (+)–bottrospicatol is an active inhibitor of lettuce seed germination, and is produced by *Streptomyces bottropensis* from (–)–*cis*–carveol. The synthesis of (+)–bottrospicatol has been effected in three steps from (–)–carvone: epoxidation with MCPBA,$NaBH_4$ reduction and H^+ catalysed cyclization.

QO-017 The synthesis of Mint–Lactone and Iso–Mint–Lactone Via Menthofuran. *Newton Antonio Varanda, Ursula Brocksom, Timothy John Brocksom* (Departamento de Química da UFSCar, Caixa Postal 676, 13560 – São Carlos (SP)).

Mint lactone and iso–mint–lactone have been isolated as minor constituents of the essential oil of *Mentha arvensis*, and contain an endocyclic α–methylene–γ–butyrolactone function. We have effected their synthesis from (–)–isopulegone by allylic chlorination and cyclization to (+)–menthofuran, followed by MCPBA oxidation to a mixture of the diastereomeric mint lactones.

QO-018 Reaction of Addition and Cooxidation of Tiophenol and Non–Conjugated Olefins. *Juanita Freer, Monica P. Peña, Cecilia Fuentealba, Jaime Baeza* (Departamento de Química, Facultad de Ciencias, Universidad de Concepción, Concepción (CHILE)).

A successful cooxidation reaction was carried out between cyclopentene and tiophenol when the mixture of neat reagents was allowed to react. The reaction was carried out under conditions either thermally or photochemically. In both cases, the reaction gave the adduct ciclopentilphenylsulfide and cooxidation products (4 compounds). The photochemical cooxidation reaction using petroleum benzine–ethyl acetate(1:1) as solvent gave the addition adduct and 2 of the cooxidation products (the hydroxisulfides). The reactions betweem α–pinene and tiophenol were also studied.

QO-020 Utilização do Ácido 1,2,3,4,5,6–*O*–Triisopropil Glucônico na Síntese de Derivados da Glicose Substituídos no Carbono Anomérico E D-Gluconamidas. *Luciene Ferreira, Takeko Nakamura* and *Vitor Francisco Ferreira* (Unidade de Programas de Sucroquímica, Divisão de Produtos Naturais, Instituto Nacional de Tecnologia).

Tetra–*O*–benzyl–δ–gluconolactone is being used very often in the syntheses of alkyl derivatives of D–glucose and in the preparation of D–gluconamides. However, gluconic acid triacetone may be used in the same manner but with advantages since it's easier to remove its protection group. In this vein, we have examined the incorporation of methyl and butyl group into carbon 1 of D–glucose and prepared several D–gluconamides. The latter compounds are being used in the syntheses of (+)–deoxynojirimycin and (+)–castanos permine.

QO-021 Anodic Oxidation of Benzyl-Phenyl-Ethers. *Gerson P. Crisci, Vera L. Pardini, Hans Viertler.* (Instituto de Química da Universidade de São Paulo, C.P. 20.780, São Paulo, S.P., Brazil).

Anodic oxidation of benzyl–(4–methyl–phenyl)ether (I) was carried out in MeOH/MeONa at constant current at carbon or platinum electrodes. The experiments with platinum anode afforded 3–benzyloxy–3,6–dimethoxy–6–methyl–1,4–cyclohexadiene as a result of a nuclear methoxylation of (I). This acetal when hydrolysed with saturated aqueous sodium hydrogen sulphite afforded 4–methyl–4–methoxy–cyclohexa–2,5–dienone and benzyl alcohol. When the electrolysis was performed with carbon anode a side chain methoxylation was observed and the dimethyl acetal of 4–benzyloxy–benzaldehyde was isolated. (CNPq, FINEP, PADCT).

QO-022 An Empirical Method for the Evaluation of Electron Affinities. *Ivan Pérsio de Arruda Campos, Daisy de Brito Rezende, Maria Eunice Ribeiro Marcondes* and *Vicente Guilherme Toscano* (Instituto de Química, Universidade de São Paulo, C.P. 20780, Brazil).

An empirical correlation among electron affinities (EA), ionization potentials (IP) and the lowest excited state singlet energy (ΔE^1_{0-0}) was obtained through a multiple regression analysis performed on the literature data of model monocyclic substituted aromatic compounds. The EA of a series of arylalkylthioethers was evaluated using this correlation. The resulting data are whithin the expected range, when compared to those of the model compounds.

QO-024 Synthesis of Trail Pheromone of *Leptogenys Diminuta*. *Rosa T. S. Frighetto* (CNPDA/EMBRAPA (Jaguariúna), *Nelson Frighetto* (CPQBA/UNICAMP (Campinas)), *Athula B. Attygalle* and *Hans J. Bestmann* (Institut für Organische Chemie (Erlangen/FRG).

Pheromones are volatile active chemical messengers which are secreted by a member of an animal species and elicit a definite behaviour in other members of the same species. The 4–Methyl–heptan–3–ol was firstly identified as aggregation pheromone of *Scolytus multistriatus* and *S. scolytus*. This same substance was found as ant trail pheromone of *Leptogenys diminuta*. We synthesised the stereoisomers by Wittig condensation between 2–Oxo–3–methylpentylidentriphenyl–phosphorane and paraformaldehyde. The biological properties were compared and the natural trail pheromone confirmed. (CNPq (RTSF) and Embrapa (NF)).

QO-025 Use of t–Butylperoxide Radical in the Preparation of γ–Lactones. *Vanilde de Faria Geronimo, Faruk Nome* and *Maria da Graça Nascimento* (Departamento de Química – UFSC – Florianópolis, SC).

In this work, γ–lactones have been prepared from aliphatic alcohols and acrilic acid via radical type reaction. More specifically, the γ–nonelactone and γ–phenyl–γ–butyrolactones were prepared by the reaction of hexyl and benzyl alcohols with acrilic acid and/or methyl acrilate at 140°C, in the presence of t–butylperoxide radical. The products were analyzed by UV, IR, and ^1H–NMR spectroscopy besides GC/MS spectra.

QO-026 Alkylation of Benzene from Alcohols Catalysed by Clay Dopped With Fe^{3+}. *Madelon B. Fagundes, Nilo Zanatta and Hugo T. S. Braibante* (Departamento de Química, Universidade Federal de Santa Maria, UFSM, RS).

On a recent communication, a novel procedure for the alkylation of aromatic molecules with alcohols catalysed by clay (K–10), was repported (XXIX Congresso Brasileiro de Química, ABQ, São Paulo, SP, outubro/89, C.3.12). This work repports a second part of this study where the alkylation were carried out by K–10 dopped with Fe^{3+} (K-10-FeIII). It was observed that the second catalyst is more efficient that the former one, in this kind of alkylation.

QO-027 Effect of Hydroxide Ion and Salt Concentration in Elimination Reactions Catalyzed by Hexadecyltrimethylammonium Hydroxide. *Ligia Catarina Mello Ferreira, Faruk Nome* and *César Zucco* (Departamento de Química, Universidade Federal de Santa Catarina, 88049 – Florianópolis, SC., Brazil).

The dehydrochlorination of 1,1–diphenyl–2,2,2–trichloroethane (DDT_H) was studied as a function of the title surfactant in the presence and absence of added KOH. The experimental data can be fitted under a wide variety of experimental conditions with only a single value for the rate constant in the micellar phase ($k_{2m} = 6.65x10^{-4}\ M^{-1}s^{-1}$) providing that the degree of dissociation of the surfactant is considered as a variable and that a term describing surfactant invasion in the micellar Phase is included.

