different tetrahedral sites: one with average Li(1)—O distance of 2·00 Å and another, larger and more mobile, with average Li(2)—O distance of 2·35 Å.

References

EFREMOV, B. A. & KALININ, B. V. (1978). *Kristallografiya*, **23**, 703–705.

FISCHER, R. X. (1985). *J. Appl. Cryst.* **18**, 258–262.

HAMDOUNE, S. (1987). Thesis. Laboratoire de Cristallographie, CNRS, Grenoble, France.

HAMDOUNE, S., TRAN QUI, D. & SCHOULER, E. J. L. (1986). *Solid State Ionics*, **19**, 587–591.

HONG, H. Y. P. (1979). International Conference on Fast-Ion Transport in Solids, Lake Geneva, Wisconsin, USA.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

LARSON, A. C., LEE, F. L., LE PAGE, Y. & GABE, E. J. (1982). NRC VAX *Crystal Structure System*. NRC, Ottawa. Canada.

LI SHI-CHUN & LIN ZU-XIANG (1983). *Solid State Ionics*, **10**, 835–838.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

TRAN QUI, D. & HAMDOUNE, S. (1987). *Acta Cryst.* C**43**, 397–399.

Acta Cryst. (1988). C**44**, 1362–1365

Structures of Trisodium Hexakis(formato)chromate(III) Tetrahydrate and Trisodium Hexakis(formato)molybdate(III)

BY ALAN HAZELL

Institute of Chemistry, Aarhus University, DK-8000 Århus C, Denmark

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Abstract. $Na_3[Cr(CHO_2)_6].4H_2O$, $M_r = 463{\cdot}15$, triclinic, $P\bar{1}$, $a = 6{\cdot}463\,(1)$, $b = 7{\cdot}678\,(2)$, $c = 8{\cdot}741\,(2)$ Å, $\alpha = 101{\cdot}46\,(1)$, $\beta = 97{\cdot}26\,(1)$, $\gamma = 78{\cdot}74\,(1)°$, $V = 415{\cdot}2\,(2)$ Å^3, $Z = 1$, $D_x = 1{\cdot}852$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 0{\cdot}824$ mm^{-1}, $F(000) = 235$, 298 K, $R(F) = 0{\cdot}023$ for 2221 reflexions [$I > 3\sigma(I)$] and 149 variables. $Na_3[Mo(CHO_2)_6]$, $M_r = 435{\cdot}02$, orthorhombic, *Pccn*, $a = 14{\cdot}630\,(6)$, $b = 6{\cdot}383\,(2)$, $c = 13{\cdot}888\,(5)$ Å, $V = 1296{\cdot}9\,(8)$ Å^3, $Z = 4$, $D_x = 2{\cdot}228$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 1{\cdot}143$ mm^{-1}, $F(000) = 852$, 293 K, $R = 0{\cdot}026$ for 1524 reflexions [$I > 3\sigma(I)$] and 113 variables. In both complexes the metal atom is bonded to six formate ions, two with *M*—O—C—O*t* in the *sp* and four in the *ap* conformation. $[Cr(CHO_2)_6]^{3-}$ is centrosymmetric with Cr—O distances of 1·973 (1), 1·979 (1) and 1·992 (1) Å; the long Cr—O bond is to an O atom which is hydrogen bonded to water. $[Mo(CHO_2)_6]^{3-}$ has twofold symmetry; the Mo—O distances are 2·116 (1), 2·113 (1) and 2·110 (1) Å. In both cases, the coordination octahedra are distorted, the r.m.s. deviations of the angles from ideality being 2·4° for CrO_6 and 5·2° for MoO_6.

Introduction. Compounds of the type $Na_3[M^{III}(CHO_2)_6]$ seem only to be known for four metals, Fe (Weinland & Reihlen, 1913), V (Barbieri, 1916), Cr (Stiasny & Walther, 1928) and Mo (Brorson & Schäffer, 1986). The structures of several formate complexes of divalent metals are known which contain $[M^{II}(CHO_2)_6]$ octahedra but in most of these (Weber, 1980, and references therein) the formates are bridges between the metal atoms. Formate groups can be bonded with *M*—O—C—O*t* (O*t* = terminal O atom) in the *sp* or *ap* conformation, *i.e.* the torsion angle can be 0 or 180°, an extreme case of the former is when the formate is bidentate as in $[Mo(CO)_2(PEt_3)_2(CHO_2)_2]$ (Brower, Winston, Tonker & Templeton, 1986). The structures of the Cr and the Mo complexes are reported here.

