Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
V	0.45478 (4)	0.82048 (4)	0.00206 (1)	1.42 (1)
Na(1)	0.2366 (1)	0.9581 (1)	0.08695 (3)	2.18 (2)
Na(2)	0.6052 (1)	0.3312 (1)	0.10246 (3)	3.22 (2)
O(1)	0.3162 (2)	0.9576 (2)	0.00619 (6)	2.23 (3)
O(2)	0.5736 (2)	0.9000 (2)	−0.05316 (5)	1.97 (3)
O(3)	0.6554 (2)	0.8628 (2)	0.03040 (5)	1.76 (3)
O(4)	0.3982 (2)	0.6244 (2)	−0.02644 (5)	1.82 (3)
O(5)	0.3914 (2)	0.6958 (2)	0.05807 (5)	1.90 (3)
O(6)	0.8084 (2)	1.0047 (2)	−0.07515 (5)	2.21 (3)
O(7)	0.3248 (2)	0.4516 (2)	0.08305 (5)	2.10 (3)
O(*W*1)	0.5022 (2)	1.0482 (2)	0.09996 (6)	2.76 (4)
O(*W*2)	0.1121 (2)	1.2009 (2)	0.07096 (7)	2.65 (4)
O(*W*3)	0.8371 (2)	0.1750 (4)	0.12499 (8)	4.08 (5)
C(1)	0.7203 (2)	0.9449 (2)	−0.04578 (7)	1.67 (3)
C(2)	0.7825 (2)	0.9141 (2)	0.00190 (7)	1.59 (4)
C(3)	0.3610 (2)	0.4924 (2)	0.00197 (7)	1.49 (3)
C(4)	0.3557 (2)	0.5475 (2)	0.05158 (6)	1.63 (4)

Table 2. *Selected geometric parameters* (Å, °)

V and Na coordination spheres			
V—O(1)	1.622 (2)	V—O(4)	1.890 (1)
V—O(2)	2.012 (1)	V—O(5)	2.016 (1)
V—O(3)	1.894 (1)	V···V^{i}	4.2929 (5)
O(4)—V—O(5)	81.74 (6)	O(3)—V—O(5)	87.94 (6)
O(3)—V—O(4)	124.98 (6)	O(2)—V—O(5)	163.04 (6)
O(2)—V—O(4)	92.61 (6)	O(2)—V—O(3)	82.21 (6)
O(1)—V—O(5)	96.53 (7)	O(1)—V—O(4)	117.44 (7)
O(1)—V—O(3)	117.38 (7)	O(1)—V—O(2)	100.25 (7)
V—O(2)—C(1)	114.9 (1)	V—O(3)—C(2)	116.8 (1)
V—O(4)—C(3)	117.3 (1)	V—O(5)—C(4)	115.6 (1)
O(1)—V···V^{i}	177.30 (9)		
Na(1)—O(1)	2.467 (2)	Na(2)—O(7)	2.596 (2)
Na(1)—O(5)	2.667 (2)	Na(2)—O(*W*3)	2.413 (2)
Na(1)—O(*W*1)	2.359 (2)	Na(2)—O(*W*1^{iv})	2.500 (2)
Na(1)—O(*W*2)	2.312 (2)	Na(2)—O(4^{i})	2.311 (2)
Na(1)—O(6^{ii})	2.316 (2)	Na(2)—O(6^{v})	2.325 (2)
Na(1)—O(7^{iii})	2.410 (2)		
Average values for the tartrate ligands			
C—C	1.53 (1)	C—O(carboxyl)	1.285 (8)
C—O(hydroxyl)	1.413 (1)	C=O	1.242 (8)
C—H	0.980 (8)		
C—C—C	106.7 (4)	C—C—O(carboxyl)	120.7 (4)
C—C—O(hydroxyl)	108.3 (3)	C—C=O	115.4 (6)
Hydrogen bonds			
O(*W*1)···O(3)	2.859 (2)	O(*W*1)···O(*W*3^{vi})	3.062 (3)
O(*W*2)···O(7^{vi})	2.751 (2)	O(*W*2)···O(1^{viii})	2.778 (3)
O(*W*3)···O(*W*2^{vii})	2.789 (3)		

Symmetry codes: (i) $y, x, -z$; (ii) $y-1, x, -z$; (iii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{4}-z$; (iv) $x, y-1, z$; (v) $\frac{3}{2}-y, x-\frac{1}{2}, \frac{1}{4}+z$; (vi) $x, 1+y, z$; (vii) $1+x, y-1, z$; (viii) $y-1, x+1, -z$.

