C(61)	0.2622 (4)	0.0225 (3)	0.0598 (3)	0.060 (5)
C(62)	0.2749	−0.0068	0.0077	0.091 (7)
C(63)	0.2633	0.0273	−0.0423	0.080 (6)
C(64)	0.2389	0.0905	−0.0402	0.071 (6)
C(65)	0.2262	0.1198	0.0119	0.065 (5)
C(66)	0.2378	0.0858	0.0619	0.042 (4)
C(71)	0.0916 (4)	0.3336 (3)	0.2666 (3)	0.069 (5)
C(72)	0.0919	0.3349	0.3256	0.090 (6)
C(73)	0.1052	0.2786	0.3560	0.086 (6)
C(74)	0.1181	0.2210	0.3274	0.071 (6)
C(75)	0.1178	0.2197	0.2684	0.054 (4)
C(76)	0.1045	0.2760	0.2380	0.051 (4)
C(81)	−0.0342 (5)	0.3099 (4)	0.1625 (3)	0.072 (6)
C(82)	−0.0915	0.3482	0.1485	0.109 (8)
C(83)	−0.0825	0.4026	0.1146	0.093 (7)
C(84)	−0.0161	0.4187	0.0948	0.087 (6)
C(85)	0.0412	0.3804	0.1088	0.060 (5)
C(86)	0.0322	0.3260	0.1426	0.047 (4)
C(100)	0.5092 (7)	0.1373 (7)	0.0465 (5)	0.083 (6)
Cl(1)	0.4363 (2)	0.0895 (2)	0.0457 (2)	0.087 (2)
Cl(2)	0.5865 (2)	0.0923 (2)	0.0550 (2)	0.115 (2)

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

Au—P(1)	2.345 (3)	Au—Cl	2.929 (3)
Au—P(2)	2.391 (3)	Au···Fe(1)	4.850 (5)
Au—P(3)	2.405 (3)	Au···Fe(2)	4.280 (5)
P(1)—Au—Cl	100.5 (1)	P(2)—Au—Cl	105.3 (1)
P(1)—Au—P(2)	124.0 (1)	P(2)—Au—P(3)	104.6 (1)
P(1)—Au—P(3)	126.2 (1)	P(3)—Au—Cl	86.1 (1)

The structure was determined by direct methods using *SHELXTL-Plus* (Sheldrick, 1992) and refined using anisotropic displacement parameters for all non-H atoms and rigid-group constraints for the phenyl rings.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving non-H atoms have been deposited with the IUCr (Reference: MU1114). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Al-Baker, S., Hill, W. E. & McAuliffe, C. A. (1985). *J. Chem. Soc. Dalton Trans.* pp. 2655–2659.

Al-Sâady, A. K., McAuliffe, C. A., Moss, K., Parish, R. V. & Fields, R. (1984). *J. Chem. Soc. Dalton Trans.* pp. 491–493.

Attar, S., Bearden, W. H., Alcock, N. W., Alyea, E. C. & Nelson, J. H. (1990). *Inorg. Chem.* **29**, 425–433.

Beusch, W., Prelati, M. & Ludwig, W. (1986). *J. Chem. Soc. Chem. Commun.* pp. 1762–1763.

Colburn, C. B., Hill, W. E., McAuliffe, C. A. & Parish, R. V. (1979). *J. Chem. Soc. Chem. Commun.* p. 218–219.

Gimeno, M. C., Laguna, A., Sarroca, C. & Jones, P. G. (1993). *Inorg. Chem.* **32**, 5926–5932.

Houlton, A., Mingos, D. M. P., Murphy, D. M., Williams, D. J., Phang, L.-Y. & Hor, T. S. A. (1993). *J. Chem. Soc. Dalton Trans.* pp. 3629–3630.

Jones, P. G. (1981). *Gold Bull.* **14**, 102–118, 159–166.

Jones, P. G. (1983). *Gold Bull.* **16**, 114–124.

Jones, P. G., Sheldrick, G. M., Muir, J. A., Muir, M. M. & Bird Pulgar, L. (1982). *J. Chem. Soc. Dalton Trans.* pp. 2123–2125.

McAuliffe, C. A., Parish, R. V. & Randall, P. D. (1979). *J. Chem. Soc. Dalton Trans.* pp. 1730–1735.

Muir, J. A., Muir, M. M., Arias, S., Jones, P. G. & Sheldrick, G. M. (1984). *Inorg. Chim. Acta*, **81**, 169 174.

