Crystal structure of 1-(4-chlorophenyl)-2-(5-methyl-1,3,4-thiadiazol-2-yl)ethanone, $C_{11}H_9ClN_2OS$

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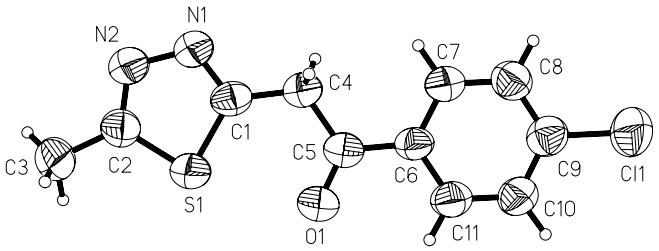

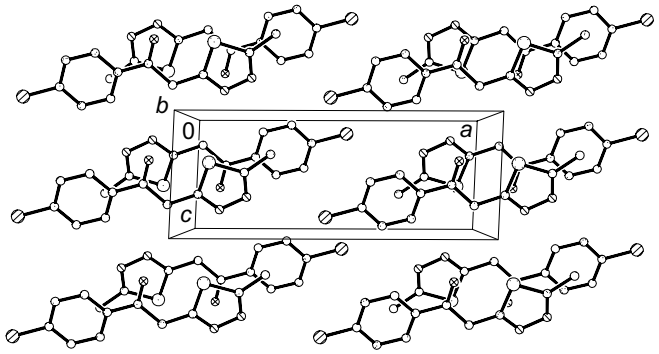

Abstract

$C_{11}H_9ClN_2OS$, monoclinic, $P12_1/c1$ (no. 14), a = 14.102(2) Å, b = 14.595(1) Å, c = 5.5101(5) Å, β = 92.309(9)°, V = 1133.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.085, $wR_{ref}(F^2)$ = 0.247, T = 293 K.

Source of material

The title compound was prepared from 2,5-dimethyl-1,3,4-thiadiazole, ethyl 4-chlorochenzoate and sodium hydride [1]. In solution ($CDCl_3$) the compound is in equilibrium with its *enol* form, in the solid state the compound exits completely as its *keto* isomer. After crystallization from ethanol the title compound forms yellow crystals (m.p. 436-437 K).

Experimental details

The large R values are caused by the poor quality of the crystals and by the omitted absorption correction.

Discussion

The molecule shows a perfectly flat conformation (figure, top). The r.m.s. deviation is 0.042 Å for all non-hydrogen atoms. The *a*,*b* view of the cell plot (figure, bottom) shows a layer-type stacking pattern with alternating polar and non-polar regions. The chlorine atoms form polar layers parallel to the *b*,*c* plane, followed by a non-polar layer built up by the phenyl moieties. Another polar layer is formed in the same way by the carbonyl functions and the thiadiazole moieties. The stacking vector is along the *a* axis.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.15 × 0.25 × 0.6 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	45.38 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	135.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2164, 1953
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1302
$N(param)_{refined}$:	182
Programs:	SHELXS-97 [2], SHELXL-97 [3], SHELXTL-plus [4]

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

Atom	Site	x	y	z	U_{iso}
H(3A)	4*e*	0.663(5)	0.106(5)	0.14(2)	0.12(3)
H(3B)	4*e*	0.736(6)	0.142(5)	0.35(2)	0.14(3)
H(3C)	4*e*	0.714(6)	0.213(6)	0.17(2)	0.15(3)
H(4A)	4*e*	1.046(4)	0.143(4)	–0.37(1)	0.09(2)
H(4B)	4*e*	1.057(6)	0.045(6)	–0.32(1)	0.14(3)
H(7)	4*e*	1.199(4)	0.054(3)	–0.463(9)	0.07(1)
H(8)	4*e*	1.361(4)	0.053(4)	–0.57(1)	0.10(2)
H(10)	4*e*	1.442(5)	0.180(4)	0.05(1)	0.10(2)
H(11)	4*e*	1.275(5)	0.198(5)	0.14(1)	0.11(2)

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}
S(1)	4*e*	0.9124(1)	0.1579(1)	0.0981(2)	0.0749(9)	0.099(1)	0.0603(8)	–0.0064(7)	0.0028(6)	–0.0155(6)
Cl(1)	4*e*	1.5291(1)	0.1172(1)	–0.3558(4)	0.068(1)	0.133(2)	0.132(2)	0.0087(8)	0.0062(9)	–0.027(1)
N(1)	4*e*	0.8775(3)	0.0742(4)	–0.2952(8)	0.068(3)	0.114(4)	0.076(3)	–0.017(2)	0.003(2)	–0.028(3)

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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(1)	4*e*	1.1056(3)	0.1709(3)	0.1158(7)	0.080(2)	0.125(3)	0.064(2)	–0.003(2)	0.002(2)	–0.029(2)
C(1)	4*e*	0.9472(4)	0.1088(3)	–0.1665(8)	0.075(3)	0.062(3)	0.052(2)	–0.007(2)	–0.005(2)	0.004(2)
N(2)	4*e*	0.7903(3)	0.0849(4)	–0.1895(9)	0.070(3)	0.120(4)	0.074(3)	–0.016(3)	0.003(2)	–0.024(3)
C(2)	4*e*	0.7986(4)	0.1279(4)	0.0163(9)	0.072(3)	0.077(3)	0.060(3)	0.001(2)	0.003(2)	0.001(2)
C(3)	4*e*	0.7152(5)	0.1494(6)	0.168(1)	0.075(4)	0.113(5)	0.075(4)	–0.001(4)	0.014(3)	–0.003(3)
C(4)	4*e*	1.0458(4)	0.1045(5)	–0.253(1)	0.068(3)	0.079(4)	0.059(3)	–0.006(3)	–0.002(2)	–0.009(3)
C(5)	4*e*	1.1244(4)	0.1367(3)	–0.0809(8)	0.074(3)	0.069(3)	0.054(3)	0.000(2)	0.001(2)	–0.004(2)
C(6)	4*e*	1.2235(3)	0.1283(3)	–0.1514(8)	0.067(3)	0.068(3)	0.050(2)	–0.001(2)	–0.006(2)	–0.005(2)
C(7)	4*e*	1.2484(4)	0.0864(4)	–0.3668(9)	0.072(3)	0.077(3)	0.061(3)	–0.003(2)	–0.005(2)	–0.009(2)
C(8)	4*e*	1.3428(4)	0.0816(4)	–0.427(1)	0.076(3)	0.082(4)	0.070(3)	0.009(3)	0.003(3)	–0.006(3)
C(9)	4*e*	1.4118(4)	0.1180(4)	–0.274(1)	0.071(3)	0.073(3)	0.081(3)	0.006(2)	–0.006(2)	–0.004(3)
C(10)	4*e*	1.3888(4)	0.1599(4)	–0.058(1)	0.065(3)	0.094(4)	0.076(3)	0.003(3)	–0.009(3)	–0.012(3)
C(11)	4*e*	1.2947(4)	0.1645(4)	0.001(1)	0.074(3)	0.081(4)	0.066(3)	0.001(3)	–0.007(2)	–0.012(3)

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