The data were corrected for Lorentz and polarization effects. The structure was determined by Patterson methods using the program *SHELXS*86 (Sheldrick, 1990) and refined by a full-matrix least-squares method using *SHELX*76 (Sheldrick, 1976). The H atoms were located from difference Fourier maps and were refined isotropically. Figures were produced using *SCHAKAL*88 (Keller, 1988) and molecular geometry calculations were performed using *BONDLA* (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) and *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1114). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Second Triclinic Form of Pyridinium μ-oxo-μ-sulfato-bis[oxotris(isothiocyanato)-molybdate(V)]

BORIS KAMENAR, MARINA CINDRIĆ AND NEVEN STRUKAN

Laboratory of General and Inorganic Chemistry, Faculty of Science, University of Zagreb, Ul. kralja Zvonimira 8, PO Box 153, 41001 Zagreb, Croatia

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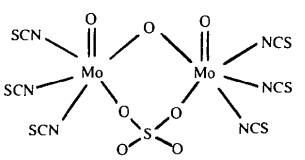
Abstract

Crystals of the new triclinic form of the title compound, $(C_5H_6N)_4[Mo_2O_3(SO_4)(NCS)_6]$, space group $P\bar{1}$, have been prepared and structurally characterized. There are two crystallographically independent complex

molecules in the asymmetric unit. Two Mo atoms in the binuclear anion are octahedrally coordinated and bridged through an oxo O atom and also through two O atoms from the bidentately bonded sulfato ligand. The structure is stabilized by N—H···O hydrogen bonds between pyridinium cations and complex anions.

Comment

The structure of one triclinic crystal form of the title compound (space group $P1$) has been reported by Yang & Yu (1984); we report here the second triclinic form (space group $P\bar{1}$). Although the unit cell of the present form is four times larger [4069 (3) *versus* 1015.5 (3) Å^3], the molecular volumes and the structures of both forms are very similar. However, significant differences occur in the molecular packing (Fig. 2).



(I)

The crystal structure is built up of pyridinium cations and $[Mo_2O_3(SO_4)(NCS)_6]^{4-}$ anions (I) held together by N—H···O hydrogen bonds [ranging from 2.62 (2) to 3.01 (3) Å] between pyridinium N and sulfato O atoms. The binuclear anion (Fig. 1) consists of two Mo atoms each coordinated in a distorted octahedral environment by two oxo ligands, one terminal and one bridging, one sulfato O atom and three N atoms from the isothiocyanato ligands. The Mo atoms are singly bridged by an oxo O atom and also by two O atoms from the bidentately bonded sulfato ion. The Mo—O(S) bonds are distinctly long [2.15 (2)–2.30 (1) Å] as a result of the *trans* influence of the terminal oxo ligands. All bond lengths within the cations and anions are close to those described for the triclinic form, reported by Yang & Yu (1984), and are comparable to the corresponding values found in similar Mo–isothiocyanato complexes (Głowiak, Rudolf, Sabat & Jezowska-Trzebiatowska, 1977; Shibahara, Kuroja, Matsumoto & Ooi, 1984; Kamenar, Kaitner & Strukan, 1991).

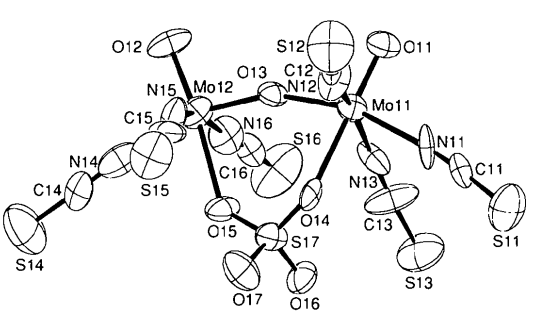


Fig. 1. View of the $[Mo_2O_3(SO_4)(NCS)_6]^{4-}$ anion showing the atom labelling for the crystallographically independent anion (1). The other independent anion (2) has essentially the same features and an analogous labelling scheme.