Experimental. The chromium compound was synthesized by M. Brorson and C. Schäffer of Copenhagen University using Stiasny & Walther's (1928) method and recrystallized by adding ethanol to an aqueous solution. A lath-shaped crystal, 0·45 × 0·22 × 0·18 mm, was mounted on a Picker FACS-1 diffractometer. Cell dimensions were determined from the setting angles of 25 reflections with $13 \leq \theta \leq 20°$. Intensities were measured out to $[(\sin\theta)/\lambda]_{\max} = 0{\cdot}7$ Å^{-1} using an ω–2θ scan and Nb-filtered Mo $K\alpha$ radiation, scan width $(2{\cdot}0 + 0{\cdot}692\tan\theta)°$, step length 0·04°, 1 s step^{-1}. Reflexions with $0 \leq h \leq 9$, $-10 \leq k \leq 10$, $-12 \leq l \leq 12$ measured giving 2421 independent reflexions of which 2221 had $I > 3\sigma_{cs}(I)$. Reflexions 400 and 222 were monitored every 60 reflexions, the overall fall off in intensity was 3·7%. No correction was made for absorption.

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Table 1. $Na_3[Cr(CHO_2)_6].4H_2O$; *fractional atomic coordinates* (× 10^4 *for* Cr, Na, O *and* C, × 10^3 *for* H) *and* U_{eq} (Å^2 × 10^{-4})

	x	*y*	*z*	U_{eq}*
Cr	0	0	0	157 (1)
Na(1)	0	5000	5000	372 (5)
Na(2)	2899 (1)	−4464 (1)	−1259 (1)	286 (3)
O(1)	949 (1)	2325 (1)	845 (1)	236 (4)
O(2)	3271 (2)	4170 (1)	1078 (1)	274 (5)
O(3)	2697 (1)	−1502 (1)	646 (1)	227 (4)
O(4)	5185 (2)	−2471 (1)	2421 (1)	350 (6)
O(5)	1024 (2)	102 (1)	−2029 (1)	251 (5)
O(6)	2309 (2)	−2722 (1)	−3224 (1)	388 (6)
O(7)	159 (2)	3793 (2)	−2690 (1)	312 (6)
O(8)	3317 (3)	3074 (2)	4584 (2)	510 (8)
C(1)	2792 (2)	2648 (2)	808 (2)	240 (6)
C(2)	3638 (2)	−1399 (2)	2038 (2)	260 (6)
C(3)	1812 (3)	−1085 (2)	−3150 (2)	314 (7)
H(1)	390 (3)	165 (2)	53 (2)	279 (42)
H(2)	318 (3)	−46 (3)	284 (2)	350 (47)
H(3)	202 (3)	−49 (3)	−414 (2)	496 (61)
H(71)	34 (4)	283 (3)	−257 (3)	593 (71)
H(72)	−82 (4)	418 (3)	−230 (3)	495 (64)
H(81)	410 (6)	341 (4)	443 (4)	1005 (146)
H(82)	380 (4)	283 (4)	548 (3)	709 (78)

* $U_{eq} = \frac{1}{3}\sum_i \sum_j U_{ij} a_i^* a_i^* \mathbf{a}_i.\mathbf{a}_j$.

