Two Crystal Forms of Bis(*O*-methoxyethylxanthato)(2,2′-bipyridyl)nickel(II)

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Abstract. (2,2′-Bipyridyl)bis(*O*-methoxyethyldithiocarbonato)nickel(II), [Ni($C_{10}H_8N_2$)($C_4H_7O_2S_2$)$_2$], M_r = 517·2, $F(000)$ = 1072, T = 295 (2) K, λ(Mo $K\alpha$) = 0·71069 Å. Two crystalline forms have been isolated, both being monoclinic. Form I, grown from diethyl ether solution, $P2_1/n$, a = 9·325 (2), b = 7·704 (1), c = 31·910 (6) Å, β = 93·74 (2)°, U = 2287 (1) Å^3, Z = 4, μ = 11·92 cm^{-1}, D_x = 1·502 (1), D_m = 1·49 (2) g cm^{-3}, R = 0·034 for 2565 unique reflections ($I \geq 2\sigma I$). Form II, grown from benzene solution, $C2/c$, a = 15·919 (2), b = 17·703 (3), c = 8·1420 (7) Å, β = 99·940 (8)°, U = 2260·1 (9) Å^3, Z = 4, μ = 12·05 cm^{-1}, D_x = 1·520 (1), D_m = 1·51 (2) g cm^{-3}, R = 0·043 for 1475 unique reflections ($I \geq 2\sigma I$). The crystal structure is found to be solvent dependent even though no solvent is present in the lattice of either form. The structures differ in the conformation of the methoxyethyl groups, and in the way in which the molecules are packed.

Introduction. The product formed on reaction of nickel(II) xanthates with heterocyclic diamines has been observed to vary with solvent and temperature (Kruger & Winter, 1971). Subsequent crystal-structure determinations have shown two types of inclusion behaviour in these compounds. One involves inclusion in open channels as seen in the 4,4′-bipyridyl adduct of Ni($S_2COC_4H_9$)$_2$ (Gable, Hoskins & Winter, 1985), whereas the second has solvent included in discrete cavities (Edwards, Hoskins & Winter, 1986; Gable, Hoskins & Winter, 1985). As a continuation of our investigation of this behaviour, crystals of Ni($S_2COCH_2CH_2OCH_3$)$_2$(2,2′-bipyridyl) [= Ni(moexa)$_2$bpy] were grown from solutions in diethyl ether and in benzene.

Experimental. The compound was prepared by combining equimolar quantities of nickel *O*-methoxyethylxanthate with 2,2′-bipyridyl in solution. Form I was obtained by reaction in warmed diethyl ether which yielded needle-like green crystals on cooling. A crystal with faces ±(10$\bar{1}$), ±(001), ±(010) separated

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by 0·43, 0·12 and 0·09 mm respectively was selected for study. Form II was obtained by reaction in hot benzene (348 K), the residue remaining on evaporation at ambient temperature and pressure was then recrystallized from benzene, yielding prismatic pleochroic crystals. Since the diffraction intensity of these crystals decreased on exposure to air the study was performed on a crystal sealed in a capillary with mother liquor. The crystal selected for study had faces ±(110), ±($\bar{1}$10), ±($\bar{1}$11) and ±(11$\bar{1}$) separated by 0·258, 0·214, 0·34 and 0·34 mm respectively.

Oscillation and Weissenberg photographs showed both crystals to be monoclinic, but of different forms. Density was measured by flotation in aqueous zinc bromide.

Data collection, structure solution and refinement for the two forms were similar and where values differ, those for form II are shown in brackets { }. An Enraf–Nonius CAD-4F diffractometer and graphite-monochromated Mo $K\alpha$ radiation were used. Cell dimensions were obtained by least-squares refinement of angular values for 25 reflections in the range $16·8 \leq 2\theta \leq 32·74°$ {$16·58 \leq 2\theta \leq 49·98°$}.

