Crystal structure of 1,2,4-trizolophenyl-(2-chloroacetamide-benzenethio)-4-chlorophenylacetone, $C_{25}H_{20}Cl_2N_4O_2S$

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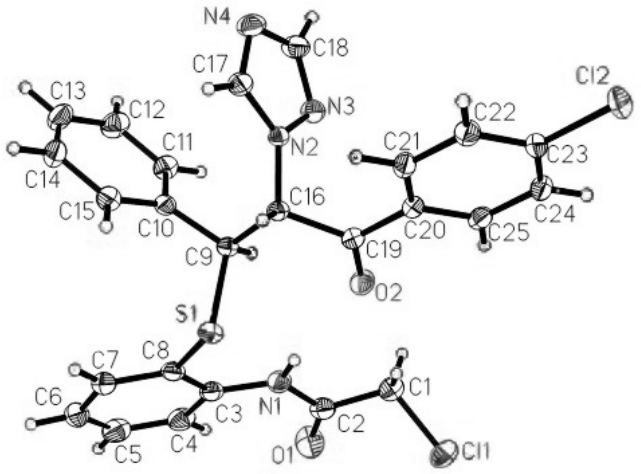

Abstract

$C_{25}H_{20}Cl_2N_4O_2S$, triclinic, $P\bar{1}$ (no. 2), a = 8.3869(3) Å, b = 11.3537(4) Å, c = 13.8221(5) Å, α = 110.927(1)°, β = 102.585(1)°, γ = 97.404(1)°, V = 1168.6 Å^3, Z = 2, $R_{gt}(F)$ = 0.032, $wR_{ref}(F^2)$ = 0.094, T = 153 K.

Source of material

To a solution of 1.2 g (0.004 mol) 1-(4-chloro-phenyl)-3- phenyl-2-(1,2,4-trizolo-1-yl)-2-2propyleno-1-one [1] in 20 ml methanol was added 0.5 g (0.004 mol) of *o*-aminothiophenol. The mixture was heated under reflux for 30 minutes. Added dropwise of 1.2 ml CF_3COOH into the mixture when it clears, the refluxing was continued for 3 hours. Then the solution was allowed to stand at room temperature for night. The crystalline solid product 2-phenyl-3-(1,2,4-trizolo)-4-(4-chloro-phenyl)-[1,5]benzothiaze-pine was filtered, washed with 2-3 ml of cool methanol and dried, then recrystallized from methanol. 0.2 g (1.5 mmol) chloracetyl chloride was added under stirring to a solution of 0.8 g (1.5 mmol) 2-phenyl-3-(1,2,4-trizolo)-4-(4-chloro-phenyl)-[1,5]benzothiaze-pine in 30 ml methylene chloride, and a solution of 0.151 g (1.5 mmol) triethylamine in the same solvent (5 ml) was added dropwise slowly. The reaction mixture was continued under stirring for 48 hours at room temperature. After complete reaction (by TLC monitoring), the solvent was evaporated at reduced pressure. The residue was purified by silica gel column chromatography with ethyl acetate/light petroleum (v/v = 1:8). Then the colorless crystals of title compound were obtained.

Experimental details

The H atoms bonded to C were positioned geometrically and refined using a riding model. The H atom bonded to N atom was found by difference Fourier methods and refined with N—H distance restrained to 0.84(2) Å.

Discussion

Triazole derivatives are used in the synthesis of antibiotics, herbicides, fungicides, plant growth hormone regulators, and potentially good corrosion inhibitors [2,3]. The 1,2,4-triazole system is also of magnetochemical interest because it is able to act as a bridge between metal centres and mediate exchange coupling [4]. The crystal structure of the title compound is built up by the $C_{25}H_{20}Cl_2N_4O_2S$ molecules, in which the bond lengths are in normal ranges. The C19—O2 (1.213(2) Å) and N1—H (0.84(2) Å) bonds are as same as the normal bonds, so hydrogen bonds are not formed, the C19—O2 and N1—H bonds are not in identical plane. The molecules are interlinked by O···H, N···H, Cl···Cl and Cl···H what generated three-dimensional network.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.40 × 0.50 × 0.60 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.99 cm^{-1}
Diffractometer, scan mode:	Rigaku R-AXIS Spider, ω
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11550, 5301
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4760
$N(param)_{refined}$:	311
Programs:	SHELXS-97 [4], SHELXL-97 [5], DIAMOND [6], ORTEP [7]