Table 2. $Na_3[Mo(CHO_2)_6]$; *fractional atomic coordinates* (× 10^5 *for* Mo, × 10^4 *for* Na, O *and* C, × 10^3 *for* H) *and* U_{eq} (Å^2 × 10^{-4} *for* Mo, Å^2 × 10^{-3} *for* Na, O, C *and* H)

	x	*y*	*z*	U_{eq}*
Mo	25000	25000	6318 (2)	138 (2)
Na(1)	2500	−2500	4247 (1)	21 (1)
Na(2)	27 (1)	2758 (1)	2193 (1)	26 (1)
O(1)	1669 (1)	3885 (2)	1704 (1)	22 (1)
O(2)	1520 (1)	6039 (3)	2960 (1)	30 (1)
O(3)	1790 (1)	−369 (2)	497 (1)	22 (1)
O(4)	547 (1)	−162 (3)	1422 (1)	38 (1)
O(5)	1734 (1)	4084 (3)	−430 (1)	26 (1)
O(6)	575 (1)	4375 (3)	−1444 (1)	37 (1)
C(1)	1984 (1)	5005 (3)	2391 (2)	23 (1)
C(2)	1062 (1)	−1096 (4)	878 (2)	24 (1)
C(3)	1023 (2)	3417 (4)	−842 (2)	30 (1)
H(1)	268 (2)	496 (6)	243 (3)	55 (11)
H(2)	92 (3)	−260 (4)	73 (2)	29 (10)
H(3)	90 (2)	187 (6)	−76 (2)	37 (9)

* $U_{eq} = \frac{1}{3}\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

Table 3. *Bond distances* (Å), *angles* (°), *torsion angles* (°) *and geometry around* Na^+ *ions*

$Na_3[Cr(CHO_2)_6].4H_2O$

Symmetry code: (i) $x, y, 1+z$; (ii) $x, 1+y, 1+z$; (iii) $x, y-1, z$; (iv) $-x, 1-y, -z$; (v) $-x, -y, -z$; (vi) $-x, 1-y, 1-z$; (vii) $1-x, -1-y, -z$; (viii) $1-x, -y, -z$.

Cr—O(1)	1·974 (1)	O(3)—C(2)	1·284 (2)
Cr—O(3)	1·978 (1)	O(4)—C(2)	1·222 (2)
Cr—O(5)	1·991 (1)	O(5)—C(3)	1·280 (2)
O(1)—C(1)	1·269 (2)	O(6)—C(3)	1·226 (2)
O(2)—C(1)	1·237 (2)		

O(1)—Cr—O(3)	95·01 (4)	O(3)—Cr—O(5)	91·92 (4)
O(1)—Cr—O(3^v)	84·99 (4)	O(3)—Cr—O(5^v)	88·08 (4)
O(1)—Cr—O(1^v)	180·00	O(5)—Cr—O(5^v)	180·00
O(1)—Cr—O(5)	87·54 (4)	Cr—O(3)—C(2)	126·3 (1)
O(1)—Cr—O(5^v)	92·46 (4)	O(3)—C(2)—O(4)	124·2 (1)
O(3)—Cr—O(3^v)	180·00	Cr—O(5)—C(3)	134·4 (1)
Cr—O(1)—C(1)	125·6 (1)	O(5)—C(3)—O(6)	128·5 (1)
O(1)—C(1)—O(2)	124·9 (1)		

Cr—O(1)—C(1)—O(2)	166·3 (1)	C(1)—O(1)—Cr—O(5)	−53·8 (1)
Cr—O(3)—C(2)—O(4)	172·1 (1)	C(3)—O(5)—Cr—O(1^v)	−31·6 (1)
Cr—O(5)—C(3)—O(6)	−5·7 (3)	C(2)—O(3)—Cr—O(1)	54·9 (1)
C(1)—O(1)—Cr—O(3)	37·9 (1)	C(3)—O(5)—Cr—O(3)	53·4 (1)
C(2)—O(3)—Cr—O(5^v)	−37·4 (1)		

Na(1)—O(7^i)	2·367 (1)	Na(2)—O(7^{iii})	2·489 (1)
Na(1)—O(8)	2·379 (1)	Na(2)—O(2^{viii})	2·511 (1)
Na(1)—O(6^{ii})	2·655 (1)	Na(2)—O(4^{vii})	2·516 (1)
Na(2)—O(6)	2·332 (1)	Na(2)—O(3)	2·529 (1)
Na(2)—O(2^{iii})	2·441 (1)	Na(2)—O(1^v)	2·722 (1)

