

hams, Hoskins, Tiekink & Winter, 1988). There is no apparent reason for this difference and the remainder of the bond lengths in both forms are comparable to those observed for Ni(moexa)₂ and Cd(moexa)₂. The conformation of the xanthate ligands of the two forms differ in the torsion angle C(1)—O(1)—C(2)—C(3) which is 91.7 (6)° in form II, whereas in form I, the analogous torsion angles have differing values, 172.6 (4)° for C(1)—O(1)—C(2)—C(3) and -94.9 (5)° for C(5)—O(3)—C(6)—C(7).

The packing of the molecules in the two forms is markedly different, as shown in Figs. 2(a,b). These are the first solvent-free crystal structures of chelating diamine nickel(II) bisxanthates to be reported, and the previously determined structures (Edwards, Hoskins & Winter, 1986; Gable, Hoskins & Winter, 1985) have included solvent molecules. At least two solvent-dependent forms are also observed for the analogous 1,10-phenanthroline adduct of nickel (*O*-methoxyethylxanthate) (Edwards, Hoskins & Winter, 1990).

Acta Cryst. (1990). **C46**, 1789–1792

Structures of Bis(*O*-methoxyethylxanthato)(1,10-phenanthroline)nickel(II) and its Benzene Solvate

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(Received 16 November 1989; accepted 2 January 1990)

Abstract. Bis(*O*-methoxyethyldithiocarbonato)(1,10-phenanthroline)nickel(II), [Ni(C₁₂H₈N₂)(C₄H₇O₂S₂)₂]. Crystals grown from acetone solution contain only the title compound, *M_r* = 541.4, monoclinic, *C*2/*c*, *a* = 15.661 (2), *b* = 18.894 (4), *c* = 8.013 (2) Å, β = 99.89 (2)°, *U* = 2338 (2) Å³, *Z* = 4, *F*(000) = 1120, μ = 11.68 cm⁻¹, *D_x* = 1.537, *D_m* = 1.52 (2) g cm⁻³, *T* = 295 (2) K, *R* = 0.042 for 1697 unique reflections (*I* > 2σ*I*). Crystals grown from benzene solution form as benzene solvate, [Ni(C₁₂H₈N₂)(C₄H₇O₂S₂)₂].C₆H₆, *M_r* = 619.5, monoclinic, *P*2₁/*a*, *a* = 10.005 (1), *b* = 19.820 (2), *c* = 15.052 (3) Å, β = 105.29 (1)°, *U* = 2879 (1) Å³, *Z* = 4, *F*(000) = 1288, λ(Mo *K*α) = 0.71069 Å, μ = 9.54 cm⁻¹, *D_x* = 1.43 g cm⁻³, *T* = 295 (2) K, *R* = 0.044 for 3532 unique reflections (*I* ≥ 2σ*I*). The crys-

The Commonwealth of Australia is thanked for a Commonwealth Post Graduate Research Award for AJE.

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tal structure of Ni(methoxyethylxanthate)(1,10-phenanthroline) is found to be solvent dependent. A striking similarity is observed between the crystal structure of this compound obtained from acetone solution and its 2,2'-bipyridyl analogue grown from benzene solution. For the present structure the difference between the molecular structures of the solvated and unsolvated forms lies in the conformation of the methoxyethylxanthate moieties.

Introduction. As a continuation of our investigation of the different forms obtained for heterocyclic diamine adducts of nickel(II) xanthates (Kruger & Winter, 1971; Gable, Hoskins & Winter, 1985; Edwards, Hoskins & Winter, 1990), crystals of Ni(S₂COCH₂CH₂OCH₃)₂.1,10-phenanthroline [Ni(moexa)₂phen] were grown from solutions in acetone and in benzene. Oscillation and Weissenberg photographs showed both crystals to be monoclinic, but of

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different forms. Earlier results had suggested that when solvent was included in the crystal structures of chelating heterocyclic diamine adducts of nickel(II) xanthates it was located in discrete cavities, with escape of the solvent requiring complete rupture of the structure.