QO-028 Single–Electron Transfer in Reactions of 2,4– and 2,6–Dinitrochlrobenzenes With Hydroxide Ion. *Radu Bacaloglu, Clifford A. Bunton* (Department of Chemistry, University of California, Santa Bárbara) and *César Zucco* (Departamento of Chemistry, UFSC, 88049 – Florianópolis, SC.)

Kinetic and H NMR measurements are reported for the reactions of 2,4– and 2,6–dinitrochlorobenzenes with HO^- in DMSO: Water. The kinetic measurements provide evidence for the existence of two Meisenheimer complexes for the 2,4 derivative but only one for the 2,6. Exchange of some aromatic protons and line broadening of the NMR proton signals are taken as evidence for the existence of a charge–transfer complex between a radical anion and HO^- or its anion in the reaction pathway. Rate and equilibrium constants are reported.

QO-029 RMN ^{13}C of Epoxide Derivatives in the Assignment of the Stereochemistry of Methyl Ent–Pimara–8,15–Dien–19–Oate. *Frederico G. Cruz* and *Nídia F. Roque* (IQ-USP).

The stereochemistry of methyl ent–pimara–8,15–dien–19–oate in carbons 5,10 and 13 could be deduced by the epoxide derivatives. The γ–effects of protection in C–5 (α–epoxide) and in C–20 (β–epoxide) were decisive in the configuration stablishment of the natural product. The desprotection in the C–17 methyl group of the α–epoxide caused by the change of the conformation of the ring C agree with the configuration proposed.

QO-030 Stereocontroled Inversion of Hydroboration Selectivity of Methyl Ent–Pimara–7,15–Dien–19–Oate. *Frederico G. Cruz; Nídia F. Roque; Helena M. C. Ferraz* (Instituto de Químicam, USP).

The obtention of methyl ent–pimara–8β, 16–diol–19–oate by hydroboration of methyl ent–pimara–7,15–dien–19–oate is explained by the borane intermediary that could only be formed between the carbons 16 and 8. The analogue diol was not observed in the hydroboration of methyl ent–pimara–8,15–dien–19–oate.

QO-031 Acid Hydrolysis of Ketals in Surfactant Solution of Sodium Monodecylphosphate (NaDP). *Maria Rosania Kretzer Walter, Faruk Nome* and *Dino Zanette* (Departamento de Química, Universidade Federal de Santa Catarina, Florianópolis, SC., Brazil).

The acid hydrolysis of 2–(p–methoxyphenyl)–1,3–dioxolane (p–MFD) and of 2–(2,4–dimethoxyphenyl)–2–ethyl–1,3–dioxolane (2,4–MFED) has been studied as a function of pH and sodium monodecylphosphate (NaDP) concentration. The reaction is effectively catalyzed by NaDP, the micellar rate constant being 4 to 6 times greater than the rate constant in water, probably reflecting the lower polarity of the micellar phase.

QO-032 Acid Hydrolysis of 2–(p–Methoxyphenyl)–1,3–dioxolane (p–MFD) in the Presence of Sodium Dodecyl Sulfate / Polyethyleneglycol (SDS-PEG 20,000) Mixed Micelles. *Angelo Ruzza, Faruk Nome* and *Dino Zanette* (Departamento de Química, Universidade Federal de Santa Catarina, 88049 – Florianópoilis, SC., Brazil).

Complexes of SDS micelles with PEG–20,000 have been characteryzed by surface tension measurements, showing two marked inflection points as a function of surfactant concentration. The surfactant concentration vs. first–order rate constant profile for the hydrolysis of p–MFD, in the presence of PEG– 20,000, shows clearly that catalysis becomes important only after the first inflection point. However, the catalytic behaviour of the agregate is not sensible to the structural change associated with the second inflection point. The pseudophase ion exchange model was used to explain the experimental data, and the agregate formed between SDS and PEG was found to be a more effective catalist.

QO-034 Synthesis of Pyranonaphthoquinone. *Maria do Carmo Maltez Miraglia, Alaíde Braga de Oliveira, Antônio Augusto Lins Mesquita* and *Délio Soares Raslan* (Instituto de Ciências Exatas, Departamento de Química, Universidade Federal de Minas Gerais).

7– or 8–methoxy–naphtho–[2,3–b]pyran–5,10–quinone were synthesized by alkylation–cyclisation of 2– or 3–hydroxy–6–methoxy–naphthoquinone in a single step.

QO-035 Synthesis of an Isostere of Loxoprofen Using Safrole from Sassafraz Oil. *Maria Aparecida César* (Instituto de Química, Dep. Química Orgânica, UFRJ) and *Eliezer J. Barreiro* (Faculdade de Farmácia, Dep. Tecnologia Farmacêutica, UFRJ).

The synthesis of a new compound structurally related to Loxoprofen, NSAI agent recently introduced in therapeutic (Loxonin, Sankyo, 1986), was developed using, as starting material, safrole, the principal allylbenzene component of the Sassafraz oil. The structure of this new indenyl analogue was designed using bioisosterism principles. (CNPq, FAPERJ, CEPG-UFRJ).

QO-036 Studies About the Utilization of 2–Methyl–5,6–Methylenedioxiindanone in Synthesis. *Lúcio M. Cabral* and *Eliezer J. Barreiro* (Departamento de Tecnologia Farmacêutica, Faculdade de Farmácia, UFRJ).

During a research programme aiming the utilization of abundant natural products in synthesis we became interested in studying the chemical reactivity of 2–methyl–5,6–methylene–dioxiindanone, obtained from natural safrole isolated from Sassafraz oil, by improving the Witiak method [Witiak, D.T. et al.; J.Org.Chem. 1974, 39, 1242]. The ring expansion Beckmann method applied to this compound furnished a quinolone, the corresponding nitrile could be prepared from the indanone by using Schmidt rearrangement, these both compounds are very useful to synthesis of nitrogen bioactive compounds. (CNPq/CEPG–UFRJ).

QO-037 Synthesis of a new Pyrazole Derivative With Potential Inhibitory Properties of Thromboxane–Synthase. *J. C. G. Passos* (Inst. Quím., UFRJ), *A. C. C. Freitas, E. J. Barreiro* and *T. M. Monteiro* (Dept. Tecnol. Farm., Fac. Farm., UFRJ).

On the way to selective inhibitors of the enzyme thromboxane synthase of the pyrazolic series, using the bioisosteric strategy, 5(N–phenyl–4–pyrazolyl)–4–oxapentanoic acid was defined as a desirable goal. Reaction of the methyl ester of N–phenyl–4–pyrazolylcarboxylic acid with LAH afforded the corresponding alcohol in 65% yield. O–Alkylation furnished the O–allyl derivative which was oxidized to the desired pyrazolic acid *via* a hydroboration–oxidation sequence in 40% overall yield. This compound is a bioisostere of 5–(N–phenyl–4–pyrazolyl)pentanoic acid, a potent antiaggregation compound(unpublished results).

QO-038 Direct and Indirect Electrochemical Oxidation of Vinyl Sulfides. *João F. Ganzerli* (Cia. Brasileira de Petróleo Ipiranga – Divisão de Produtos Químicos), *Vera L. Pardini* and *Hans Viertler* (Instituto de Química – USP, São Paulo – S.P.).

The controlled potential electrolyses of 1–methylthiocyclohexene (I), 1–phenylthiocyclohexene (II) and their 2–methyl– and 4–t–butyl–derivatives were studied in aqueous MeCN using a Pt anode. Tris–(4–bromophenyl–)amine was used as mediator in the indirect process. The α–sulfenilated cyclohexanones were the main products with compounds (II) whereas compounds (I) were hydrolysed to the corresponding ketones or yielded vinyl sulfoxides (CNPq, FINEP, PADCT).