$Na_3[Mo(CHO_2)_6]$

Symmetry code: (i) $0{\cdot}5-x, 0{\cdot}5-y, z$; (ii) $-x, 1-y, z$; (iii) $x, y-1, z$; (iv) $0{\cdot}5-x, y, 0{\cdot}5+z$; (v) $0{\cdot}5-x, y-1, 0{\cdot}5+z$; (vi) $-x, 0{\cdot}5+y, 0{\cdot}5-z$; (vii) $-x, y-0{\cdot}5, 0{\cdot}5-z$; (viii) $x, 0{\cdot}5-y, 0{\cdot}5+z$; (ix) $x, -0{\cdot}5-y, 0{\cdot}5+z$.

Mo—O(1)	2·116 (1)	O(3)—C(2)	1·277 (3)
Mo—O(3)	2·114 (1)	O(4)—C(2)	1·222 (3)
Mo—O(5)	2·110 (1)	O(5)—C(3)	1·261 (3)
O(1)—C(1)	1·279 (3)	O(6)—C(3)	1·226 (3)
O(2)—C(1)	1·233 (3)		

O(1)—Mo—O(1^i)	90·57 (8)	O(1)—Mo—O(3^i)	89·02 (6)
O(1)—Mo—O(5)	89·21 (6)	O(3)—Mo—O(5^i)	77·51 (6)
O(1)—Mo—O(5^i)	175·68 (7)	O(5)—Mo—O(5^i)	91·33 (9)
O(1)—Mo—O(3)	98·17 (6)	Mo—O(3)—C(2)	133·5 (1)
O(3)—Mo—O(5)	95·28 (6)	O(3)—C(2)—O(4)	126·4 (2)
O(3)—Mo—O(3^i)	169·80 (7)	Mo—O(5)—C(3)	126·4 (2)
Mo—O(1)—C(1)	123·5 (1)	O(5)—C(3)—O(6)	125·6 (2)
O(1)—C(1)—O(2)	125·4 (2)		

Mo—O(1)—C(1)—O(2)	170·0 (2)	C(1)—O(1)—Mo—O(3^i)	−45·4 (2)
Mo—O(3)—C(2)—O(4)	3·9 (4)	C(3)—O(5)—Mo—O(1)	−89·6 (2)
Mo—O(5)—C(3)—O(6)	178·4 (2)	C(2)—O(3)—Mo—O(1)	3·4 (4)
C(1)—O(1)—Mo—O(1^i)	52·8 (2)	C(3)—O(5)—Mo—O(3)	8·5 (2)
C(2)—O(3)—Mo—O(5)	−86·6 (2)		

Na(1)—O(3)	2·438 (2)	Na(2)—O(6^{viii})	2·465 (2)
Na(1)—O(2^i)	2·473 (2)	Na(2)—O(4^{vi})	2·483 (2)
Na(1)—O(5^v)	2·493 (2)	Na(2)—O(2^{vii})	2·524 (2)
Na(2)—O(4)	2·280 (2)	Na(2)—O(1)	2·598 (2)
Na(2)—O(6^{ii})	2·282 (2)		

The structure was determined from Patterson and Fourier syntheses, H-atom positions were determined from a difference map. Least-squares refinement (on *F*), all non-H atoms refined anisotropically. Anomalous-dispersion factors were included for Cr, no extinction correction was necessary. The weighting scheme was $w = 1/\sigma(F)^2$, where $\sigma(F) = [\sigma_{cs}(F^2) + 1{\cdot}02F^2]^{1/2} - |F|$ and $\sigma_{cs}(F^2)$ is the standard deviation of F^2 from counter statistics. Final $R = 0{\cdot}023$,* $wR = 0{\cdot}035$, $S = 1{\cdot}76$, $(\Delta/\sigma)_{max} = 0{\cdot}02$, $\Delta\rho$ 0·29 (3) to −0·29 (5) e Å^{-3}. Fractional coordinates are listed in Table 1, bond distances and angles in Table 3.

