Crystal structure of 3-(2-chlorobenzoyl)-1,1-diphenylthiourea, $(C_6H_5)_2N(CS)(NH)(CO)(C_6H_4Cl)$

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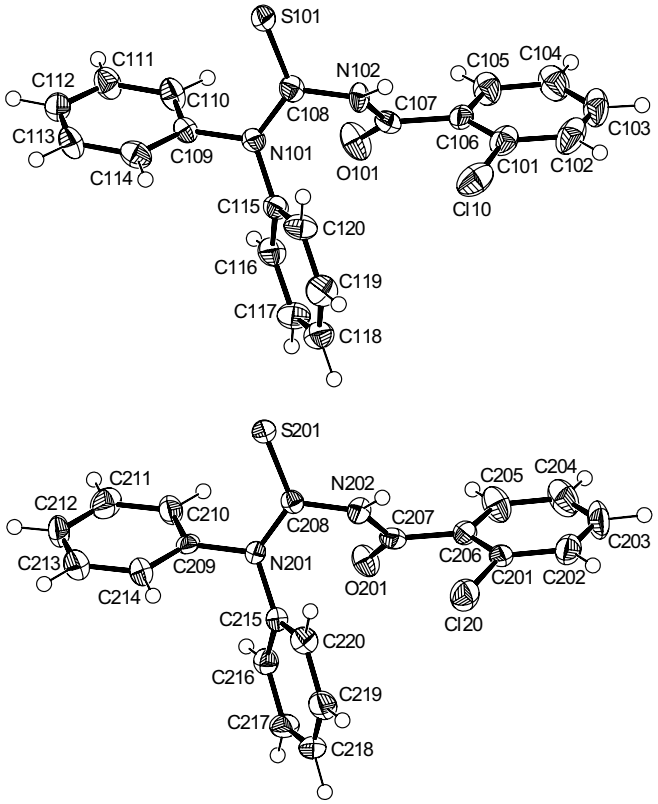
Received June 28, 2004, accepted and available on-line October 6, 2004; CCDC no. 1267/1359



Abstract

$C_{20}H_{15}ClN_2OS$, triclinic, $P\bar{1}$ (no. 2), a = 9.9962(8) Å, b = 12.911(1) Å, c = 14.670(1) Å, α = 85.568(2)°, β = 70.610(2)°, γ = 82.639(2)°, V = 1770.1 Å^3, Z = 4, $R_{gt}(F)$ = 0.046, $wR_{ref}(F^2)$ = 0.079, T = 173 K.

Source of material

All chemicals used for the preparation of the title compound were of reagent grade quality. The compound was prepared by a procedure similar to that reported in [1,2]. A solution of an appropriate 2-chlorobenzoyl chloride (10 mmol) in acetone (50 mL) was added dropwise to a suspension of potassium thiocyanate (10 mmol) in acetone (30 mL). The reaction mixture was heated under reflux for 30 min, and then cooled to room temperature. A solution of diphenylamine (10 mmol) in acetone (10 mL) was added and the resulting mixture was stirred for 2 h. 0.1 N Hydrochloric acid (300 mL) was added and the solution filtered. The solid product was washed with water and purified by recrystallization from ethanol.

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Discussion

The biological activities of thiourea derivatives have been successfully screened for various biological actions, and some derivatives have been used in commercial fungicides [1]. Also these derivatives have been found to be useful compounds for the enrichment and potential determination of transition metals from different matrices [3]. Recently, we discussed a novel series of thiourea derivatives and their metal complexes [4-7]. One of these new derivatives is also the title compound.

There are two independent, but identical molecules per asymmetric unit. The geometric parameters of the second molecule are very close to those of the first one, so the following discussion focuses only on the first molecule. The bond lengths of the carbonyl and thiocarbonyl groups have typical double-bond character d(C107—O101) = 1.214(4) Å, d(C108—S101) 1.657(4) Å. The bond lengths of all C—N bonds d(C115—N101) = 1.445(4) Å, d(C109—N101) = 1.444(4) Å, d(C108—N101) = 1.347(4) Å, d(C108—N102) = 1.388(4) Å, d(C107—N102) = 1.387(4) Å are shorter than the normal C—N single-bond (1.48 Å) and longer than normal C=N double bond (1.25 Å). These results are in agreement with expected delocalization in the molecule and confirmed by ∠C107–N102–C108 = 123.9(3)°, ∠C108–N101–C109 = 120.4(3)° showing an sp^2 hybridization on N101 and N102 atoms. The conformation of the molecule with respect to the thiocarbonyl and carbonyl moieties is twisted, as reflected by the torsion angles ∠O101–C107–N102–C108 and ∠C107–N102–C108–N101 of −4.2(6)° and 48.8(5)°, respectively.