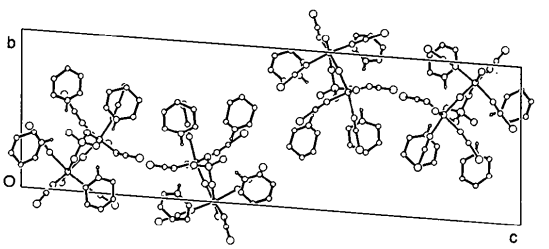


Fig. 2. The crystal structure in projection down *b*. Hydrogen bonds are indicated by broken lines.

Experimental

The title compound was prepared by mixing 0.5 g (0.5 mmol) of $(C_4H_6N)_4[Mo_2O_4(NCS)_6]$ with 0.7 g (3.15 mmol) of 1-(2-thenoyl)-3,3,3-trifluoracetone in 10 cm^3 of CH_3OH. Dark red–brown crystals were grown from the mother liquor. Details of the synthetic procedure will be published elsewhere.

Crystal data

$(C_5H_6N)_4[Mo_2O_3(SO_4)(NCS)_6]$	Mo $K\alpha$ radiation
M_r = 1004.85	λ = 0.71069 Å
Triclinic	Cell parameters from 12 reflections
$P\bar{1}$	θ = 5–11°
a = 8.220 (1) Å	μ = 1.029 mm^{-1}
b = 12.491 (6) Å	T = 293 (2) K
c = 40.138 (21) Å	Prism
α = 93.45 (2)°	0.46 × 0.21 × 0.14 mm
β = 95.13 (4)°	Dark red–brown
γ = 96.44 (2)°	
V = 4069 (3) Å^3	
Z = 4	
D_x = 1.640 Mg m^{-3}	

Data collection

Philips PW1100 diffractometer	R_{int} = 0.296
ω–2θ scans	θ_{max} = 26.12°
Absorption correction: none	h = −10 → 10
17 372 measured reflections	k = −15 → 15
14 446 independent reflections	l = −38 → 36
3231 observed reflections [$I > 2\sigma(I)$]	4 standard reflections frequency: 70 min intensity variation: 10%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max}$ = −0.109
$R(F)$ = 0.0844	$\Delta\rho_{max}$ = 0.886 e Å^{-3}
$wR(F^2)$ = 0.348	$\Delta\rho_{min}$ = −0.770 e Å^{-3}
S = 1.227	Extinction correction: none

