Crystal structure of α-(4-(hydroxyimino)-2,5-dichloro-2,5-cyclohexadien-1-ylidene)-benzeneacetonitrile, $C_{14}H_8Cl_2N_2O$

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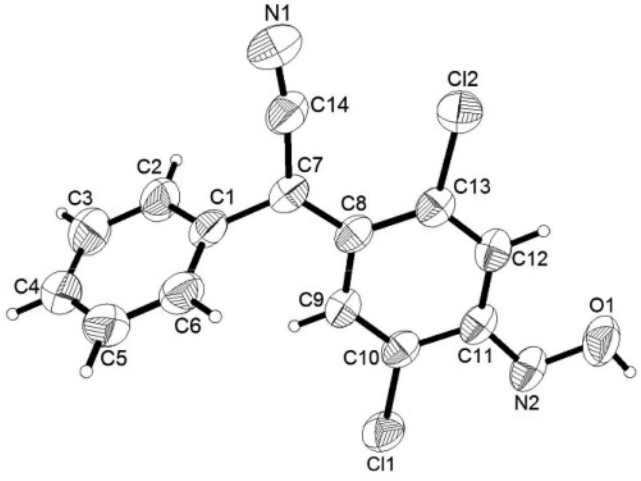

Abstract

$C_{14}H_8Cl_2N_2O$, monoclinic, $P2_1/c$ (no. 14), a = 18.692(4) Å, b = 5.8557(13) Å, c = 12.575(3) Å, β = 106.875(3)°, V = 1317.1 Å^3, Z = 4, $R_{gt}(F)$ = 0.0392, $wR_{ref}(F^2)$ = 0.1127, T = 296 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.42×0.45×0.50 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	4.84 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	54.93°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10842, 2971
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2175
$N(param)_{refined}$:	173
Programs:	SHELX [10], DIAMOND [11]

Source of material

The title compound was obtained according to the literature methods [1–3]. Colourless crystals suitable for X-ray diffraction were obtained by slow evaporation of the dichloromethane solution at room temperature.

Experimental details

Hydrogen atoms were placed geometrically and refined using a riding model with d(C–H) = 0.93 Å (aromatic), U_{iso}(H) = 1.2 U_{eq}(C) for CH groups. The hydroxyl group of oxime was idealized and refined using rigid group allowed to rotate about the N–O bond (AFIX 147 option of the SHELXL-2013 program [10]).

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Discussion

The oximes and their derivatives have been playing an important role in organic chemistry due to their various applications in deoxidant [4], anti-skinning agent [5], material of nylon resin and fibre [6], medicinal chemistry and are intermediates of organic synthesis [7]. Over the past decades, lots of arylcyanomethylenequinone oximes had been prepared, including 4-chloro-α-(2-chloro-4-(hydroxyimino)-5-methyl-2,5-cyclohexadien-1-ylidene)-benzeneacetonitrile, which was used as one of the key intermediates in the synthetic protocol of the synthesis of anthelmintic Closantel sodium [8, 9]. Taking these results into account, we report the synthesis and crystal structure of the arylcyanomethylenequinone oxime compound, α-(4-(hydroxyimino)-2,5-dichloro-2,5-cyclohexadien-1-ylidene)benzeneacetonitrile. The molecular structure of the title compound is given in the figure, in which all bond lengths are in normal ranges. The C10–Cl1 bond length is 1.728(2) Å. The N1, C14 and C7 atoms form a bond angle of 171.0(2)°. The molecular conformation is stabilized by an inter-molecular O–H···N hydrogen bond (O1–H1···N2^i): d(H1···N2^i) = 2.16 Å, d(O1···N2^i) = 2.817(2) Å, and ∠O1–H1···N2^i = 137° (Symmetry code i: –x+2, –y–1, –z+2).