Parish, R. V., Parry, O. & McAuliffe, C. A. (1981). *J. Chem. Soc. Dalton Trans.* pp. 2098–2104.

Sheldrick, G. M. (1992). *SHELXTL-Plus*. PC version. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Shieh, S. J., Peng, S.-M. & Che, C.-M. (1993). *J. Chem. Soc. Dalton Trans.* pp. 195–196.

Togni, A., Pastor, S. D. & Rihs, G. (1990). *J. Organomet. Chem.* **381**, C21–C25.

Acta Cryst. (1995). **C51**, 32–34

Disodium μ-(Ethylenediaminetetraacetato-*N*,*O*,*O*′:*N*′,*O*″,*O*‴)-di-μ-oxo-bis[oxomolybdate(V)](*Mo*—*Mo*) Tetrahydrate

K. MUKHERJEE AND P. ROYCHOWDHURY*

Department of Physics, University College of Science, 92 A.P.C. Road, Calcutta 700009, India

K. ROY AND D. MUKHERJEE

Department of Chemistry, University College of Science, 92 A.P.C. Road, Calcutta 700009, India

M. KUSUNOKI

Institute for Protein Research, Osaka University, 3-2, Yamadaoka, Suita, Osaka 565, Japan

(*Received* 4 *January* 1993; *accepted* 2 *November* 1993)

Abstract

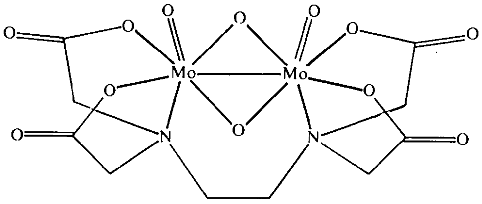
In the title compound, $2Na[Mo_2O_4(C_{10}H_{12}N_2O_8)].4H_2O$, the hexadentate ethylenediaminetetraacetate (edta) ligand coordinates to each Mo atom through two carboxylate O atoms and an N atom while two bridging O atoms and a terminal O atom complete the coordination sphere around each Mo centre.

Comment

The $[Mo_2O_4(edta)]^{2}$ anion, (I), consists of two Mo atoms each coordinated in a distorted octahedral environment by five O atoms and an N atom. The hexadentate ethylenediaminetetraacetate (edta) ligand coordinates to each Mo atom through two carboxylate O atoms and an N atom. The Mo═O(carboxylate) distances are within the expected range for O atoms σ-bonded to Mo and are similar to those found in the structure $Cs_2[Mo^{V}(\mu\text{-}S)_2(\mu\text{-}edta)O_2]^{2}$ (Spivack & Dori, 1973). They are also comparable to the value of 2.12 A found in $Ba[Mo_2O_4(C_2O_4)_2(H_2O)_2]$ (Cotton & Morehouse, 1965). The

Acta Crystallographica Section C
ISSN 0108-2701 ©1995

edta carboxylate groups in the title compound have bond lengths comparable to those found in the carboxylic group of uncoordinated H_4edta (Cotrait, 1972). The distance between the two ethylene C atoms [1.542 (8) Å], is larger than that found in uncoordinated H_4edta [1.512 (2) Å] (Cotrait, 1972). A similarly large value was found by Spivack & Dori (1973).



(I)

The Mo atoms are displaced by 0.392 (1) and 0.376 (1) Å, respectively, from the plane containing O(5), O(6), O(7), O(8), O(1) and O(2) towards the terminal O atoms. Similar behaviour was observed by Spivack & Dori (1973). The multiple bonding between the Mo and the terminal O atoms is probably responsible for this type of tetragonal distortion and the short Mo—O distances of 1.691 (5) and 1.696 (4) Å. There is direct bonding between the two Mo atoms [2.554 (1) Å] which indicates coupling of electron spins on adjacent Mo^V ions through metal–metal bonding (Kay & Mitchell, 1968). This bond is comparable to that (2.54 Å) in the compound studied by Cotton & Morehouse (1965).

The hydrogen bonding in the crystal is confined to three weak interactions [O(1*W*)···O(4) = 2.967 (8), O(2)···O(2*W*) = 2.94 (1), O(6)···O(3*W*) = 2.99 (1) Å] and strong interactions between different water molecules [O(1*W*)···O(2*W*) = 2.88 (2), O(3*W*)···O(4*W*) = 2.73 (2) Å].