QO-040 Apparent Partition Coefficient Determination of *N,N*–[(Dimethylamino)Ethyl]Benzoate Hydrochloride. *Y. Miyazaki, A. S. Stachissini* and *A. T. do Amaral* (Chemistry Dept., Chemistry Institute, USP – C.P. 20780, CEP 01498, São Paulo, Brazil).

In order to apply the classical Q.S.A.R. approach P_{app} was determined experimentally in octanol/water system at 25°C, $\mu = 0.10$ mol/l from pH 2.0 to 8.9. The plot of log P_{app} v s. pH showed a profile that complies well with the theoretical curve obtained by calculation using an equation which takes into account the pK_a (8.3), P_u and P_i for the compound.

QO-041 Determination of the Lifetime of Upper Triplet States of Anthracenes: Substituent Effect. *Cornélia Bohne*[1], *Steve Kennedy*[1], *Ron Boch*[2], *Wilem Siebrand*[1] and *J. C. Scaiano*[1,2]. [1](Division of Chemistry, National Research Council of Canada: Ottawa, Ontario, Canada K1A OR6) and [2](Department of Chemistry, University of Ottawa, Ottawa, Ontario, Canada K1N 6N5).

Intersystem crossing in anthracenes is known to proceed through the T_2 state, but literature values show discrespancies of an order of magnitude in the T_2 lifetimes. The efficiency of T_1 formation was measured after laser excitation. This efficiency is decreased in the presence of octadiene, a quencher with energy lower than T_2 but higher than T_1. Quenching efficiencies were corrected for singlet quenching. T_2 lifetimes of antracenes are not altered by substitutions at the 9–position, but they are significantly shortened for 1– or 2–substituents.

QO-042 Exploratory Studies of the Photochemistry of N–Hydroxypyridine–2–Thione Esters. Generation of Excited Radicals by Laser Flash Photolysis and in a Conventional Fluorescence Spectrometer. *Cornélia Bohne*[1], *Ron Boch*[2] and *J. C. Scaiano*[1,2]. ([1]Division of Chemistry, National Research Council of Canada, Ottawa, Ontario, Canada K1A OR6 and 2, Department of Chemistry, University of Ottawa, Ottawa, Ontario, Canada K1N 6N5).

The photodecomposition of 2–hydroxypyridine–2–thione esters derived from the diphenylacetic (I), p–methoxybenzoic (II) and the 2,2–dimethylvaleric (III) acids was examined using laser flash photolysis techniques. In all cases the formation of the thiylradical is a very rapid (10ns) process. In the case of II, where decarboxylation is slow, the p–methoxybenzoyloxyl radical can be readily detected. In contrast, in the case of I loss of CO_2 is fast leading to the formation of diphenylmethyl radicals that are reexcited by the laser pulse to its fluorescent excited state. The effeciency of cleavage and excitation and the high fluorescence yield combine to make the detection of the excited radical possible even with a commercial spectrofluorimeter.

QO-043 Preparation of N–Nitroso–N–Methyl–P–Toluenesulfonamide. *A. A. Craveiro. I. G. P. Vieira* and *A. C. Craveiro* (Fundação Centro de Pesquisa e Desenvolvimento da Indústria Química do Estado do Ceará – Laboratório de Produtos Naturais – Universidade Federal do Ceará).

The preparation of p–Tolyl Sulfonylmethylnitrosamide starting from tosyl chloride was done in continuation of the UMBRAL I project (Utilization of Brazilian Raw Material). Tosyl chloride, was prepared from p–Toluenosulfonic acid, and was allowed to react to give N–Methyl–N–Nitroso p–Toluenosulfonamide, in 67% yield. (PADCT/FINEP/CNPq/RHAE).

QO-044 Synthesis of a Analogous of Capsaicin from the Eucaliptus Essencial Oil. *Afrânio Aragão Craveiro* and *Danilo Caldas de Queiróz* (Laboratório de Produtos Naturais – Departamento de Química Orgânica e Inorgânica – Universidade Federal do Ceará).

A analogous compound of capsaicin, N–Vanillil–3,7–dimethyl–n–octanamide, has been synthesized from citronellal and vanillilamine. The hidrogenation of citronellal gives 3,7–Dimethyl–octanal, which is oxidized by $K_2Cr_2O_7/H_2SO_4$ to the respective acid. The acid is condensed with vanillilamine which is produced from vanillin via vanillil–oxime. (UFC/CNPq).

QO-046 An Improved Method to Calculate First Order Rate Constants When Infinity Readings Are Not Possible. *Eduardo Humeres* (Departamento de Química, Universidade Federal de Santa Catarina, Florianópolis, SC, Brasil) and *Jairo Quijano* (Departamento de Química, Universidad de Antioquia, Medellín, Colombia).

There are a number of situations where the calculation of the infinity reading of a first order reaction is important. A typical example is a consecutive reaction that competes with the main reaction. In general least squares regression analysis is applied, but this method does not provide necessarily a correct value. Guggenheim method requires a constant time increment equal at least to one half life of the reaction. The proposed method considers three consecutive readings with a time increment that can as low as 0.2 half life units. Calculation with several triads of readings would give an insight a deviation of a first order kinetics.

QO-047 Mechanism of Formation of Dithiocarbamates. *Eduardo Humeres* and *Olímpio Scalco* (Departamento de Química, Universidade Federal de Santa Catarina, Florianópolis, SC, Brasil).

The reaction of amines and CS_2 at 25°C was studied, changing the pKa in the range 1 to 11. The second order rate constant with respect to the free amine showed a nonlinear Bronsted plot, that is an evidence for a change of structure of the transition state. These results are consistent with the proposed mechanism of the acid decomposition of dithiocarbamates (E. Humeres *et al.*, Atualidades de Físico-Química Orgânica, *1983*, 204). The rate determining step is the formation of *1* for weakly basic amines, and the proton transfer from *1*, for strongly basic amines.

QO-049 Generation of Reactive Intermediates During Autoxidation of Biologically Important β–Diketones and β–Ketoesters. *M. Christoff, L. E. Soares Neto, G. P. Crisci,* and *E. J. H. Bechara* (Instituto de Química, Universidade de São Paulo, São Paulo, Brazil).

Easily enolisable endogenous carbonyl compounds such as α–aminoketones, α–hydroxyketones and β–diketones undergo aerobic oxidation, catalyzed by iron complexes, leading to deleterious free radicals, peroxides and excited species. In metabolic disorders linked to Ile and Met, large amounts of 2–methyl–3–oxybutyrate and methylmalonate, respectively, are excreted. This work describes the mechanism of aerobic oxidation of 3–methylacetoacetone (MAA) and ethyl 2–methylacetoacetate as model substrates, in the presence of hemoglobin, myoglobin and horseradish peroxidase. Triplet biacetyl is shown to be the main product from MAA and ESR spin trapping studies demonstrates the intermediacy of carbon–centered radicals. These reactive species may be implicated in the physiopathology of the metabolic defects mentioned above.

QO-051 Mechanistic Considerations on the Reduction of ω–Haloacetophenones by "S. cerevisiae". *Paulo José Samenho Moran, José Augusto R. Rodrigues, Márcia de Carvalho* and *Marina Toshiko Okamoto* (Instituto de Química, Universidade Estadual de Campinas).