14 370 reflections
698 parameters
Only H-atom U's refined
Calculated weights
$w = 1/[\sigma^2(F_o^2) + (0.1633P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Mo11	0.4376 (2)	0.33716 (13)	0.15558 (5)	0.0416 (5)
Mo12	0.2758 (2)	0.12019 (14)	0.09230 (5)	0.0510 (6)
O11	0.6186 (14)	0.3185 (10)	0.1728 (3)	0.053 (4)
O12	0.4235 (18)	0.0469 (12)	0.0796 (4)	0.080 (5)
O13	0.3955 (13)	0.2192 (9)	0.1227 (3)	0.043 (4)
O14	0.1895 (15)	0.3751 (8)	0.1311 (3)	0.041 (3)
O15	0.0546 (14)	0.2033 (10)	0.1052 (4)	0.054 (4)
O16	−0.0576 (16)	0.2967 (10)	0.1493 (3)	0.055 (4)
O17	−0.0570 (17)	0.3680 (11)	0.0945 (4)	0.073 (5)
N11	0.4150 (22)	0.4832 (13)	0.1831 (4)	0.051 (5)
C11	0.3938 (24)	0.5674 (18)	0.1933 (5)	0.049 (6)
S11	0.3515 (10)	0.6833 (5)	0.2071 (2)	0.094 (2)
N12	0.5274 (24)	0.4379 (13)	0.1214 (4)	0.060 (6)
C12	0.5807 (29)	0.5060 (19)	0.1053 (6)	0.063 (7)
S12	0.6518 (10)	0.6049 (6)	0.0840 (2)	0.101 (3)
N13	0.2979 (21)	0.2623 (14)	0.1898 (5)	0.057 (5)
C13	0.1969 (32)	0.2523 (18)	0.2087 (7)	0.085 (9)
S13	0.0702 (9)	0.2418 (6)	0.2363 (2)	0.094 (2)
N14	0.0795 (27)	0.0287 (14)	0.0632 (5)	0.071 (6)
C14	−0.0418 (32)	−0.0258 (18)	0.0498 (5)	0.055 (7)
S14	−0.1948 (10)	−0.0977 (6)	0.0316 (2)	0.118 (3)
N15	0.2777 (24)	0.2332 (14)	0.0563 (5)	0.060 (6)
C15	0.2511 (25)	0.3011 (21)	0.0413 (6)	0.057 (7)
S15	0.2023 (10)	0.3934 (5)	0.0160 (2)	0.090 (2)
N16	0.2106 (23)	0.0268 (13)	0.1319 (5)	0.065 (5)
C16	0.1769 (26)	−0.0133 (17)	0.1575 (6)	0.054 (6)
S16	0.1247 (12)	−0.0680 (6)	0.1879 (2)	0.121 (3)
S17	0.0373 (7)	0.3129 (4)	0.1209 (2)	0.0489 (15)
Mo21	0.9984 (2)	0.2416 (2)	0.34725 (5)	0.0580 (6)
Mo22	0.8242 (3)	0.0016 (2)	0.38508 (5)	0.0686 (7)
O21	1.1708 (15)	0.2245 (12)	0.3303 (4)	0.075 (5)
O22	0.9656 (19)	−0.0861 (12)	0.3930 (4)	0.092 (6)
O23	0.9505 (16)	0.1088 (9)	0.3658 (3)	0.053 (4)
O24	0.7751 (13)	0.2811 (10)	0.3712 (3)	0.048 (4)
O25	0.6350 (18)	0.1067 (11)	0.3808 (4)	0.071 (5)
O26	0.5487 (22)	0.2740 (12)	0.4029 (4)	0.097 (6)
O27	0.5192 (17)	0.2227 (12)	0.3435 (4)	0.080 (5)
N21	0.9871 (24)	0.3997 (17)	0.3344 (5)	0.074 (6)
C21	0.9492 (38)	0.4798 (27)	0.3305 (9)	0.119 (13)
S21	0.8862 (14)	0.6006 (7)	0.3229 (3)	0.153 (4)
N22	1.1003 (26)	0.3124 (17)	0.3899 (6)	0.079 (7)
C22	1.1845 (30)	0.3670 (22)	0.4129 (8)	0.080 (9)
S22	1.2812 (12)	0.4466 (8)	0.4446 (2)	0.143 (4)
N23	0.6310 (31)	−0.0890 (17)	0.4052 (6)	0.099 (9)
C23	0.5232 (44)	−0.1514 (21)	0.4095 (7)	0.096 (10)
S23	0.3671 (12)	−0.2351 (8)	0.4180 (3)	0.151 (4)
N24	0.8705 (23)	0.0900 (18)	0.4316 (6)	0.084 (7)
C24	0.8784 (32)	0.1551 (25)	0.4550 (6)	0.081 (9)
S24	0.8783 (12)	0.2429 (8)	0.4829 (2)	0.133 (4)
N25	0.8344 (22)	0.1946 (15)	0.3043 (5)	0.065 (5)
C25	0.7254 (24)	0.1983 (18)	0.2860 (6)	0.064 (7)
