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

Compound (I)		Compound (II)	
Br1—Cu1	2·504(2)	Cu1—Cl1	2·498(2)
Br1—Cu1'	2·553(2)	Cu1—N1	2·013(5)
Cu1—N11	2·048(8)	Cu1—N2	2·014(4)
Cu1—N21	2·077(8)	Cu1—Cu1'	3·008(1)
		Cu1—Cl1'	2·443(2)
Cu1—Br1—Cu1'	85·02(6)	Cu1—Cl1—Cu1'	74·98(6)
Br1—Cu1—N11	119·6(2)	Cl1—Cu1—N1	103·1(1)
Br1—Cu1—N21	104·5(3)	Cl1—Cu1—N2	103·2(1)
Br1—Cu1—Br1'	94·98(6)	Cl1—Cu1—Cl1'	105·03(6)
N11—Cu1—N21	116·8(3)	N1—Cu1—N2	135·0(2)
N11—Cu1—Br1'	104·7(2)	N1—Cu1—Cl1'	104·0(1)
N21—Cu1—Br1'	114·5(2)	N2—Cu1—Cl2'	103·5(1)

Pakawatchai, Patrick & White, 1985), while binuclear species may be obtained by direct recrystallization of the copper(I) halide from solution in a pyridine base with carbon substituent in the 2-position only, as with 2-methylpyridine or quinoline (Healy, Pakawatchai & White, 1983; Dyason, Engelhardt, Healy, Pakawatchai & White, 1985), or with a 1:2 stoichiometry from an appropriate supporting solvent. In exploring further the role and volume of the 2-substituent in controlling stoichiometry and structure in the adducts, we have determined the structures of these further adducts with 2-substituted pyridine ligands, the substituents being benzyl and bromo moieties, and record the structure determinations hereunder, with copper(I) chloride and bromide respectively, as 1:2 binuclear dimers. By comparison with selected studies in the above references, the $N_2CuX_2CuN_2$ molecular core geometries (Table 2) are unremarkable.

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Structure of [LFe(μ-MoO4)3FeL].CH2Cl2.H2O (L = 1,4,7-Trimethyl-1,4,7-triazacyclononane)
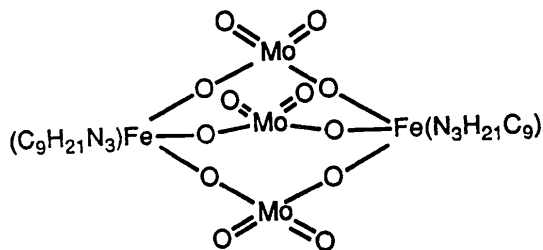
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Abstract. Tri-μ-molybdato(2−)-bis[(1,4,7-trimethyl-1,4,7-triazacyclononane)iron(III)]–dichloromethane–water (1/1/1), $[Fe_2(MoO_4)_3(C_9H_{21}N_3)_2].CH_2Cl_2.H_2O$, $M_r = 1037·0$, orthorhombic, *Pbca*, $a = 15·239$ (2), $b = 16·376$ (3), $c = 28·964$ (4) Å, $V = 7227·9$ (17) Å^3, $D_x = 1·91$ g cm^{-3}, $Z = 8$, $\lambda(Mo\ K\alpha) = 0·71073$ Å, $\mu = 19·8$ cm^{-1}, $F(000) = 4144$, $T = 293$ K, $R = 0·047$, $wR = 0·051$ for 2457 observed reflections. The structure consists of a tri-μ-molybdato-diiron(III) molecular cluster, similar to that of [LFe(CrO4)3-FeL].H2O   (L = 1,4,7-trimethyl-1,4,7-triazacyclononane) [Chaudhuri, Winter, Wieghardt, Gehring, Haase, Nuber & Weiss (1988). *Inorg. Chem.* **27**, 1564–1569]. The synthesis from acetonitrile affords a convenient preparation of crystalline complex and avoids the use of perchlorate salts. The complex provides an unusual example of an early-transition-metal polyoxoanion which is neutral, rather than anionic as is the case with the vast majority of polyoxometalate species [Pope (1983). *Heteropoly and Isopoly Oxometalates.* Berlin: Springer-Verlag].