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	0.6701	0.5553	0.7337	0.075
H(3)	4*e*	0.5761	0.5041	0.5703	0.088
H(4)	4*e*	0.4971	0.1938	0.5491	0.084
H(5)	4*e*	0.5148	–0.0713	0.6902	0.081
H(6)	4*e*	0.6101	–0.0262	0.8520	0.068
H(9)	4*e*	0.7424	–0.0246	0.7945	0.053
H(12)	4*e*	0.9364	0.0383	1.1244	0.058
H(1)	4*e*	1.0346	–0.3998	1.1029	0.106

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}
C(1)	4*e*	0.6507(1)	0.2691(3)	0.8102(2)	0.0435(9)	0.038(1)	0.060(1)	0.0100(8)	0.0164(8)	0.0054(9)
C(2)	4*e*	0.6392(1)	0.4278(4)	0.7250(2)	0.058(1)	0.047(1)	0.075(1)	0.0015(9)	0.007(1)	0.015(1)
C(3)	4*e*	0.5826(1)	0.3982(4)	0.6275(2)	0.079(2)	0.061(2)	0.070(2)	0.010(1)	0.004(1)	0.019(1)
C(4)	4*e*	0.5358(1)	0.2125(4)	0.6145(2)	0.068(1)	0.058(1)	0.070(1)	0.011(1)	–0.001(1)	–0.004(1)
C(5)	4*e*	0.5463(1)	0.0545(4)	0.6988(2)	0.060(1)	0.052(1)	0.086(2)	–0.003(1)	0.014(1)	–0.003(1)
C(6)	4*e*	0.6033(1)	0.0817(4)	0.7957(2)	0.056(1)	0.043(1)	0.071(1)	0.0031(9)	0.019(1)	0.008(1)
C(7)	4*e*	0.7134(1)	0.2992(3)	0.9147(2)	0.0461(9)	0.037(1)	0.055(1)	0.0062(8)	0.0195(8)	0.0064(8)
C(8)	4*e*	0.7754(1)	0.1606(3)	0.9405(2)	0.0456(9)	0.037(1)	0.046(1)	0.0056(7)	0.0203(8)	0.0091(8)
C(9)	4*e*	0.7802(1)	–0.0149(3)	0.8614(2)	0.0453(9)	0.045(1)	0.044(1)	0.0083(8)	0.0160(8)	0.0070(8)
C(10)	4*e*	0.8371(1)	–0.1637(3)	0.8813(2)	0.050(1)	0.041(1)	0.048(1)	0.0079(8)	0.0224(8)	0.0060(8)
C(11)	4*e*	0.8984(1)	–0.1558(3)	0.9828(2)	0.0459(9)	0.046(1)	0.050(1)	0.0120(8)	0.0216(8)	0.0112(8)
C(12)	4*e*	0.8969(1)	0.0239(3)	1.0597(2)	0.046(1)	0.055(1)	0.046(1)	0.0092(9)	0.0162(8)	0.0105(9)
C(13)	4*e*	0.8395(1)	0.1718(3)	1.0401(2)	0.050(1)	0.044(1)	0.044(1)	0.0064(8)	0.0209(8)	0.0074(8)
C(14)	4*e*	0.7007(1)	0.4871(4)	0.9803(2)	0.053(1)	0.049(1)	0.069(1)	0.013(1)	0.016(1)	0.001(1)
Cl(1)	4*e*	0.83904(3)	–0.37152(9)	0.78467(5)	0.0677(3)	0.0564(3)	0.0636(3)	0.0178(2)	0.0215(3)	–0.0071(2)
Cl(2)	4*e*	0.84366(3)	0.3817(1)	1.13808(5)	0.0729(4)	0.0627(4)	0.0561(3)	0.0135(3)	0.0140(3)	–0.0105(2)
N(1)	4*e*	0.6819(1)	0.6411(4)	1.0216(2)	0.087(2)	0.069(1)	0.101(2)	0.029(1)	0.021(1)	–0.018(1)
N(2)	4*e*	0.94986(9)	–0.3124(3)	0.9958(1)	0.0495(9)	0.059(1)	0.058(1)	0.0193(8)	0.0177(8)	0.0130(8)
O(1)	4*e*	1.00565(8)	–0.2919(3)	1.0956(1)	0.0572(9)	0.081(1)	0.0677(9)	0.0301(8)	0.0072(7)	0.0084(9)

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