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

Atom	Site	x	y	z	U_{iso}
H(1B)	2*i*	0.3316	1.1379	0.4156	0.042
H(1C)	2*i*	0.1863	1.1690	0.3381	0.042
H(4B)	2*i*	0.4061	0.9580	0.0636	0.036
H(5A)	2*i*	0.5086	0.8032	–0.0540	0.041
H(6A)	2*i*	0.5924	0.6350	–0.0119	0.040
H(7A)	2*i*	0.5567	0.6139	0.1447	0.033
H(9A)	2*i*	0.1727	0.7659	0.2703	0.021
H(11A)	2*i*	–0.0550	0.6109	0.1428	0.030
H(12A)	2*i*	–0.1902	0.3981	0.0241	0.039
H(13A)	2*i*	–0.0565	0.2295	0.0218	0.040
H(14A)	2*i*	0.2109	0.2728	0.1371	0.037
H(15A)	2*i*	0.3459	0.4852	0.2574	0.030
H(16A)	2*i*	0.2980	0.6936	0.4463	0.021
H(17A)	2*i*	0.0909	0.4754	0.4025	0.028
H(18A)	2*i*	–0.3289	0.5840	0.3437	0.033
H(21A)	2*i*	0.2285	0.7336	0.6067	0.026
H(22A)	2*i*	0.1840	0.7929	0.7781	0.029
H(24A)	2*i*	0.0929	1.1322	0.7688	0.030
H(25A)	2*i*	0.1445	1.0756	0.6000	0.026
H(1A)	2*i*	0.362(2)	0.960(2)	0.309(2)	0.032(5)

