STRUCTURAL AND REACTIVITY ANALYSES OF 2-BENZYLAMINO-1,4-NAPHTHOQUINONE BY X-RAY CHARACTERIZATION, ELECTROCHEMICAL MEASUREMENTS, AND DFT SINGLE-MOLECULE CALCULATIONS[#]

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Table 1S. Crystal data and structure refinement

Identification code	rlso5b
Empirical formula	$C_{17}H_{13}NO_2$
Formula weight	263.28
Temperature	293(2) K
Wavelength	1.54180 A
Crystal system, space group	?, ?
Unit cell dimensions	a = 10.858(2) A alpha = 90 deg.
	b = 24.209(4) A beta = 90 deg.
	c = 5.0380(10) A gamma = 90 deg.
Volume	1324.3(4) A^3
Z, Calculated density	4, 1.321 Mg/m^3
Absorption coefficient	0.700 mm^{-1}
F(000)	552
Crystal size	0.35 x 0.15 x 0.10 mm
Theta range for data collection	3.65 to 67.85 deg.
Limiting indices	-13<=h<=0, -29<=k<=1, 0<=l<=6
Reflections collected / unique	1421 / 1339 [R(int) = 0.0330]
Completeness to theta = 67.85	99.7 %
Max. and min. transmission	0.9333 and 0.7917
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1339 / 1 / 182
Goodness-of-fit on F^2	1.142
Final R indices [I>2sigma(I)]	R1 = 0.0665, wR2 = 0.1726
R indices (all data)	R1 = 0.1042, wR2 = 0.2428
Absolute structure parameter	-10(10)
Extinction coefficient	0.024(4)
Largest diff. peak and hole	0.314 and -0.329 $e.A^{-3}$

Table 2S. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3). U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

	x	y	z	U(eq)
O(1)	1534(5)	2839(2)	-840(14)	83(2)
O(2)	5673(4)	3257(2)	4504(15)	69(2)
N(1)	4404(5)	2358(2)	5880(14)	59(2)
C(1)	2494(6)	2936(3)	428(18)	60(2)
C(2)	3242(6)	3433(3)	-203(16)	57(2)
C(3)	2858(8)	3794(4)	-2319(18)	77(3)
C(4)	3596(11)	4233(4)	-2840(30)	96(3)
C(5)	4657(11)	4341(4)	-1600(20)	91(3)
C(6)	5046(8)	3994(3)	420(20)	77(3)
C(7)	4326(6)	3536(3)	1066(17)	58(2)
C(8)	4749(5)	3164(3)	3285(15)	51(2)
C(9)	3966(5)	2675(3)	3864(15)	49(2)
C(10)	2906(6)	2576(3)	2526(16)	55(2)
C(11)	3771(7)	1880(3)	6980(20)	68(2)
C(12)	3901(6)	1363(3)	5195(18)	55(2)
C(13)	3089(7)	932(3)	5660(20)	69(2)
C(14)	3175(8)	445(3)	4360(30)	90(3)
C(15)	4081(9)	371(3)	2410(20)	82(3)
C(16)	4915(9)	797(3)	1970(20)	84(3)
C(17)	4806(7)	1284(3)	3380(20)	69(2)

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[#]This work is dedicated to Prof. Hans Viertler, pioneer in electrochemistry applied to organic synthesis in Brazil, on the occasion of his 70th birthday