The fermenting baker's yeast reduction of ω–haloacetophenones without sucrose addition gave (–)–(R)–halohydrins for the ω–fluoro–, ω–chloro– and ω–bromoacetophenone. For ω–iodoacetophenone reduction the products were acetophenone and (–)–(s)–1–phenylethanol. Based on the reduction products a electron transfer mechanism is proposed for the ω–iodoacetophenone reduction while a hydride transfer mechanism is proposed for the other ω–haloacetophenones reductions. However, the addition of m–dinitrobenzene in the experiments with ω–iodoacetophenone did not inhibit the production of acetophenone.

QO-052 Regioselectivity in the Reactions of Silyl-Cuprate With Tertiary Allylic Acetates. *Décio Marchi Jr., Marco Aurélio M. Mouta* and *Amauri F. do Patrocínio* (Instituto de Química da Universidade Estadual de Campinas).

The dimethylphenylsilyl–cuprate reagent reacts with a variety of open chain tertiary allylic acetates giving allylsilanes exclusively by allylic substitution. Where are the possibility of formation of geometric isomers of allylsilanes the E isomer predominates but there was a case where just the Z isomer was obtained.

QO-053 Reductive Reaction of Methanesulfonic Ester With NaI/Zn° in DMF. *P. M. Imamura* (Instituto de Química da Universidade Estadual de Campinas – S.P.) and *A. R. Araújo* (Departamento de Química – FUE de Maringá – PR).

Deoxygenation reaction is one of the important tools for the synthetic chemists and we are particulary interested in such reaction applied to the system as *1* and *2* to obtain the corresponding deoxy compounds. Submitting the methanesulfonic esters of *1* and *2* according to the Tatsuno's reduction condition *(Tetrahedron Lett.*, 1976, *37*,3325) i.e, NaI/Zn° in DMF, we obtained ring contracted rearrangement products in excellent yields. On the other hand, sulfonic esters of hydrogenated compound of *2* and lanosterol (neopentylic alcohols) gave us a product of elimination and for homoallylic alcohol like isocopalol, a conjugated diene is obtained in excellent yield. [$\Delta^{1(9)}$ – 8,8–dimethyl-7-hydroxy–octaline (1) and $\Delta^{1(9)}$ – 8,8,10–trimethyl–7–hidroxy–octaline (2)].

QO-054 Chiral Homoketals I. Enantiomerically pure α–bromo–ketones. *Lourivaldo da S. Santos* (Depº de Química – UFPA – Belém – PA) and *Lauro E. S. Barata* (Instituto de Química – UNICAMP – Campinas – SP).

Two enantiomeric α–bromo–ketones, (S)– and (R)–α–bromo–3,4–dimethoxy–propiophenone, have been synthesized in the optically pure state by use of (+) and (–)–dimethyltartrate as chiral auxiliar. These are importants intermediates to the enantioselective synthesis of biologically actives neolignans.

QO-055 Biomarkers. *Lucia Rebouças* and *Anita Jocelyne Marsaioli* (Instituto de Química, UNICAMP CP 6154, 13081 Campinas, SP).

Motivated by the discovery of acidic petroleum in Campos Basin, we began a program envolving the analysis of naphthenic acids. We have soon realized that one of our major problems was the lack of authentic samples to help our GC/MS analysis. We have now some 20 authentic samples from various sources but we particularly want to report the synthesis of the (1R, 2S, 7S, 8R, 11R)–carbomethoxy–3,3,11–trimethyltricyclo(6,3,0,$0^{2,4}$) undecane. This trycyclic carbomethoxy terpene was visualized as a useful sample to test the maturation processes due to the presence of the cyclopropane ring.

QO-056 Diastereoselection in the Addition of Silylenolethers to Activated Imines. *R. A. Pilli* and *D. Russowsky* (Instituto de Química, UNICAMP, 13081, Campinas, SP).

The simple diastereoselection in the addition of silylenolethers to activated imines is described. Trimethylsilyl triflate ($TMSOT_F$), dicyclopentylboron triflate, Me_2AlCl and $TiCl_4$ were employed and *syn*–β–amino–ketones were obtained as the major isomer (d.e. > 70%) when Me_2AlCl and $TiCl_4$ were employed in catalytic and stoichiometric amounts in CH_2Cl_2 at –78°C.

QO-057 1,3–Asymmetric Induction in the Reduction of Acyclic N–Alkyl–β–Aryl–β–Aminoketones. *R. A. Pilli* and *L. C. Dias* (Instituto de Química, UNICAMP, 13081, Campinas, SP).

The stereoselective reduction of acyclic N–aryl–β–aryl–β–aminoketones with $LiBH(C_2H_5)_3$ and $Zn(BH_4)_2$ to afford the corresponding *anti*– and *syn*–γ–aminoalcohols, respectively, has been reported (R.A. Pilli, D. Russowsky and L.C. Dias, *J. Chem. Soc. Perkin Commun.*, in press). Here we describe the reduction of acyclic N–alkyl–β–aryl–β–aminoketones with $LiBH(C_2H_5)_3$ and $Zn(BH_4)_2$: in contrast to the N–aryl series, secondary and tertiary N–alkyl–β–aminoketones afforded mixture of γ–aminoalcohols (d.e.: 0–60%) with $LiBH(C_2H_5)_3$ while $Zn(BH_4)_2$ afforded good (d.e. > 80%) to moderate (d.e. 42–66%) excess of *syn*–γ–aminoalcohols with tertiary and secondary alkylamino substituents, respectively.

QO-058 Stereoselective Synthesis of (*3SR, 4SR*) – 4 – Methyl – Heptan – 3 – Ol: the Aggregation Pheromone of *Scolytus Multistriatus* Marsham. *R. A. Pilli* and *M. M. Murta* (Instituto de Química, UNICAMP, 13081 – Campinas, SP).

The stereosective total synthesis of (*3SR, 4SR*) – 4 – methyl – heptan – 3 – ol, the aggregation pheromone of the elm bark beetle *Scolytus Multistriatus* Marsham, is described featuring a stereoselective aldol addition of 2 – methyl – 2 – trimethylsilyloxy – 3 – pentanone to propionaldehyde and a Cu (II) catalysed coupling reaction.

QO-059 Monoterpene Synthon of Cariocal. *Lídia Nascimento* and *Anita J. Marsaioli* (Instituto de Química, UNICAMP CP 6154, 13081 Campinas, SP).

Our retrosynthetic analysis of cariocal, a seco–abietane diterpene, led to synthons A (containing the terpene moiety) and B (containing the aromatic portion). We were first interested in developing a synthetic route to obtain A (1–carbomethoxy–2–caboxy–6,6dimethyl–lcyclohexene) and compounds C (1,2–dimethylene–6,6–dimethylcyclohexane), D (1–carbomethoxy–2–oxirane–6,6–dimethylcyclohexane) and E (1–carbomethoxy–2–cyano–2–hydroxy–6,6–dimethylcyclohexane) were visualized as intermediates but unfortunately the reactions were not reproducible and in most cases the final steps presented low yields. We are working on new intermediates.

QO-060 Simultaneous Ocurrence of π–Methane and Di–π–Methane Rearrangements. *A. B. B. Ferreira* and *F. A. da Silva* (Depto. de Química, Universidade Federal Rural do Rio de Janeiro). partamento de Química, Universidade Federal Rural do Rio de Janeiro).