Experimental. The compound was prepared by combining equimolar quantities of nickel *O*-methoxyethylxanthate with 1,10-phenanthroline in solution. The unsolvated form was obtained by reaction in hot acetone (323 K). The residue remaining on evaporation at ambient temperature and pressure was recrystallized from acetone and yielded green crystals. The crystal selected for study had faces $\pm(110)$, $\pm(1\bar{1}0)$ and $\pm(001)$ separated by 0.07, 0.14, and 0.64 mm respectively. Density was measured by flotation in aqueous zinc bromide solution.

The benzene solvate was obtained by reaction in hot benzene (348 K). The residue remaining on evaporation at ambient temperature and pressure was recrystallized from benzene. A dark-green crystal with faces $\pm(100)$, $\pm(010)$, $\pm(001)$ separated by 0.64, 0.31 and 0.24 mm respectively was selected for study, the data were collected with the crystal sealed in a tube in the presence of mother liquor as immediate deterioration was observed for crystals which had been exposed to the atmosphere, the density of the solvated crystals could not be accurately measured due to the rapidity of this deterioration.

An Enraf-Nonius CAD-4F diffractometer and graphite-monochromated Mo *K* α radiation were used. Data collection, structure solution and refinement for the two forms were similar and where values differ, those for the benzene solvate are shown in brackets {}. Cell dimensions were obtained by least-squares refinement of angular values for 25 reflections in the range $14.8 \leq 2\theta \leq 39.8^\circ$ $\{22.2 \leq 2\theta \leq 40.8^\circ\}$.

Intensity data were collected using the ω - 2θ scan technique, $2 \leq 2\theta \leq 55^\circ$ ($-2 \leq h \leq 20$, $-5 \leq k \leq 24$, $-10 \leq l \leq 10$ $\{-3 \leq h \leq 12$, $0 \leq k \leq 25$, $-19 \leq l \leq 19\}$), no decomposition of either crystal was observed during the data collection based on three reference reflections remeasured every 3600 s throughout the data collection. Corrections were applied for Lorentz and polarization effects and for absorption (*SHELX76*; Sheldrick, 1976), maximum and minimum transmission factors 0.9253 and 0.8349 {0.8064 and 0.7361} respectively. Equivalent reflections in the 4175 {8365} measured intensities were merged ($R_{\text{int}} = 0.018$ {0.022}) giving 2684 {6576} unique reflections of which 1696 {3532} had $I \geq 2\sigma I$. Both structures were solved by Patterson interpretation (*SHELXS86*; Sheldrick, 1985) which gave the Ni- and S-atom coordinates. The remaining non-H atoms were located in subsequent difference

maps. All non-H atoms were refined using a least-squares procedure based on *F* (*SHELX76*; Sheldrick, 1976) with anisotropic thermal parameters. Evidence of all H atoms was observed in a difference map and these were included in the model at their calculated positions with a common isotropic thermal parameter being refined for each H-type. A weighting scheme of the form $w = k[\sigma^2(F_o) + 0.0005F_o^2]$ was used and refinement of 147 {344} parameters, converged with $R = 0.042$ {0.044}, $wR = 0.042$ {0.042}, $S = 1.44$ {1.22}, $k = 2.198$ {1.2329}. Residual electron density in the range -0.33 to 0.38 $\{-0.43$ to $0.53\}$ e \AA^{-3} , max. $\Delta/\sigma = 0.01$ {0.003}. An analysis of variance showed no special features. The neutral-atom scattering factors used for Ni were from *International Tables for X-ray Crystallography* (1974) and those for the remaining atoms were as included in *SHELX76* (Sheldrick, 1976). All calculations were performed on the University of Melbourne VAX 11/780 and VAX 11/8650 computers. Fractional atomic coordinates are given in Table 1 for both crystal forms. Bond distances and angles are given in Table 2.