Intensity data were collected using the ω–2θ scan technique in the range $2 \leq 2\theta \leq 50°$ ($-7 \leq h \leq 11$, $-5 \leq k \leq 10$, $-38 \leq l \leq 38$) {$2 \leq 2\theta \leq 55°$ ($-17 \leq h \leq 20$, $-2 \leq k \leq 22$, $-10 \leq l \leq 10$)}, the crystal of form I was weakly scattering, and no significant data could be measured above $2\theta = 50°$. No significant variation in the diffraction intensity of either crystal was observed during the data collection, based on the remeasurement of three check reflections every 3600 s of X-ray exposure time. Corrections were applied for Lorentz and polarization effects and for absorption (*SHELX*76; Sheldrick, 1976); maximum and minimum transmission factors 0·8988 and 0·8517 {0·7616 and 0·6847} respectively, no correction was made for extinction. Equivalent reflections in the 6853 {3746} measured intensities were merged (R_{int} = 0·025 {0·016}) giving 4015 {2553} unique reflections of which 2565 {1475} had $I \geq 2\sigma I$.

Both structures were solved by Patterson methods which gave the Ni- and S-atom coordinates (*SHELXS*86; Sheldrick, 1985). The remaining

Table 1. *Fractional atomic coordinates for Ni(moexa)₂bpy, (a) form I and (b) form II*

$$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
(a) Form I				
Ni	0·01570 (5)	0·05232 (8)	0·13020 (2)	2·86 (4)
S(1)	0·0195 (1)	0·3514 (2)	0·10588 (4)	3·76 (6)
S(2)	−0·1445 (1)	0·0659 (2)	0·06611 (4)	3·83 (5)
S(3)	−0·0557 (1)	−0·2364 (2)	0·15281 (4)	3·67 (5)
S(4)	−0·1867 (1)	0·0924 (2)	0·17430 (4)	3·79 (5)
C(1)	−0·0988 (5)	0·2784 (6)	0·0678 (1)	3·5 (2)
O(1)	−0·1612 (4)	0·3799 (5)	0·0383 (1)	4·4 (2)
C(2)	−0·1386 (6)	0·5657 (7)	0·0410 (2)	4·6 (2)
C(3)	−0·2040 (6)	0·6428 (7)	0·0014 (2)	4·7 (3)
O(2)	−0·3551 (4)	0·6265 (6)	0·0009 (1)	5·6 (2)
C(4)	−0·4253 (7)	0·6675 (9)	−0·0380 (2)	6·6 (4)
C(5)	−0·1811 (4)	−0·1264 (7)	0·1773 (1)	3·3 (2)
O(3)	−0·2791 (3)	−0·2007 (5)	0·2003 (1)	4·7 (2)
C(6)	−0·2908 (6)	−0·3900 (8)	0·2019 (2)	5·3 (3)
C(7)	−0·4043 (6)	−0·4509 (8)	0·1700 (2)	5·6 (3)
O(4)	−0·5395 (4)	−0·3947 (6)	0·1806 (1)	5·6 (2)
C(8)	−0·6536 (6)	−0·4644 (9)	0·1532 (2)	6·1 (3)
N(1)	0·1735 (3)	0·0947 (5)	0·1779 (1)	2·9 (2)
N(2)	0·1965 (3)	−0·0320 (5)	0·1017 (1)	2·9 (2)
C(9)	0·1552 (5)	0·1647 (6)	0·2155 (1)	3·5 (2)
C(10)	0·2673 (5)	0·1969 (7)	0·2447 (2)	4·3 (2)
C(11)	0·4031 (5)	0·1533 (8)	0·2348 (2)	4·9 (3)
C(12)	0·4253 (5)	0·0791 (7)	0·1966 (1)	4·0 (2)
C(13)	0·3076 (4)	0·0534 (6)	0·1681 (1)	2·8 (2)
C(14)	0·3214 (4)	−0·0172 (6)	0·1255 (1)	2·8 (2)
C(15)	0·4517 (5)	−0·0643 (7)	0·1100 (1)	3·7 (2)
C(16)	0·4538 (5)	−0·1330 (7)	0·0702 (2)	4·4 (2)
C(17)	0·3261 (5)	−0·1527 (7)	0·0466 (2)	4·5 (2)
C(18)	0·2005 (5)	−0·0993 (7)	0·0632 (1)	3·8 (2)
(b) Form II				
Ni	0·00000	0·20548 (5)	0·25000	3·51 (4)
S(1)	0·1130 (1)	0·30146 (8)	0·2910 (2)	5·78 (7)
S(2)	0·05097 (9)	0·21960 (7)	−0·0145 (1)	4·67 (6)
C(1)	0·1163 (4)	0·2843 (3)	0·0894 (6)	4·5 (2)
O(1)	0·1746 (3)	0·3239 (2)	0·0235 (5)	5·6 (2)
C(2)	0·1843 (5)	0·3111 (3)	−0·1479 (7)	6·7 (4)
C(3)	0·1317 (4)	0·3610 (4)	−0·2614 (7)	6·2 (3)
O(2)	0·1565 (3)	0·4357 (2)	−0·2425 (5)	6·2 (2)
C(4)	0·1060 (5)	0·4829 (4)	−0·3606 (9)	7·3 (5)
N	0·0764 (2)	0·1153 (2)	0·3432 (5)	4·1 (2)
C(5)	0·0417 (3)	0·0469 (3)	0·3043 (7)	4·7 (2)
C(6)	0·0841 (5)	−0·0172 (6)	0·3693 (9)	7·4 (4)
C(7)	0·1613 (6)	−0·0117 (5)	0·473 (1)	9·4 (6)
C(8)	0·1954 (5)	0·0580 (4)	0·5129 (9)	8·1 (5)
C(9)	0·1510 (4)	0·1205 (4)	0·4456 (7)	5·8 (3)

