

VALIDATION OF AN ANALYTICAL METHODOLOGY FOR THE QUANTITATIVE ANALYSIS OF PETROLEUM HYDROCARBONS IN MARINE SEDIMENT SAMPLES

Eloy Yordad Companioni Damas*, Miriam Odette Cora Medina, Ana Catalina Núñez Clemente and Miguel Ángel Díaz Díaz

Environmental Chemistry Laboratory, Petroleum Research Center, 169 Washington Road, Havana, Cuba

Luis González Bravo

Center of Nuclear Technological Applications and Development, 30 Ave., Havana, Cuba

Rolando Marbot Ramada

Analytical Chemistry Laboratory, Petroleum Research Center, 169 Washington Road, Havana, Cuba

Rodny Montes de Oca Porto

Antidoping Laboratory, 100 Street, Havana, Cuba

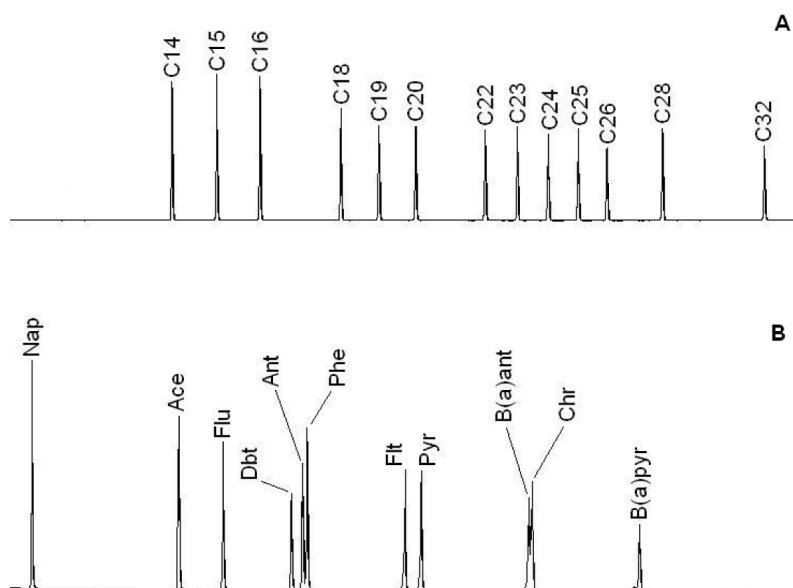


Figure 1S. Separation of *n*-alkanes (A) and PAH (B) obtained from the GC-FID analysis of standard solutions. See Instrumentation and Apparatus for chromatographic conditions. Nap: naphthalene, Ace: acenaphthene, Flu: fluorene, Dbt: dibenzothiophene, Ant: anthracene, Phe: phenanthrene, Flt: fluoranthene, Pyr: pyrene, B(a)ant: benzo(a)anthracene, Chr: chrysene, B(a)pyr: benzo(a)pyrene

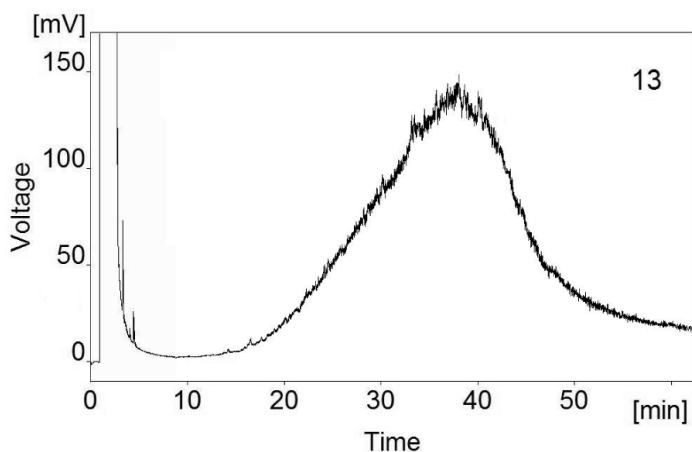


Figure 2S. Characteristic gas chromatogram of aromatic hydrocarbon fractions extracted from collected marine sediments

*e-mail: elocompa@yahoo.com

Table 1S. Aliphatic compounds identified in the total ion chromatogram displayed in Figure 4A