Experimental. The title compound (I) was obtained from the reaction of $(C_9H_{21}N_3)FeCl_3$ (Chaudhuri, Winter, Wieghardt, Gehring, Haase, Nuber & Weiss, 1988) and $[(C_4H_9)_4N]_2[Mo_2O_7]$ (Day, Fredrich, Klemperer & Shum, 1977) (1:2·5) in refluxing acetonitrile, followed by recrystallization from dichloromethane/methanol (2:1).

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$[Fe_2(MoO_4)_3(C_9H_{21}N_3)_2].CH_2Cl_2.H_2O$

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

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

	x	y	z	U_{eq}
Mo1	3819 (1)	3253 (1)	603 (1)	28 (1)
Mo2	5912 (1)	1548 (1)	1259 (1)	29 (1)
Mo3	4565 (1)	3661 (1)	2101 (1)	27 (1)
Fe1	3651 (1)	1858 (1)	1568 (1)	27 (1)
Fe2	5936 (1)	3726 (1)	1089 (1)	32 (1)
O1	3071 (7)	4010 (6)	568 (4)	74 (5)
O2	3879 (8)	2801 (7)	79 (3)	80 (5)
O3	6197 (6)	921 (6)	808 (4)	65 (4)
O4	6526 (7)	1308 (6)	1724 (3)	66 (4)
O5	5005 (8)	3618 (6)	2641 (3)	68 (4)
O6	3815 (7)	4422 (6)	2095 (4)	71 (5)
O7	3466 (6)	2537 (5)	1028 (3)	47 (4)
O8	4786 (5)	1450 (6)	1387 (3)	47 (4)
O9	4041 (6)	2714 (5)	1975 (3)	50 (4)
O10	4852 (6)	3690 (6)	739 (3)	56 (4)
O11	6144 (7)	2574 (5)	1106 (4)	62 (4)
O12	5405 (6)	3864 (6)	1687 (3)	50 (4)
N1	2275 (7)	2012 (6)	1773 (3)	33 (4)
N2	3046 (7)	797 (6)	1233 (3)	32 (4)
N3	3584 (7)	1002 (7)	2158 (3)	38 (4)
N4	6031 (6)	5044 (6)	993 (4)	33 (4)
N5	6772 (8)	3796 (6)	456 (4)	42 (4)
N6	7227 (7)	3956 (6)	1391 (3)	35 (4)
C1	2051 (10)	2893 (9)	1777 (5)	59 (5)
C2	3386 (11)	700 (10)	761 (5)	67 (5)
C3	4416 (10)	1002 (9)	2405 (5)	62 (5)
C4	1760 (12)	1629 (10)	1393 (6)	74 (5)
C5	2098 (11)	855 (10)	1245 (6)	73 (5)
C6	3322 (12)	61 (11)	1500 (5)	78 (5)
C7	3356 (11)	162 (10)	2001 (5)	71 (5)
C8	2884 (11)	1369 (11)	2452 (6)	78 (6)
C9	2108 (11)	1648 (10)	2235 (5)	68 (5)
C11	5192 (10)	5431 (9)	1108 (5)	61 (5)
C12	6451 (11)	3188 (10)	120 (6)	70 (5)
C13	7392 (11)	3447 (9)	1800 (5)	66 (5)
C14	6206 (12)	5158 (11)	491 (6)	78 (6)
C15	6725 (12)	4629 (11)	270 (6)	80 (6)
C16	7672 (11)	3582 (11)	627 (6)	76 (5)
C17	7916 (13)	3853 (12)	1071 (6)	91 (6)
C18	7187 (12)	4805 (11)	1543 (6)	87 (6)
C19	6720 (11)	5353 (10)	1281 (5)	65 (5)
O13	9525 (9)	2414 (8)	1616 (4)	102 (4)
Cl1	−68 (11)	2729 (10)	681 (6)	61 (5)
Cl2	1196 (19)	3812 (18)	211 (10)	145 (10)
C20	−112 (73)	4274 (66)	535 (37)	173 (41)

Mo(1) through N(6) refined anisotropically; all other atoms refined isotropically.

