observed in solution and in the solid state for [$Rh\{CN(2,6\text{-}Me_2C_6H_3)\}_4$]$^+$ cations with various anions (Baumann, Keller, Nöthe, Rupp & Uhlmann, 1976). The conformations of the aryl rings with respect to the coordination plane are in general agreement with the conformation predicted on the basis of calculated 'fan angles' for 2,6-$Me_2C_6H_3NC$ ligands (Yamamoto *et al.*, 1979).

The geometry of the BPh_4 anion is as expected and shows no unusual features. The disordered ethanol molecule is very poorly defined and no significance can be placed on the observed geometry of this molecule.

Intensity data were collected at the National Physical Research Laboratory, CSIR.

References

BALCH, A. L. & OLMSTEAD, M. M. (1979). *J. Am. Chem. Soc.* **101**, 3128–3129.

BAUMANN, D., KELLER, H. J., NÖTHE, D., RUPP, H. H. & UHLMANN, G. (1976). *Z. Naturforsch. Teil B*, **31**, 912–921.

Cambridge Structural Database (CSD) (1983). Univ. Chemical Laboratory, Lensfield Road, Cambridge, England. As implemented on the CDC Cyber 750 computer, CSIR, Pretoria, update of May 1983.

CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, pp. 71–147. Birmingham: Kynoch Press.

ENDRES, H., GOTTSTEIN, N., KELLER, H. J., MARTIN, R., RODEMER, W. & STEIGER, W. (1979). *Z. Naturforsch. Teil B*, **34**, 827–833.

HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502–510.

MANN, K. R., LEWIS, N. S., WILLIAMS, R. M., GRAY, H. B. & GORDON, J. G. II (1978). *Inorg. Chem.* **17**, 828–834.

OLMSTEAD, M. M. & BALCH, A. L. (1978). *J. Organomet. Chem.* **148**, C15–C18.

PAULING, L. (1960). In *The Nature of the Chemical Bond*, 3rd ed. Ithaca: Cornell Univ. Press.

SHELDRICK, G. M. (1978). *Computing in Crystallography*, pp. 34–42. Delft Univ. Press.

SINGLETON, E. & OOSTHUIZEN, H. E. (1983). *Adv. Organomet. Chem.* **22**, 209–310.

YAMAMOTO, Y., AOKI, K. & YAMAZAKI, H. (1979). *Inorg. Chem.* **18**, 1681–1687.

Acta Cryst. (1984). C**40**, 1172–1175

Structures of Bis(1,5-dithiacyclooctan-3-ol-*O*,*S*,*S*′)nickel(II) Diperchlorate Dimethanol, [$Ni(C_6H_{12}OS_2)_2$](ClO_4)$_2$.2CH_4O, and Bis(1,5-dithiacyclooctan-3-ol-*O*,*S*,*S*′)iron(II) Diperchlorate, [$Fe(C_6H_{12}OS_2)_2$](ClO_4)$_2$

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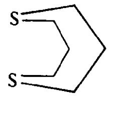
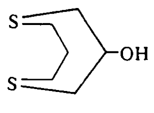
Abstract. [$Ni(C_6H_{12}OS_2)_2$](ClO_4)$_2$.2CH_3OH: M_r = 650·28, $P2_1/c$, a = 10·734 (2), b = 11·853 (3), c = 10·392 (2) Å, β = 103·62 (2)°, V = 1285·0 (5) Å^3, Z = 2, D_m(298 K) decomp., D_x(140 K) = 1·68 g cm^{-3}, Mo $K\alpha$, λ = 0·71069 Å, μ = 13·3 cm^{-1}, F(000) = 676, T = 140 K, R = 0·037, 1960 unique observed reflections, recrystallization from methanol. The complex is centrosymmetric with Ni–S of 2·398 (1) and 2·396 (1) Å, Ni–O of 2·046 (3) Å. A molecule of methanol present in the lattice is hydrogen-bonded to the hydroxyl group of the ligand and to an oxygen atom of an uncoordinated perchlorate ion. [$Fe(C_6H_{12}OS_2)_2$](ClO_4)$_2$: M_r = 583·33, $P2_1/c$, a = 7·990 (2), b = 8·573 (2), c = 15·681 (3) Å, β = 95·50 (2)°, V = 1069·2 (4) Å^3, Z = 2, D_m(298 K) decomp., D_x(140 K) = 1·81 g cm^{-3}, Mo $K\alpha$, λ = 0·71069 Å, μ = 13·8 cm^{-1}, F(000) = 600, T = 140 K, R = 0·046, 1601 unique observed reflections, recrystallization from methanol. The complex resembles the nickel species except that the thioether–metal bonds are much longer: Fe–S 2·565 (1) and 2·514 (1) Å, Fe–O 2·062 (3) Å.