Discussion. Crystals of Ni(moexa)₂phen and its benzene solvate are composed of discrete molecules. The molecule is shown with its numbering scheme in Fig. 1(a) for the unsolvated form, in which the asymmetric unit is one half molecule of the complex, the Ni atom being located on a twofold axis. The benzene solvate has one molecule of the complex plus one molecule of benzene in the asymmetric unit as shown in Fig. 1(b). The molecular structure of Ni(moexa)₂phen is similar in both structures. The Ni atom is coordinated by a distorted octahedral arrangement of four S atoms from the two xanthate ligands and two N atoms from the 1,10-phenanthroline moiety. All bond lengths and angles lie in the range typically observed in nickel xanthate diamine adducts; the distance C(2)—C(3) of 1.466 (6) \AA in the unsolvated form is slightly shorter than the values observed for the analogous bonds in the benzene solvate but is longer than the 1.438 (8) \AA observed in the *C2/c* form of the 2,2'-bipyridyl analogue (Edwards, Hoskins & Winter, 1990). The major variation between the molecular structures of the two forms here, as was the case in the 2,2'-bipyridyl analogue, is found in the conformation of the methoxyethylxanthate moiety. In the benzene solvate the torsion angles C(1)—O(1)—C(2)—C(3) and C(5)—O(3)—

* Tables of anisotropic thermal parameters, calculated H-atom coordinates, observed and calculated structure factors, intermolecular contacts and mean planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52895 (48 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates for Ni-(moexa)₂phen: (a) unsolvated form (b) solvated form
$$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

(a) Unsolvated form				
	x	y	z	$B_{eq}(\text{\AA}^2)$
Ni	0.50000	0.71956 (3)	0.25000	3.25 (3)
S(1)	0.6138 (1)	0.80845 (6)	0.3066 (1)	2.72 (6)
S(2)	0.55844 (8)	0.73520 (5)	-0.0122 (1)	4.02 (5)
C(1)	0.6235 (3)	0.7948 (2)	0.1027 (4)	4.0 (2)
O(1)	0.6838 (2)	0.8330 (1)	0.0442 (3)	4.9 (2)
C(2)	0.6956 (3)	0.8242 (2)	-0.1298 (5)	5.1 (2)
C(3)	0.6382 (3)	0.8699 (2)	-0.2471 (5)	5.0 (2)
O(2)	0.6626 (2)	0.9405 (2)	-0.2246 (4)	5.6 (2)
C(4)	0.6120 (4)	0.9852 (3)	-0.3447 (6)	6.5 (3)