The simultaneous ocurrence of $2\pi + 2\sigma$ (–methane) and $2\pi + 2\sigma + + 2\pi$ (di–π–methane) rearrangements on 1,1–dicyano–3–phenyl–but–1–ene allows direct comparison of the rates of photochemical reaction by these two channels. The second channel is 4.8 times more efficient than the first. This is to be compared with Zimmerman's results, comparing the reactivity of tetraphenyl–diene and it's mono–unsaturated analogue, when the reactivity ratio is 6×10^4 (*J. Amer. Chem. Soc.*, 1974, *96, 5143).*

QO-061 Mechanism and Catalysis in the Hydrolysis of N–p–n'–Phenylsuphamoylphenylsuccinimide. *A. Xavier, R. Correia, R. J. Nunes, R. A. Yunes* (Depto. de Química, Universidade Federal de Santa Catarina, Florianópolis, SC, 88049).

The imido ring opening reaction of N–p–N'–phenyl–sulphamoylphenylsuccinimide was studied in different buffer solutions at 25°C and ionic strength = 1. The beta Bronsted value = 0.78 and the point corresponding to the hydroxyde ion lies on the Bronsted. This fact strongly suggests that the line hydroxide ion is acting as a general base and indicates that the reaction mechanism is general base catalysed.

QO-062 Two Reaction Rates in the Condensation of Chitosan with Salycylaldehyde. *L. H. Callegaro, M. Laranjeira, R. J. Nunes, R. A. Yunes* (Deptº de Química, UFSC, Florianópolis, SDC, 88049).

The reaction of chitosan with salycylaldehyde in methanol/water (80/20v/v) and in pure water in the presence of borax showed two distinct rates with the second greater than the first. A possible explanation for this behaviour is that a change in the acessibility of the amino groups on the chitosan occurs during the course of the reaction due to a gel formation. This causes coil expansion which has the effect of making the remaining amine groups more accessible to salycylaldehyde. This is an evidence that the mentioned behaviour is not related to a solvent effect as previously reported.

QO-063 Special Behaviour of Phenylhydroxylamine as Nucleophile in its Reaction with Benzaldehydes. *Rosane M. Budal, Inês M. C. Brighente, Rosendo A. Yunes.* (Depto. de Química, UFSC, 88049, Florianópolis - SC).

The reaction between benzaldehydes and nitrogen bases shows as rate determining step in acidic pH, the attack of the nucleophile on the carbonyl group. There is a linear correlation between the catalytic hidronium rate constant and the pKa of the nitrogen bases, however, the experimental value of the catalytic hidronium rate constant for phenylhydroxylamine is 3.7 times greater than the obtained from the correlation line and have been demonstrated that in this conditions, the rate determining step of the reaction of phenylhydroxylamine and benzaldehydes is the dehydration of an addition intermediate. This fact indicate a special behaviour of phenylhydroxylamine as nucleophile.

QO-064 Photoredox Reactions in Alkylammonium Dodecyl Sulfate Micelles. *J. B. S. Bonilha; P. M. Nassar; D. M. A. Ferraz; A. C. Tedesco* (Departamento de Química da Faculdade de Filosofia, Ciências e Letras de Ribeirão Preto – USP).

The aggregation number of hexamethylethylammonium dodecyl sulfate $[(SD)_2HED]$ formed in water solutions was determined from fluorescence quenching experiments, employing ruthenium tris(bipyridyl) chloride as donor and 9–methyl anthracene as quencher (N. J. Turro, and A. Yekta, J. Amer. Chem. Soc. *1978*, 100, 5951). It was found that $(SD)_2HED$ agregates comprise 124 monomers when formed on water. As shown in previous studies, the quenching of the luminescent anionic ruthenium complex RuL_3^{4-}, by organic cations such as N,N'–dibenzyl–4,4'–bipyridinium (+2) (MV^{2+}) and N–methyl–4–cyanopyridinium (CMP^+) (J. B. S. Bonilha; R. M. Z. Georgetto; A. C. Tedesco; L. Miola; D. G. J. Whitten; Phys. Chem. *1989*, 93,367). In the present study we focus on the behaviour of $(SD)_2HED$ micelles in the presence of the cations Na^+ and MV^{2+}. The quenching of extramicellar probe luminescence has been developed to provide a sensitive indication of the critical micelle concentration (CMC) values for the several detergent under a variety of conditions.

QO-065 Photohydrolysis of 2–Halo–5–Nitroanisole: *A. C. Tedesco, J. C. Carreiro, J. B. S. Bonilha* (Departamento de Química – Faculdade de Filosofia, Ciências e Letras de Ribeirão Preto – USP).

Quantum yields have been determined for the photohydrolysisof 2–chloro–, 2–bromo–and 5–nitroanisole in alkaline aqueous or micellar N–hexadecyl–N,N,N–trimethylammonium chloride (CTAC1). Hydroxide ion reacts with 2–halo–5–nitroanisole in its lowest triplet state in both aqueous and micellar soluton with formatin of 2–halo 5–nitrophenolate íon. The quantum yields increased in the presence of micellar CTAC1 (0–80mM), reaching a plateau at high CTAC1 concentration (50–80mM). The apparent catalysis by cationic surfactant is reduced by added NaBr. The results suggest that the increase in the quantum yield in the presence of CTAC1 micelles reflects reaction of the triplet state with OH^- in the aqueous phase to give a long–lived σ complex which preferencially decomposes to product in the micellar phase.

QO-067 Synthesis and Spectroscopic Properties of New Substituted Triaril–Amidines. *Lúcia Helena S. Santos, Aurea Echevarria* (Dept. Química, Universidade Federal Rural do Rio de Janeiro) and *Joseph Miller* (LTF, Universidade Federal da Paraíba).

The amidines are nucleophilic intermediates in the synthesis of heterocyclic systems and are known to be biologically active. The triaril–amidines were synthesized $Ph{-}N{=}C(NHPh)C_6H_4{-}X$, X = H, OCH_3, CH_3, C1, Br, NO_2 via two methods depending on the electronic effect of the substituents according to the scheme below : acyl chlorides → anilides → imidoyl chlorides → amidines utilizing $SOCl_2$ or PCl_5 in appropriate reaction conditions. The chemical shifts of amidino carbon are in the range 165,16 to 153,13 ppm in ^{13}C NMR and the stretching frequency of N–C=N bond around 1660 to 1625 cm^{-1} in infrared spectra depending mainly on the substitution in the aromatic ring and to some extent also on the substitution at both nitrogen atoms.

QO-068 Reaction of o–Phenylene Phosphorochloridite with Aliphatic Amines, *Marcos Costa de Souza* and *Whei Oh Lin* (Seção de Química, Instituto Militar de Engenharia, Rio de Janeiro).

In order to introduce phosphorous atom into the potencial complexation agents developed in our Institute we have investigated the preparation of amino phenylenedioxyphosphorane (II). The reaction of o–phenylene phosphorochloridite with approprite amine in ether in the presence of triethyl amine gave an unexpected ethoxy phenylenedioxy–phosphorane (III) as main product. The detailed studies of this reaction in different solvent, base and temperature proved that ether plays an important role in the reaction.

QO-069 Nitration of Guanidine in the Mixture of Nitric Acid and Sulfuric Acid. *Whei Oh Lin* and *Édio Pereira de Oliveira*, (Seção de Química, Instituto Militar de Engenharia, Rio de Janeiro).

Nitration of guanidine nitrate in the mixture of concentrated nitric acid and sulfuric acid is little known. We have investigated this reaction in the acid range, $n_{HNO_3} / (n_{HNO_3} + n_{H_2SO_4})$, between 0.0 and 1.0, at different temperature (0°C, 25°C, 45°C, 60°C and 80°C) with three hours reaction time. The results suggest that nitration itself is a rapid process, decomposition of the nitrated product–nitroguanidine becomes more and more important while temperature of the reaction is increased.