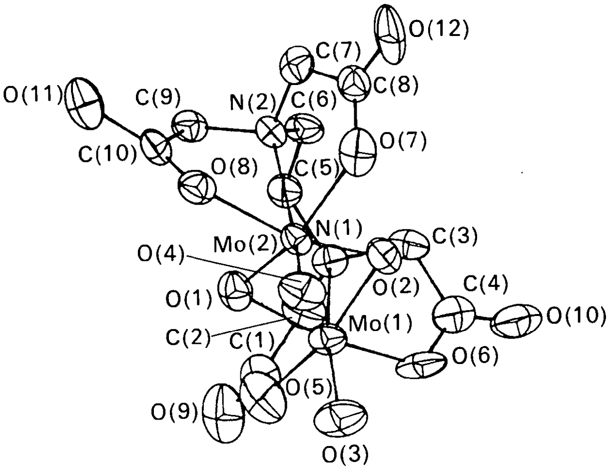


Fig. 1. View of the anion of the title compound. Displacement ellipsoids are shown at the 75% probability level.

Experimental

Crystals were prepared from a dilute solution of a pure sample of $2Na[Mo_2O_4(edta)]$ by allowing a 50 cm^3 solution to evaporate for about 45 days at room temperature.

Crystal data

$2Na[Mo_2O_4(C_{10}H_{12}N_2O_8)].4H_2O$
$M_r = 662.13$
Monoclinic
Pa
$a = 7.540$ (3) Å
$b = 16.761$ (6) Å
$c = 8.650$ (3) Å
$\beta = 99.56$ (2)°
$V = 1077.99$ (69) $Å^3$
$Z = 2$
$D_x = 2.0399$ Mg m^{-3}
$D_m = 2.01$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 22 reflections
$\theta = 12.8$–17.2°
$\mu = 1.25$ mm^{-1}
$T = 293$ K
Needle
0.20 × 0.20 × 0.15 mm
Red

Data collection

Rigaku AFC-4 four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: none
8573 measured reflections
8223 independent reflections
6864 observed reflections [$I \geq 2.5\sigma(I)$]

$R_{int} = 0.0101$
$\theta_{max} = 33°$
$h = -11 \rightarrow 0$
$k = -25 \rightarrow 25$
$l = -12 \rightarrow 13$
3 standard reflections intensity variation: <2%

Refinement

Refinement on F
$R = 0.043$
$wR = 0.051$
$S = 1.1$
6864 reflections
287 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F) + 0.008381F^2]$
$(\Delta/\sigma)_{max} = 0.151$
$\Delta\rho_{max} = 1.23$ e $Å^{-3}$
$\Delta\rho_{min} = -1.652$ e $Å^{-3}$
Atomic scattering factors from Cromer & Mann (1968) and Cromer & Liberman (1970)

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

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

	x	y	z	U_{eq}
Mo(1)	−0.99280	−0.18079 (3)	−0.92590	0.0234 (1)
Mo(2)	−0.96286 (8)	−0.33120 (3)	−0.96167 (7)	0.0188 (1)
O(1)	−0.9047 (6)	−0.2420 (2)	−1.0857 (5)	0.026 (1)
O(2)	−0.9443 (6)	−0.2667 (2)	−0.7743 (5)	0.026 (1)
O(3)	−1.2176 (7)	−0.1682 (3)	−0.9755 (8)	0.040 (2)
O(4)	−1.1806 (6)	−0.3575 (3)	−1.0220 (6)	0.032 (1)
O(5)	−0.9423 (6)	−0.0793 (3)	−1.0569 (7)	0.038 (1)
O(6)	−0.9722 (7)	−0.1089 (3)	−0.7247 (7)	0.039 (2)
O(7)	−0.9195 (6)	−0.4288 (2)	−0.8054 (5)	0.027 (1)
O(8)	−0.8765 (6)	−0.3985 (3)	−1.1398 (5)	0.031 (1)
O(9)	−0.7735 (7)	−0.0245 (3)	−1.2091 (7)	0.043 (2)
O(10)	−0.8216 (8)	−0.0248 (3)	−0.5544 (7)	0.044 (2)
O(11)	−0.6715 (8)	−0.4644 (4)	−1.2364 (6)	0.044 (2)
O(12)	−0.7586 (7)	−0.4846 (3)	−0.5934 (6)	0.036 (1)
N(1)	−0.6792 (6)	−0.1434 (3)	−0.8631 (6)	0.021 (1)
N(2)	−0.6460 (6)	−0.3494 (2)	−0.8789 (5)	0.019 (1)
C(1)	−0.7914 (9)	−0.0680 (4)	−1.1021 (8)	0.032 (2)

