Acta Cryst. (1992). C**48**, 1843–1844

Structure of Bis(trifluoracetamidinium) Di-μ-chloro-hexachlorodioxodimolybdate(V)

BY REGINE HERBST-IRMER

Institut für Anorganische Chemie der Universität Göttingen, Tammannstrasse 4, *W*-3400 *Göttingen, Germany*

AND ERNST EGERT*

Institut für Organische Chemie der Universität Frankfurt, Niederurseler Hang, W-6000 *Frankfurt am Main* 50, *Germany*

(*Received* 6 *January* 1992; *accepted* 24 *February* 1992)

Abstract. $[C_2H_4F_3N_2]_2[Mo_2Cl_8O_2]$, $M_r = 733.6$, triclinic, $P\bar{1}$, $a = 6.701$ (1), $b = 8.674$ (1), $c = 9.179$ (1) Å, $\alpha = 83.50$ (1), $\beta = 89.37$ (1), $\gamma = 74.35$ (1)°, $V = 510.3$ (1) Å^3, $Z = 1$, $D_x = 2.387$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 2.32$ mm^{-1}, $F(000) = 350$, $T = 298$ K, $R = 0.021$ for 1699 observed reflections. The dimeric anion shows approximate C_{2h} symmetry with asymmetric chlorine bridges and lies on a crystallographic inversion centre. The amino groups from four hydrogen bonds to the Cl atoms of three symmetry-related anions: N(1)···Cl(1^{i}) 3.323 (3), N(1)···Cl(2^{ii}) 3.434 (3), N(1′)···Cl(1^{ii}) 3.299 (3), N(1′)···Cl(4^{iii}) 3.348 (3) Å [(i) $-x$, $1-y$, $-z$; (ii) $1-x$, $1-y$, $-z$; (iii) $1-x$, $1-y$, $1-z$].

Experimental. Green prisms from 1,2-dichloroethane, crystal size 0.7 × 0.4 × 0.1 mm. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode involving variable scan width and speed (Clegg, 1981); cell constants refined from $\pm 2\theta$ values of 82 reflections in the range 20–25°, 3927 reflections measured, $2\theta_{max} = 50°$ ($-7 \le h \le 4$, $-10 \le k \le 10$, $-10 \le l \le 10$). Three check reflections with no significant intensity variation, 1786 unique reflections ($R_{int} = 0.019$) of which 1699 with $|F| > 4\sigma(F)$ were used for all calculations (*SHELXTL*; Sheldrick, 1978), 400 azimuthal scan reflections used to correct the data for absorption (range of relative transmission factors 0.52–0.98). Structure solution by Patterson interpretation. Refinement on F to $R = 0.021$, $wR = 0.030$, all non-H atoms anisotropic, H refined with restrained N—H bond lengths (r_{N-H} 0.93 Å) and $U(H) = 1.2 \times U_{eq}(N)$, 131 parameters refined, $S = 1.11$, weighting scheme $w = [\sigma^2(F) + 0.0005F^2]^{-1}$ which led to a featureless analysis of variance in terms of sinθ and F_o, maximum $\Delta/\sigma = 0.008$ in last cycle, maximum and minimum heights in final $\Delta\rho$ map 0.32 and -0.47 e Å^{-3} respectively, atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å$^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Mo	979 (1)	2840 (1)	1414 (1)	26 (1)
Cl(1)	1148 (1)	1626 (1)	−795 (1)	34 (1)
Cl(2)	4566 (1)	1786 (1)	1619 (1)	40 (1)
Cl(3)	−2247 (1)	4705 (1)	548 (1)	30 (1)
Cl(4)	1207 (1)	4575 (1)	3183 (1)	38 (1)
O	105 (3)	1515 (2)	2461 (2)	44 (1)
C(1)	4452 (4)	7590 (3)	3237 (3)	31 (1)
C(2)	2943 (4)	7928 (3)	4485 (3)	34 (1)
N(1)	3710 (4)	7874 (3)	1908 (3)	43 (1)
N(1′)	6401 (4)	7070 (4)	3606 (3)	50 (1)
F(2)	1009 (2)	8159 (2)	4045 (2)	48 (1)
F(2′)	3386 (3)	6714 (2)	5544 (2)	55 (1)
F(2″)	3082 (3)	9241 (2)	5030 (2)	58 (1)

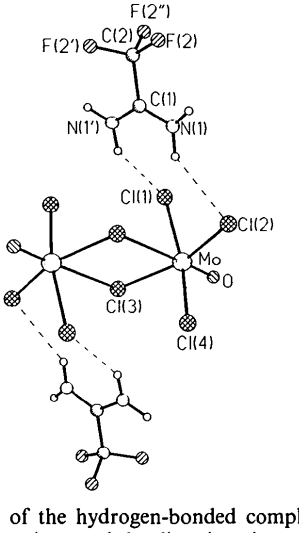

Fig. 1. Structure of the hydrogen-bonded complex between two cations and the dimeric anion.

* Author to whom correspondence should be addressed.

