Crystal structure of dimethyl 4-(4-chlorophenyl)-1,4-dihydropyridine-3,5-dicarboxylate, $C_{15}H_{14}ClNO_4$

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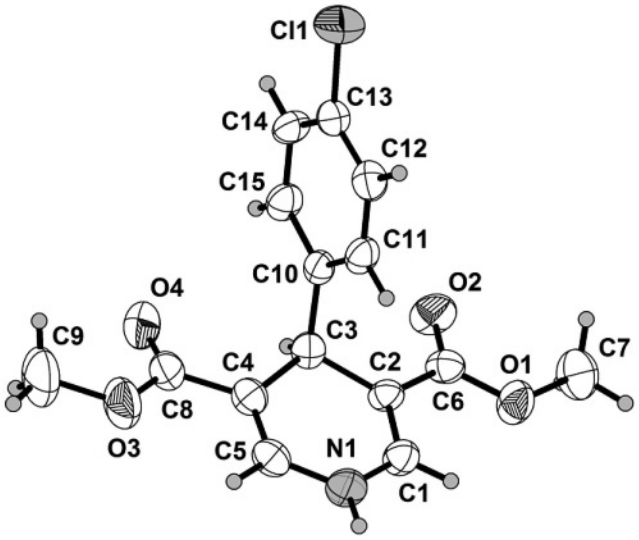
Received November 22, 2013, accepted March 17, 2014, available online April 10, 2014, CCDC no. 1267/4076



Abstract

$C_{15}H_{14}ClNO_4$, monoclinic, $P2_1/c$ (no. 14), a = 8.3515(6) Å, b = 7.4351(5) Å, c = 23.931(2) Å, β = 96.655(1)°, V = 1476.0 Å^3, Z = 4, $R_{gt}(F)$ = 0.0503, $wR_{ref}(F^2)$ = 0.1412, T = 298 K.

Table 1. Data collection and handling.

Crystal:	colourless irregular, size 0.37×0.39×0.43 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.73 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7109, 2601
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1422
$N(param)_{refined}$:	192
Programs:	SHELX [3], DIAMOND [4]

Source of material

Dimethyl 4-(4-chlorophenyl)-1,4-dihydropyridine-3,5-dicarboxylate is easily available by a literature known synthesis [1]. A mixture of 4-chlorobenzaldehyde (1 mmol), methyl propiolate (2 mmol), ammonium acetate (10 mmol) and acetic acid (1 ml) were warmed together on a steam-bath for 10-15 min. The product crystallized on cooling; it was filtered off and crystallized from methanol.

Discussion

The title compound was synthesized to be used as an organic calcium channel modulator [1, 2]. The values of the geometric parameters of the title structure are within normal ranges and experimental errors. In the crystal structure, weak intermolecular N–H···O interactions link the molecules (chains along the b direction), in which they may be effective in the stabilization of the structure. **IR** (KBr, cm^{-1}): 3331, 2985, 2922, 1598, 1487, 1360, 872, 758. **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm): 3.53 (s, 6H, OCH_3), 4.92 (s, 1H, CH), 6.35 (brs, 1H, NH), 7.32 (s, 1H, CH), 7.34 (s, 1H, CH), 7.51 (d, 2H, J=8.2Hz, Ar-H), 7.53(d, 2H, J=8.2Hz, Ar-H). **MS**: 308 ($M+H^+$). **Elemental Anal.** calcd for $C_{15}H_{14}ClNO_4$: C, 58.55; H, 4.59; N, 4.55 %. Found: C, 58.49; H, 4.58; N, 4.55.

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.2483	0.1721	0.5050	0.074
H(1A)	4*e*	0.3061	0.4214	0.4594	0.061
H(3)	4*e*	0.0737	0.6967	0.5645	0.054
H(5)	4*e*	0.1160	0.1726	0.5813	0.065
H(7A)	4*e*	0.2885	0.9474	0.3946	0.132
H(7B)	4*e*	0.4396	0.8465	0.3769	0.132
H(7C)	4*e*	0.4540	0.9585	0.4327	0.132
H(9A)	4*e*	–0.0317	0.3384	0.7380	0.179
H(9B)	4*e*	–0.0962	0.1459	0.7207	0.179
H(9C)	4*e*	–0.1942	0.3165	0.6986	0.179
H(11)	4*e*	0.4758	0.5351	0.5987	0.059
H(12)	4*e*	0.6653	0.6482	0.7136	0.067
H(14)	4*e*	0.3403	0.9946	0.7136	0.068
H(15)	4*e*	0.1516	0.8849	0.6438	0.061