C(2)	−0.6323 (8)	−0.1141 (3)	−1.0136 (7)	0.026 (1)
C(3)	−0.6705 (9)	−0.0758 (4)	−0.7531 (8)	0.030 (2)
C(4)	−0.8304 (9)	−0.0694 (4)	−0.6715 (8)	0.031 (2)
C(5)	−0.5502 (7)	−0.2050 (3)	−0.7901 (7)	0.023 (1)
C(6)	−0.5220 (7)	−0.2799 (3)	−0.8867 (7)	0.024 (1)
C(7)	−0.6237 (8)	−0.3797 (3)	−0.7157 (6)	0.023 (1)
C(8)	−0.7758 (9)	−0.4366 (3)	−0.6999 (7)	0.026 (1)
C(9)	−0.5885 (8)	−0.4137 (3)	−0.9782 (7)	0.024 (1)
C(10)	−0.7176 (8)	−0.4266 (3)	−1.1329 (7)	0.025 (1)
Na(1)	−0.4750 (4)	−0.5115 (2)	−1.4014 (3)	0.0376 (9)
Na(2)	−1.0541 (4)	−0.0066 (2)	−1.3683 (4)	0.044 (1)
O(1*W*)	−0.3542 (11)	−0.3696 (4)	−1.3551 (8)	0.058 (2)
O(2*W*)	−0.5370 (2)	−0.6567 (6)	−1.4927 (1)	0.094 (5)
O(3*W*)	−1.1051 (1)	−0.1491 (5)	−1.4268 (2)	0.097 (5)
O(4*W*)	−0.9460 (1)	0.1317 (5)	−1.3636 (1)	0.101 (5)

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

Mo(1)—Mo(2)	2.554 (1)	O(10)—C(4)	1.252 (9)
Mo(1)—O(1)	1.926 (4)	O(11)—C(10)	1.195 (8)
Mo(1)—O(2)	1.941 (4)	O(11)···Na(1)	2.357 (7)
Mo(1)—O(3)	1.691 (5)	O(12)—C(8)	1.218 (8)
Mo(1)—O(5)	2.113 (5)	N(1)—C(2)	1.488 (8)
Mo(1)—O(6)	2.101 (6)	N(1)—C(3)	1.474 (8)
Mo(1)—N(1)	2.419 (5)	N(1)—C(5)	1.485 (7)
Mo(2)—O(1)	1.932 (4)	N(2)—C(6)	1.502 (6)
Mo(2)—O(2)	1.934 (4)	N(2)—C(7)	1.483 (7)
Mo(2)—O(4)	1.696 (4)	N(2)—C(9)	1.487 (7)
Mo(2)—O(7)	2.113 (4)	C(1)—C(2)	1.522 (8)
Mo(2)—O(8)	2.098 (5)	C(3)—C(4)	1.50 (1)
Mo(2)—N(2)	2.397 (4)	C(5)—C(6)	1.542 (8)
O(5)—C(1)	1.277 (9)	C(7)—C(8)	1.515 (8)
O(6)—C(4)	1.277 (8)	C(9)—C(10)	1.534 (8)
O(7)—C(8)	1.302 (7)	Na(1)···O(1*W*)	2.554 (8)
O(8)—C(10)	1.279 (7)	Na(1)···O(2*W*)	2.58 (1)
O(9)—C(1)	1.203 (9)	Na(2)···O(3*W*)	2.458 (9)
O(9)···Na(2)	2.344 (9)	Na(2)···O(4*W*)	2.455 (9)
O(6)—Mo(1)—N(1)	74.4 (2)	Mo(1)—O(1)—Mo(2)	82.9 (2)
O(5)—Mo(1)—N(1)	69.8 (2)	Mo(1)—O(2)—Mo(2)	82.5 (1)
Mo(1)—O(5)—C(1)	122.2 (4)	Mo(1)—O(6)—C(4)	122.6 (5)
O(3)—Mo(1)—O(6)	94.1 (3)	Mo(2)—O(7)—C(8)	123.3 (3)
O(3)—Mo(1)—O(5)	91.5 (2)	Mo(2)—O(8)—C(10)	124.5 (4)
Mo(1)—N(1)—C(5)	117.5 (3)	O(2)—Mo(1)—O(3)	109.4 (2)
Mo(1)—N(1)—C(3)	106.3 (4)	Mo(1)—N(1)—C(2)	104.7 (3)
O(1)—Mo(1)—O(3)	109.6 (2)	O(1)—Mo(1)—O(2)	92.3 (2)
Mo(2)—N(2)—C(9)	106.9 (3)	Mo(2)—N(2)—C(7)	106.1 (3)
Mo(2)—N(2)—C(6)	119.0 (3)	O(9)—C(1)—C(2)	120.1 (6)
O(5)—C(1)—O(9)	123.0 (7)	O(5)—C(1)—C(2)	116.0 (6)
N(1)—C(2)—C(1)	109.0 (5)	N(1)—C(3)—C(4)	113.9 (5)
O(10)—C(4)—C(3)	119.0 (6)	O(6)—C(4)—C(3)	119.0 (6)
O(8)—Mo(2)—N(2)	74.7 (2)	O(6)—C(4)—O(10)	122.0 (7)
O(7)—Mo(2)—N(2)	70.7 (2)	N(1)—C(5)—C(6)	118.3 (5)
N(2)—C(6)—C(5)	117.6 (4)	N(2)—C(7)—C(8)	109.5 (4)
O(4)—Mo(2)—O(8)	92.1 (2)	O(12)—C(8)—C(7)	119.9 (5)
O(4)—Mo(2)—O(7)	92.0 (2)	O(7)—C(8)—C(7)	115.3 (5)
O(7)—C(8)—O(12)	124.8 (6)	N(2)—C(9)—C(10)	113.7 (4)
O(11)—C(10)—C(9)	120.5 (6)	O(2)—Mo(2)—O(4)	109.6 (2)
O(8)—C(10)—C(9)	116.3 (5)	O(8)—C(10)—O(11)	123.2 (6)
O(1)—Mo(2)—O(4)	109.1 (2)	O(1)—Mo(2)—O(2)	92.4 (2)