S25	0.5844 (9)	0.2047 (7)	0.2569 (2)	0.102 (3)
N26	0.7382 (24)	−0.0625 (17)	0.3396 (5)	0.071 (6)
C26	0.6793 (34)	−0.0998 (20)	0.3119 (9)	0.092 (9)
S26	0.6166 (10)	−0.1488 (7)	0.2745 (2)	0.107 (3)
S27	0.6118 (7)	0.2218 (4)	0.3736 (2)	0.054 (2)
C31	0.8879 (33)	0.5438 (24)	0.1689 (7)	0.096 (9)
N32	0.8995 (23)	0.4768 (16)	0.1899 (5)	0.078 (6)
C33	0.8847 (37)	0.5001 (26)	0.2236 (8)	0.119 (11)
C34	0.8521 (34)	0.6079 (24)	0.2350 (7)	0.104 (9)
C35	0.8425 (38)	0.6809 (25)	0.2098 (9)	0.122 (11)
C36	0.8427 (38)	0.6452 (26)	0.1747 (9)	0.123 (11)
N41	0.3311 (26)	0.0348 (16)	0.3117 (6)	0.087 (6)
C42	0.3054 (31)	−0.0347 (21)	0.3342 (7)	0.080 (8)
C43	0.1903 (32)	−0.1201 (21)	0.3311 (7)	0.087 (8)
C44	0.0862 (39)	−0.1260 (25)	0.3019 (9)	0.120 (11)
C45	0.1010 (38)	−0.0566 (25)	0.2776 (8)	0.114 (10)
C46	0.2339 (37)	0.0240 (23)	0.2849 (8)	0.103 (9)
N51	0.7694 (22)	0.0970 (15)	0.1545 (5)	0.071 (6)
C52	0.7134 (36)	0.0770 (25)	0.1836 (8)	0.114 (10)
C53	0.6223 (37)	−0.0294 (26)	0.1843 (8)	0.115 (10)
C54	0.6110 (36)	−0.0994 (24)	0.1591 (8)	0.107 (10)
C55	0.6819 (38)	−0.0768 (25)	0.1321 (8)	0.112 (10)
C56	0.7658 (30)	0.0270 (21)	0.1287 (7)	0.082 (8)
N61	0.1723 (30)	0.5710 (20)	0.0905 (7)	0.116 (8)
C62	0.2248 (29)	0.6222 (21)	0.1162 (7)	0.077 (7)
C63	0.2031 (37)	0.7332 (25)	0.1176 (8)	0.114 (10)
C64	0.1230 (40)	0.7740 (26)	0.0908 (9)	0.129 (12)
C65	0.0562 (30)	0.7032 (21)	0.0638 (6)	0.080 (8)
C66	0.0656 (34)	0.5971 (23)	0.0634 (7)	0.099 (9)
N71	0.4153 (28)	0.4149 (19)	0.3179 (7)	0.108 (8)
C72	0.4078 (45)	0.4409 (33)	0.2861 (11)	0.156 (14)
C73	0.3922 (50)	0.5469 (38)	0.2842 (12)	0.176 (16)
C74	0.3898 (41)	0.6145 (29)	0.3107 (10)	0.134 (12)
C75	0.3888 (45)	0.5759 (33)	0.3428 (10)	0.155 (14)
C76	0.4188 (42)	0.4683 (31)	0.3460 (10)	0.137 (12)
N81	0.7575 (24)	0.2914 (17)	0.0399 (5)	0.080 (6)
C82	0.6746 (31)	0.3536 (20)	0.0253 (7)	0.082 (8)
C83	0.5720 (34)	0.3089 (24)	−0.0101 (7)	0.105 (9)
C84	0.5982 (36)	0.2116 (25)	−0.0205 (8)	0.110 (10)
C85	0.7174 (49)	0.1435 (32)	−0.0011 (11)	0.170 (16)
C86	0.7888 (41)	0.1927 (28)	0.0279 (9)	0.127 (11)
N91	0.7623 (33)	0.4696 (21)	0.4259 (8)	0.116 (9)
C92	0.7395 (68)	0.5091 (52)	0.4551 (17)	0.253 (26)
C93	0.7984 (68)	0.6160 (52)	0.4651 (15)	0.246 (25)
C94	0.8513 (55)	0.6761 (37)	0.4399 (14)	0.178 (17)
C95	0.9011 (52)	0.6361 (39)	0.4163 (12)	0.176 (17)
C96	0.8426 (48)	0.5150 (35)	0.4084 (10)	0.146 (14)
C101	0.2727 (38)	0.0473 (26)	0.4951 (8)	0.115 (10)
C102	0.3865 (47)	0.1374 (31)	0.5027 (10)	0.152 (14)
C103	0.4415 (44)	0.1969 (30)	0.4766 (11)	0.149 (14)
N104	0.3883 (28)	0.1579 (20)	0.4460 (6)	0.100 (7)
C105	0.3040 (43)	0.0714 (30)	0.4387 (9)	0.133 (12)
C106	0.2340 (37)	0.0084 (25)	0.4624 (9)	0.116 (11)