Table 3S. Anisotropic displacement parameters (A^2 x 10^3. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a^{*2} U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	58(3)	104(4)	87(5)	-17(4)	-29(3)	15(3)
O(2)	46(2)	62(3)	100(4)	-9(3)	-13(3)	-4(2)
N(1)	50(3)	50(3)	77(4)	-9(3)	-16(3)	0(2)
C(1)	44(3)	73(4)	62(5)	-12(4)	-9(4)	15(3)
C(2)	60(4)	55(3)	57(4)	-10(4)	-2(4)	21(3)
C(3)	89(5)	79(5)	64(5)	-22(5)	-13(5)	29(5)
C(4)	123(8)	69(5)	95(8)	10(6)	-7(8)	34(6)
C(5)	125(8)	63(5)	84(7)	2(5)	17(7)	14(5)
C(6)	81(5)	54(4)	97(7)	-9(5)	-3(6)	-2(4)
C(7)	55(4)	47(3)	71(5)	-15(4)	3(4)	8(3)
C(8)	41(3)	52(3)	58(4)	-18(3)	-3(3)	6(3)
C(9)	41(3)	49(3)	57(4)	-11(3)	-7(3)	5(2)
C(10)	41(3)	66(4)	60(4)	-18(4)	1(3)	-1(3)
C(11)	62(4)	61(4)	80(6)	-1(4)	-1(5)	1(3)
C(12)	48(3)	52(3)	66(5)	9(4)	-1(4)	6(3)
C(13)	60(4)	59(4)	89(6)	0(5)	-1(5)	0(3)
C(14)	83(5)	52(4)	136(10)	5(6)	-2(7)	-6(4)
C(15)	102(6)	55(4)	88(7)	-10(5)	-15(6)	16(4)
C(16)	98(6)	66(5)	89(7)	-2(5)	8(6)	15(4)
C(17)	68(4)	61(4)	77(6)	4(4)	6(5)	1(3)

Table 4S. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3

	x	y	z	U(eq)
H(1)	5105	2446	6553	71
H(3)	2140	3730	-3278	92
H(4)	3346	4476	-4168	115
H(5)	5127	4647	-2076	109
H(6)	5775	4064	1329	93
H(10)	2434	2269	2972	66
H(11A)	2904	1967	7198	81
H(11B)	4106	1799	8725	81
H(13)	2466	979	6910	83
H(14)	2632	160	4750	109
H(15)	4125	45	1440	98
H(16)	5542	755	728	101
H(17)	5371	1566	3076	83

Table 5S. Hydrogen bonds [A and deg.]

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(2)	0.86	2.30	2.668(7)	105.7
C(17)-H(17)...N(1)	0.93	2.60	2.921(10)	100.7
N(1)-H(1)...O(1)#1	0.86	2.15	2.882(8)	143.3
C(10)-H(10)...O(2)#2	0.93	2.42	3.307(9)	158.6

Symmetry transformations used to generate equivalent atoms: #1 x+1/2,-y+1/2,z+1 #2 x-1/2,-y+1/2,z

Table 6S. Torsion angles [deg

O(1)-C(1)-C(2)-C(7)	-176.9(7)
C(10)-C(1)-C(2)-C(7)	3.4(10)
O(1)-C(1)-C(2)-C(3)	-1.2(10)
C(10)-C(1)-C(2)-C(3)	179.1(6)
C(7)-C(2)-C(3)-C(4)	-2.5(11)
C(1)-C(2)-C(3)-C(4)	-178.3(8)
C(2)-C(3)-C(4)-C(5)	2.0(15)
C(3)-C(4)-C(5)-C(6)	-1.2(17)
C(4)-C(5)-C(6)-C(7)	0.8(15)
C(3)-C(2)-C(7)-C(6)	2.4(11)
C(1)-C(2)-C(7)-C(6)	178.1(7)
C(3)-C(2)-C(7)-C(8)	-179.9(6)
C(1)-C(2)-C(7)-C(8)	-4.2(10)
C(5)-C(6)-C(7)-C(2)	-1.5(13)
C(5)-C(6)-C(7)-C(8)	-179.3(7)
C(2)-C(7)-C(8)-O(2)	-176.9(7)
C(6)-C(7)-C(8)-O(2)	0.8(10)
C(2)-C(7)-C(8)-C(9)	3.5(9)
C(6)-C(7)-C(8)-C(9)	-178.8(7)
C(11)-N(1)-C(9)-C(10)	-3.0(11)
C(11)-N(1)-C(9)-C(8)	175.1(7)
O(2)-C(8)-C(9)-C(10)	178.4(7)
C(7)-C(8)-C(9)-C(10)	-1.9(9)
O(2)-C(8)-C(9)-N(1)	0.2(9)
C(7)-C(8)-C(9)-N(1)	179.9(6)
N(1)-C(9)-C(10)-C(1)	179.2(7)
C(8)-C(9)-C(10)-C(1)	1.2(10)
O(1)-C(1)-C(10)-C(9)	178.5(7)
C(2)-C(1)-C(10)-C(9)	-1.8(10)
C(9)-N(1)-C(11)-C(12)	77.8(8)
N(1)-C(11)-C(12)-C(17)	22.1(11)
N(1)-C(11)-C(12)-C(13)	-163.7(7)
C(17)-C(12)-C(13)-C(14)	-0.4(13)
C(11)-C(12)-C(13)-C(14)	-175.1(9)
C(12)-C(13)-C(14)-C(15)	-1.9(15)
C(13)-C(14)-C(15)-C(16)	3.1(16)
C(14)-C(15)-C(16)-C(17)	-2.0(15)
C(13)-C(12)-C(17)-C(16)	1.6(13)
C(11)-C(12)-C(17)-C(16)	175.7(9)
C(15)-C(16)-C(17)-C(12)	-0.3(15)