N	0.4234 (2)	0.6353 (2)	0.1496 (4)	3.3 (2)
C(5)	0.3482 (3)	0.6364 (3)	0.0425 (5)	4.7 (2)
C(6)	0.3054 (3)	0.5746 (3)	-0.0212 (6)	6.2 (3)
C(7)	0.3399 (3)	0.5111 (3)	0.0279 (6)	6.1 (3)
C(8)	0.4194 (3)	0.5074 (2)	0.1396 (5)	4.7 (2)
C(9)	0.4590 (2)	0.5716 (2)	0.1946 (4)	3.2 (2)
C(10)	0.4618 (3)	0.4423 (2)	0.1990 (6)	6.5 (2)
(b) Solvated form				
	x	y	z	$B_{eq}(\text{\AA}^2)$
Ni	0.11345 (5)	-0.00460 (3)	0.27813 (3)	2.78 (2)
S(1)	-0.03856 (5)	0.03856 (6)	0.38068 (8)	3.53 (5)
S(2)	0.1671 (1)	0.11519 (6)	0.30041 (7)	3.51 (5)
S(3)	-0.0830 (1)	-0.00081 (6)	0.14082 (7)	3.28 (5)
S(4)	0.0161 (1)	-0.1177 (5)	0.25702 (8)	3.41 (5)
C(1)	0.0638 (4)	0.1106 (2)	0.3730 (3)	3.2 (2)
O(1)	0.0574 (3)	0.1656 (1)	0.4220 (2)	4.6 (2)
C(2)	-0.0336 (5)	0.1659 (2)	0.4834 (3)	5.1 (2)
C(3)	0.0002 (8)	0.2302 (3)	0.5401 (4)	7.3 (4)
O(2)	-0.0415 (5)	0.2834 (2)	0.4843 (3)	8.0 (2)
C(4)	-0.0165 (9)	0.3461 (3)	0.5348 (5)	10.6 (5)
C(5)	-0.0963 (4)	-0.0835 (2)	0.1646 (3)	3.0 (2)
O(3)	-0.1985 (3)	-0.1176 (1)	0.1088 (2)	3.5 (2)
C(6)	-0.2103 (5)	-0.1893 (2)	0.1260 (3)	4.2 (2)
C(7)	-0.3335 (5)	-0.2149 (3)	0.0565 (4)	4.9 (3)
O(4)	-0.4523 (4)	-0.1880 (2)	0.0760 (3)	5.8 (2)
C(8)	-0.5777 (6)	-0.2126 (3)	0.0189 (5)	7.1 (4)
N(1)	0.2641 (3)	-0.0213 (2)	0.2078 (2)	3.0 (2)
N(2)	0.2763 (3)	-0.0385 (2)	0.3857 (2)	2.8 (2)
C(9)	0.2567 (5)	-0.0109 (2)	0.1194 (3)	3.9 (2)
C(10)	0.3690 (6)	-0.0224 (3)	0.0825 (3)	5.0 (3)
C(11)	0.4909 (5)	-0.0455 (2)	0.1377 (4)	4.8 (2)
C(12)	0.5023 (5)	-0.0570 (2)	0.2313 (3)	3.7 (2)
C(13)	0.3857 (4)	-0.0441 (2)	0.2630 (3)	3.0 (2)
C(14)	0.2782 (5)	-0.0496 (2)	0.4727 (3)	3.4 (2)
C(15)	0.3911 (5)	-0.0783 (2)	0.5356 (3)	4.0 (2)
C(16)	0.5080 (6)	-0.0942 (2)	0.5091 (3)	4.0 (2)
C(17)	0.5098 (5)	-0.0827 (2)	0.4172 (3)	3.5 (2)
C(18)	0.3903 (4)	-0.0556 (2)	0.3573 (3)	2.8 (2)
C(19)	0.6285 (5)	-0.0947 (3)	0.3834 (4)	4.7 (2)
C(20)	0.6253 (5)	-0.0816 (3)	0.2952 (4)	5.1 (2)
C(1BZ)	0.4915 (9)	0.2070 (5)	0.2337 (5)	8.9 (6)
C(2BZ)	0.5163 (9)	0.1435 (4)	0.2153 (5)	7.7 (5)
C(3BZ)	0.641 (1)	0.1252 (4)	0.2115 (5)	7.8 (5)
C(4BZ)	0.7455 (8)	0.1674 (5)	0.2267 (4)	7.2 (5)
C(5BZ)	0.728 (1)	0.2329 (6)	0.2476 (5)	9.5 (6)
C(6BZ)	0.592 (1)	0.2536 (4)	0.2500 (5)	9.8 (6)