Table 1. Data collection and handling.

Crystal:	yellow prism, size 0.12 × 0.15 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.44 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	51.36°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9926, 6594
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2600
$N(param)_{refined}$:	451
Program:	SHELXTL [8]

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

Atom	Site	x	y	z	U_{iso}
H(10A)	2*i*	0.6035	0.1013	0.4613	0.031
H(10B)	2*i*	1.0248	0.0911	0.5246	0.062
H(10E)	2*i*	0.9719	0.1081	0.6897	0.062
H(10D)	2*i*	0.7423	0.1710	0.7832	0.058
H(10C)	2*i*	0.5715	0.2272	0.7104	0.049
H(11D)	2*i*	0.1780	0.3186	0.5523	0.042

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(11E)	2*i*	–0.0530	0.3810	0.5542	0.045
H(11I)	2*i*	–0.1050	0.4052	0.4110	0.042
H(11B)	2*i*	0.0727	0.3731	0.2650	0.044
H(11A)	2*i*	0.3055	0.3113	0.2609	0.042
H(11F)	2*i*	0.4431	0.4649	0.4198	0.040
H(11G)	2*i*	0.6345	0.5560	0.3301	0.047
H(11H)	2*i*	0.8222	0.4733	0.2062	0.045
H(11C)	2*i*	0.8163	0.3014	0.1746	0.048
H(12A)	2*i*	0.6287	0.2092	0.2649	0.042
H(20A)	2*i*	0.0072	0.4021	–0.0733	0.030
H(20B)	2*i*	–0.2672	0.4553	–0.2705	0.049
H(20D)	2*i*	–0.5027	0.4613	–0.1681	0.060

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(20E)	2*i*	–0.5546	0.3864	–0.0148	0.061
H(20C)	2*i*	–0.3734	0.3052	0.0412	0.051
H(21D)	2*i*	0.0833	0.1669	0.1735	0.041
H(21H)	2*i*	0.2093	0.0898	0.2746	0.052
H(21I)	2*i*	0.4563	0.0497	0.2123	0.054
H(21G)	2*i*	0.5752	0.0882	0.0519	0.051
H(21F)	2*i*	0.4507	0.1653	–0.0494	0.041
H(21B)	2*i*	0.2654	0.2752	–0.1912	0.037
H(21E)	2*i*	0.3016	0.1769	–0.3274	0.044
H(21C)	2*i*	0.2582	0.0020	–0.3074	0.042
H(21A)	2*i*	0.1748	–0.0740	–0.1532	0.041
H(22A)	2*i*	0.1365	0.0235	–0.0168	0.034