The molybdenum complex was synthesized by Brorson & Schäffer (1986). A tabular crystal, 0·45 × 0·40 × 0·10 mm, was mounted on a Huber diffractometer. Cell dimensions were determined from the setting angles of 30 reflexions, $8 \leq \theta \leq 16°$. Intensities were measured out to $[(\sin\theta)/\lambda]_{max} = 0{\cdot}7$ Å^{-1} using an ω–2θ

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* Lists of structure factors, anisotropic thermal parameters and some bond distances and angles for both compounds have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44903 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

scan and Nb-filtered Mo $K\alpha$ radiation, scan width $(1{\cdot}2 + 0{\cdot}692\tan\theta)^\circ$ with 50 steps scan^{-1}. A data set was first collected with 1 s step^{-1}, reflexions with l odd (to which Mo does not contribute) were remeasured with 2 s step^{-1}. Reflexions with $0 \leq h \leq 20$, $0 \leq k \leq 8$, $-19 \leq l \leq 19$ were measured giving 1896 independent reflexions of which 1524 had $I > 3\sigma_{cs}(I)$, R_{int} was 0·028. Reflexions 400 and 006 were monitored every 50 reflexions, the overall fall off in intensity was 3·0%. The data were corrected for absorption, transmission factors ranged from 0·659 to 0·889.

The structure was determined by the heavy-atom method, H-atom positions were located from a difference map. Least-squares refinement (on F), all non-H atoms refined anisotropically. Anomalous-dispersion factors were included for Mo, no extinction correction was necessary. The weighting scheme was $w = 1/\sigma(F)^2$ where $\sigma(F) = [\sigma_{cs}(F^2) + 1{\cdot}03F^2]^{1/2} - |F|$ and $\sigma_{cs}(F^2)$ is the standard deviation of F^2 from counter statistics. Final $R = 0{\cdot}026$,* $wR = 0{\cdot}040$, $S = 1{\cdot}28$, $(\Delta/\sigma)_{max} = 0{\cdot}08$, $\Delta\rho$ 0·8 (1) to −1·5 (1) e Å^{-3}. Fractional coordinates are listed in Table 2, bond distances and angles in Table 3.

Computations were carried out on a VAX 11/780 computer with the following programs: *INTEG* based on the Nelmes (1975) algorithm for integration of intensities of the Cr complex, *COLL5* based on the Lehmann & Larsen (1974) algorithm for integrating the intensities of the Mo complex; *DATAP* and *DSORT* (State University of New York) for data processing; modified *ORFLS* (Busing, Martin & Levy, 1962) for least-squares refinement; *ORFFE* (Busing, Martin & Levy, 1964) for geometry; *ORTEP* (Johnson, 1965) for drawings. Scattering curves those of Cromer & Mann (1968) for Mo, Cr, Na, O, and C; those of Stewart, Davidson & Simpson (1965) for H; anomalous-dispersion corrections for Cr and Mo from Cromer & Liberman (1970).

Discussion. In the Cr complex (Fig. 1), the Cr atom which is situated on a symmetry centre is bonded to six unidentate formate ions. The six O atoms of the inner coordination sphere form a somewhat distorted octahedron† ($\Delta = 2{\cdot}4^\circ$). The Cr—O distances are 1·973 (1), 1·979 (1) and 1·992 (1) Å. O(5) is hydrogen bonded to a water molecule, O(5)···O(7) is 2·937 (1) Å and the angle O(5)···H(71)—O(7) is 175 (2)°. Cr, O(5), C(3), O(6), H(71) and O(7) are nearly coplanar. The long Cr—O bond is that to the hydrogen-bonded O(5) atom. The hydrogen-bonded formate has Cr—O—C—Ot in the sp conformation. The complex anion is surrounded by 12 Na atoms which are in layers at $y \simeq 0{\cdot}5$. Na(1) which is on a symmetry centre is surrounded by a distorted octahedron ($\Delta = 3{\cdot}5^\circ$) of O atoms, Na(2) by seven O atoms which form a distorted pentagonal bipyramid ($\Delta = 7{\cdot}2^\circ$). The Na—O distances range from 2·334 (1) to 2·722 (1) Å. The only O atom for which there is no short contact to Na is O(5) which is hydrogen bonded to a water molecule.