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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)	2*i*	0.41725(5)	1.34014(3)	0.41755(3)	0.0420(2)	0.0228(2)	0.0373(2)	0.0021(2)	0.0070(2)	0.0124(2)
Cl(2)	2*i*	0.09551(5)	1.00935(4)	0.91202(3)	0.0529(2)	0.0319(2)	0.0279(2)	0.0047(2)	0.0253(2)	0.0053(2)
S(1)	2*i*	0.45790(4)	0.76289(3)	0.32315(2)	0.0176(2)	0.0246(2)	0.0207(2)	0.0024(1)	0.0055(1)	0.0102(1)
O(1)	2*i*	0.3915(2)	1.1420(1)	0.1988(1)	0.0604(8)	0.0364(6)	0.0454(7)	0.0194(6)	0.0257(6)	0.0276(5)
O(2)	2*i*	0.2403(1)	0.94822(9)	0.44265(8)	0.0391(6)	0.0187(4)	0.0266(5)	0.0047(4)	0.0138(4)	0.0111(4)
N(1)	2*i*	0.3738(2)	0.9768(1)	0.2562(1)	0.0293(6)	0.0226(6)	0.0241(6)	0.0025(5)	0.0102(5)	0.0107(5)
N(2)	2*i*	0.0465(1)	0.6450(1)	0.39600(8)	0.0190(5)	0.0164(5)	0.0193(5)	0.0045(4)	0.0077(4)	0.0084(4)
N(3)	2*i*	–0.0982(1)	0.6855(1)	0.3737(1)	0.0211(5)	0.0220(5)	0.0307(6)	0.0072(4)	0.0091(5)	0.0124(5)
N(4)	2*i*	–0.1528(2)	0.4829(1)	0.3695(1)	0.0280(6)	0.0238(6)	0.0410(7)	0.0041(5)	0.0129(5)	0.0178(5)
C(1)	2*i*	0.3085(2)	1.1751(1)	0.3606(1)	0.0389(8)	0.0218(7)	0.0441(9)	0.0029(6)	0.0191(7)	0.0104(6)
C(2)	2*i*	0.3638(2)	1.0982(1)	0.2629(1)	0.0240(7)	0.0256(7)	0.0320(7)	0.0036(5)	0.0077(6)	0.0132(6)
C(3)	2*i*	0.4260(2)	0.8823(1)	0.1787(1)	0.0209(6)	0.0232(6)	0.0221(6)	–0.0015(5)	0.0060(5)	0.0080(5)
C(4)	2*i*	0.4389(2)	0.8896(2)	0.0815(1)	0.0349(8)	0.0289(7)	0.0256(7)	–0.0024(6)	0.0085(6)	0.0128(6)
C(5)	2*i*	0.4996(2)	0.7971(2)	0.0114(1)	0.0419(9)	0.0352(8)	0.0215(7)	–0.0063(7)	0.0126(6)	0.0084(6)
C(6)	2*i*	0.5472(2)	0.6960(2)	0.0354(1)	0.0360(8)	0.0314(7)	0.0273(7)	–0.0008(6)	0.0170(6)	0.0026(6)
C(7)	2*i*	0.5283(2)	0.6848(1)	0.1292(1)	0.0264(7)	0.0245(6)	0.0287(7)	0.0009(6)	0.0113(6)	0.0069(5)
C(8)	2*i*	0.4678(2)	0.7768(1)	0.2008(1)	0.0187(6)	0.0237(6)	0.0212(6)	–0.0008(5)	0.0071(5)	0.0082(5)
C(9)	2*i*	0.2311(2)	0.7066(1)	0.2960(1)	0.0166(5)	0.0185(6)	0.0183(5)	0.0025(5)	0.0057(4)	0.0082(5)
C(10)	2*i*	0.1584(2)	0.5694(1)	0.2130(1)	0.0215(6)	0.0200(6)	0.0166(5)	0.0027(5)	0.0079(5)	0.0076(5)
C(11)	2*i*	–0.0008(2)	0.5423(1)	0.1425(1)	0.0255(7)	0.0246(6)	0.0228(6)	0.0040(5)	0.0048(5)	0.0097(5)
C(12)	2*i*	–0.0812(2)	0.4158(2)	0.0715(1)	0.0311(7)	0.0312(8)	0.0244(7)	–0.0022(6)	0.0006(6)	0.0070(6)
C(13)	2*i*	–0.0017(2)	0.3160(1)	0.0702(1)	0.0433(9)	0.0208(6)	0.0282(7)	–0.0019(6)	0.0119(6)	0.0024(5)
C(14)	2*i*	0.1565(2)	0.3416(1)	0.1387(1)	0.0404(8)	0.0202(6)	0.0347(8)	0.0083(6)	0.0174(7)	0.0083(6)
C(15)	2*i*	0.2369(2)	0.4681(1)	0.2102(1)	0.0239(6)	0.0242(6)	0.0272(6)	0.0058(5)	0.0086(5)	0.0104(5)
C(16)	2*i*	0.2064(2)	0.7229(1)	0.4076(1)	0.0173(6)	0.0171(5)	0.0184(6)	0.0019(5)	0.0053(4)	0.0077(5)
C(17)	2*i*	0.0101(2)	0.5243(1)	0.3914(1)	0.0271(7)	0.0199(6)	0.0280(6)	0.0068(5)	0.0114(5)	0.0134(5)
C(18)	2*i*	–0.2125(2)	0.5850(1)	0.3597(1)	0.0221(6)	0.0277(7)	0.0372(8)	0.0047(6)	0.0100(6)	0.0164(6)
C(19)	2*i*	0.2150(2)	0.8659(1)	0.4778(1)	0.0188(6)	0.0177(6)	0.0206(6)	0.0021(5)	0.0053(5)	0.0069(5)
C(20)	2*i*	0.1892(2)	0.8982(1)	0.5864(1)	0.0198(6)	0.0164(6)	0.0188(6)	0.0021(5)	0.0057(5)	0.0049(5)
C(21)	2*i*	0.2016(2)	0.8145(1)	0.6398(1)	0.0259(6)	0.0177(6)	0.0209(6)	0.0053(5)	0.0082(5)	0.0060(5)
C(22)	2*i*	0.1746(2)	0.8491(1)	0.7411(1)	0.0301(7)	0.0216(6)	0.0223(6)	0.0036(5)	0.0095(5)	0.0090(5)
C(23)	2*i*	0.1338(2)	0.9671(1)	0.7868(1)	0.0248(6)	0.0229(6)	0.0199(6)	0.0000(5)	0.0100(5)	0.0022(5)
C(24)	2*i*	0.1212(2)	1.0519(1)	0.7357(1)	0.0239(6)	0.0180(6)	0.0271(7)	0.0037(5)	0.0089(5)	0.0029(5)
C(25)	2*i*	0.1506(2)	1.0177(1)	0.6355(1)	0.0218(6)	0.0166(6)	0.0251(6)	0.0022(5)	0.0051(5)	0.0072(5)

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