QO-070 Studies on the Synthesis of Compounds Isolated From *Dyalyanthera Parvifolia*. Part II – Carbonation and Hydration of Acetylenic Derivatives. *Ivone Carvalho* (Faculdade de Ciências Farmacêuticas de Ribeirão Preto – USP) and *Mauricio Gopmes Constantino* (Departamento de Química da Faculdade de Filosofia, Ciências e Letras de Ribeirão Preto – USP).

Carbonation and hydration of acetylenic compounds containing a cyclopentane ring and OH groups (with and without THP protection) are the object of these studies. Carbonation is effected either with CO_2 or with ethyl chloroformate. The best hydration method involves conjugated addition of piperidine to an acetylenic ester.

QO-071 Synthesis and Ozonation of Allylic Derivatives. *Milton Beltrame Junior* and *Mauricio Gomes Constantino* (Deptº Química – Fac. de Filosofia, Ciências e Letras de Ribeirão Preto – USP).

Allylic derivatives usually give "abnormal" products in ozonation reaction. The object of this work is the synthesis and ozonation of an allylic spiro-lactone. Depending on the results obtained this ozonation reaction can be used in a synthesis of natural biologically active peroxides.

QO-072 Phenolic Standars Synthesis for Gas Cromatograph and Mass Spectrometry. Study of the NMR-13C of Acylphenols Intermediates. *Luzia Koike*, *Joana Félix de Souza* and *Maurício H. Ishikawa* (Chemitry Institut of Campinas State University).

No commercial phenolic compounds was synthesized for to use as standars in GC–MS analysis of the acidic fraction from Aviation Kerosene. In our previous studies about this fraction, we could see that it was rich in phenolic components. We concluded this studies using coinjection technic with 22 phenolic componds from synthetic to commercial. Otherwise we could study the NMR–13C of the intermediate phenol synthesis.

QO-073 NMR Studies of Derivatives of 6α, 7β–Dihydroxyvouacapan–17–β–Oic Acid.I. *Jeannette de Magalhães Moreira*, *Guglielmo Marconi Stefani* and *Dorila Piló Veloso* (Departamento de Química, UFMG) and *Dalton L. Ferreira Alves* (Departamento de Farmacologia, UFMG).

The employement of seed alcoholic infusions from *Pterodon*, Leguminosae, by brazylian folk medicin in rheumatic affections tratment is well known. It was previously reported that 6α, 7β–dihydroxy vouacapan–17–β–oic acid.I., isolated from *P.polygalaeflorus* Benth, presents antiinflammatory activity (Ferreira Alves et alli, Brazilian J. Med. Exp. Sc. *15*: 450, 1982). We synthesized the methyl(II) and the tetra–O–acetylglycosyl(III) ester derivatives of I to pharmacological studies. III was available by reaction of I with α–ABG catalysed by either $HgBr_2$ or Ag_2CO_3. From comparison of both ^{13}C and ^{1}H NMR spectra of I, II, and III, by assignments made on the basis of the chemical shift theory and ^{13}C DEPT, it was shown that I is a β–glycoside derivative (CNPq, CAPES, FINEP).

QO-075 Synthesis of Derivatives of Sistem Pentacycle $[6.2.1.1^{3,5}.0^{2,7}.0^{5,9}]$ Dodecan. *Valentim Emilio Uberti Costa* (Instituto de Química – Universidade Federal do Rio Grande do Sul) and *Peter Rudolf Seidl* (Seção de Química – Instituto Militar de Engenharia).

From dechlorination reaction of the 1,8,9,10,11,11–hexachloro–tetracycle $[6.2.1.1^{3,6}.0^{2,7}]$ dodec 4,5–epoxi–9–en ("endrin") we got a mixture of two isomers : insaturated tetracyclic alcohol and a pentacycle alcohol. The treatment of the mixture with saturated solution of $AgNO_3$ chromatography in SiO_2. $AgNO_3$ 10% gave us the pentacyclic system pure. Some chemical reaction were made to get functional derivatives of pentacyclic compound. Their Carbon–13 NMR spectra (1–D and 2–D) were analysed.

QO-076 Cycle (Bond C–C) Adition Effect on the Carbon–13 Chemical Shifts on the Bicycle [2.2.1] Heptan (Norbornane). *Valentim Emílio Uberti Costa* (Instituto de Química – Universidade Federal do Rio Grande do Sul) and *Peter Rudolf Seidl* (Seção de Química – Instituto Militar de Engenharia).

A family of polycyclic compounds were prepared and their Carbon–13 NMR spectra were analysed. We research the effect of sequential adition of chemical bonds on the $\sigma - {}^{13}C$ in the norbornane. Tables construction permited us to propose a method for the prediction of Carbon–13 Chemical shifts for a polycycles family exhibiting the same chemical function.

QO-077 Total Synthesis of Parvakone (2–Cyclohexyl–3–Hydroxy–1,4–Naphthoquinone). *Alaíde Braga de Oliveira*, *Délio Soares Raslan* and *Marcos Antonio Fernandes Brandão* Univ. Federal de Minas Gerais, Instituto de Ciências Exatas, Depto. de Química).

The biological activity of 2–alkyl–3–hydroxy–1,4–naphthoquinone has led to important discoveries in various areas of chemotherapy and pesticide science. The parvakone(I) has been used against *Theileria parva* the causative agent of the East African cattle disease – East Coast Fever. We describe now the total synthesis of parvakone(I) by alkylation of lawsone(II) with cyclohexane and benzoyl peroxide. The lawsone(II) was prepared by oxidation of α–tetralone(III) with oxygen in alkaline condition. The α–tetralone(III) was obtained by reactin of –butyrolactone with benzene in the presence of aluminum chloride.

QO-078 A Simple Preparation of 5,6–Dihidro–4H–Piranone–4 Compounds From D–Glucal. *Yvone Memória* (Laboratório Síntese Orgânica, Depto. de Química Orgânica e Inorgânica, Universidade Federal do Ceará). *Anne Bouillot*, *Duc Do Khac*, *Fréderic Guir* (Dept. de Chimie Organique, Ecole Polytechnique, Université Paris XI).

A selective oxidation study of D–glucal is described in this work. We report three differents methods to prepare 5,6–dihidro–4–piranone–4 the chiral precursors of trichothecenes, of our synthetic scheme proposed. Oxidation of allylic acetates by NBS, allylic alcohols by Ag_2CO_3/celite and by PDC. This work was supported by – CNPq.

QO-079 Study Kinetic of Reaction Between Poly(Phenylene Ether) and Triazolinediones. *Ivonete Oliveira Barcellos*, *Marines Bastianel*, *Marco Aurélio de Araújo*. (Instituto de Química – UFRGS–POA/RS).

In this work, the kinetics of the reaction between poly[oxy(2,6–dimethyl–1,4–phenylene)] (PPE) and 4–phenyl–1,2,4–triazoline–3,5 dione (PhTD) were investigated by spectroscopical methods.Kinetics experiments were performed under pseudo–first order with respect to polymer concentratin and using chorobenzene as solvent. Pseudo–first order rate constants, second order rate constants and activation parameters such as Ea, $\Delta H^{\neq}$, $\Delta G^{\neq}$ and $\Delta S^{\neq}$ were determined for this reaction.

QO-080 Dichloromethane: An Alternative Solvent in Friedel-Crafs Reactions. *Janilce R. Martins* and *Angelo C. Pinto* (IQ/UFRJ).