In the Mo complex (Fig. 2) the Mo atom is situated on a twofold axis and is bonded to six unidentate formate ions. The six O atoms of the inner coordination sphere form a somewhat distorted octahedron ($\Delta = 5{\cdot}2^\circ$). The Mo—O distances are 2·116 (1), 2·113 (1) and 2·110 (1) Å, *cf.* 2·089 (1) Å in $Cs[Mo(H_2O)_6](SO_4)_2.6H_2O$ (Brorson & Gajhede, 1987). As in the Cr complex, two formate groups have M—O—C—Ot in the sp conformation; here also they are *trans* to each other although this is not required by symmetry unlike the centrosymmetric Cr complex. The anion and the Na$^+$ ions are in layers at $y \simeq 0{\cdot}25$.

The bond lengths in the formate group are very similar for the two complexes, Table 4, and are much closer to those in the free acid than to those in the formate ion. Here the terminal C—Ot group is significantly shorter than that joined to the metal. Cotton and co-workers (Cotton, Darensbourg & Kolthammer, 1981*a*,*b*; Cotton, Darensbourg, Kolthammer & Kudarski, 1982) have shown that the geometry of carboxyl groups depends on the electrophilic character of the acceptor to which they are bonded: for good electrophiles C—Ot is short whereas for poor electrophiles, *e.g.* $Cr(CO)^{5+}$, the two C—O bonds are almost

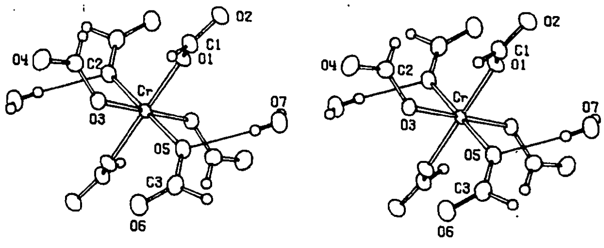

Fig. 1. Stereoscopic drawing of $[Cr(CHO_2)_6]^{3-}.2H_2O$.

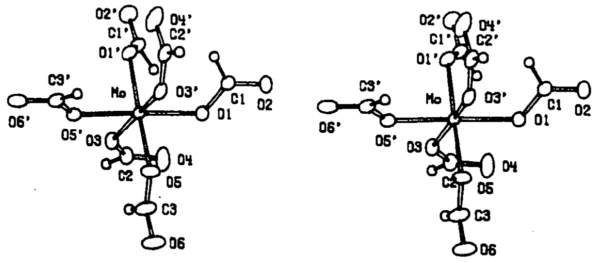

Fig. 2. Stereoscopic drawing of $[Mo(CHO_2)_6]^{3-}$.

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* See deposition footnote.

† The deviation, Δ, of a polyhedron MX_n from the ideal polyhedron MY_n is measured by considering the angles $\delta\theta$ between MX_i and MY_i and rotating MY_n so that $n\Delta^2 = \sum(\delta\theta)^2$ is a minimum. The centres of the two polyhedra are assumed to coincide. This one-parameter measure of the deviation is closely related to that proposed by Dollase (1974), *i.e.* the r.m.s. distance between the vertices of the observed and ideal polyhedra.

Table 4. *Bond lengths* (Å) *in the two complexes compared with those in the free acid and in the formate ion*

	Cr[a]	Mo[a]	Free acid[b]	Formate ion[c]
C–O–	1·278 (1)	1·272 (2)	1·309 (2)	1·2561 (4)
C–O*t*	1·228 (1)	1·227 (1)	1·222 (2)	1·2561 (4)

References: (*a*) this work; (*b*) Nahringbauer (1978); (*c*) Fuess & Bats (1982).

identical. C—H distances range from 0·93 (2) to 1·09 (2) Å in the Cr complex and from 1·01 (4) to 1·02 (4) Å in the Mo complex.