C(6)—C(7) are 170.9 (4) and 178.5 (4)° respectively, close to the value observed for one of the two methoxyethylxanthate moieties in the $P2_1/n$ form of the 2,2'-bipyridyl analogue, while the same angle in the unsolvated form C(1)—O(1)—C(2)—C(3) has a value of 87.2 (4)°, close to the value observed for the analogous bond in the $C2/c$ form of the 2,2'-bipyridyl compound.

The packing of molecules in the unsolvated crystals is shown in Fig. 2(a). It bears a striking similarity to that observed in the $C2/c$ form of the 2,2'-bipyridyl analogue (Edwards, Hoskins & Winter, 1990). The manner in which the molecules are packed in crystals of the benzene solvate is shown in

Table 2. Bond lengths (Å) and angles (°) for Ni-(moexa)₂phen: (a) unsolvated form (b) solvated form

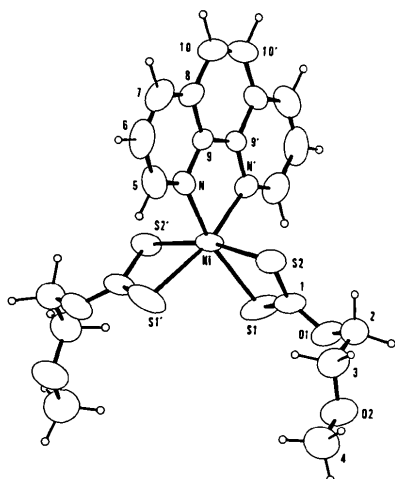
(a) Unsolvated form			
Ni—S(1)	2.436 (1)	N—C(5)	1.334 (5)
Ni—S(2)	2.450 (1)	N—C(9)	1.349 (5)
Ni—N	2.072 (3)	C(5)—C(6)	1.399 (7)
S(1)—C(1)	1.687 (4)	C(6)—C(7)	1.347 (8)
S(2)—C(1)	1.683 (4)	C(7)—C(8)	1.406 (6)
C(1)—O(1)	1.337 (5)	C(8)—C(9)	1.399 (5)
O(1)—C(2)	1.447 (5)	C(8)—C(10)	1.440 (6)
C(2)—C(3)	1.466 (6)	C(9)—C(9')	1.432 (4)
C(3)—O(2)	1.391 (5)	C(10)—C(10')	1.329 (6)
O(2)—C(4)	1.417 (6)		
S(1)—Ni—S(2)	72.9 (1)	O(1)—C(2)—C(3)	112.6 (3)
S(1)—Ni—S(1)'	92.8 (1)	C(2)—C(3)—O(2)	110.9 (3)
S(1)—Ni—S(2)'	97.3 (1)	C(3)—O(2)—C(4)	112.0 (3)
S(1)—Ni—N	163.6 (2)	Ni—N—C(5)	128.8 (3)
S(1)—Ni—N'	95.7 (2)	Ni—N—C(9)	113.3 (3)
S(2)—Ni—S(2)	166.1 (2)	C(5)—N—C(9)	117.7 (3)
S(2)—Ni—N	92.1 (2)	N—C(5)—C(6)	122.5 (4)
S(2)—Ni—N'	98.5 (2)	C(5)—C(6)—C(7)	119.6 (4)
N(1)—Ni—N'	79.6 (2)	C(6)—C(7)—C(8)	119.9 (4)
Ni—S(1)—C(1)	84.2 (2)	C(7)—C(8)—C(9)	117.0 (3)
Ni—S(2)—C(1)	83.8 (2)	C(7)—C(8)—C(10)	124.2 (3)
S(1)—C(1)—S(2)	119.0 (3)	C(9)—C(8)—C(10)	118.8 (3)
S(1)—C(1)—O(1)	116.4 (3)	N—C(9)—C(8)	123.2 (3)
S(2)—C(1)—O(1)	124.6 (3)	N—C(9)—C(9')	116.9 (3)
C(1)—O(1)—C(2)	119.4 (3)	C(8)—C(10)—C(10')	121.3 (3)
(b) Solvated form			
Ni—S(1)	2.458 (1)	N(1)—C(13)	1.357 (5)
Ni—S(2)	2.438 (1)	N(2)—C(14)	1.323 (6)
Ni—S(3)	2.449 (1)	N(2)—C(18)	1.362 (6)
Ni—S(4)	2.431 (1)	C(9)—C(10)	1.397 (8)
Ni—N(1)	2.084 (3)	C(10)—C(11)	1.361 (7)
Ni—N(2)	2.082 (3)	C(11)—C(12)	1.402 (8)
S(1)—C(1)	1.684 (4)	C(20)—C(19)	1.345 (9)
S(2)—C(1)	1.693 (5)	C(20)—C(12)	1.433 (6)