Following our studies in Friedel–Crafs reactions, we wish to report a more general use of dichloromethane (DCM) in these reactions. Since carbondissulfide is considered to be very toxic and nitrobenzene difficult to remove from the reaction medium, the search for other solvents is justified. Considering our previous experiment, in which DCM was used for isatine formation via Friedel–Crafts reaction, we applied this solvent in a more general sense, so investigating the acetylation of cumene and metoxybenzene. The reactions were successfully accomplished, so yielding the desired *para* – acetylated products regioselectively. This result showed that DCM can be used to replace the commonly used solvents in Friedel–Crafs reactions, even in reactions involving arenes substituted with electron donating groups.

QO-081 On the Cyclization of 1–Acetyl–2–Acetonyl–Cyclohexane in Micellar Reverse Systems. *Sandro Azeredo*, *Rogerio Perez*, *Joel Jones Júnior*, *Ricardo Bicca de Alencastro* (Inst. Química – U. Fed. Rio de Janeiro – CEP 21945 – RJ – Brazil).

We have studied the cyclization of 1–acetyl–2–acetonyl–cyclohexane in micellar reverse phase (AOT –isooctane $-H_2O$ –HCl and AOT–isooctane $-H_2O$ $-NaHCO_3$). In both systems we were able to observe positive catalysis on AOT micelle formation. In acidic media extensive dehydration of the products occurs, as expected. In $NaHCO_3$ media, we were able to obtain good yield of alcohols intermediaries. Better yields are obtained at lower temperatures (ca.40°C).

QO-082 Synthesis of Arylethanolamines N–Substituted in *Orto* Position. *Rosângela Bezerra da Silva* and *Ângelo C. Pinto* (Inst. Química – Univ. Fed. Rio de Janeiro – 21945 – RJ – Brazil).

The reaction of N–Acetylisatin with isopropylamine produce N–isopropyl–2–(2–acetamidophenyl)–2–oxo–acetamide in 93% crude yield. Its reduction with BH_3/THF in different conditions, showed high selectivity for both amides function. At r.t. or 0°C we obtained the N–Isopropyl–2–(2–Ethylamino–phenyl)–2–hydroxy–acetamide in 94% crude yield. When refluxed for 2 h we obtained the N–Isopropyl–2–(2–Ethylaminophenyl)–2–hydroxi–amino–ethanol in 78% crude yield.

QO-083 Monocyclofarnesate as a Precursor for Terpenoids: Investigation of the Cyclization Through Ketene Intermediates. *Fernando A. S. Coelho* (Salgema, AL), *Vania G. Bernardes* (IQ, UFRJ) and *Anibal L. Pereira* (Fac. Farm., Dept. Tecnol. Farm., UFRJ).

The intramolecular cycloaddition reaction of ketenes is one of the most useful methods for the synthesis of cyclobutanones and compounds derived hereby. On the way to drimanic compounds we prepared monocyclofarnesic acid from *beta*–ionone (4 steps, 68% yield) and submitted it to ketene generation conditions through Snider ($(COCl)_2$, NEt_3, arom. solv, refl.) or Lynch ($(COCl)_2$, $N(i\text{–}Pr)_2Et$, CH_2Cl_2, –20°C) procedures. In both cases, we obtained monocyclofarnesic acid anhydride as the single product, as evidenced by spectral data and conversion to esters by alcoholysis catalyzed by DMAP.

QO-084 Synthesis of N–[9–(1–p–Menthenyl)]–N–Benzyl–Amine From Limonene and Benzylazide. *Denilson Ferreira de Oliveira* and *W. Bruce Kover* (Inst. Química – U. Fed. Rio de Janeiro – 21945 – Rio de Janeiro – RJ – Brazil).

Limonene and benzylazide were heated to produce N–[9–(1–p–menthenilidene)]–N–benzyl–amine in 100% crude yield. After its reduction with $NaBH_4$/i–PrOH we obtained the N–[9–(1–p–menthenyl)]–N–benzyl–amine in 92% crude yield.

QO-085 Effect of PH Variation on Solvolysis of N–Tosyl–N–Benzyl–Benzohydroxamate in Aqueous Dioxane. *Giselle Medeiros* and *W. Bruce Kover* (Inst. Química – Univ. Fed. do Rio de Janeiro – CEP 21945 – Brazil).

The rearrangement of alkyl-acyl– nitrenium ion could provide a new methode of preparing secondary amines in high purity. Trying to favour the breaking of N–O bond, relative to A_{AC^2} hidrolysis, we examed the N–tosyl–N–benzyl–benzohydroxamate solvolysis in buffered aqueous dioxane at specific pH value. The analysis of those reactions by HPLC, show that the benzoic acid yield decreases with pH reduction.

QO-086 Carvenone Tribromide. Reaction With NaI. *Florinda do N. Cersosimo* & *W. Bruce Kover*. (Departamento de Química Orgânica – Instituto de Química da Univers. Fed. do Rio de Janeiro, Rio de Janeiro, 21945, Brasil).

The treatment of carvenone tribromide with an equimolar quantity of NaI leads to the production 6–bromo–carvenone but with excess iodide the reduction to carvenone is observed and addition of methyl iodide traps the intermediate / enolate.

QO-087 Reaction of the α–α–Dichloro–Cyclo–Butanone With Benzylamina in DMF. *Márcia Cristina C. Veloso* and *W. Bruce Kover* (Inst. Química U. Fed. Rio de Janeiro – RJ – CEP 21945 – Brazil).

The reaction of 2,2–dichloro–(3R,S)–methyl–(3R,S)–[(4R)–methyl–cyclo–hexene–3–enyl]–cyclo–butanone obtained by cyclo-addition of dichloro–ketene to limonene with benzylamine in DMF at 70°C affords surprisingly products of Favorskii.

QO-088 Cycloaddition of Nitrile Oxides to Limonene. *Márcio Contrucci S. de Mattos* & *Warner Bruce Kover* (Inst. de Química, Univ. Federal do Rio de Janeiro, Rio de Janeiro, 21945, Brazil).

The cycloaddition of RCNO generated *in situ* to limonene (R = Me,Et,*i*–Pr,Bu,Ph and crotyl) is chemo– and regiospecific, occuring at the acyclic double bond of the substrate. The isoxazolines were obtained with 40–75% yields (isolated).

QO-089 Oxidative Diamino–Mercuration of Limonene: Investigation of the Reaction Pathway. *M. C. S. de Mattos, W. B. Kover* (Inst. de Química, Univ. Federal do Rio de Janeiro, Rio de Janeiro, 21945, Brazil), *F. Aznar* and *J. Barluenga* (Departamento de Química Organometalica, Universidad de Oviedo, Oviedo, 33071, Spain).

The solvomercuration–demercuration of limonene in the presence of aniline *(I)* occurs at the acyclic double bond. The oxidative mercuratin–demercuration in the presence of excess *(I)* led exclusively to the diamination of the cyclic double bond *via* opening of the 1,8–diamino–2,9–dialkylmercury compound.

QO-090 Oxidation of Limonene With Dimethyldioxirane. *José Renato Cagnon, Joel Jones Júnior* and *W. Bruce Kover*. (Inst. Quím. – U. Fed. Rio de Janeiro – RJ – 21945 – Brazil).

Limonene when oxidized with acetone/$KHSO_5$ (dimethyldioxirane) produced two diastereoisomers of 1,2–epoxy–8,9–dihydroxy–limonene. This conclusion based on CG/MS, shows diepoxy production followed by preferential at the 8,9 position.