Introduction. The coordination mode of 1,5-dithiacyclooctane in transition-metal complexes varies considerably. Either one or two sulfurs can coordinate to the metal, and thus bidentate (Hirschon & Musker, 1980), bridging (Hill & Hope, 1974) or monodentate (Olmstead, Williams & Musker, 1982) modes are observed. Substitution of a hydroxyl group in the 3-position of the ring



1,5-dithiacyclooctane



1,5-dithiacyclooctan-3-ol

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does not alter the reduction potential of the free ligand, but it does cause different reactivities towards metals that apparently stem from geometric factors. For example, reduction of $Cu(ClO_4)_2$ by 1,5-dithiacyclooctane in methanol proceeds too rapidly to isolate a Cu^{II} product (Musker, Olmstead & Kessler, 1984). However, the rate of reduction is retarded by substitution of a hydroxyl group in the 3-position and a Cu^{II} complex can be isolated easily. Also, no complex of $Fe(ClO_4)_2$ with 1,5-dithiacyclooctane forms, but unstable purple crystals with 1,5-dithiacyclooctan-3-ol can be isolated. In order to understand the detailed coordination geometry of the substituted ligand, crystal structure determinations were carried out on the series of Fe^{II}, Ni^{II}, Cu^{II} and Cu^{I} perchlorates. Results for the Cu^{I} and Cu^{II} complexes have been reported (Musker, Olmstead & Kessler, 1984). We believe that the Fe^{II} structure reported here represents the first determination of an Fe^{II}—S(thioether) bond length.

Experimental. $[Ni(C_6H_{12}OS_2)_2](ClO_4)_2.2CH_3OH$ synthesized by combining methanolic solutions of 0·10 mol dm^{-3} $Ni(ClO_4)_2$ and 0·10 mol dm^{-3} 1,5-dithiacyclooctan-3-ol in a ratio of one to two. Purple parallelepipeds obtained from methanol recrystallization; decomposition during density measurement; crystal selected 0·130 × 0·325 × 0·375 mm, Syntex $P2_1$ diffractometer, fourteen reflections with $2\theta > 30°$ used in determination of lattice parameters; conditions $h0l$, $l = 2n$; $0k0$, $k = 2n$; no absorption correction applied, $A^*_{\rm min} = 1{\cdot}15$, $A^*_{\rm max} = 1{\cdot}35$; data collection to $2\theta_{\rm max} = 50°$ with h 0 to 13, k 0 to 15 and l −13 to 13; 3 check reflections measured every 100 reflections displayed <2% average fluctuation; 2465 reflections measured; 2265 unique reflections, $R_{\rm int} = 0{\cdot}033$; 305 unobserved $[F < 5\sigma(F)]$; structure solved by placing Ni at center of inversion; blocked-cascade least-squares refinement (based on F); all non-hydrogen atoms assigned anisotropic thermal parameters; hydroxyl hydrogens of methanol solvate and the ligand allowed to refine freely, remaining hydrogens included at calculated positions riding on the bonded carbon with $U_{\rm iso} = 1{\cdot}2U_{\rm eq}({\rm C})$; $R_w = 0{\cdot}041$ with $w = [\sigma^2(F) + 0{\cdot}0007F^2]^{-1}$, $S = 1{\cdot}40$, max. Δ/σ 0·016 for overall scale and an av. Δ/σ 0·003; max. and min. heights of 0·82 and −0·66 e Å^{-3} in final difference map; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974); computer programs from the *SHELXTL* (Sheldrick, 1981) package.