Table 2. *Bond lengths (Å) and angles (°) for Ni(moexa)₂bpy, (a) form I (b) form II*

(a) Form I

Ni—S(1)	2·432 (2)	C(6)—C(7)	1·495 (8)
Ni—S(2)	2·455 (1)	C(7)—O(4)	1·395 (7)
Ni—S(3)	2·444 (4)	O(4)—C(8)	1·436 (7)
Ni—S(4)	2·446 (1)	N(1)—C(9)	1·336 (5)
Ni—N(1)	2·073 (3)	N(1)—C(13)	1·347 (5)
Ni—N(2)	2·072 (3)	N(2)—C(14)	1·353 (5)
S(1)—C(1)	1·684 (4)	N(2)—C(18)	1·336 (5)
S(2)—C(1)	1·684 (4)	C(9)—C(10)	1·377 (7)
S(3)—C(5)	1·678 (4)	C(10)—C(11)	1·367 (7)
S(4)—C(5)	1·689 (6)	C(11)—C(12)	1·374 (7)
C(1)—O(1)	1·328 (5)	C(12)—C(13)	1·393 (5)
O(1)—C(2)	1·449 (7)	C(13)—C(14)	1·478 (5)
C(2)—C(3)	1·490 (8)	C(14)—C(15)	1·390 (6)
C(3)—O(2)	1·414 (7)	C(15)—C(16)	1·377 (7)
O(2)—C(4)	1·401 (7)	C(16)—C(17)	1·375 (7)
C(5)—O(3)	1·338 (5)	C(17)—C(18)	1·379 (7)
O(3)—C(6)	1·464 (7)		