No.	Compounds	RT (min)	Confirmation ions (m/z)
1	1-Tetradecene	23.545	83/196
2	n-Hexadecane	23.798	85/226
3	2, 6, 10-trimethylpentadecane (norpristane)	25.528	85/238
4	1-Pentadecene	27.093	83/210
5	n-Heptadecane	27.369	85/240
6	2, 6, 10, 14 – tetrametil-pentadecane (pristane)	27.543	85/268
7	1-Heptadecene	28.879	83/238
8	4- Methylheptadecane	29.313	85/254
9	3-Methylheptadecane	29.748	85/254
10	1-Octadecene	30.530	83/252
11	n-Octadecane	30.807	85/252
12	2, 6, 10, 14-tetramethylpentadecane (phytane)	31.084	85/282
13	7, 11-Dimethyloctadecane	32.166	85/282
14	3-Methyloctadecane	33.075	85/268
15	2, 6, 10, 14-tetramethylheptadecane	33.794	85/296
16	n-Nonadecane	34.110	85/268
17	4-Cyclohexyltridecane	35.849	85/288
18	4-Methylnonadecane	36.054	85/282
19	3-Methylnonadecane	36.283	85/282
20	n-Eicosane	37.271	85/282
21	7-Propyltridecane	38.401	85/226
22	4-Propyltridecane	38.733	85/226
23	3-Methyleicosane	39.318	85/226
24	n-Heneicosane	40.290	85/296
25	n-Docosane	43.174	85/310
26	n-Tricosane	45.932	85/324
27	n-Tetracosane	48.579	85/338
28	n-Pentacosane	51.124	85/352
29	Diisooctylfthalate ^a	52.017	149/390
30	n-Hexacosane	53.573	85/366
31	5 α , 8 α , 14 β Cholestane	54.316	217/218
32	3-Ethyltetracosane	54.980	85/366
33	n-Heptacosane	55.944	85/380
34	5 α , 3 Cholestene	57.951	217/218
35	n-Octacosane	58.212	85/394
36	n-Nonacosane	60.425	85/408
37	17 α (H), 21 β (H) Norhopane	61.800	191
38	n-Triacontane	62.519	85/464
39	17 β (H), 21 α (H) Norhopane	63.554	191
40	n-Hentriacontane	63.720	85/436
41	n-Dotriacontane	64.581	85/450
42	n-Tritriacontane	66.557	85/464
43	n-Tetratriacontane	68.524	85/478
44	n-Pentatriacontane	70.413	85/492

RT: Retention time; ^a contamination derived from the laboratory plastic materials.

Table 2S. Concentrations of hydrocarbons determined in representative marine sediment samples

Compounds	1	5	9	10	13
C ₁₄ (ng/g)	N.D	N.D	N.D	N.D	N.D
C ₁₅	N.D	N.D	N.D	N.Q	N.D
C ₁₆	N.D	N.D	N.D	61.4 ± 9.5	78.4 ± 10.2
iC ₁₈	N.D	N.D	N.D	56.2 ± 9.3	65.3 ± 9.7
C ₁₇	43.3 ± 13.0 ^{aP}	39.6 ± 12.8	35.5 ± 12.5	93.0 ± 16.2	321.4 ± 30.5
iC ₁₉	39.3 ± 12.8	N.D	32.2 ± 12.3	93.8 ± 16.2	236.7 ± 25.2
C ₁₈	75.3 ± 15.0	N.D	77.6 ± 15.2	112.1 ± 17.4	128.6 ± 18.4
iC ₂₀	66.3 ± 14.5	N.D	56.0 ± 13.8	125.6 ± 18.2	171.8 ± 21.1
C ₁₉	91.3 ± 13.5	38.5 ± 6.39	151.1 ± 21.6	199.5 ± 28.1	201.4 ± 28.4
C ₂₀	113.0 ± 21.7	61.8 ± 13.1	165.0 ± 30.4	185.8 ± 33.9	163.3 ± 30.1
C ₂₁	122.6 ± 23.3	89.3 ± 17.7	139.8 ± 26.2	128.3 ± 24.2	151.3 ± 28.1
C ₂₂	127.1 ± 23.5	110.0 ± 20.9	150.0 ± 27.0	118.8 ± 22.3	174.6 ± 30.7
C ₂₃	142.6 ± 15.2	105.3 ± 13.4	121.5 ± 14.2	109.5 ± 13.6	184.5 ± 17.3
C ₂₄	167.5 ± 21.0	96.8 ± 16.8	106.7 ± 17.4	83.0 ± 16.0	193.8 ± 22.6
C ₂₅	159.9 ± 19.5	91.0 ± 14.8	98.8 ± 15.3	59.4 ± 12.6	245.7 ± 25.4
C ₂₆	155.5 ± 12.5	67.7 ± 7.9	91.0 ± 9.1	66.7 ± 7.9	147.8 ± 12.1
C ₂₇	148.7 ± 20.8	77.1 ± 11.4	103.5 ± 14.9	N.D	229.4 ± 31.4
C ₂₈	152.3 ± 21.3	102.9 ± 14.8	208.0 ± 28.6	N.D	149.9 ± 21.0
C ₂₉	125.0 ± 17.7	56.1 ± 8.7	116.2 ± 16.5	N.D	N.D
C ₃₀	89.2 ± 13.0	38.1 ± 6.3	112.3 ± 16.0	N.D	N.D
C ₃₁	84.0 ± 12.3	50.2 ± 7.9	116.7 ± 16.6	N.D	N.D
C ₃₂	69.7 ± 16.0	N.D	81.6 ± 17.0	N.D	N.D
C ₃₃	62.0 ± 9.4	N.D	78.0 ± 11.5	N.D	N.D
C ₃₄	35.2 ± 5.9	N.D	N.D	N.D	N.D
NA (µg/g)	1.96 ± 0.16	1.02 ± 0.11	1.95 ± 0.16	1.22 ± 0.12	2.37 ± 0.19
AH (µg/g)	14.0 ± 0.8	6.3 ± 0.4	17.5 ± 1.0	76.4 ± 4.3	105.1 ± 5.9
ArH (µg/g)	10.5 ± 2.1	1.1 ± 0.2	6.8 ± 1.3	21.4 ± 4.2	38.4 ± 7.6

iC₁₈: norpristane; iC₁₉: pristane; iC₂₀: phytane; NA: n-alkanes total concentration; AH: aliphatic hydrocarbon total concentration; ArH: aromatic hydrocarbon total concentration; N.D.: not detected; N.Q: detected but not quantified; ^a Estimated uncertainty