0108-2701/92/101843-02$06.00

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Table 2. *Bond lengths* (Å) *and angles* (°)

Mo—Cl(1)	2.376 (1)	Mo—Cl(2)	2.331 (1)
Mo—Cl(3)	2.404 (1)	Mo—Cl(4)	2.368 (1)
Mo—O	1.640 (2)	Mo—Cl(3^i)	2.928 (1)
		C(1)—C(2)	1.521 (4)
C(1)—N(1)	1.294 (3)	C(1)—N(1′)	1.296 (3)
C(2)—F(2)	1.317 (3)	C(2)—F(2′)	1.322 (3)
C(2)—F(2″)	1.320 (4)		
Cl(1)—Mo—Cl(2)	87.1 (1)	Cl(1)—Mo—Cl(3)	89.0 (1)
Cl(2)—Mo—Cl(3)	156.8 (1)	Cl(1)—Mo—Cl(4)	163.6 (1)
Cl(2)—Mo—Cl(4)	88.5 (1)	Cl(3)—Mo—Cl(4)	88.8 (1)
Cl(1)—Mo—O	97.5 (1)	Cl(2)—Mo—O	103.2 (1)
Cl(3)—Mo—O	100.0 (1)	Cl(4)—Mo—O	98.9 (1)
Cl(1)—Mo—Cl(3^i)	81.5 (1)	Cl(2)—Mo—Cl(3^i)	80.6 (1)
Cl(3)—Mo—Cl(3^i)	76.2 (1)	Cl(4)—Mo—Cl(3^i)	82.2 (1)
O—Mo—Cl(3^i)	176.0 (1)	Mo—Cl(3)—Mo^i	103.8 (1)
C(2)—C(1)—N(1)	118.2 (2)	C(2)—C(1)—N(1′)	116.3 (2)
N(1)—C(1)—N(1′)	125.5 (3)	C(1)—C(2)—F(2)	112.0 (2)
C(1)—C(2)—F(2′)	110.5 (2)	F(2)—C(2)—F(2′)	108.4 (2)
C(1)—C(2)—F(2″)	109.5 (2)	F(2)—C(2)—F(2″)	108.0 (2)
F(2′)—C(2)—F(2″)	108.4 (2)		

Symmetry code: (i) $-x, 1-y, -z$.

Atomic parameters are given in Table 1, bond distances and angles in Table 2.* Fig. 1 shows a plot with the atom numbering.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55230 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SE1001]

Related literature. For structures containing the monomeric $[MoOCl_4]^-$ anion, see Garner, Hill, Mabbs, McFadden & McPhail (1977), Knopp, Lörcher & Strähle (1977) and Weller, Müller, Weiher & Dehnicke (1980); for a structure containing the dimeric anion, see Klinzing, El-Kholi, Müller, Dehnicke & Findeisen (1989).

We thank the Fonds der Chemischen Industrie for financial support and Professor Dr H. W. Roesky for providing us with the sample.

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Acta Cryst. (1992). C**48**, 1844–1846

Tri-μ-chloro-bis(1,4,7-trithiacyclononane)dinickel(II) Tetrafluoroborate Acetonitrile Solvate

By Alexander J. Blake,* Malcolm A. Halcrow and Martin Schröder

Department of Chemistry, University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, Scotland

(*Received* 13 *November* 1991; *accepted* 21 *February* 1992)

Abstract. $[Ni_2(C_6H_{12}S_3)_2Cl_3][BF_4].C_2H_3N$, M_r = 712.28, monoclinic, $P2_1/c$, a = 12.379 (5), b = 13.790 (6), c = 16.031 (6) Å, β = 99.73 (4)°, V = 2697 Å^3, Z = 4, D_x = 1.754 Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, μ = 2.184 mm^{-1}, $F(000)$ = 1448, T = 150.0 (1) K, R = 0.0562 for 2173 unique observed reflections. Each Ni atom is approximately octahedrally coordinated to all three S atoms of one ligand molecule and to the three Cl atoms. Unusually, the Ni^{II} centres are triply chloride bridged, giving a cation which consists of two face-sharing octahedra in which the Ni···Ni distance is 2.9211 (20) Å.

Experimental. Compound prepared by reaction of $NiCl_2$ with 1,4,7-trithiacyclononane and $NaBF_4$ in CH_3NO_2; crystals obtained from CH_3CN/Et_2O. To prevent crystal degradation resulting from the loss of solvent, a pale-green lath with dimensions 0.08 × 0.21 × 0.70 mm was transferred from its cold mother liquor into a drop of mineral oil and then onto a Stoe Stadi-4 four-circle diffractometer equipped with an Oxford Cryosystems low-temperature device (Cosier & Glazer, 1986) operating at 150 K. Graphite-monochromated Mo $K\alpha$ radiation, cell parameters from 2θ values of 23 reflections measured at $\pm\omega$ ($20 < 2\theta < 22°$). For data collection, ω–2θ scans with ω-scan width $(1.32 + 0.35\tan\theta)°$, $2\theta_{max}$ = 45°, h −13→13, k 0→14, l 0→17, three standard reflections, no significant crystal decay or movement, no absorption correction, 4448 reflections collected, 3084 unique (R_{int} = 0.05), giving 2173 with $F > 4\sigma(F)$ for structure solution (from a Patterson synthesis

* To whom correspondence should be addressed.

0108-2701/92/101844-03$06.00

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