$[Fe(C_6H_{12}OS_2)_2](ClO_4)_2$ synthesized by the same method used for the Ni^{II} complex. (*Note: in one instance this product detonated during work-up.*) Purple parallelepipeds obtained from methanol recrystallization; decomposition during density measurement; crystal selected 0·250 × 0·325 × 0·370 mm; Syntex $P2_1$ diffractometer, 14 reflections with $2\theta > 30°$ used in determination of lattice parameters; conditions $h0l$, $l = 2n$; $0k0$, $k = 2n$; no absorption correction; $A^*_{\rm min} = 1{\cdot}33$, $A^*_{\rm max} = 1{\cdot}44$; data collection to $2\theta_{\rm max} = 50°$ with h 0 to 10, k 0 to 11, l −19 to 19; 2 check reflections measured every 100 reflections displayed <2% average fluctuation; 2164 reflections measured; 1876 unique reflections, $R_{\rm int} = 0{\cdot}008$; 275 unobserved $[F < 5\sigma(F)]$; structure solved by placing Fe at center of inversion; blocked-cascade least-squares refinement (based on F); two of perchlorate oxygens disordered and assigned partial occupancies; ordered non-hydrogen atoms assigned anisotropic thermal parameters; hydroxyl hydrogen allowed to refine freely, remaining hydrogens included at calculated positions riding on the bonded carbon with $U_{\rm iso} = 1{\cdot}2\ U_{\rm eq}({\rm C})$; $R_w = 0{\cdot}050$ with $w = [\sigma^2(F) + 0{\cdot}00031F^2]^{-1}$, $S = 2{\cdot}05$, max. Δ/σ −0·293 for $U_{\rm iso}$ of O(8) (part of disordered ClO_4^-) and av. Δ/σ 0·028; max. and min. heights of 0·83 and −0·54 e Å^{-3} in final difference map; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974); computer programs from the *SHELXTL* (Sheldrick, 1981) package.

The perchlorate group presented a severe disorder problem. After considerable trial and error with several models for perchlorate disorder, the most persistent oxygen features were refined using occupancy factors totalling four. Thermal parameters ranged from 0·026 to 0·075 Å^2 after refinement. The Cl—O distances ranged from 1·28 (3) to 1·535 (6) Å. Two of the oxygen atoms, O(2) and O(3), seemed normal, thus any model that includes these at full occupancy requires at least three orientations for the group. The method used does not result in a physically reasonable model, but fortunately the cation distances and angles are not sensitive to the model employed.

Discussion. A drawing of the $[Ni(3\text{-}OH\text{-}1,5\text{-}dithiacyclooctane)_2]^{2+}$ species is presented in Fig. 1. Final atomic parameters are given in Table 1.† Bond distances and angles are given in Table 2. The coordination geometry for nickel is rhombohedrally distorted tetragonal, with a donor set involving four thioether sulfurs and two hydroxyl oxygens. Each ligand coordinates in a facial manner, contributing two chelating sulfurs and an oxygen from the hydroxyl group. Since the complex lies on a center of symmetry, the nickel and four sulfurs describe a plane. The direction of the Ni—O bonds is 10·0 (1)° from perpendicular to this plane. The conformation of the eight-membered ring is boat-chair, which is probably

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† Lists of structure factors, anisotropic thermal parameters, hydrogen coordinates and torsion angles for both compounds have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39330 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

the most common for this ligand. The four Ni—S bonds [2·396 (1), 2·398 (1) Å] are longer than the two Ni—O bonds [2·046 (3) Å]. Since these are normal bond lengths for nickel, the geometry may be considered to be derived from octahedral geometry for nickel. This contrasts with the Cu$^{\mathrm{II}}$ complex, in which the two Cu—O bonds are 2·350 (1) Å, 0·016 Å longer than the Cu—S bonds (Musker, Olmstead & Kessler, 1984). In this latter case, the geometry is derived from elongated tetragonal.

The crystal structure of the nickel complex also contains an ionic perchlorate and a molecule of methanol in the asymmetric unit. No disorder is present in either of these species. There are two hydrogen bonds between the 3-hydroxyl hydrogen atom, HO(1), and the methanolic oxygen, O(6), and between the hydroxyl hydrogen of methanol, HO(6), and O(4) of the ionic perchlorate. Both HO(1) and HO(6) were clearly located on a difference Fourier map and were allowed to refine. Bond distances and angles for these interactions are included in Table 2.

The [Fe(3-OH-1,5-dithiacyclooctane)$_2$]$^{2+}$ species is depicted in Fig. 2. Final atomic parameters are given in Table 3; bond distances and angles are in Table 4. It is immediately apparent that it is similar in coordination geometry to the nickel species, but differs in the conformation of the eight-membered ring. Rather than being boat-chair, the conformation is the slightly less common chair-chair. Subtle changes in crystal packing are probably sufficient to bring about this change. Torsion angles are given in the supplementary material* for both structures.