Single crystals of dimensions $0.36 \times 0.34 \times 0.33$ mm were mounted on a Nicolet $R3m/V$ diffractometer, cell parameters were obtained from the setting angles of 35 reflections with $25 \le 2\theta \le 30°$ (Sparks, 1976); data were collected using $\theta/2\theta$ scans at $3.00–10.00°$ min^{-1}, with a scan range of $1.0°$ below $K\alpha_1$ to $1.0°$ above $K\alpha_2$; stationary background counts for half of the scan time at each end of the scan range; $2\theta_{max} = 45°$; $0 \le h \le 14$, $0 \le k \le 15$, $0 \le l \le 27$; two standards were monitored every 250 reflections and measurements were within $\pm 2\%$; 3820 unique reflections collected of which 2463 with $|F_o| \ge 5\sigma(|F_o|)$, where $\sigma(F_o^2) = [C + 1/4(t_c/t_b)^2(b_1 + b_2) + (pI)^2]^{1/2}$ (C = total counts, t_c = total peak count time, t_b = count time for one background, b_1 = low-angle background, b_2 = high-angle background, p = 0.03) were used in the structure analysis (Sheldrick, 1982); R_{int} = 0.059. The structure was solved by the Patterson method using *SHELXTL* (Sheldrick,

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Mo1—O1	1·687 (9)	Fe1—O7	1·940 (8)
Mo1—O2	1·691 (10)	Fe1—O8	1·926 (8)
Mo1—O7	1·783 (8)	Fe1—O9	1·928 (8)
Mo1—O10	1·774 (9)	Fe1—N1	2·195 (10)
Mo2—O3	1·717 (10)	Fe1—N2	2·193 (10)
Mo2—O4	1·686 (9)	Fe1—N3	2·123 (10)
Mo2—O8	1·762 (8)	Fe2—O10	1·939 (9)
Mo2—O11	1·774 (9)	Fe2—O11	1·913 (9)
Mo3—O5	1·702 (9)	Fe2—O12	1·925 (9)
Mo3—O6	1·691 (9)	Fe2—N4	2·181 (11)
Mo3—O9	1·781 (8)	Fe2—N5	2·235 (11)
Mo3—O12	1·787 (8)	Fe2—N6	2·186 (10)

O1—Mo1—O2	107·8 (6)	O7—Fe1—O8	96·4 (4)
O1—Mo1—O7	108·7 (5)	O7—Fe1—O9	97·0 (4)
O1—Mo1—O10	108·4 (5)	O7—Fe1—N1	90·8 (4)
O2—Mo1—O7	110·4 (5)	O7—Fe1—N2	92·1 (4)
O2—Mo1—O10	109·1 (5)	O7—Fe1—N3	168·3 (4)
O7—Mo1—O10	112·3 (4)	O8—Fe1—O9	98·2 (4)
O3—Mo2—O4	109·2 (5)	O8—Fe1—N1	166·3 (4)
O3—Mo2—O8	110·6 (5)	O8—Fe1—N2	89·1 (4)
O3—Mo2—O11	109·0 (5)	O8—Fe1—N3	91·9 (4)
O4—Mo2—O8	110·5 (5)	N1—Fe1—N2	79·0 (4)
O4—Mo2—O11	108·1 (5)	N1—Fe1—N3	79·6 (4)
O8—Mo2—O11	109·4 (4)	N2—Fe1—N3	79·7 (4)
O5—Mo3—O6	107·9 (5)	O9—Fe1—N1	92·5 (4)
O5—Mo3—O9	109·2 (5)	O9—Fe1—N2	167·7 (4)
O5—Mo3—O12	110·0 (5)	O9—Fe1—N3	90·1 (4)
O6—Mo3—O9	109·7 (5)	O10—Fe2—O11	97·1 (4)
O6—Mo3—O12	109·8 (5)	O10—Fe2—O12	96·6 (4)
O9—Mo3—O12	110·2 (4)	O10—Fe2—N4	91·1 (4)
		O10—Fe2—N5	93·4 (4)
		O10—Fe2—N6	168·9 (4)
O11—Fe2—O12	99·3 (4)	O12—Fe2—N5	166·3 (4)
O11—Fe2—N4	165·5 (4)	O12—Fe2—N6	89·9 (4)
O11—Fe2—N5	88·7 (4)	N4—Fe2—N5	78·9 (4)
O11—Fe2—N6	90·6 (4)	N4—Fe2—N6	79·7 (4)
O12—Fe2—N4	91·5 (4)	N5—Fe2—N6	78·9 (4)