Symmetry transformations used to generate equivalent ato ms

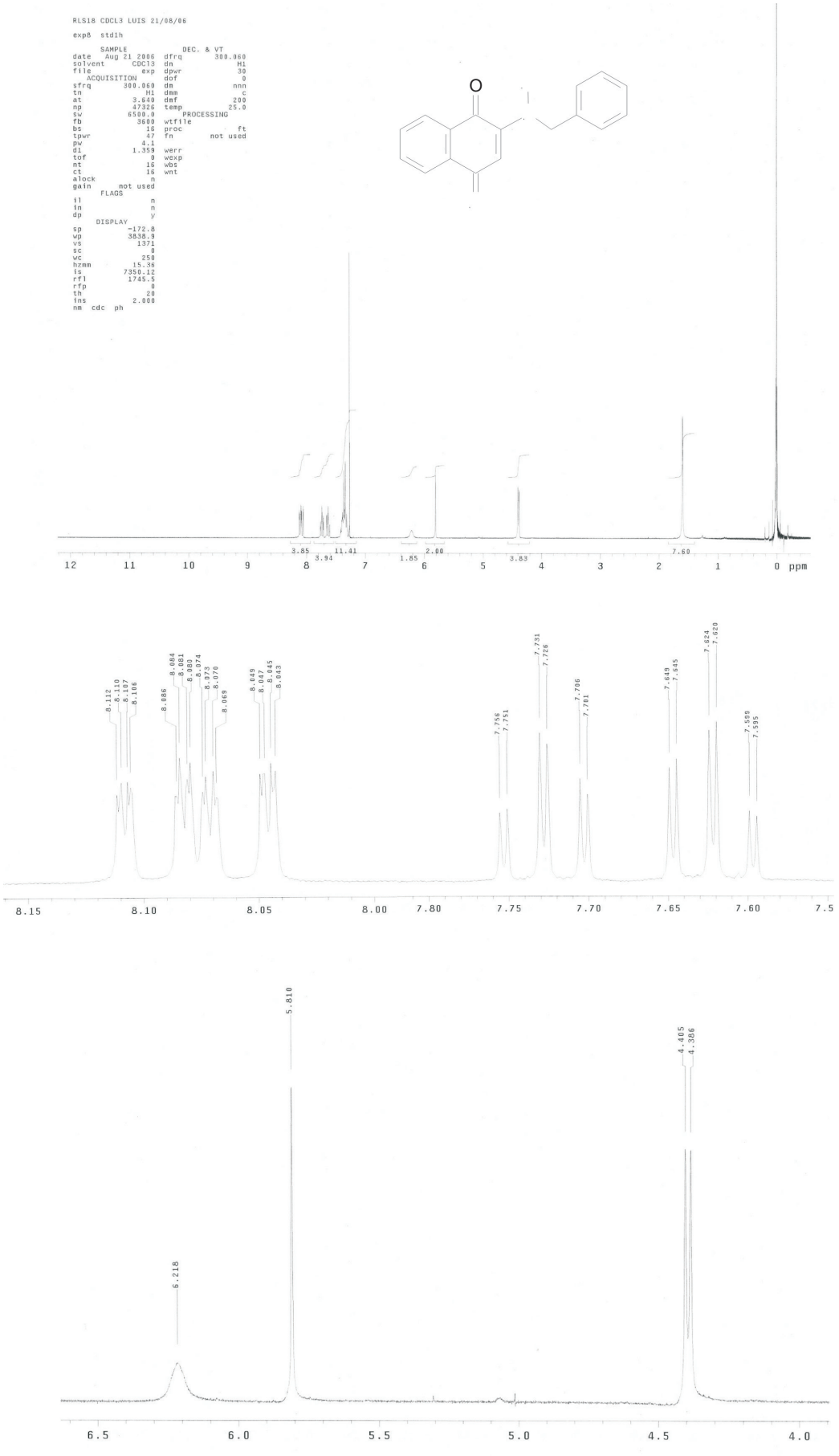

Figure 1S. *^{1}H NMR spectrum (and expansions) of compound **1** ($CDCl_3$)*

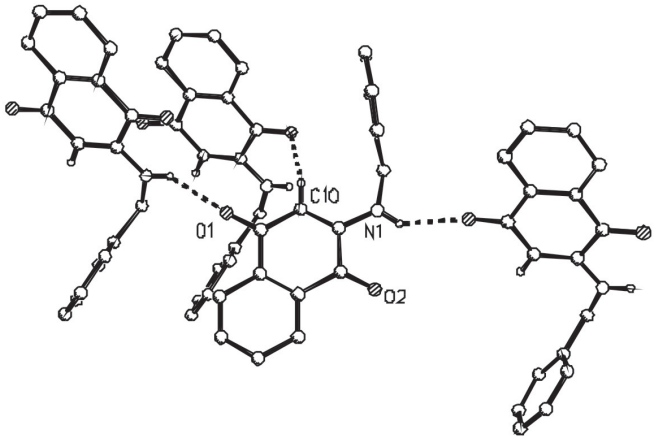

Figure 2S. *Intermolecular hydrogen bonding of compound* ***1***