Table 2. *Selected geometric parameters* (Å, °)

Mo11—O11	1.63 (1)	Mo21—O21	1.66 (1)
Mo11—O13	1.90 (1)	Mo21—O23	1.88 (1)
Mo11—N12	2.05 (2)	Mo21—N22	1.95 (3)
Mo11—N13	2.07 (2)	Mo21—N21	2.08 (2)
Mo11—N11	2.11 (2)	Mo21—N25	2.10 (2)
Mo11—O14	2.30 (1)	Mo21—O24	2.24 (1)
Mo12—O12	1.70 (1)	Mo22—O22	1.71 (2)
Mo12—O13	1.82 (1)	Mo22—O23	1.85 (1)
Mo12—N14	2.09 (2)	Mo22—N26	1.99 (2)
Mo12—N15	2.08 (2)	Mo22—N24	2.09 (3)
Mo12—N16	2.10 (2)	Mo22—N23	2.09 (2)
Mo12—O15	2.28 (1)	Mo22—O25	2.15 (2)
O11—Mo11—O13	101.3 (6)	O21—Mo21—O23	101.8 (7)
O11—Mo11—N12	94.6 (7)	O21—Mo21—N22	97.0 (8)
O13—Mo11—N12	91.6 (6)	O23—Mo21—N22	93.0 (7)
O11—Mo11—N13	97.9 (6)	O21—Mo21—N21	96.4 (8)
O13—Mo11—N13	94.5 (6)	N22—Mo21—N21	83.1 (8)
O11—Mo11—N11	97.4 (6)	O21—Mo21—N25	97.6 (7)
N12—Mo11—N11	83.4 (6)	O23—Mo21—N25	92.5 (7)
N13—Mo11—N11	86.2 (7)	N21—Mo21—N25	86.7 (8)
O13—Mo11—O14	81.2 (4)	O23—Mo21—O24	82.7 (5)
N12—Mo11—O14	82.4 (6)	N22—Mo21—O24	79.6 (7)
N13—Mo11—O14	84.6 (5)	N21—Mo21—O24	79.0 (6)
N11—Mo11—O14	79.9 (5)	N25—Mo21—O24	85.2 (6)
O12—Mo12—O13	101.4 (7)	O22—Mo22—O23	100.6 (7)
O12—Mo12—N14	96.0 (7)	O22—Mo22—N26	96.9 (8)
O12—Mo12—N15	99.9 (7)	O23—Mo22—N26	89.4 (7)
O13—Mo12—N15	90.5 (6)	O22—Mo22—N24	96.0 (8)
N14—Mo12—N15	88.6 (7)	O23—Mo22—N24	90.0 (7)
O12—Mo12—N16	97.1 (7)	O22—Mo22—N23	95.8 (9)
O13—Mo12—N16	89.1 (6)	N26—Mo22—N23	89.6 (9)
N14—Mo12—N16	86.6 (7)	N24—Mo22—N23	87.3 (9)
O13—Mo12—O15	85.8 (5)	O23—Mo22—O25	85.1 (5)