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(10)	2*i*	0.8766(1)	0.13886(9)	0.39492(9)	0.0336(7)	0.0553(8)	0.0573(9)	–0.0087(6)	–0.0016(6)	–0.0109(7)
S(101)	2*i*	0.3242(1)	0.07885(7)	0.46145(8)	0.0301(6)	0.0271(6)	0.0453(8)	–0.0041(5)	–0.0179(6)	0.0054(6)
O(101)	2*i*	0.5051(3)	0.3113(2)	0.5564(2)	0.060(2)	0.031(2)	0.043(2)	0.013(2)	–0.025(2)	–0.012(2)
N(101)	2*i*	0.4028(3)	0.2690(2)	0.4067(2)	0.024(2)	0.023(2)	0.033(2)	–0.001(2)	–0.008(2)	0.004(2)
N(102)	2*i*	0.5447(3)	0.1586(2)	0.4784(2)	0.021(2)	0.022(2)	0.036(2)	0.009(2)	–0.013(2)	–0.008(2)
C(101)	2*i*	0.8288(4)	0.1487(3)	0.5200(3)	0.032(3)	0.029(3)	0.051(3)	–0.004(2)	–0.015(3)	–0.008(2)
C(102)	2*i*	0.9319(4)	0.1185(3)	0.5627(4)	0.037(3)	0.035(3)	0.090(5)	–0.004(2)	–0.029(3)	–0.015(3)
C(103)	2*i*	0.9005(5)	0.1280(3)	0.6602(4)	0.056(3)	0.033(3)	0.089(5)	–0.004(3)	–0.053(3)	–0.006(3)
C(104)	2*i*	0.7653(5)	0.1665(3)	0.7152(3)	0.066(3)	0.039(3)	0.054(4)	–0.007(3)	–0.038(3)	–0.002(3)
C(105)	2*i*	0.6637(4)	0.1983(3)	0.6724(3)	0.050(3)	0.036(3)	0.047(3)	–0.006(2)	–0.030(3)	–0.005(3)
C(106)	2*i*	0.6942(4)	0.1886(3)	0.5737(3)	0.026(2)	0.021(2)	0.040(3)	–0.004(2)	–0.014(2)	0.000(2)
C(107)	2*i*	0.5760(4)	0.2278(3)	0.5343(3)	0.033(3)	0.022(2)	0.030(3)	–0.004(2)	–0.011(2)	0.005(2)
C(108)	2*i*	0.4262(4)	0.1743(3)	0.4476(3)	0.025(2)	0.025(2)	0.029(3)	0.004(2)	–0.006(2)	–0.003(2)
C(109)	2*i*	0.2616(4)	0.3086(3)	0.4055(3)	0.029(2)	0.019(2)	0.025(3)	0.002(2)	–0.010(2)	–0.004(2)
C(110)	2*i*	0.1565(4)	0.3294(3)	0.4936(3)	0.038(3)	0.040(3)	0.029(3)	0.006(2)	–0.015(2)	–0.002(2)
C(111)	2*i*	0.0200(4)	0.3661(3)	0.4945(3)	0.035(3)	0.038(3)	0.036(3)	0.009(2)	–0.010(2)	–0.001(2)
C(112)	2*i*	–0.0105(4)	0.3811(3)	0.4098(3)	0.033(3)	0.032(3)	0.044(3)	–0.004(2)	–0.018(2)	0.002(2)
C(113)	2*i*	0.0943(4)	0.3616(3)	0.3236(3)	0.043(3)	0.041(3)	0.031(3)	0.002(2)	–0.020(2)	0.003(2)
C(114)	2*i*	0.2324(4)	0.3249(3)	0.3209(3)	0.047(3)	0.032(3)	0.026(3)	–0.005(2)	–0.012(2)	0.000(2)
C(115)	2*i*	0.5187(4)	0.3283(3)	0.3521(3)	0.027(2)	0.027(2)	0.026(3)	–0.002(2)	–0.007(2)	0.003(2)
C(116)	2*i*	0.5201(4)	0.4311(3)	0.3703(3)	0.031(2)	0.024(2)	0.040(3)	0.005(2)	–0.008(2)	–0.003(2)
C(117)	2*i*	0.6331(4)	0.4854(3)	0.3167(3)	0.047(3)	0.024(2)	0.047(3)	–0.010(2)	–0.015(3)	–0.001(2)
C(118)	2*i*	0.7442(4)	0.4366(3)	0.2434(3)	0.034(3)	0.038(3)	0.038(3)	–0.007(2)	–0.008(2)	0.002(2)
C(119)	2*i*	0.7405(4)	0.3348(3)	0.2252(3)	0.035(3)	0.041(3)	0.034(3)	0.004(2)	–0.002(2)	–0.005(2)
C(120)	2*i*	0.6295(4)	0.2798(3)	0.2784(3)	0.042(3)	0.023(2)	0.033(3)	–0.004(2)	–0.005(2)	–0.003(2)
Cl(20)	2*i*	–0.0061(1)	0.37259(8)	–0.25259(8)	0.0429(7)	0.0558(8)	0.0304(7)	–0.0002(6)	–0.0124(6)	0.0004(6)
