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Key indicators

Single-crystal X-ray study
T = 120 K
Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$
R factor = 0.040
wR factor = 0.097
Data-to-parameter ratio = 16.0

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

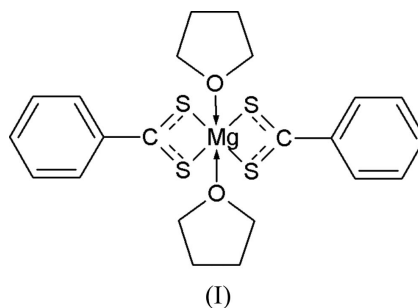
Bis(dithiobenzoato- $\kappa^2\text{S,S}'$)bis(tetrahydrofuran- κO)magnesium(II)

The title compound, $[\text{Mg}(\text{C}_7\text{H}_5\text{S}_2)_2(\text{C}_4\text{H}_8\text{O})_2]$, was obtained by the reaction of PhMgBr with an excess of CS_2 . The magnesium(II) cation is six-coordinated by two dithiobenzoate ligands coordinating through their S atoms in a bidentate chelating fashion and two O atoms from two tetrahydrofuran ligands, in a distorted octahedral geometry.

Received 19 July 2006
Accepted 2 August 2006

Comment

In the course of our studies on silicon-sulfur chemistry, we have synthesized two magnesium derivatives of dithiobenzoic acid, *viz.* $\text{PhCS}_2\text{MgS}_2\text{CPh}\cdot 2\text{THF}$, (I), and $\text{PhCS}_2\text{MgBr}\cdot 3\text{THF}$, (II), by the reaction of PhMgBr with an excess of CS_2 (Ramadan *et al.*, 1983). The formation of (I) can be rationalized in terms of an equilibrium involving (II) or in terms of a Schlenk equilibrium (Schlenk *et al.*, 1929) involving PhMgBr . In the second case, the formation of (I) can be described as the reaction of magnesium diphenyl with CS_2 . To the best of our knowledge, compound (I) is the first example of a magnesium salt with two dithiobenzoate ligands.



In complex (I) (Fig. 1), the magnesium(II) cation is six-coordinated by four S atoms from two dithiobenzoate anions and two O atoms from two tetrahydrofuran molecules, in a distorted octahedral geometry. Deviations from the ideal geometry are imposed by the S—C—S angle of the chelating dithiobenzoate ligands (Table 1). Few metal complexes of Ni (Tang *et al.*, 1984; Fackler *et al.*, 1984), Fe (Coucovanis *et al.*, 1969), Cr (Tang *et al.*, 1985) and Co (Lu *et al.*, 1995), with two or more ligands containing the dithiocarboxy group and with geometry close to octahedral, have been reported. Among them, an Ni complex containing two α -naphthylthiocarboxylate ligands and two molecules of pyridine (Tang *et al.* 1984) is similar to complex (I). The C—S distances are between those of a typical C—S single bond (1.819 Å) and a C=S double bond (1.599 Å) (Prince, 2004), indicating electron delocalization in the dithiocarboxy groups. No classical hydrogen bonds were observed in the crystal structure.

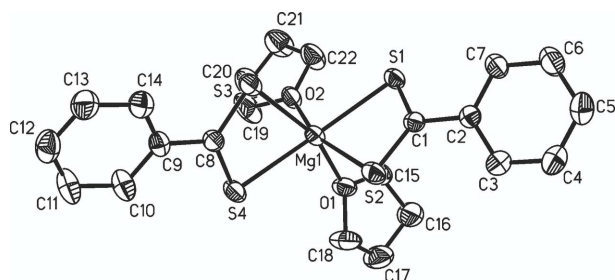


Figure 1
The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

Experimental

The work was carried out using standard vacuum-nitrogen line and Schlenk techniques. PhMgBr (100 ml, 0.06 mol) was added dropwise into CS₂ (26 ml, 0.36 mol) at room temperature and the resulting red solution was concentrated under vacuum. A small amount of oily residue was obtained which was diluted in toluene (2 ml). Pentane vapours were diffused into the toluene solution and the solution was left to stand at room temperature for two months, after which time red–orange crystals of (I) were deposited.

Crystal data

[Mg(C ₇ H ₅ S ₂) ₂ (C ₄ H ₈ O) ₂]	Z = 4
<i>M_r</i> = 474.98	<i>D_x</i> = 1.306 Mg m ^{−3}
Monoclinic, <i>P</i> 2 ₁ / <i>n</i>	Mo <i>K</i> α radiation
<i>a</i> = 8.3846 (9) Å	<i>μ</i> = 0.44 mm ^{−1}
<i>b</i> = 14.3899 (9) Å	<i>T</i> = 120 (2) K
<i>c</i> = 20.0275 (15) Å	Block, red–orange
<i>β</i> = 90.287 (11)°	0.55 × 0.45 × 0.3 mm
<i>V</i> = 2416.4 (4) Å ³	

Data collection

Stoe IPDS diffractometer	10590 measured reflections
φ scans	4201 independent reflections
Absorption correction: numerical	2373 reflections with <i>I</i> > 2σ(<i>I</i>)
[<i>X-RED32</i> (Stoe & Cie, 1997);	<i>R</i> _{int} = 0.039
crystal shape approximated by a	<i>θ</i> _{max} = 25.1°
polyhedron with 13 faces]	
<i>T</i> _{min} = 0.781, <i>T</i> _{max} = 0.876	

Refinement

Refinement on <i>F</i> ²	H-atom parameters constrained
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)] = 0.040	<i>w</i> = 1/[σ ² (<i>F</i> _o ²) + (0.0526 <i>P</i>) ²]
<i>wR</i> (<i>F</i> ²) = 0.097	where <i>P</i> = (<i>F</i> _o ² + 2 <i>F</i> _c ²)/3
<i>S</i> = 0.85	(Δ/σ) _{max} < 0.001
4201 reflections	Δρ _{max} = 0.31 e Å ^{−3}
262 parameters	Δρ _{min} = −0.19 e Å ^{−3}

Table 1

Selected geometric parameters (Å, °).

S1–Mg1	2.5614 (11)	S4–Mg1	2.5753 (11)
S2–Mg1	2.6086 (13)	O1–Mg1	2.065 (2)
S3–Mg1	2.5831 (14)	O2–Mg1	2.074 (2)
S2–C1–S1	121.37 (16)	O2–Mg1–S3	92.78 (8)
S3–C8–S4	120.57 (19)	S1–Mg1–S3	97.95 (4)
O1–Mg1–O2	87.59 (10)	S4–Mg1–S3	69.30 (4)
O1–Mg1–S1	99.35 (7)	O1–Mg1–S2	90.61 (8)
O2–Mg1–S1	94.35 (6)	O2–Mg1–S2	163.13 (7)
O1–Mg1–S4	93.55 (7)	S1–Mg1–S2	69.39 (3)
O2–Mg1–S4	100.94 (6)	S4–Mg1–S2	95.91 (4)
S1–Mg1–S4	160.38 (5)	S3–Mg1–S2	93.90 (5)
O1–Mg1–S3	162.61 (7)		

H atoms were placed in idealized positions and allowed to ride on their parent atoms, with C–H = 0.95–0.99 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C).

Data collection: *IPDS Software* (Stoe & Cie, 1997); cell refinement: *IPDS Software*; data reduction: *X-RED32* (Stoe & Cie, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Financial support to RG from the Polish Ministry of Research and Information (project No. 3T09A 120 28) is gratefully acknowledged.

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