Crystal structure of 4-chloro-3'-methoxy-[1,1'-biphenyl]-3-carbaldehyde, $C_{14}H_{11}ClO_2$

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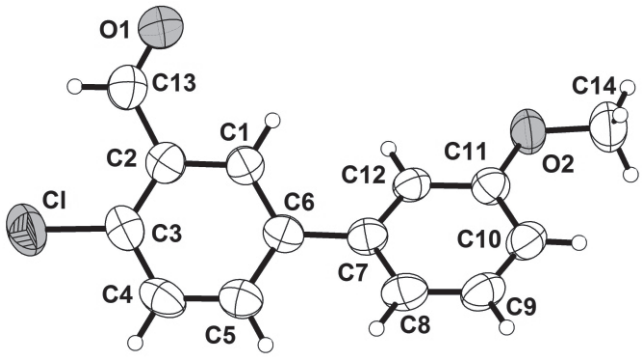

Abstract

$C_{14}H_{11}ClO_2$, orthorhombic, $P2_12_12_1$ (no. 19), a = 4.1210(8) Å, b = 11.363(2) Å, c = 24.982(5) Å, V = 1169.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.0572, $wR_{ref}(F^2)$ = 0.1287, T = 293 K.

Table 1. Data collection and handling.

Crystal:	colourless needles, size 0.10×0.10×0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.11 cm^{-1}
Diffractometer, scan mode:	Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	50.72°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2543, 2130
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1336
$N(param)_{refined}$:	154
Programs:	SHELX [2]

Source of material

To a mixture of 3-methoxybenzyl boronic acid (2.863 g, 18.8 mmol), 5-bromo-2-chlorobenzaldehyde (4.0 g, 18.4 mmol), Pd(0)(PPh_3)$_4$ (0.698 g, 0.6 mmol) and sodium carbonate (2.430 g, 22.9 mmol) protected by a N_2 atmosphere was added degassed THF (125 ml), toluene (125 ml) and H_2O (4 ml). This was heated to 98 °C and stirred for 27 h. The solution was allowed to cool, then condensed under reduced pressure and re-dissolved in DCM (200 ml). The dichlormethane (DCM) solution was washed with 10% $NaHCO_3$(aq) (100 ml), brine (100 ml), dried with Na_2SO_4, condensed under reduced pressure and purified on silica eluting with DCM/Hexane. The product was isolated as a clear oil: yield 1.958 g (43.27%). **Elemental analysis** calcd. for $C_{14}H_{11}Cl_1O_2$: C, 68.16; H, 4.49; Cl, 14.37; O, 12.97%; found: C, 68.18; H, 4.47; N, 14.35; O, 12.99%.

Experimental details

In the crystal, H atoms were positioned geometrically with C–H = 0.93 Å for aromatic, and methyl, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 (or 1.5 for methyl groups) times U_{eq}(C). According to the quality of the crystals and the low redundancy of the data collection strategy the absolute structure could not be reliable determined.

Discussion

4-chloro-3'-methoxy-[1,1'-biphenyl]-3-carbaldehyde is an important synthon for the modification of porphyrin [1]. Until now, there are no synthesized of this Structure. All bond lengths and angels are in the expected ranges. The two rings are twisted against each other by roughly 30°. The shortest intermolecular O···H distance falls with 2.69 Å in the range of the O/H van der Waals distance. Therefore, significant non-classical hydrogen bonds can be ruled out.

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4*a*	0.6284	0.4591	0.6510	0.056
H(4A)	4*a*	0.0417	0.7377	0.5573	0.071
H(5A)	4*a*	0.2684	0.7836	0.6387	0.066
H(8A)	4*a*	0.6810	0.8327	0.6920	0.065
H(9A)	4*a*	0.9290	0.8768	0.7718	0.068
H(10A)	4*a*	0.9826	0.7388	0.8381	0.064
H(12A)	4*a*	0.5766	0.4979	0.7415	0.052
H(13A)	4*a*	0.3373	0.3459	0.5282	0.078
H(14A)	4*a*	0.9888	0.4679	0.9022	0.107
H(14B)	4*a*	0.8869	0.6006	0.8987	0.107
H(14C)	4*a*	1.2112	0.5582	0.8721	0.107

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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	4*a*	0.0421(3)	0.5407(1)	0.49180(4)	0.0771(9)	0.104(1)	0.0464(6)	0.0120(8)	–0.0059(6)	0.0042(6)
C(1)	4*a*	0.510(1)	0.5153(3)	0.6323(2)	0.047(2)	0.046(2)	0.048(2)	–0.003(2)	–0.001(2)	0.002(2)
O(1)	4*a*	0.630(1)	0.2976(3)	0.5795(1)	0.126(4)	0.060(2)	0.077(2)	0.018(2)	–0.026(2)	–0.008(2)
O(2)	4*a*	0.8222(9)	0.5137(2)	0.8317(1)	0.089(2)	0.054(2)	0.047(2)	–0.005(2)	–0.019(2)	0.002(1)
C(2)	4*a*	0.379(1)	0.4864(4)	0.5824(2)	0.049(3)	0.056(2)	0.044(2)	–0.001(2)	0.009(2)	0.003(2)
C(3)	4*a*	0.210(1)	0.5691(4)	0.5539(2)	0.038(2)	0.071(3)	0.043(2)	–0.002(2)	0.005(2)	0.008(2)
C(4)	4*a*	0.162(1)	0.6816(4)	0.5758(2)	0.058(3)	0.067(3)	0.053(3)	0.018(3)	0.009(2)	0.020(2)
C(5)	4*a*	0.295(1)	0.7082(4)	0.6250(2)	0.056(3)	0.051(3)	0.059(3)	0.003(2)	0.003(2)	0.007(2)
C(6)	4*a*	0.466(1)	0.6263(3)	0.6544(2)	0.047(3)	0.041(2)	0.049(2)	–0.005(2)	0.005(2)	0.010(2)
C(7)	4*a*	0.608(1)	0.6579(3)	0.7070(2)	0.046(3)	0.045(2)	0.051(3)	0.001(2)	0.005(2)	0.000(2)
C(8)	4*a*	0.711(1)	0.7741(3)	0.7175(2)	0.064(3)	0.035(2)	0.065(3)	–0.005(2)	0.009(3)	0.008(2)
C(9)	4*a*	0.855(1)	0.8006(4)	0.7659(2)	0.056(3)	0.041(2)	0.074(3)	–0.004(2)	–0.001(3)	–0.012(2)
C(10)	4*a*	0.893(1)	0.7183(4)	0.8053(2)	0.053(3)	0.051(3)	0.057(3)	0.004(2)	0.001(2)	–0.011(2)
C(11)	4*a*	0.796(1)	0.6042(3)	0.7953(2)	0.048(3)	0.042(2)	0.050(3)	–0.002(2)	–0.001(2)	0.000(2)
C(12)	4*a*	0.649(1)	0.5746(3)	0.7470(2)	0.045(2)	0.035(2)	0.050(2)	0.000(2)	0.000(2)	–0.003(2)
C(13)	4*a*	0.445(1)	0.3674(4)	0.5595(2)	0.080(4)	0.065(3)	0.050(3)	0.004(3)	–0.004(3)	–0.003(2)
C(14)	4*a*	0.991(1)	0.5370(4)	0.8801(2)	0.088(4)	0.075(3)	0.052(3)	–0.005(4)	–0.016(3)	–0.001(2)

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