S(201)	2*i*	0.1433(1)	0.40552(7)	0.06473(8)	0.0300(6)	0.0259(6)	0.0336(7)	0.0001(5)	–0.0150(5)	–0.0065(5)
O(201)	2*i*	–0.1080(2)	0.1935(2)	–0.0015(2)	0.032(2)	0.027(2)	0.045(2)	–0.004(1)	–0.012(2)	0.008(2)
N(201)	2*i*	0.1771(3)	0.2181(2)	–0.0090(2)	0.028(2)	0.025(2)	0.025(2)	0.001(2)	–0.014(2)	–0.003(2)
N(202)	2*i*	0.0058(3)	0.3415(2)	–0.0412(2)	0.030(2)	0.020(2)	0.029(2)	–0.008(2)	–0.014(2)	0.005(2)
C(201)	2*i*	–0.1798(4)	0.3780(3)	–0.1733(3)	0.028(2)	0.026(2)	0.038(3)	0.002(2)	–0.016(2)	–0.011(2)
C(202)	2*i*	–0.2887(4)	0.4254(3)	–0.2068(3)	0.054(3)	0.031(3)	0.050(3)	–0.003(2)	–0.033(3)	0.000(2)
C(203)	2*i*	–0.4277(4)	0.4283(3)	–0.1463(4)	0.036(3)	0.046(3)	0.080(4)	0.000(2)	–0.039(3)	0.001(3)
C(204)	2*i*	–0.4581(4)	0.3844(3)	–0.0557(4)	0.024(3)	0.051(3)	0.075(4)	–0.002(2)	–0.013(3)	–0.002(3)
C(205)	2*i*	–0.3506(4)	0.3365(3)	–0.0221(3)	0.029(3)	0.040(3)	0.056(3)	–0.004(2)	–0.011(3)	0.008(3)
C(206)	2*i*	–0.2096(4)	0.3344(3)	–0.0810(3)	0.023(2)	0.025(2)	0.034(3)	–0.005(2)	–0.012(2)	–0.005(2)
C(207)	2*i*	–0.1006(4)	0.2801(3)	–0.0394(3)	0.028(2)	0.028(2)	0.024(3)	0.001(2)	–0.004(2)	–0.007(2)
C(208)	2*i*	0.1096(3)	0.3159(3)	0.0030(3)	0.019(2)	0.025(2)	0.021(2)	–0.002(2)	–0.003(2)	0.000(2)
C(209)	2*i*	0.2558(4)	0.1739(3)	0.0532(3)	0.033(2)	0.017(2)	0.023(3)	–0.002(2)	–0.013(2)	–0.003(2)
C(210)	2*i*	0.1840(4)	0.1511(3)	0.1486(3)	0.032(2)	0.036(3)	0.034(3)	0.004(2)	–0.014(2)	0.001(2)
C(211)	2*i*	0.2586(4)	0.1051(3)	0.2085(3)	0.065(3)	0.035(3)	0.033(3)	–0.007(2)	–0.023(3)	0.007(2)
C(212)	2*i*	0.4047(5)	0.0816(3)	0.1717(4)	0.051(3)	0.035(3)	0.061(4)	0.010(2)	–0.040(3)	–0.004(3)
C(213)	2*i*	0.4746(4)	0.1042(3)	0.0770(3)	0.035(3)	0.043(3)	0.052(3)	0.006(2)	–0.020(3)	–0.008(3)
C(214)	2*i*	0.4010(4)	0.1502(3)	0.0168(3)	0.028(2)	0.038(3)	0.039(3)	0.005(2)	–0.015(2)	–0.005(2)
C(215)	2*i*	0.1956(4)	0.1588(3)	–0.0931(3)	0.023(2)	0.026(2)	0.026(3)	–0.000(2)	–0.010(2)	–0.002(2)
C(216)	2*i*	0.2459(4)	0.2043(3)	–0.1835(3)	0.029(2)	0.032(2)	0.029(3)	–0.004(2)	–0.006(2)	0.001(2)
C(217)	2*i*	0.2683(4)	0.1457(3)	–0.2643(3)	0.034(3)	0.045(3)	0.025(3)	0.005(2)	–0.004(2)	–0.001(2)
C(218)	2*i*	0.2422(4)	0.0422(3)	–0.2524(3)	0.035(3)	0.041(3)	0.029(3)	0.007(2)	–0.011(2)	–0.014(2)
C(219)	2*i*	0.1930(4)	–0.0028(3)	–0.1609(3)	0.038(3)	0.028(2)	0.040(3)	–0.004(2)	–0.014(2)	–0.010(2)
C(220)	2*i*	0.1696(3)	0.0547(3)	–0.0800(3)	0.026(2)	0.031(2)	0.028(3)	–0.003(2)	–0.008(2)	0.000(2)

Acknowledgment. This work was supported by the Mersin University Research Fund (project nos.: FEF.K.HA.99.1, ECZ.F.TB.HA.2003.1 and FBE.KB.GP.01).

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