Data were collected by the rotating-anode method. No absorption correction was necessary on account of the very small crystal size. The two Mo atoms were obtained from a Patterson map. Subsequent Fourier maps based on these two atoms located the rest of the non-H atoms of the assymetric unit. All the H-atom positions were generated. The structure was refined by full-matrix least-squares methods using *SHELX*76 (Sheldrick, 1976). The *x* and *z* coordinates of Mo(1) were fixed to define the origin. The absolute structure of the individual crystal was checked by refinement with the opposite hand, resulting in $R = 0.045$.

We are grateful to the Council of Scientific & Industrial Research (CSIR) for financing the research project.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AL1041). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cotrait, M. (1972). *Acta Cryst.* B**28**, 781–785.
Cotton, F. A. & Morehouse, S. M. (1965). *Inorg. Chem.* **4**, 1377–1381.
Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.
Kay, A. & Mitchell, P. C. H. (1968). *Nature (London)*, **219**, 267–268.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* Univ. of Cambridge, England.
Spivack, B. & Dori, Z. (1973). *J. Chem. Soc. Dalton Trans.* pp. 1173–1177.

Acta Cryst. (1995). C**51**, 34–37

$[\{\mu\text{-}C(OC_2H_5)\}(CO)_4Fe_2C\{(OC_2H_5)(p\text{-}CF_3C_6H_4)(\eta\text{-}C_7H_7)\}]$

JIAN CHEN AND PEI-JU ZHENG*

Research Center of Analysis and Measurement,
Fudan University, Shanghai 200433,
People's Republic of China

YONG YU AND JIA-BI CHEN

Shanghai Institute of Organic Chemistry,
Chinese Academy of Sciences, Shanghai 200032,
People's Republic of China

(*Received* 25 *May* 1993; *accepted* 17 *January* 1994)

Abstract

The crystal structure of tetracarbonyl-1κ^2C,2κ^2C-μ-{2-[α-ethoxy-4-(trifluoromethyl)benzylidene-1κC^{α}]-1(1,2-η):2(3,4,5,6-η)-1,3,5-cycloheptatriene}-μ-ethoxymethylidyne-1:2κ^2C-diiron has been determined by X-ray diffraction analysis. The Fe atoms coordinated by carbonyl and cycloheptatriene groups are unsymmetrically bridged by a carbyne ligand. The extensively delocalized bonding may be found over the entire metal–ligand framework.

Comment

The structural study of the title compound is one of a series of studies of olefin-coordinated transition metal