S(1)—Ni—S(2)	73·4 (1)	N(2)—C(18)—C(17)	123·0 (3)
S(1)—Ni—S(3)	165·1 (1)	S(2)—Ni—S(3)	97·0 (1)
S(1)—Ni—S(4)	95·3 (1)	S(2)—Ni—S(4)	91·3 (1)
S(1)—Ni—N(1)	93·5 (1)	S(2)—Ni—N(1)	165·8 (1)
S(1)—Ni—N(2)	97·3 (1)	S(2)—Ni—N(2)	96·7 (1)
S(3)—Ni—N(1)	96·9 (1)	S(3)—Ni—S(4)	73·1 (1)
S(4)—Ni—N(1)	95·5 (1)	S(3)—Ni—N(2)	95·1 (1)
N(1)—Ni—N(2)	79·2 (2)	S(4)—Ni—N(2)	166·6 (1)
Ni—S(2)—C(1)	82·9 (2)	Ni—S(1)—C(1)	83·8 (2)
Ni—S(4)—C(5)	83·4 (2)	Ni—S(3)—C(5)	83·6 (2)
S(1)—C(1)—O(1)	123·4 (3)	S(1)—C(1)—S(2)	119·9 (2)
S(2)—C(1)—O(1)	116·7 (3)	C(1)—O(1)—C(2)	119·0 (3)
O(1)—C(2)—C(3)	107·1 (4)	C(2)—C(3)—O(2)	109·1 (4)
C(3)—O(2)—C(4)	113·4 (4)	S(3)—C(5)—S(4)	119·8 (3)
S(3)—C(5)—O(3)	124·1 (3)	S(4)—C(5)—O(3)	116·0 (3)
C(5)—O(3)—C(6)	120·0 (3)	O(3)—C(6)—C(7)	109·9 (4)
C(6)—C(7)—O(4)	110·4 (4)	Ni—N(1)—C(9)	126·6 (3)
C(7)—O(4)—C(8)	112·4 (4)	Ni—N(2)—C(14)	115·0 (3)
Ni—N(1)—C(13)	114·7 (3)	C(9)—N(1)—C(13)	118·6 (3)
Ni—N(2)—C(18)	126·7 (3)	N(1)—C(9)—C(10)	123·0 (3)
C(14)—N(2)—C(18)	118·3 (3)	C(10)—C(11)—C(12)	120·2 (4)
C(9)—C(10)—C(11)	118·2 (4)	N(1)—C(13)—C(12)	121·2 (3)
C(11)—C(12)—C(13)	118·7 (4)	C(12)—C(13)—C(14)	122·7 (3)
N(1)—C(13)—C(14)	116·0 (3)	N(2)—C(14)—C(15)	121·3 (3)
N(2)—C(14)—C(13)	115·1 (3)	C(14)—C(15)—C(16)	119·6 (3)
C(13)—C(14)—C(15)	123·7 (3)	C(16)—C(17)—C(18)	118·9 (4)
C(15)—C(16)—C(17)	118·9 (4)		

(b) Form II

Ni—S(1)	2·455 (2)	C(3)—O(2)	1·381 (8)
Ni—S(2)	2·442 (1)	O(2)—C(4)	1·415 (8)
Ni—N	2·070 (4)	N—C(5)	1·346 (6)
S(1)—C(1)	1·679 (5)	N—C(9)	1·331 (7)
S(2)—C(1)	1·675 (5)	C(5)—C(5)′	1·463 (7)
C(1)—O(1)	1·346 (8)	C(5)—C(6)	1·389 (8)
O(1)—C(2)	1·448 (7)	C(6)—C(7)	1·37 (1)
C(2)—C(3)	1·438 (8)	C(7)—C(8)	1·36 (1)
		C(8)—C(9)	1·38 (1)

S(1)—Ni—S(1)′	92·4 (1)	C(1)—O(1)—C(2)	119·9 (4)
S(1)—Ni—S(2)	72·7 (1)	O(1)—C(2)—C(3)	112·1 (4)
S(1)—Ni—S(2)′	99·0 (1)	C(2)—C(3)—O(2)	113·1 (4)
S(1)—Ni—N	96·5 (1)	C(3)—O(2)—C(4)	111·8 (4)
S(1)—Ni—N′	162·5 (2)	Ni—N—C(5)	114·6 (3)
S(2)—Ni—S(2)′	168·2 (1)	Ni—N—C(9)	125·5 (3)
S(2)—Ni—N	98·0 (1)	C(5)—N—C(9)	119·7 (3)
S(2)—Ni—N′	91·1 (1)	N—C(5)—C(5)′	115·8 (4)
N—Ni—N′	79·1 (2)	N—C(5)—C(6)	120·1 (4)
Ni—S(1)—C(1)	83·6 (2)	C(5)—C(6)—C(7)	119·6 (5)
Ni—S(2)—C(1)	84·1 (2)	C(6)—C(7)—C(8)	119·8 (6)
S(1)—C(1)—S(2)	119·7 (3)	C(7)—C(8)—C(9)	118·5 (6)
S(1)—C(1)—O(1)	115·8 (4)	C(8)—C(9)—N	122·4 (5)
S(2)—C(1)—O(1)	124·4 (4)	C(5)′—C(5)—C(6)	124·1 (4)