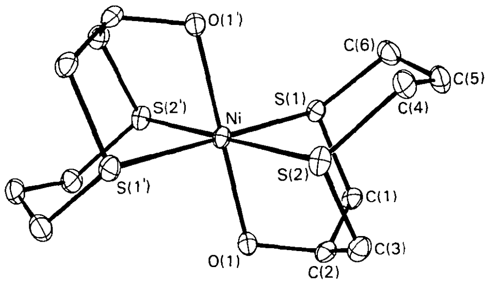

Fig. 1. A drawing of [Ni(3-OH-1,5-dithiacyclooctane)$_2$]$^{2+}$ showing the atom-numbering scheme and anisotropic thermal ellipsoids at the 50% level.

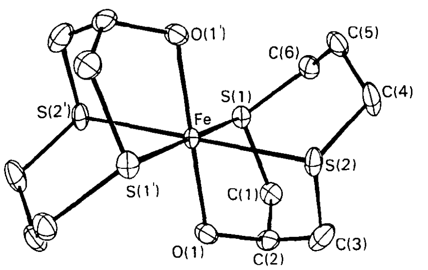

Fig. 2. A drawing of [Fe(3-OH-1,5-dithiacyclooctane)$_2$]$^{2+}$ showing the atom-numbering scheme and anisotropic thermal ellipsoids at the 50% level.

Table 1. *Atomic coordinates* (× 10^4) *and isotropic thermal parameters* (Å^2 × 10^3) *for* [Ni(3-OH-1,5-*dithiacyclooctane*)$_2$](ClO$_4$)$_2$.2CH$_3$OH

	x	y	z	$U_{\mathrm{eq}}/U_{\mathrm{iso}}$
Ni	0	0	0	13 (1)*
Cl	−3160 (1)	227 (1)	3209 (1)	21 (1)*
S(1)	1224 (1)	−1704 (1)	102 (1)	16 (1)*
S(2)	1021 (1)	369 (1)	2271 (1)	17 (1)*
O(1)	−1166 (2)	−975 (2)	842 (2)	16 (1)*
O(2)	−2939 (3)	884 (2)	2121 (3)	36 (1)*
O(3)	−2059 (3)	315 (3)	4301 (3)	41 (1)*
O(4)	−3335 (3)	−933 (2)	2807 (3)	42 (2)*
O(5)	−4258 (3)	619 (3)	3607 (4)	53 (1)*
O(6)	2963 (2)	2283 (2)	414 (3)	37 (1)*
C(1)	470 (3)	−2425 (3)	1273 (3)	18 (1)*
C(2)	−371 (3)	−1645 (3)	1879 (3)	17 (1)*
C(3)	293 (3)	−815 (3)	2937 (3)	19 (1)*
C(4)	2733 (3)	55 (3)	2678 (4)	21 (1)*
C(5)	3137 (3)	−1127 (3)	2429 (3)	22 (1)*
C(6)	2887 (3)	−1505 (3)	994 (3)	22 (1)*
C(7)	4215 (4)	2149 (4)	198 (4)	40 (1)*
HO(1)	−1673 (40)	−1284 (37)	476 (41)	28 (13)
HO(6)	3022 (35)	2699 (33)	803 (38)	34 (11)

* $U_{\mathrm{eq}} = (U_{11} + U_{22} + U_{33})/3$.

Table 2. *Interatomic distances* (Å) *and angles* (°) *for* [Ni(3-OH-1,5-*dithiacyclooctane*)$_2$](ClO$_4$)$_2$.2CH$_3$OH