S(3)—C(5)	1.690 (4)	C(19)—C(17)	1.430 (8)
S(4)—C(5)	1.684 (4)	C(16)—C(15)	1.368 (8)
C(1)—O(1)	1.327 (5)	C(16)—C(17)	1.407 (7)
O(1)—C(2)	1.458 (6)	C(15)—C(14)	1.390 (6)
C(2)—C(3)	1.521 (7)	C(18)—C(17)	1.401 (6)
C(3)—O(2)	1.344 (7)	C(18)—C(13)	1.426 (7)
O(2)—C(4)	1.444 (7)	C(12)—C(13)	1.396 (7)
C(5)—O(3)	1.325 (4)	C(1BZ)—C(2BZ)	1.33 (1)
O(3)—C(6)	1.455 (5)	C(1BZ)—C(6BZ)	1.34 (1)
C(6)—C(7)	1.480 (6)	C(2BZ)—C(3BZ)	1.32 (1)
C(7)—O(4)	1.403 (7)	C(3BZ)—C(4BZ)	1.31 (1)
O(4)—C(8)	1.408 (7)	C(4BZ)—C(5BZ)	1.36 (1)
N(1)—C(9)	1.329 (6)	C(5BZ)—C(6BZ)	1.43 (1)
S(1)—Ni—S(2)	73.3 (1)	C(1)—O(1)—C(2)	119.0 (3)
S(1)—Ni—S(3)	93.9 (1)	O(1)—C(2)—C(3)	106.0 (3)
S(1)—Ni—S(4)	97.8 (1)	C(2)—C(3)—O(2)	108.7 (4)
S(1)—Ni—N(1)	164.9 (1)	C(3)—O(2)—C(4)	111.4 (4)
S(1)—Ni—N(2)	94.1 (1)	S(3)—C(5)—S(4)	119.5 (2)
S(2)—Ni—S(3)	100.7 (1)	S(3)—C(5)—O(3)	116.8 (3)
S(2)—Ni—S(4)	169.1 (1)	S(3)—C(5)—O(3)	123.7 (3)
S(2)—Ni—N(1)	93.6 (1)	C(5)—O(3)—C(6)	118.1 (3)
S(2)—Ni—N(2)	96.2 (1)	O(3)—C(6)—C(7)	107.5 (3)
S(3)—Ni—S(4)	73.3 (1)	C(6)—C(7)—O(4)	108.4 (4)
S(3)—Ni—N(1)	95.9 (1)	C(7)—O(4)—C(8)	114.1 (4)
S(3)—Ni—N(2)	162.8 (1)	Ni—N(1)—C(9)	128.8 (3)
S(4)—Ni—N(1)	96.1 (1)	Ni—N(1)—C(13)	113.2 (3)
S(4)—Ni—N(2)	90.5 (1)	C(9)—N(1)—C(13)	118.0 (3)
N(1)—Ni—N(2)	79.7 (2)	Ni—N(2)—C(14)	128.9 (3)
Ni—S(1)—C(1)	83.1 (2)	Ni—N(2)—C(18)	112.6 (3)
Ni—S(2)—C(1)	83.5 (2)	C(14)—N(2)—C(18)	118.3 (3)
Ni—S(3)—C(5)	83.2 (2)	N(1)—C(9)—C(10)	122.3 (3)
Ni—S(4)—C(5)	83.9 (2)	C(9)—C(10)—C(11)	119.8 (4)
S(1)—C(1)—S(2)	119.8 (2)	C(10)—C(11)—C(12)	119.4 (4)
S(1)—C(1)—O(1)	124.0 (3)	C(19)—C(20)—C(12)	121.4 (4)
S(2)—C(1)—O(1)	116.2 (3)	C(20)—C(19)—C(17)	120.9 (4)
C(15)—C(16)—C(17)	118.9 (4)	C(20)—C(12)—C(13)	118.7 (3)
C(16)—C(15)—C(14)	120.0 (3)	N(1)—C(13)—C(18)	116.8 (3)
N(2)—C(14)—C(15)	122.6 (3)	N(1)—C(13)—C(12)	123.2 (3)
N(2)—C(18)—C(17)	122.4 (3)	C(18)—C(13)—C(12)	120.0 (3)
N(2)—C(18)—C(13)	117.6 (3)	C(2BZ)—C(1BZ)—C(6BZ)	121.3 (6)
C(17)—C(18)—C(13)	120.0 (3)	C(1BZ)—C(2BZ)—C(3BZ)	120.2 (6)
C(19)—C(17)—C(16)	123.4 (4)	C(2BZ)—C(3BZ)—C(4BZ)	122.6 (6)
C(19)—C(17)—C(18)	118.9 (4)	C(3BZ)—C(4BZ)—C(5BZ)	120.3 (6)
C(16)—C(17)—C(18)	117.7 (3)	C(4BZ)—C(5BZ)—C(6BZ)	117.5 (7)
C(11)—C(12)—C(20)	123.9 (4)	C(1BZ)—C(6BZ)—C(5BZ)	118.1 (7)
C(11)—C(12)—C(13)	117.4 (3)		