In the hexaformato complexes, the *M*—O—C—O geometry can be characterized by (*a*) the planarity of *M*—O—C—O*t*, (*b*) its rotation about the *M*—O bond, (*c*) an in-plane deformation of the *M*—O—C angle and (*d*) whether *M*—O—C—O*t* is *sp* or *ap*. The *M*—O—C—O torsion angles are close to zero or 180°, *i.e.* the atoms are almost coplanar. The torsion angles τ = C—O—*M*—O might be expected to be 0 or 180°, *i.e.* the formate group to lie close to an O—*M*—O plane or to be about 45°, *i.e.* the plane of the formate group bisecting two O—*M*—O planes. For the Cr complex, all formates are 'bisecting', for the Mo complex two are 'bisecting' and four are 'in-plane'. In $Sr[Cu(CHO_2)_6].8H_2O$ (Baggio, de Perazzo & Polla, 1985) four are 'bisecting'. The in-plane deformation is greatest for the *sp* formate groups with *M*—O—C being 5–10° larger than for the *ap* formates.

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References

Baggio, R. F., de Perazzo, P. K. & Polla, G. (1985). *Acta Cryst.* **C41**, 194–197.
Barbieri, G. A. (1916). *Atti R. Accad. Naz. Lincei Rend. Cl. Sci. Fis. Mat. Nat.* **25**, 723–730.
Brorson, M. & Gajhede, M. (1987). *Inorg. Chem.* **26**, 2109–2112.
Brorson, M. & Schäffer, C. (1986). *Acta Chem. Scand. Ser. A*, **40**, 358–360.
Brower, D. C., Winston, P. B., Tonker, T. L. & Templeton, J. L. (1986). *Inorg. Chem.* **25**, 2883–2888.
Busing, W. R., Martin, K. O. & Levy, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee, USA.
Busing, W. R., Martin, K. O. & Levy, H. A. (1964). *ORFFE*. Report ORNL-TM-306. Oak Ridge National Laboratory, Tennessee, USA.
Cotton, F. A., Darensbourg, D. J. & Kolthammer, B. W. S. (1981*a*). *J. Am. Chem. Soc.* **103**, 398–405.
Cotton, F. A., Darensbourg, D. J. & Kolthammer, B. W. S. (1981*b*). *Inorg. Chem.* **20**, 1287–1291.
Cotton, F. A., Darensbourg, D. J., Kolthammer, B. W. S. & Kudarski, R. (1982). *Inorg. Chem.* **21**, 1656–1662.
Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
Dollase, W. A. (1974). *Acta Cryst.* **A30**, 513–517.
Fuess, H. & Bats, J. W. (1982). *Acta Cryst.* **B38**, 736–743.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Lehmann, M. S. & Larsen, F. K. (1974). *Acta Cryst.* **A30**, 580–584.
Nahringbauer, I. (1978). *Acta Cryst.* **B34**, 315–318.
Nelmes, R. J. (1975). *Acta Cryst.* **A31**, 273–279.
Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
Stiasny, E. & Walther, G. (1928). *Collegium* (*Haltingen*), pp. 389–425.
Weber, G. (1980). *Acta Cryst.* **B36**, 3107–3109.
Weinland, R. F. & Reihlen, H. (1913). *Chem. Ber.* **46**, 3144–3150.

Acta Cryst. (1988). C**44**, 1365–1368

Structure of *trans*-Dioxo(oxoacetato)dipyridineosmium(VI)–Methanol (2:1)

By P. D. Robinson

Department of Geology, Southern Illinois University, Carbondale, IL 62901, *USA*

and C. C. Hinckley* and P. A. Kibala

Department of Chemistry and Biochemistry, Southern Illinois University, Carbondale, IL 62901, *USA*

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Abstract. $[OsO_2(C_2H_2O_3)(C_5H_5N)_2]_2.CH_3OH$, M_r = 940·92, monoclinic, $P2_1/n$, a = 11·014 (2), b = 16·938 (4), c = 15·343 (4) Å, β = 92·68 (2)°, V = 2859 (1) Å^3, Z = 4, D_x = 2·19 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 89·58 cm^{-1}, $F(000)$ = 1784, T = 295 K, R = 0·037, 2994 unique observed reflections. The compound was prepared by combining dipotassium tetramethylosmate(VI) with glycolic acid and

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* To whom all correspondence should be addressed.