Ni—S(1)	2·398 (1)	Cl—O(2)	1·438 (3)
Ni—S(2)	2·396 (1)	Cl—O(3)	1·437 (3)
Ni—O(1)	2·046 (3)	Cl—O(4)	1·437 (3)
S(1)—C(1)	1·824 (4)	Cl—O(5)	1·416 (4)
S(1)—C(6)	1·823 (3)		
S(2)—C(3)	1·820 (3)		
S(2)—C(4)	1·824 (3)		
O(1)—C(2)	1·445 (4)	O(1)—HO(1)	0·69 (4)
O(6)—C(7)	1·423 (5)	O(6)—HO(6)	0·63 (4)
C(1)—C(2)	1·528 (5)	O(1)···O(6$^{\mathrm{i}}$)	2·577 (4)
C(2)—C(3)	1·522 (4)	HO(1)···O(6$^{\mathrm{i}}$)	1·89 (4)
C(4)—C(5)	1·506 (5)	O(6)···O(4$^{\mathrm{ii}}$)	2·776 (4)
C(5)—C(6)	1·519 (5)	HO(6)···O(4$^{\mathrm{ii}}$)	2·15 (4)
S(1)—Ni—S(2)	89·5 (1)		
S(1)—Ni—O(1)	83·6 (1)		
S(2)—Ni—O(1)	82·3 (1)	O(2)—Cl—O(3)	108·9 (2)
Ni—S(1)—C(6)	112·0 (1)	O(2)—Cl—O(4)	108·9 (2)
Ni—S(1)—C(1)	95·7 (1)	O(3)—Cl—O(4)	109·1 (2)
Ni—S(2)—C(3)	95·5 (1)	O(2)—Cl—O(5)	110·8 (2)
Ni—S(2)—C(4)	113·1 (1)	O(3)—Cl—O(5)	109·4 (2)
Ni—O(1)—C(2)	108·4 (2)	O(4)—Cl—O(5)	109·8 (2)
C(3)—S(2)—C(4)	104·7 (2)		
C(1)—S(1)—C(6)	104·9 (1)		
S(1)—C(1)—C(2)	112·9 (2)		
S(1)—C(6)—C(5)	117·6 (2)	Ni—O(1)—HO(1)	128 (4)
S(2)—C(3)—C(2)	113·2 (2)	C(2)—O(1)—HO(1)	112 (3)
S(2)—C(4)—C(5)	117·7 (2)	C(7)—O(6)—HO(6)	103 (4)
O(1)—C(2)—C(1)	109·1 (3)		
O(1)—C(2)—C(3)	106·1 (2)		
C(1)—C(2)—C(3)	117·8 (3)	O(6)—HO(6)···O(4$^{\mathrm{ii}}$)	176 (2)
C(4)—C(5)—C(6)	116·8 (3)	O(1)—HO(1)···O(6$^{\mathrm{i}}$)	173 (2)

Symmetry code: (i) $-x, -y, -z$; (ii) $-x, \frac{1}{2} + y, \frac{1}{2} - z$.

The most interesting aspect of the Fe structure is the Fe—S bond distance. No other Fe—S(thioether) distances have been reported, and the rather long distances

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* See deposition footnote.

Table 3. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* (Å$^2 \times 10^3$) *for* [Fe(3-OH-1,5-*dithiacyclooctane*)$_2$](ClO$_4$)$_2$

	x	y	z	U_{eq}/U_{iso}
Fe	0	0	0	13 (1)*
Cl	4928 (2)	2849 (2)	1328 (1)	40 (1)*
S(1)	−357 (1)	232 (1)	1603 (1)	19 (1)*
S(2)	103 (2)	−2907 (1)	210 (1)	21 (1)*
O(1)	2455 (4)	−269 (4)	516 (2)	23 (1)*
O(2)	5205 (6)	2118 (6)	2145 (3)	65 (2)*
O(3)	3120 (7)	3009 (6)	1129 (4)	75 (2)*
O(4)	5177 (7)	1447 (7)	745 (4)	34 (1)
O(5)	5904 (13)	3979 (12)	1149 (7)	41 (2)
O(6)	5066 (12)	4516 (11)	1746 (6)	44 (2)
O(7)	6607 (20)	2832 (19)	1036 (10)	26 (4)
O(8)	4118 (36)	2518 (33)	611 (18)	72 (7)
O(9)	5372 (32)	4120 (31)	748 (16)	61 (7)
C(1)	1674 (6)	−655 (6)	1951 (3)	24 (1)*
C(2)	2611 (6)	−1319 (6)	1240 (3)	24 (1)*
C(3)	2077 (6)	−2908 (6)	887 (3)	31 (2)*
C(4)	−1426 (7)	−3546 (5)	930 (3)	32 (2)*
C(5)	−2618 (6)	−2316 (5)	1231 (3)	26 (1)*
C(6)	−1913 (6)	−1183 (5)	1913 (3)	24 (1)*
HO(1)	3193 (80)	349 (73)	575 (41)	55 (21)

* $U_{eq} = (U_{11} + U_{22} + U_{33})/3$.

found here [2·565 (1) and 2·514 (1) Å] are no doubt a reflection of the low bond strength. Previous values for Fe—S(thiolate) are typically near 2·27 Å (Lane, Ibers, Frankel & Holm, 1975). The Fe—O(1) bond is tilted 14·6 (1)° from the normal to the FeS$_4$ plane. An ill-defined (owing to disorder in the ClO$_4^-$ group) hydrogen-bonding interaction exists between the hydrogen atom of the hydroxyl group and O(4) and O(8) of the perchlorate anion.