N14—Mo12—O15	76.8 (6)	N26—Mo22—O25	87.3 (7)
N15—Mo12—O15	80.5 (6)	N24—Mo22—O25	79.7 (7)
N16—Mo12—O15	82.4 (6)	N23—Mo22—O25	78.6 (7)
Mo12—O13—Mo11	157.9 (7)	Mo22—O23—Mo21	156.7 (8)
N11—C11—S11	176 (2)	N21—C21—S21	177 (4)
N12—C12—S12	177 (2)	N22—C22—S22	174 (2)
N13—C13—S13	176 (3)	N23—C23—S23	176 (3)
N14—C14—S14	178 (2)	N24—C24—S24	175 (3)
N15—C15—S15	174 (3)	N25—C25—S25	174 (2)
N16—C16—S16	176 (2)	N26—C26—S26	175 (3)

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
N32—H32···O16^{i}	1.97	2.77 (2)	154
N41—H41···O27	2.07	2.83 (2)	148
N51—H51···O16^{i}	1.91	2.76 (2)	170
N61—H61···O17	2.66	3.01 (3)	106
N71—H71···O27	2.08	2.86 (3)	150
N81—H81···O17^{i}	1.79	2.62 (2)	162
N91—H91···O26	2.06	2.90 (3)	165
N104—H104···O26	1.85	2.67 (3)	159

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

Data collection and cell refinement: *DIF4* (Stoe & Cie, 1992*a*). Data reduction: *REDU4S* (Stoe & Cie, 1992*b*). The structure was solved by Patterson and Fourier methods using *SHELXS86* (Sheldrick, 1985) and refined by full-matrix least-squares on F using *SHELX76* (Sheldrick, 1976) and on F^2 (in order to have a better ratio between the number of observations and the number of refined parameters) using *SHELXL93* (Sheldrick, 1993). Residual indices obtained in the F^2 refinement were $R1 = 0.084$ for 3231 reflections with $F > 4\sigma(F)$ and 698 parameters (scale factor, positional and anisotropic displacement parameters for all atoms of the complex anions, positional and isotropic displacement parameters for C and N atoms of the pyridinium cations and an overall isotropic displacement parameter for H atoms), $wR2 = 0.348$ for 14370 independent reflections and $wR2 = 0.389$ for all 14446 reflections (76 reflections with $\Delta/\sigma > 5$ were omitted). The H atoms attached to the pyridinium cations were positioned geometrically (C—H 0.93 and N—H 0.86 Å) and included as riding atoms in the structure-factor calculations. All structural parameters discussed in the *Comment* are from the F^2 refinement. Software used to prepare molecular graphics and material for publication: *PLUTON* (Spek, 1982); *SHELXL93*; *CSU* (Vicković, 1988).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: NA1097). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Lithium Tris(oxalato-*O*,*O*′)chromate(III) Hexahydrate

AKIKO SEKINE, TAKUHIRO OTSUKA, YUJI OHASHI AND YOUKOH KAIZU

Department of Chemistry, Tokyo Institute of Technology, 2-12-1 O-okayama Meguro-ku, Tokyo 152, Japan

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Abstract

In the title compound, $3Li[Cr(C_2O_4)_3].6H_2O$, the Li cations are coordinated by O atoms of both the oxalato ligands and the solvent water molecules. Two of the six water molecules are not bonded to the Li cations but are instead hydrogen bonded to other water molecules or oxalato ligand molecules. The tris(oxalato)chromate(III) anion takes the form of a distorted octahedron. The average Cr—O distance and O—Cr—O angle of the chelate rings are 1.979 Å and 82.3°, respectively.

Comment

The effect of the environment around the Cr^{3+} ion on its luminescence and relaxation has been studied extensively using various ligands. The relationship between the complex distortion and the relative intensity of the 0–0 band and the vibronic band has been reported (Flint, 1974). We have examined the effect of the solvent water molecules in tris(oxalato)chromate(III) complex crystals on the luminescence intensity of relaxation from ^2E_g to $^4A_{2g}$. In order to elucidate the mechanism, the structure of the title complex has been determined.

The chromate ion (I) is octahedrally coordinated by three oxalato ligands. The averages of the six Cr—O bonds and the three O—Cr—O angles of the chelate rings are 1.979 Å and 82.3°, respectively, which are very similar to the corresponding values for the