Crystal structure of 2-chloro-3-(*N*-acetyl)phenylamino-1,4-naphthoquinone, $C_{18}H_{12}ClNO_3$

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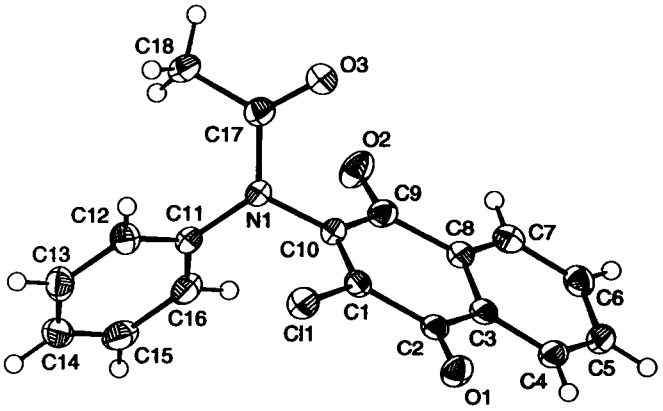

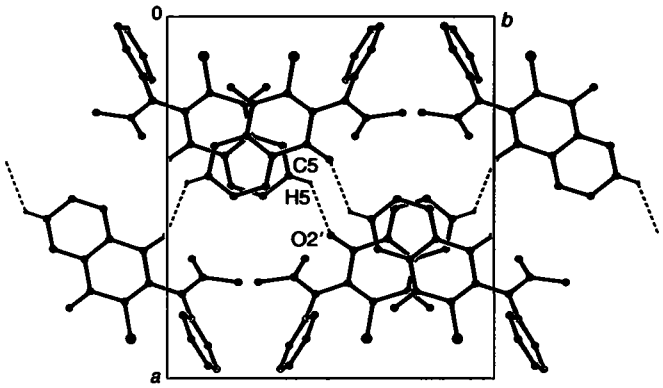

Abstract

$C_{18}H_{12}ClNO_3$, monoclinic, $P12_1/c1$ (no. 14), $a = 15.196(3)$ Å, $b = 13.430(3)$ Å, $c = 7.360(2)$ Å, $\beta = 97.83(3)°$, $V = 1488.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.042$, $wR_{ref}(F^2) = 0.109$, $T = 293$ K.

Source of material

During our search for biological active naphthoquinones [1] we have synthesized the title compound by acetylation of 2-chloro-3-phenylamino-1,4-naphthoquinone with acetic anhydride and concentrate sulfuric acid. The yellow product was purified by double crystallization from a mixture of the solvents dichloromethane and ethanol. Pure single crystals, suitable for the X-ray structure analysis, were obtained from dichloromethane as yellow needles (m.p. 407-408 K).

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Discussion

The title compound possesses three distinct units: an acetyl group, a *N*-phenyl ring, and a naphthoquinone ring. These units do not lie in the same molecular plane, as can be clearly seen in the ORTEP diagram (figure, top). The *N*-phenyl ring has a C11–N1–C10 angle of 117.5(2)° with the naphthoquinone ring. The C17–N1–C10 angle between the acetyl group and the naphthoquinone ring is 116.8(2)°. The C17–N1–C11 angle between the acetyl group and the *N*-phenyl ring is 125.2(2)°. The torsion angle C11–N1–C10–C1 between the *N*-phenyl ring and naphthoquinone ring is –71.0(3)°. In the unit cell the molecules form layers via nonlinear intermolecular hydrogen bonds, and they are packed in a crossed arrangement when viewed along *a* axis (figure, bottom). The weak nonlinear intermolecular hydrogen bonds are formed between the C5–H5 of the naphthoquinone ring and one of the quinonic oxygens (O2) in the second molecule. The C–H···O bond length and bond angle are 3.255 Å and 128.82°, respectively, being in the range known from literature data [2-4].

Table 1. Data collection and handling.

Crystal:	yellow needle, size 0.1 × 0.1 × 0.3 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.71 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, ω/φ
$2\theta_{max}$:	50.7°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2715, 2715
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2153
$N(param)_{refined}$:	209
Programs:	SHELXS-97 [5], SHELXL-97 [6], SHELXTL-Plus [7]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(4)	4*e*	0.3200	0.0935	–0.1147	0.029
H(5)	4*e*	0.4592	0.0617	–0.1976	0.033
H(6)	4*e*	0.5526	0.1923	–0.2474	0.034
H(7)	4*e*	0.5064	0.3555	–0.2226	0.031
H(12)	4*e*	0.0830	0.6421	–0.0937	0.034
H(13)	4*e*	–0.0234	0.6888	–0.3349	0.040
H(14)	4*e*	–0.0083	0.6431	–0.6310	0.044
H(15)	4*e*	0.1155	0.5554	–0.6893	0.044
H(16)	4*e*	0.2252	0.5132	–0.4506	0.036
H(18A)	4*e*	0.2125	0.7403	0.0407	0.052
H(18B)	4*e*	0.2587	0.7331	–0.1368	0.052
H(18C)	4*e*	0.3155	0.7556	0.0530	0.052