Atoms marked ′ are related to the given atom by $-x, y, \frac{1}{2}-z$.

non-H atoms were located in subsequent difference maps. All non-H atoms were refined using a least-squares procedure based on *F* (*SHELX*76; Sheldrick, 1976) with anisotropic thermal parameters. Evidence of all H atoms was observed in the 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, and unit weights were used throughout. Refinement converged with $R = 0·034$ {0·043}, $wR = 0·036$ {0·042} for 265 {138} parameters, the goodness of fit $S = 1·521$ {1·772}. Residual electron density was in the range $-0·26$ to $0·29$ {$-0·25$ to $0·47$} e Å^{-3}, max. $\Delta/\sigma = 0·002$ {0·001} in the final refinement cycle. An analysis of variance showed no special features. The neutral-atom scattering factors used for Ni were from *International Tables for X-ray Crystallography* (Ibers & Hamilton, 1974) and for the remaining
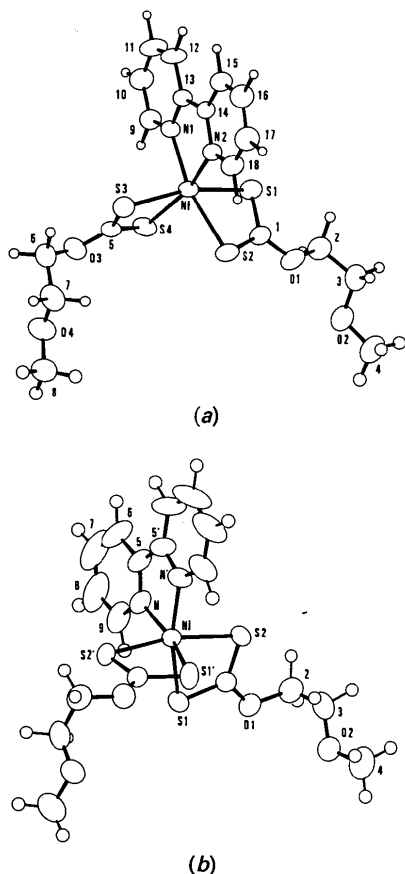
atoms were those included in *SHELX*76 (Sheldrick, 1976). All calculations were performed on the University of Melbourne VAX 11/780 and VAX 11/8650 computers. Fractional atomic coordinates

for both forms are given in Table 1* and bond lengths and angles are given in Table 2.

Discussion. Crystals of both forms of Ni-(moexa)₂bpy are composed of discrete molecules of the compound. The asymmetric unit for form I consists of one complete molecule which is shown with its numbering scheme in Fig. 1(*a*). In form II the asymmetric unit is half a molecule, the Ni atom being located on a twofold axis and the numbering scheme used is indicated in Fig. 1(*b*).