QO-091 Solvolysis of Tosylate the (1R,2R,3R,5R)–2,3–Epoxy–5–Isopropenyl–2–Methyl–Cyclohexyl. Part II. *J. Jones Jr.* and *W. B. Kover* (Inst. de Química da Univ. Federal do Rio de Janeiro).

Solvolysis of the epoxytosylate I in water/dioxane produced carvone(II) and ketone(III) in 45% yield. When proton scavengers are used the epoxy–alcohol IV and the hydroxyketone V were obtained. Under the conditions of acidic hidrolise those were converted to II and III respectivity, indicating that IV and V are intermediates in the conversion of I to II and III.

QO-092 Photochemical Study of the Degradation of Tetra–Phenyl–Porphine (TPP) in Photo–Oxigenation Reactions. *Marco Antonio a. da Silva* & *David E. Nicodem* (Organic Chemistry Department of the Institute of Chemistry of the Federal University of Rio de Janeiro).

It was observed that TPP was degradated when used as a dye in photo–oxigenation reactions of alkenes. Possible reasons could involve: temperature, luminous intensity and wavelength. Tests with monochromatic light (405 nm), in an elliptical reactor "*Fire–fly*", changing the luminous intensity and the reaction temperature show the product is thermo–instable and that the reaction of degradation depends on luminous intensity.

QO-093 Calculation of Values of λ_{max} for Substituted Stilbenes. *David E. Nicodem* and *Eudes R. Oliveira* (Departamento de Química Orgânica do Instituto de Química da UFRJ).

Ten stilbenes were synthesized with substituents in the 4 and 4' positions and absorption spectra were taken. It was possible to correlate the energy of the absorption maxima with values of sigma + and sigma – in a way that takes into account electron demand and merostabilization.

QO-094 Biantrone Cyclization Sensitized by 4–Phenyl–Acetophenone. *David E. Nicodem* and *Maria Fernanda V. da Cunha* (Depto. de Química Orgânica do Instituto de Química da UFRJ).

Irradiation of 4–phenylacetophenone at 313 nm in solutions containing biantrone gives helioanthone. This cyclization is a singlet state reaction, while 4–phenyl–acetophenone is generally considered a selective triplet state sensitizer. The accurance of singlet sensitization indicated that this sensitizer should be used with caution.

QO-095 Synthesis and Characterization of New Derivatives of Methylene Blue. *David E. Nicodem* and *Leila S. Rocha* (Departamento de Química Orgânica do Instituto de Química da UFRJ).

Two counterion containing derivatives of methylene blue –[3–(N,N–metil–undecanoic acidamino)–7–(N,N,–dimethylemino) phenothiazin–5–ium chloride] and [3–(N,N–acetic acid–methylamino–7–(N,N,–dimethylamino) phenothiazin–5–ium chloride] –were synthesized by condensation of the corresponding aniline derivatives and N,N–dimethyl–p–phenylenediamine and were characterized by spectroscopic methods.

QO-096 Quantum Yields of the Individual Photoprocesses Involved in the Transformation of 7–Dehydrocholesterol in Vitamin D_3. *David E. Nicodem and Rosaly S. da Silva* (Departamento de Química Orgânica do Instituto de Química da UFRJ).

7–dehydrocholesterol (7–DHC) was irradiated in ether using 254nm light. The concentration of 7–DHC, P_3 e T_3 were determined by HPLC at varying conversions, while the light absorbed was also followed using potassium ferrioxalate actinometry. Data analysis using a computer program created for this purpose gave quantum yields for the individual processes. It was observed that lowering the temperature from 0°C to –70°C slightly reduced the efficiency of the photoprocesses.

QO-098 Quantitative Analysis of Propionic Acid Chlorination Products by Proton Nuclear Magnetic Resonance. *Rosane Aguiar da Silva San Gil* & *Adelina Costa Neto* (Dep. Org. Chem., Fed. Univ. of Rio de Janeiro, Brazil).

Proton NMR integration values of certain absorption peaks in chosen chemical shift ranges may be used to characterize the possible products in propionic acid chlorination reactions. The chosen chemical shift ranges for propionic acid, 2–chloro–, 2,3–dichloro–, 3,3–dichloro–, 3–chloro– and 2,2–dichloro–propionic acids where (ppm): 0,50–1,55, 1,55–2,00, 4,00–5,00, 5,40–6,20, 3,20–4,00 and 2,00–3,20 respec.

QO-099 Synthesis of Some Derivatives of 3,4–Dimethoxy– and 3,4–Methylenedioxy–Amphetamine. *Milton A. Torres, Marcos C. Rezende* (Departamento de Química, Universidade Federal de S. Catarina) and *Bruce K. Cassels* (Departamento de Quimica, (Faculdad de Ciencias, Universidad de Chile).

The synthesis of 2–nitro–4,5–dimethoxyamphetamine, 2–amino–4,5–dimethoxyamphetamine, 2–nitro–4,5–methylenedioxyamphetamine and 2–amino–4,5–methylenedioxyamphetamine are described.

QO-100 Synthesis of 8–Membered Cyclic Sulphites and of Their Dimeric Forms. *Afonso Celso Guimarães* (Departamento de Química, ICEx, UFMG) and *Jean Bernard Robert* (Laboratoire de Chimie Organique Physique, DRF, Grenoble, France).

The cyclisation reaction of 2–mercaptoethyl sulfide $(S(CH_2CH_2OH)_2$ or 2–hydroxyethyl ether $(O(CH_2CH_2OH)_2$ with thionyl chloride $(O=SCl_2)$ leads in addition to the expected eight–membered ring sulfites to the formation of the corresponding sixteen–membered ring dimers.

QO-101 Synthesis of 5–Metoxy–Naphtho(2,3–b)Furano–4,9–Dione. *Alaíde Braga de Oliveira, Carlos Leomar Zani* and *Sérgia Maria Starling* (Departamento de Química – ICEx – UFMG. Centro de Pesquisas René Rachou).

The 5–metoxy–naphtho(2,3–b)furano–4,9–dione was previously synthesized by a tandem *ortho* metalation tactics with a total yield of 13%. We describe now the synthesis of this compound by the usual *ortho* litiation methodology. Starting from 0–methoxy–N,N–diethyl–benzamide *1*, followed by addition to 2–furfuraldehyde affording the ftalide *2* (80% yield). Hydrogenolysis of the ftalide *2* (Zn, $CuSO_4$, NH_4OH) gave the carboxylic acid *3* (95% yield). The furanonaphthoquinone *4* was obtained (60% yield) from the carboxylic acid *3* by reflux with Ac_2O/AcOH followed by oxidation with CrO_3. All the compounds were caracterized by the usual spectroscopic methods. Compound *4* was tested against *P. falciparum* and *T. cruzi*. (CAPES, CNPq, FINEP).

QO-102 Conversion of Glucal to α,β– Insaturated Chiral α and γ – Pyrone as a Intermediate of a Forskoline and Trichothecenes Synthesis. *Yvone Memória* (Laboratório Síntese Orgânica, Depto. Química Orgânica e Inorgânica, Universidade Federal do Ceará); *Nguien Hien, Duc Do Khac, Marcel Fetizon* (Dept. Chimie Organique, École Polytechnique, Université de Paris XI).

Our plan for construction of a chiral intermediate to preparing natural products begins by the regioselective protection of the primary alcohol group as a silyl ether. Subsequent selective oxidation or protection of the allylic alcohol of glucal has been developped. At this point the C–4 hydroxyl was acetylated and reduced photochemically to the corresponding disilyl or monosilylether 4 – deoxy glucal. Regioselective oxidation is also studied. (CNPq).