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Bromo(dithiobenzoato-κ^2S,S')tris(tetrahydrofuran-κO)magnesium(II): a redetermination

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The crystal structure of the title compound, [$MgBr(C_6H_5CS_2)(C_4H_8O)_3$], has been reported previously by Chang, Yang, Guo, Huang, Wang, Lee & Peng [*J. Chin. Chem. Soc. (Taipei)* (2003), **50**, 965–971] using intensity data collected at room temperature. However, no coordinates are available. We present here a redetermination of this structure using new intensity data collected at 120 K. The Mg^{II} atom is six-coordinate in an octahedral geometry.

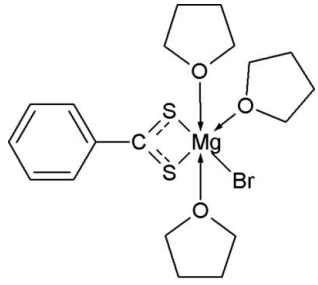
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.009 Å
R factor = 0.070
wR factor = 0.181
Data-to-parameter ratio = 16.7

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

Comment

The crystal structure of the title compound, (I), has previously been reported by Chang *et al.* (2003), with an R value of 0.048, using intensity data collected at room temperature; however, no coordinates are available in the Cambridge Structural Database (Version 5.27; Allen, 2002). We report here a redetermination of (I) using new intensity data collected at 120 K.



(I)

The Mg^{II} atom is six-coordinated by two S atoms from a dithiobenzoate anion, three O atoms from three tetrahydrofuran ligands and a bromide anion (Fig. 1) in a distorted octahedral geometry. All the bond lengths and angles in (I) (Table 1) are comparable with those observed in the previous determination (Chang *et al.*, 2003).

In the previous work, Chang *et al.* (2003) obtained colourless crystals of (I) but we obtained red crystals of (I), which is a typical colour for compounds containing the dithiocarboxy group. We observed that this compound is not stable in air.

Experimental

PhMgBr (100 ml, 0.06 mol) was added dropwise to CS_2 (26 ml, 0.36 mol) at room temperature. The resulting red solution was concentrated under vacuum, giving a small amount of oily residue. The residue was diluted in tetrahydrofuran (2 ml), covered with a layer of pentane and left at room temperature for six months. Large red crystals of (I) were deposited.

Crystal data

$[MgBr(C_7H_5S_2)(C_4H_8O)_3]$
M_r = 473.76
Monoclinic, $P2_1/c$
a = 11.901 (4) Å
b = 14.720 (3) Å
c = 13.0391 (17) Å
β = 105.154 (18)°
V = 2204.8 (9) Å^3
Z = 4
D_x = 1.427 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 2.10 mm^{-1}
T = 120 (2) K
Plate, red
0.38 × 0.22 × 0.06 mm

Data collection

Kuma KM-4-CCD diffractometer
ω scans
Absorption correction: numerical (*CrysAlis RED*; Oxford Diffraction, 2005)
T_{min} = 0.432, T_{max} = 0.796
14329 measured reflections
3932 independent reflections
3418 reflections with $I > 2\sigma(I)$
R_{int} = 0.110
θ_{max} = 25.1°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.181
S = 1.07
3932 reflections
235 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0913P)^2 + 8.1437P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.08 e Å^{-3}
$\Delta\rho_{min}$ = −0.69 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—Mg1	2.5644 (19)	Mg1—O3	2.113 (4)
Mg1—O1	2.061 (4)	Mg1—S1	2.578 (2)
Mg1—O2	2.100 (4)	Mg1—S2	2.720 (2)
S2—C1—S1	120.3 (3)	O2—Mg1—S1	97.31 (13)
O1—Mg1—O2	84.77 (16)	O3—Mg1—S1	91.96 (13)
O1—Mg1—O3	84.28 (16)	Br1—Mg1—S1	97.42 (7)
O2—Mg1—O3	168.51 (17)	O1—Mg1—S2	94.32 (13)
O1—Mg1—Br1	101.23 (13)	O2—Mg1—S2	86.91 (12)
O2—Mg1—Br1	92.95 (12)	O3—Mg1—S2	90.46 (12)
O3—Mg1—Br1	92.57 (12)	Br1—Mg1—S2	164.38 (8)
O1—Mg1—S1	161.11 (14)	S1—Mg1—S2	67.15 (6)

All H atoms were treated as riding on their parent atoms, with C—H = 0.95–0.99 Å and U_{iso}(H) = 1.2U_{eq}(C). The highest residual density peak is located 0.03 Å from Br1.

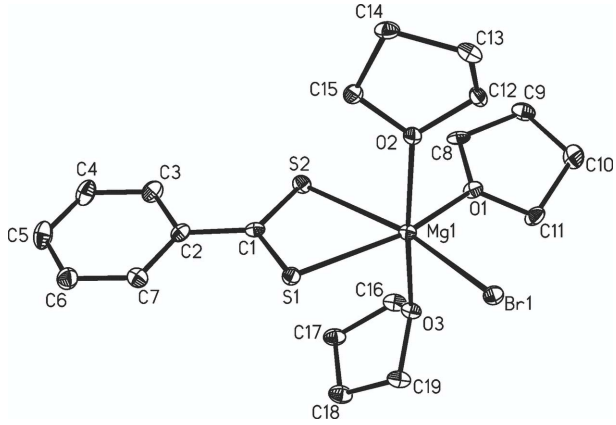


Figure 1
The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2005); 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).

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