The molecular structure of Ni(moexa)₂bpy is similar in both forms. 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 2,2′-bipyridyl moiety. All bond lengths and angles in the coordination sphere of the metal atom and in the bipyridyl moiety are in the range observed in comparable compounds (Edwards, Hoskins & Winter, 1986; Gable, Hoskins & Winter, 1985). There are two significant differences between the geometry of molecules of form I and form II. The bond length of 1·438 (8) Å for C(2)—C(3) in form II is shorter than the comparable distances C(2)—C(3) and C(6)—C(7) in form I which are 1·490 (8) and 1·495 (8) Å respectively. These latter two values are in close agreement with the values reported for analogous bonds in Ni(moexa)₂ and Cd(moexa)₂ (Abra-
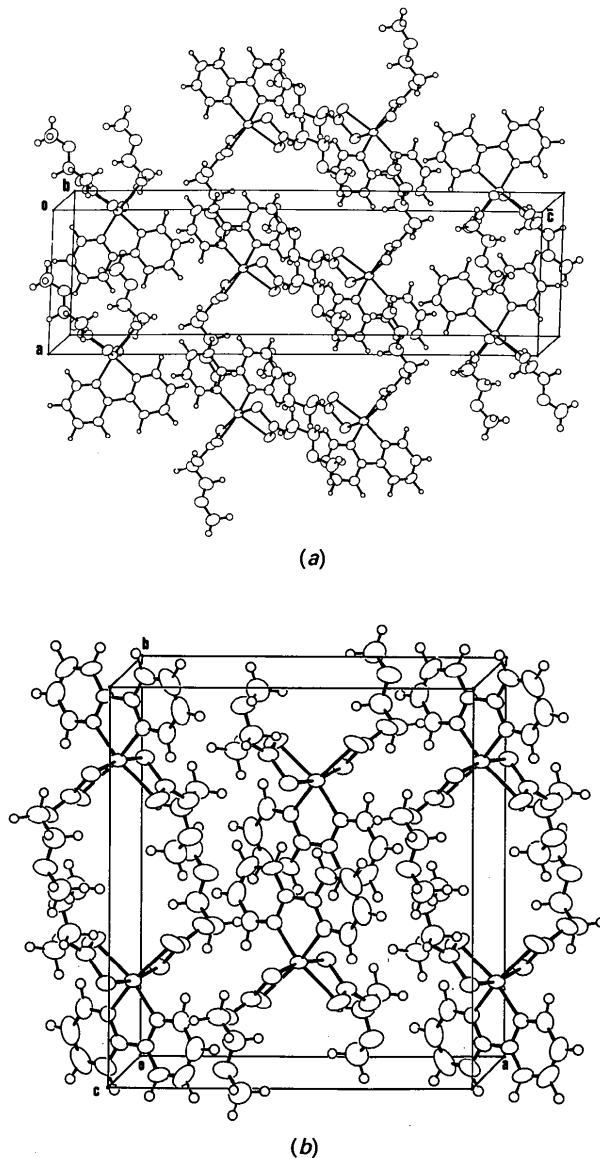
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* Tables of anisotropic thermal parameters, calculated H-atom coordinates, observed and calculated structure factors, inter-molecular contacts and mean planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52893 (40 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(*a*)

(*b*)

Fig. 1. (*a*) A molecule of Ni(moexa)₂bpy, form I, and (*b*) a molecule of Ni(moexa)₂bpy, form II, showing the numbering scheme employed. Atoms marked ′ are related to the reported coordinates by (−x, y, ½ − z) (*ORTEP*II; Johnson, 1971).

(*a*)

(*b*)

Fig. 2. The packing in crystals of (*a*) form I and (*b*) form II (*ORTEP*II; Johnson, 1971).

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).

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

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Abstract. Bis(O-methoxyethyldithiocarbonato)(1,10-phenanthroline)nickel(II), [Ni(C$_{12}$H$_8$N$_2$)(C$_4$H$_7$O$_2$S$_2$)$_2$]. Crystals grown from acetone solution contain only the title compound, $M_r = 541\cdot4$, monoclinic, C2/c, $a = 15\cdot661$ (2), $b = 18\cdot894$ (4), $c = 8\cdot013$ (2) Å, $\beta = 99\cdot89$ (2)°, $U = 2338$ (2) Å^3, $Z = 4$, $F(000) = 1120$, $\mu = 11\cdot68$ cm^{-1}, $D_x = 1\cdot537$, $D_m = 1\cdot52$ (2) g cm^{-3}, $T = 295$ (2) K, $R = 0\cdot042$ for 1697 unique reflections ($I > 2\sigma I$). Crystals grown from benzene solution form as benzene solvate, [Ni(C$_{12}$H$_8$N$_2$)(C$_4$H$_7$O$_2$S$_2$)$_2$].C$_6$H$_6$, $M_r = 619\cdot5$, monoclinic, P2$_1$/a, $a = 10\cdot005$ (1), $b = 19\cdot820$ (2), $c = 15\cdot052$ (3) Å, $\beta = 105\cdot29$ (1)°, $U = 2879$ (1) Å^3, $Z = 4$, $F(000) = 1288$, λ(Mo Kα) = 0·71069 Å, $\mu = 9\cdot54$ cm^{-1}, $D_x = 1\cdot43$ g cm^{-3}, $T = 295$ (2) K, $R = 0\cdot044$ for 3532 unique reflections ($I \geq 2\sigma I$). The crystal 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(S$_2$COCH$_2$CH$_2$OCH$_3$)$_2$.1,10-phenanthroline [Ni(moexa)$_2$phen] 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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