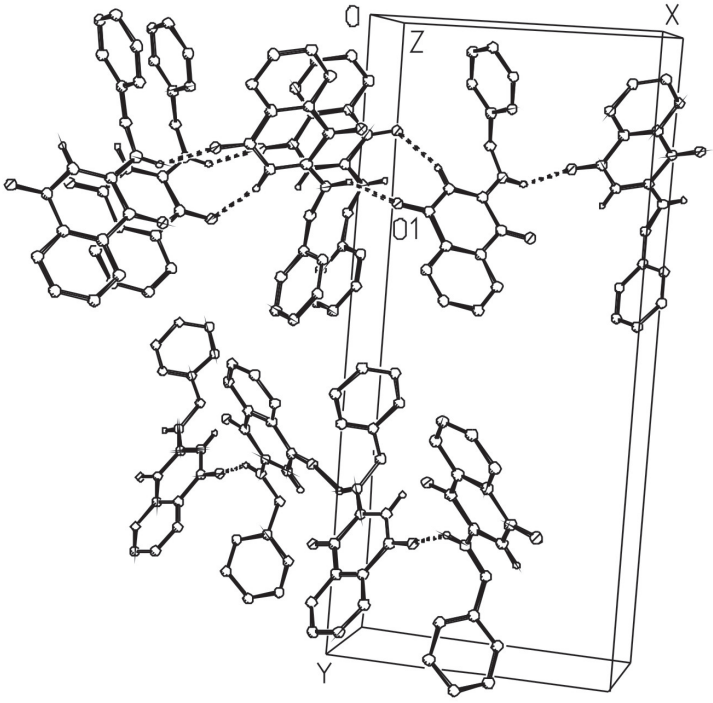

Figure 3S. *Molecular packing of* ***1*** *in the c-axis. The top chain is in the [1 0 2] direction, and bottom chain is in the [-1 0 2] direction. For clarity, only two molecules in one upper parallel chain are indicated, and only the hydrogens in H-bond are presented*