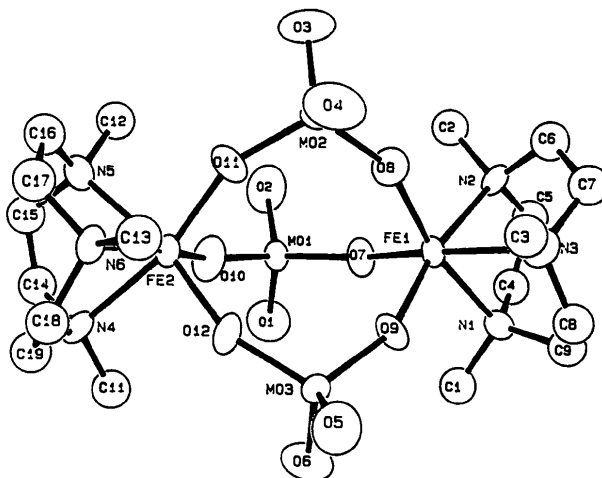Fig. 1. Perspective view of the structure of $[Fe_2(MoO_4)_3(C_9H_{21}N_3)_2]$ showing the atom-labeling scheme, and 50% probability ellipsoids.

1982). The Mo, Fe, O and N atoms were refined anisotropically. All H atoms were included as fixed contributors with the C—H distances set at 0·96 Å and with fixed isotropic temperature factors. The final R factor was 0·047 (296 variables), $wR = 0.051$, $S = 1.541$; the function minimized was $\sum w(|F_o| - |F_c|)^2$ with $w = 1/[\sigma^2(F_o) + 0.001(F_o)^2]$. $(\Delta/\sigma)_{max} = 0.004$, extrema in final difference map

$+ 0.73$, $- 0.57$ e Å^{-3}. The atomic scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed on a MicroVAX II computer. Atomic parameters are listed in Table 1,* and selected bond distances and angles in Table 2. A view of the molecule, with the atom-labeling scheme, is shown in Fig. 1.

Related literature. The structure of the analogous chromate bridged complex [(C$_9$H$_{21}$N$_3$)Fe(CrO$_4$)$_3$-Fe(C$_9$H$_{21}$N$_3$)] has been reported (Chaudhuri *et al.*, 1988), as well as μ-phosphato [(C$_9$H$_{21}$N$_3$)Fe-(O)(C$_6$H$_5$OPO$_3$)$_2$Fe(C$_9$H$_{21}$N$_3$)] (Drueke, Wieghardt, Nuber, Weiss, Fleischhauer, Gehring & Haase, 1989) and μ-carbonato species (Drueke, Wieghardt, Nuber & Weiss, 1989). A complex containing three-[(C$_9$H$_{21}$N$_3$)Fe] moieties bridging a [Mo$_4$O$_{14}$(μ-

OMe)$_3$] core has also been described (Chaudhuri, Wieghardt, Nuber & Weiss, 1987).

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53998 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Diaqua-1,6-bis(benzimidazol-2-yl)-2,5-dithiahexanecobalt(II) Iodide Monohydrate at 233 K

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Abstract. [Co(C$_{18}$H$_{18}$N$_4$S$_2$)(H$_2$O)$_2$]I$_2$.H$_2$O, M_r = 721.29, monoclinic, $C2/c$, $a = 18.804$ (4), $b = 11.101$ (5), $c = 12.326$ (5) Å, $\beta = 109.86$ (3)°, $V = 2420$ Å^3, $Z = 4$, $D_x = 1.980$ g cm^{-3}, λ(Mo $K\alpha$) = 0.70930 Å, $\mu = 34.246$ cm^{-1}, $F(000) = 1396$, $T = 233$ K, $R = 0.043$ for 1913 observed reflections. The Co atom of the complex is surrounded by two N atoms from the benzimidazole groups in a *trans* arrangement [Co—N = 2.122 (5) Å] and two sulfur atoms of the thioether groups with a *cis* geometry [Co—S = 2.521 (2) Å]. The O atoms of the two symmetrically related water molecules [Co—O = 2.087 (6) Å] complete the coordination around the metal ion to form a distorted octahedron. The packing in the crystal results in hydrogen-bond interactions between the water molecules, and between the N—H groups of the imidazoles and the I atoms.

Experimental. Colorless prisms of the title compound were obtained by slowly evaporating solvent from a solution of [Co(BBDH)(OH$_2$)$_2$]I$_2$ [BBDH = 1,6-bis-(benzimidazol-2-yl)-2,5-dithiahexane] in ethanol. Crystal size 0.08 × 0.08 × 0.16 mm, Enraf–Nonius CAD-4 four-circle single-crystal automated diffrac-