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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(1)	4*e*	0.6584(1)	0.9065(2)	0.75121(4)	0.0916(8)	0.1015(9)	0.0756(7)	–0.0253(7)	–0.0062(6)	–0.0201(6)
N(1)	4*e*	0.2231(4)	0.2741(3)	0.5185(1)	0.085(2)	0.032(2)	0.069(2)	–0.001(1)	0.020(2)	–0.004(1)
O(1)	4*e*	0.3423(3)	0.7248(3)	0.4386(1)	0.095(2)	0.054(2)	0.058(2)	–0.005(1)	0.028(1)	0.007(1)
O(2)	4*e*	0.2353(3)	0.9034(3)	0.4992(1)	0.115(2)	0.034(1)	0.068(2)	0.003(1)	0.019(2)	0.000(1)
O(3)	4*e*	–0.0009(3)	0.2729(4)	0.6596(1)	0.091(2)	0.092(2)	0.074(2)	–0.021(2)	0.024(2)	0.024(2)
O(4)	4*e*	–0.0416(3)	0.5699(4)	0.6546(1)	0.079(2)	0.095(2)	0.086(2)	–0.011(2)	0.040(2)	–0.012(2)
C(1)	4*e*	0.2602(4)	0.4284(4)	0.4929(1)	0.065(2)	0.042(2)	0.047(2)	–0.000(2)	0.012(2)	–0.001(2)
C(2)	4*e*	0.2333(4)	0.5900(4)	0.5140(1)	0.056(2)	0.033(2)	0.041(2)	–0.001(2)	0.005(1)	–0.002(1)
C(3)	4*e*	0.1667(4)	0.6156(4)	0.5699(1)	0.048(2)	0.038(2)	0.051(2)	0.006(2)	0.010(2)	0.002(2)
C(4)	4*e*	0.1115(4)	0.4331(4)	0.5900(1)	0.050(2)	0.045(2)	0.052(2)	–0.001(2)	0.005(2)	0.006(2)
C(5)	4*e*	0.1458(4)	0.2803(4)	0.5657(1)	0.059(2)	0.039(2)	0.063(2)	–0.005(2)	0.007(2)	0.005(2)
C(6)	4*e*	0.2681(4)	0.7536(4)	0.4841(1)	0.059(2)	0.042(2)	0.049(2)	–0.002(2)	0.001(2)	–0.002(2)
C(7)	4*e*	0.3847(5)	0.8826(5)	0.4081(2)	0.121(4)	0.072(3)	0.073(3)	–0.021(3)	0.025(3)	0.026(2)
C(8)	4*e*	0.0158(4)	0.4358(6)	0.6374(2)	0.051(2)	0.074(3)	0.055(2)	–0.014(2)	0.009(2)	–0.001(2)
C(9)	4*e*	–0.0881(6)	0.2680(8)	0.7083(2)	0.124(4)	0.156(5)	0.084(3)	–0.043(4)	0.041(3)	0.027(3)
C(10)	4*e*	0.2929(4)	0.6957(4)	0.6142(1)	0.050(2)	0.034(2)	0.044(2)	–0.000(1)	0.014(2)	0.001(1)
C(11)	4*e*	0.4475(4)	0.6276(4)	0.6218(1)	0.059(2)	0.042(2)	0.050(2)	0.005(2)	0.018(2)	–0.004(2)
C(12)	4*e*	0.5611(4)	0.6943(5)	0.6631(1)	0.050(2)	0.061(2)	0.058(2)	–0.002(2)	0.012(2)	0.002(2)
C(13)	4*e*	0.5187(4)	0.8288(5)	0.6974(1)	0.063(2)	0.054(2)	0.047(2)	–0.015(2)	0.013(2)	0.000(2)
C(14)	4*e*	0.3671(5)	0.9015(4)	0.6904(1)	0.076(3)	0.047(2)	0.049(2)	–0.008(2)	0.017(2)	–0.014(2)
C(15)	4*e*	0.2545(4)	0.8353(4)	0.6488(1)	0.057(2)	0.044(2)	0.054(2)	0.008(2)	0.014(2)	–0.005(2)

Acknowledgments. This work was supported financially by Scientific Research and Development Guidance Plan Project of Shijiazhuang No. 12120453.

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