This work was supported by National Science Foundation grant CHE-8015489.

Table 4. *Interatomic distances* (Å) *and angles* (°) *for* [Fe(3-OH-1,5-*dithiacyclooctane*)$_2$](ClO$_4$)$_2$

Fe–S(1)	2·565 (1)		
Fe–S(2)	2·514 (1)		
Fe–O(1)	2·062 (3)		
S(1)–C(1)	1·828 (5)		
S(1)–C(6)	1·835 (5)		
S(2)–C(3)	1·815 (5)		
S(2)–C(4)	1·825 (6)		
O(1)–C(2)	1·445 (6)		
C(1)–C(2)	1·513 (7)		
C(2)–C(3)	1·516 (7)	O(1)–HO(1)	0·79 (6)
C(4)–C(5)	1·526 (7)	O(1)···O(4)	2·62 (2)
C(5)–C(6)	1·513 (6)	O(1)···O(8)	2·73 (3)
S(1)–Fe–S(2)	87·4 (1)	C(3)–S(2)–C(4)	103·5 (2)
S(1)–Fe–O(1)	79·3 (1)	C(1)–S(1)–C(6)	104·5 (2)
S(2)–Fe–O(1)	79·6 (1)	S(1)–C(1)–C(2)	115·1 (3)
Fe–S(1)–C(6)	110·5 (1)	S(1)–C(6)–C(5)	116·9 (3)
Fe–S(1)–C(1)	94·7 (1)	S(2)–C(3)–C(2)	114·3 (3)
Fe–S(2)–C(3)	95·3 (2)	S(2)–C(4)–C(5)	117·3 (3)
Fe–S(2)–C(4)	111·3 (2)	O(1)–C(2)–C(1)	109·5 (4)
Fe–O(1)–C(2)	112·7 (3)	O(1)–C(2)–C(3)	105·8 (4)
		C(1)–C(2)–C(3)	117·8 (4)
		C(4)–C(5)–C(6)	117·5 (4)
Fe–O(1)–HO(1)	130 (5)		
C(2)–O(1)–HO(1)	109 (5)		

References

HILL, N. L. & HOPE, H. (1974). *Inorg. Chem.* **13**, 2079–2082.

HIRSCHON, A. & MUSKER, W. K. (1980). *Transition Met. Chem.* **5**, 191–194.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

LANE, R. W., IBERS, J. A., FRANKEL, R. B. & HOLM, R. H. (1975). *Proc. Natl Acad. Sci. USA*, **72**, 2868–2874.

MUSKER, W. K., OLMSTEAD, M. M. & KESSLER, R. M. (1984). *Inorg. Chem.* In the press.

OLMSTEAD, M. M., WILLIAMS, K. A. & MUSKER, W. K. (1982). *J. Am. Chem. Soc.* **104**, 5567–5568.

SHELDRICK, G. M. (1981). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Obtained from Nicolet Instruments, Madison, Wisconsin.

Acta Cryst. (1984). C**40**, 1175–1177

Structure of *catena*-Tetraaqua-di-μ_3-(*N*-salicylidene-DL-glutamato)-tricopper(II) Heptahydrate, [Cu$_3$(C$_{12}$H$_{10}$NO$_5$)$_2$(H$_2$O)$_4$].7H$_2$O

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Abstract. $M_r = 885{\cdot}23$, triclinic, $P\bar{1}$, $a = 7{\cdot}874\ (2)$, $b = 8{\cdot}702\ (3)$, $c = 13{\cdot}278\ (6)$ Å, $\alpha = 97{\cdot}32\ (4)$, $\beta = 104{\cdot}06\ (3)$, $\gamma = 97{\cdot}83\ (3)°$, $V = 862{\cdot}0$ Å^3, $Z = 1$, $D_m = 1{\cdot}68$ (flotation), $D_x = 1{\cdot}705$ Mg m^{-3}, $\mu(\text{Mo } K\alpha) = 1{\cdot}99$ mm^{-1}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $F(000) = 455$, room temperature, $R = 0{\cdot}037$ for 3054 reflections with $F_o \geq 6{\cdot}0\sigma(F_o)$. Each Schiff-base ligand is hexadentate, coordinated through N and four O atoms to two Cu atoms in the trimeric unit and through one carboxyl O atom to Cu in the adjacent trimeric unit, thus forming a polymeric structure. The coordination geometries around Cu atoms are square-pyramidal (4 + 1) and

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