Atoms marked ' are related to the reported asymmetric unit by $1-x, y, \frac{1}{2}-z$.

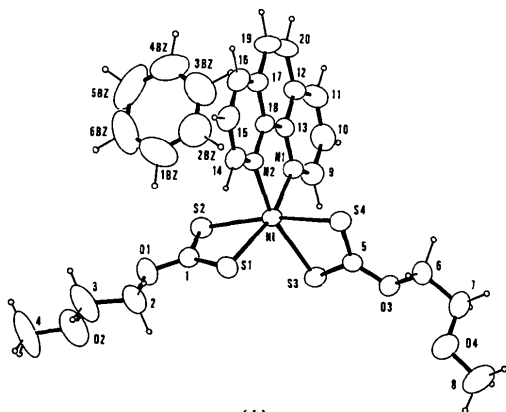
Fig. 2(b). It is apparent that the benzene molecules lie in an interrupted channel extending through the structure, the interruptions being caused by the terminal atoms of the methoxyethyl moieties. The open nature of this cavity provides an explanation for the speed with which the crystals deteriorate on exposure to the atmosphere.

The structures reported here verify that solvent inclusion behaviour is not a general occurrence in nickel(II) xanthate diamine adducts, and where such inclusion behaviour is observed for chelating diamines, it is not restricted to inclusion in discrete cavities.

The Commonwealth of Australia is thanked for a Commonwealth Post Graduate Research Award for AJE.

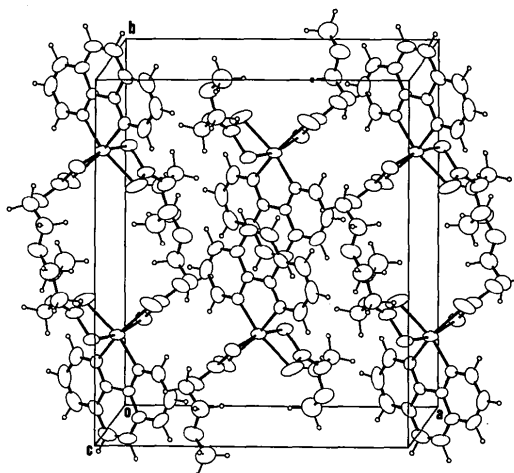


(a)

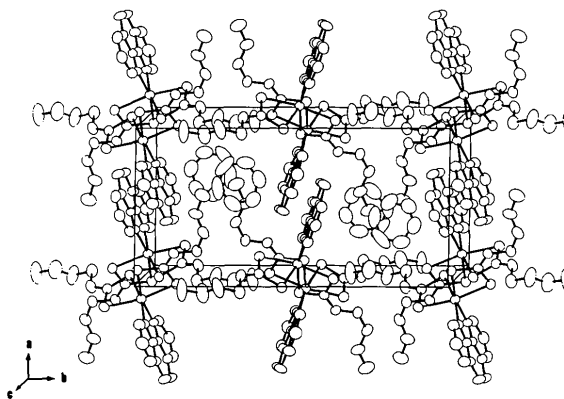


(b)

Fig. 1. (a) A molecule of $\text{Ni}(\text{moexa})_2\text{phen}$ (unsolvated form), atoms indicated ' are related to the reference asymmetric unit by $1-x, y, \frac{1}{2}-z$ (ORTEPII; Johnson, 1971). (b) The asymmetric unit for $\text{Ni}(\text{moexa})_2\text{phen}$ (benzene solvate) (ORTEPII; Johnson, 1971).



(a)



(b)

Fig. 2. The packing of the molecules in (a) the unsolvated form and (b) the solvated form (ORTEPII; Johnson, 1971).

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