Crystal structure of 3-chloro-6-ethyl-5-hydroxy-7-methoxy-1,4-naphthoquinone, $C_{13}H_{11}ClO_4$

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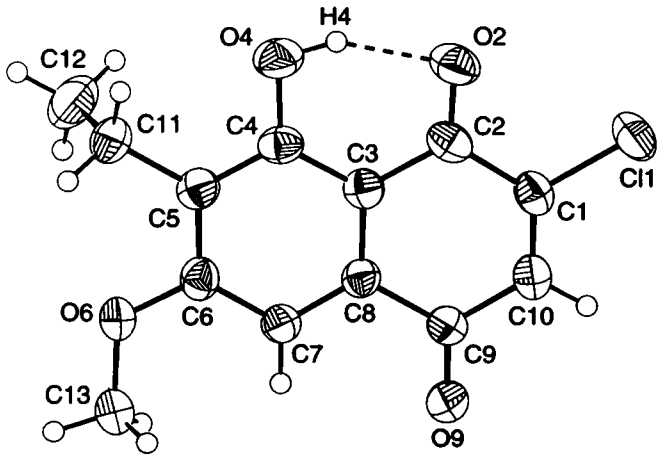

Abstract

$C_{13}H_{11}ClO_4$, monoclinic, $P12_1/c1$ (no. 14), $a = 11.322(2)$ Å, $b = 5.255(1)$ Å, $c = 19.718(2)$ Å, $\beta = 91.87(1)°$, $V = 1172.5$ Å^3, $Z = 4$, $R_{gt}(F) = 0.058$, $wR_{ref}(F^2) = 0.154$, $T = 293$ K.

Source of material

The title compound was prepared in analogy to the literature [1] by reacting 2,6-dichlorobenzoquinone with freshly prepared 1,3-dimethoxy-2-ethyl-1-(trimethylsiloxy)-1,3-diene.

Discussion

In the crystal the molecule is probably stabilized by a strong intramolecular hydrogen bond between the OH group O4—H4 and O2 of naphthoquinone kernel (d(H4···O2) = 1.787 Å, d(O4···O2) = 2.607 Å, ∠O4–H4···O2 = 150.5°).

Table 1. Data collection and handling.

Crystal:	orange lath, size 0.02 × 0.15 × 0.7 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.29 cm^{-1}
Diffractometer, scan mode:	Bruker AXS P4, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2814, 2683
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1601
$N(param)_{refined}$:	169
Programs:	SHELXS-97 [2], SHELXL-97 [3]

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

Atom	Site	x	y	z	U_{iso}
H(4)	4e	0.708(3)	0.059(8)	0.801(2)	0.08(1)
H(7)	4e	0.6403	0.8492	0.9706	0.08
H(10)	4e	0.2868	0.5011	0.8850	0.08
H(11A)	4e	0.9627	0.2555	0.8666	0.08
H(11B)	4e	0.9954	0.4632	0.9212	0.08
H(12A)	4e	1.0807	0.5689	0.8185	0.08
H(12B)	4e	0.9879	0.7780	0.8362	0.08
H(12C)	4e	0.9558	0.5698	0.7817	0.08
H(13A)	4e	0.9099	1.0596	1.0354	0.08
H(13B)	4e	0.7954	0.9033	1.0496	0.08
H(13C)	4e	0.7925	1.1158	0.9937	0.08

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)	4e	0.29636(7)	0.0894(2)	0.79931(4)	0.0550(5)	0.0636(6)	0.0533(5)	–0.0162(4)	–0.0089(4)	–0.0135(4)
C(1)	4e	0.3988(2)	0.2772(6)	0.8413(1)	0.040(2)	0.042(2)	0.036(2)	–0.006(1)	–0.005(1)	0.003(1)
C(2)	4e	0.5255(3)	0.2133(6)	0.8306(1)	0.052(2)	0.035(2)	0.031(1)	–0.002(1)	–0.004(1)	0.003(1)
O(2)	4e	0.5507(2)	0.0321(4)	0.7943(1)	0.061(1)	0.045(1)	0.050(1)	0.004(1)	–0.005(1)	–0.014(1)
C(3)	4e	0.6140(2)	0.3716(5)	0.8654(1)	0.038(1)	0.037(2)	0.032(1)	0.003(1)	–0.002(1)	0.004(1)
C(4)	4e	0.7356(2)	0.3290(6)	0.8567(1)	0.044(2)	0.038(2)	0.034(2)	0.008(1)	0.002(1)	0.000(1)
O(4)	4e	0.7734(2)	0.1415(5)	0.8158(1)	0.052(1)	0.052(2)	0.053(1)	0.015(1)	0.002(1)	–0.012(1)
C(5)	4e	0.8216(2)	0.4771(6)	0.8902(1)	0.039(1)	0.044(2)	0.036(2)	0.007(1)	–0.001(1)	0.003(1)
C(6)	4e	0.7838(2)	0.6689(6)	0.9334(1)	0.033(1)	0.045(2)	0.039(2)	0.002(1)	–0.003(1)	0.003(1)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(6)	4*e*	0.8718(2)	0.8031(5)	0.9660(1)	0.032(1)	0.066(2)	0.055(1)	−0.002(1)	−0.0024(9)	−0.018(1)
C(7)	4*e*	0.6637(2)	0.7189(6)	0.9421(1)	0.035(1)	0.043(2)	0.034(2)	0.003(1)	−0.001(1)	−0.002(1)
C(8)	4*e*	0.5812(2)	0.5721(6)	0.9078(1)	0.033(1)	0.039(2)	0.033(1)	0.004(1)	−0.000(1)	0.000(1)
C(9)	4*e*	0.4538(2)	0.6311(6)	0.9156(1)	0.038(2)	0.040(2)	0.037(2)	−0.000(1)	−0.000(1)	−0.004(1)
O(9)	4*e*	0.4219(2)	0.8103(5)	0.9494(1)	0.038(1)	0.068(2)	0.074(2)	0.002(1)	0.004(1)	−0.032(1)
C(10)	4*e*	0.3668(2)	0.4686(6)	0.8801(2)	0.036(1)	0.047(2)	0.046(2)	−0.004(1)	−0.003(1)	−0.000(2)
C(11)	4*e*	0.9510(2)	0.4321(7)	0.8791(2)	0.035(2)	0.061(2)	0.058(2)	0.012(2)	−0.002(1)	−0.007(2)
C(12)	4*e*	0.9982(3)	0.6030(9)	0.8238(2)	0.047(2)	0.095(3)	0.071(2)	0.008(2)	0.015(2)	−0.001(2)
C(13)	4*e*	0.8398(3)	0.9851(6)	1.0151(2)	0.039(2)	0.055(2)	0.051(2)	−0.005(2)	−0.002(1)	−0.007(2)

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