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(1)	4*e*	0.10922(3)	0.38826(4)	0.01227(7)	0.0238(3)	0.0281(3)	0.0334(3)	0.0000(2)	0.0085(2)	−0.0014(2)
O(1)	4*e*	0.19456(9)	0.1960(1)	−0.0127(2)	0.0289(8)	0.0226(8)	0.0385(9)	−0.0043(6)	0.0069(6)	0.0034(6)
O(2)	4*e*	0.3949(1)	0.4975(1)	−0.1983(2)	0.0314(8)	0.0226(8)	0.058(1)	−0.0037(7)	0.0161(7)	0.0039(7)
O(3)	4*e*	0.3298(1)	0.5768(1)	0.1530(2)	0.0349(8)	0.0299(8)	0.0351(9)	0.0018(7)	−0.0061(7)	−0.0008(7)
N(1)	4*e*	0.2326(1)	0.5454(1)	−0.1003(2)	0.0236(9)	0.0172(9)	0.0276(9)	0.0016(7)	0.0001(7)	−0.0019(7)
C(1)	4*e*	0.2117(1)	0.3689(2)	−0.0552(3)	0.020(1)	0.023(1)	0.021(1)	0.0014(8)	0.0005(8)	−0.0002(8)
C(2)	4*e*	0.2409(1)	0.2626(2)	−0.0597(3)	0.024(1)	0.022(1)	0.018(1)	−0.0020(8)	−0.0017(8)	0.0010(8)
C(3)	4*e*	0.3292(1)	0.2439(2)	−0.1179(3)	0.024(1)	0.023(1)	0.018(1)	−0.0006(8)	−0.0010(8)	0.0004(8)
C(4)	4*e*	0.3571(1)	0.1460(2)	−0.1358(3)	0.029(1)	0.021(1)	0.023(1)	−0.0023(8)	0.0015(8)	0.0006(9)
C(5)	4*e*	0.4406(1)	0.1270(2)	−0.1853(3)	0.033(1)	0.023(1)	0.026(1)	0.0036(9)	0.0003(9)	−0.0024(9)
C(6)	4*e*	0.4964(1)	0.2055(2)	−0.2163(3)	0.025(1)	0.034(1)	0.027(1)	0.0050(9)	0.0020(8)	−0.0037(9)
C(7)	4*e*	0.4689(1)	0.3033(2)	−0.2012(3)	0.023(1)	0.029(1)	0.026(1)	−0.0024(9)	0.0021(8)	−0.0005(9)
C(8)	4*e*	0.3849(1)	0.3228(2)	−0.1539(3)	0.022(1)	0.022(1)	0.020(1)	0.0002(8)	0.0004(8)	0.0005(8)
C(9)	4*e*	0.3529(1)	0.4276(2)	−0.1509(3)	0.023(1)	0.022(1)	0.028(1)	−0.0024(9)	0.0039(8)	−0.0006(9)
C(10)	4*e*	0.2625(1)	0.4450(1)	−0.0962(3)	0.024(1)	0.018(1)	0.023(1)	0.0016(8)	0.0009(8)	−0.0007(8)
C(11)	4*e*	0.1651(1)	0.5741(2)	−0.2487(3)	0.026(1)	0.017(1)	0.027(1)	−0.0015(8)	0.0010(8)	0.0016(8)
C(12)	4*e*	0.0902(1)	0.6256(2)	−0.2135(3)	0.027(1)	0.029(1)	0.028(1)	0.0009(9)	0.0045(9)	0.0021(9)
C(13)	4*e*	0.0261(1)	0.6526(2)	−0.3579(3)	0.029(1)	0.027(1)	0.042(1)	0.0010(9)	0.0008(9)	0.008(1)
C(14)	4*e*	0.0354(2)	0.6259(2)	−0.5349(3)	0.041(1)	0.027(1)	0.037(1)	−0.007(1)	−0.012(1)	0.008(1)
C(15)	4*e*	0.1094(2)	0.5735(2)	−0.5697(3)	0.058(2)	0.025(1)	0.026(1)	−0.006(1)	−0.001(1)	−0.002(1)
C(16)	4*e*	0.1750(2)	0.5478(2)	−0.4267(3)	0.040(1)	0.020(1)	0.031(1)	0.0020(9)	0.0059(9)	−0.0019(9)
C(17)	4*e*	0.2792(1)	0.6106(2)	0.0242(3)	0.027(1)	0.024(1)	0.027(1)	−0.0004(9)	0.0049(9)	−0.0025(9)
C(18)	4*e*	0.2652(2)	0.7196(2)	−0.0076(3)	0.045(1)	0.022(1)	0.035(1)	−0.000(1)	−0.004(1)	−0.0058(9)

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