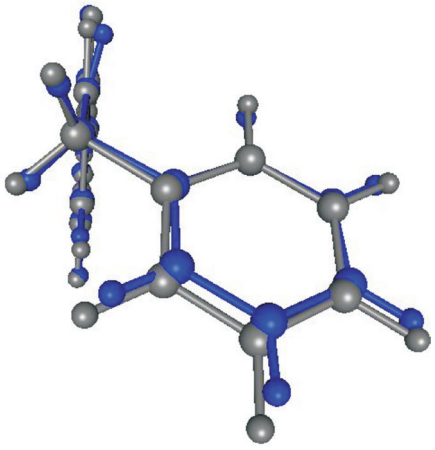

Figure 4S. *Superposition of X-ray (blue) structure of 2-benzylamino-1,4-naphthoquinone and optimized structure (gray) at the B3PW91/6-31+G(d) level*

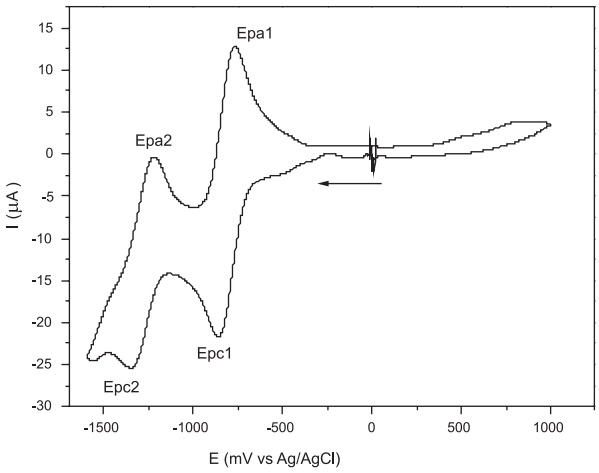

Figure 5S. *Cyclic voltammogram of* ***1*** *in acetonitrile with* Bu_4NBF_4 *(0.1 mol* L^{-1}*),* $v = 100\ mV\ s^{-1}$

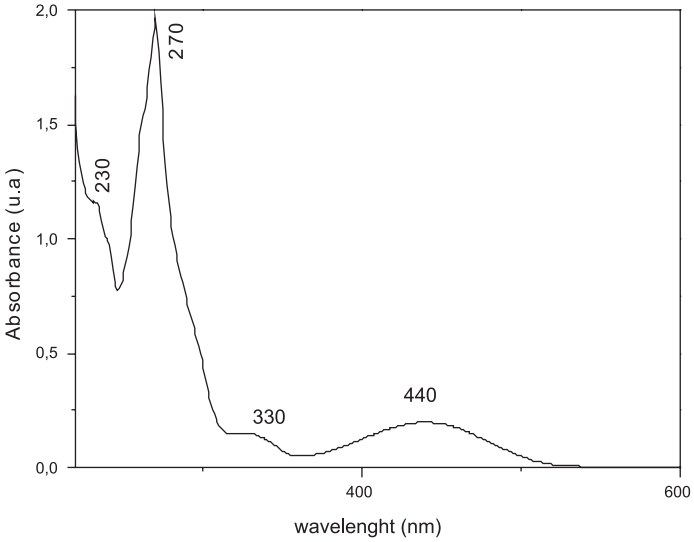

Figure 6S. *Molecular absorption spectrum of compound* ***1***

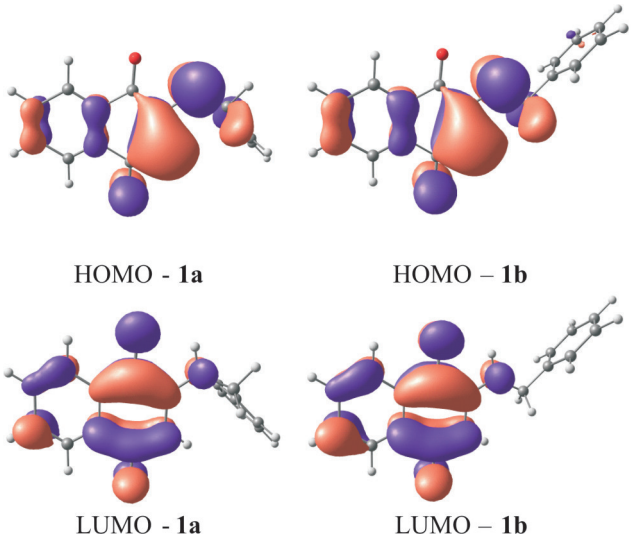

Figure 7S. *HOMO/LUMO molecular orbital for conformers* ***1a-1b***