MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
ROBERTS, P. & SHELDRICK, G. M. (1975). *XANADU*. Program for crystallographic calculations. Univ. of Cambridge, England.
SHANNON, R. D. (1976). *Acta Cryst.* A**32**, 751–767.
SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
SHELDRICK, G. M. (1984). Personal communication.
VÖGTLE, F. & WEBER, E. (1979). *Angew. Chem.* **91**, 813–837.
WEBER, G., HIRAYAMA, F., SAENGER, W. & SHELDRICK, G. M. (1984). *Acta Cryst.* C**40**, 1570–1572.

Acta Cryst. (1987). C**43**, 1080–1083

Oligoether Complexes of Alkaline-Earth Metal Ions. II. Structures of 2,5,8,11-Tetraoxadodecane ('Triglyme') Complexed With Calcium and Strontium Thiocyanates

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Abstract. (1) 2,5,8,11-Tetraoxadodecane–calcium thiocyanate monohydrate, $[Ca(SCN)_2(C_8H_{18}O_4)].H_2O$, $M_r = 352{\cdot}49$, monoclinic, $P2_1/n$, $a = 14{\cdot}672\,(10)$, $b = 16{\cdot}306\,(8)$, $c = 7{\cdot}286\,(2)$ Å, $\beta = 92{\cdot}89\,(4)°$, $V = 1741\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}35$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 5{\cdot}97$ cm^{-1}, $F(000) = 744$, $T = 291$ K, $R = 0{\cdot}048$ for 1474 reflections. (2) 2,5,8,11-Tetraoxadodecane–strontium thiocyanate dihydrate, $[Sr(SCN)_2(C_8H_{18}O_4)].2H_2O$, $M_r = 418{\cdot}04$, monoclinic, $P2_1/c$, $a = 7{\cdot}608\,(6)$, $b = 13{\cdot}612\,(14)$, $c = 17{\cdot}393\,(39)$ Å, $\beta = 93{\cdot}46\,(14)°$, $V = 1798\,(5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}54$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 33{\cdot}79$ cm^{-1}, $F(000) = 856$, $T = 291$ K, $R = 0{\cdot}058$ for 1339 reflections. The two triglyme complexes are very different. In the Ca^{2+} complex, the triglyme chain is equatorially coordinated in the familiar, loosely coiled conformation characterized by $ag^{\pm}a$ units with CC *gauche* bonds of alternating sign. In the Sr^{2+} complex, the chain is tightly folded so as to coordinate entirely from one side. The conformation of the triglyme chain contains two $ag^{\pm}g^{\pm}$ units, and all *gauche* bonds are of the same sign.

Introduction. The complexation of a series of oligoethylene glycol dimethyl ethers (tri-, tetra-, penta-, hexa- and heptaglyme) with alkali- and alkaline-earth metal thiocyanates has been studied by ^{13}C NMR spectroscopy (Dale, Krane & Thomassen, 1987). We have undertaken to study the X-ray structures of these complexes in order to compare their conformation in the solid state with that observed in solution. In the first paper we reported the structures of 2,5,8,11,14-pentaoxapentadecane ('tetraglyme') complexed with Ca, Sr and Ba thiocyanates (Wei, Tinant, Declercq, Van Meerssche & Dale, 1987*a*). Here we report the structures of the complexes of triglyme with calcium thiocyanate monohydrate (1) and strontium thiocyanate dihydrate (2).

Experimental. Crystals obtained by slow evaporation from (1) acetone, (2) ethyl acetate–acetone 1:1. Crystal sizes: (1) $0{\cdot}15 \times 0{\cdot}15 \times 0{\cdot}20$ mm, (2) $0{\cdot}06 \times 0{\cdot}08 \times 0{\cdot}20$ mm. Lattice parameters refined using 15 reflections in the range $5 \le 2\theta \le 20°$. Syntex $P2_1$ diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, ω scan, scan width 1·3°, scan rate 1·3–30° min^{-1} $[(\sin\theta)/\lambda]_{\max} = 0{\cdot}561$ Å^{-1}. For (1) 2570 $h\ k\ \pm l$ independent reflections measured ($0 \le h \le 16$, $0 \le k \le 18$, $-8 \le l \le 8$), 1474 observed [criterion $I \ge 2{\cdot}5\sigma(I)$]. For (2) 2671 $h\ k\ \pm l$ measured ($0 \le h \le 8$, $0 \le k \le 15$, $-18 \le l \le 18$), 1339 observed. Standard reflections: one standard reflection [202 for (1), $21\bar{2}$ for (2)] checked every 50 reflections for each measurement: no significant deviation. Absorption correction: none for (1), numerical corrections for crystal defined by its faces for (2) using *SHELX76* (Sheldrick, 1976). Max./min. transmission: 0·829 and 0·757.

Structure solution: in both structures the position of the cation was found from an $E \times F$ Patterson map (*SHELX84*; Sheldrick, 1984) and the positions of the remaining non-H atoms from subsequent difference Fourier synthesis. Anisotropic least-squares refinement with *SHELX76* (Sheldrick, 1976) on F. Hydrogen atoms [except H of the water molecule in (1) located

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from a difference Fourier synthesis] included in the refinement in idealized positions (C—H = 1·08 Å, H—C—H = 109·4°). 188 for (1) and 191 for (2) LS parameters. Weighting scheme $w = [\sigma^2(F)+gF^2]^{-1}$ with $g = 0{\cdot}0001$ for (1) and 0·0007 for (2). (1) $R = 0{\cdot}048$, $wR = 0{\cdot}041$ for 1474 observed reflections. (2) $R = 0{\cdot}058$, $wR = 0{\cdot}052$ for 1339 observed reflections. $(\Delta/\sigma)_{\max}$ in final refinement cycle: (1) 0·75 [U_{22} of atom C(7)]; (2) 0·40 (U_{33} of atom Sr). $S = 1{\cdot}44$ (1) and 1·23 (2). Max. and min. heights in final Fourier synthesis (1) 0·34, −0·37, (2) 0·68 −0·68 e Å^{-3}; largest peak close to the cation in both structures. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Final atomic parameters for the two structures are given in Tables 1 and 2.* A comparison of bond lengths and coordination distances is presented in Table 3 while bond and torsion angles are listed in Table 4.

Table 1. *Atomic coordinates* (×10^4) *and equivalent isotropic temperature factors* (Å^2) *for* (1)

$$B_{\rm eq} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
Ca	3070 (1)	3689 (1)	1942 (2)	3·11 (2)
S(1)	4145 (1)	767 (1)	694 (3)	5·35 (4)
S(2)	6319 (1)	3432 (1)	4594 (2)	4·54 (4)
C(1)	4874 (4)	3903 (4)	−871 (9)	5·6 (2)
C(2)	3538 (4)	4505 (4)	−2148 (8)	5·4 (2)
C(3)	2658 (5)	4874 (4)	−1654 (9)	5·1 (2)
C(4)	1258 (5)	4508 (4)	−368 (10)	5·9 (2)
C(5)	836 (4)	3840 (4)	675 (11)	5·9 (2)
C(6)	1074 (5)	3105 (5)	3538 (12)	6·8 (2)
C(7)	1659 (7)	3002 (7)	5038 (12)	11·1 (3)
C(8)	3103 (7)	2292 (5)	5487 (11)	9·7 (3)
C(9)	3743 (4)	1706 (4)	770 (8)	3·6 (1)
C(10)	5245 (4)	3642 (4)	4061 (7)	3·7 (1)
N(1)	3473 (4)	2359 (3)	839 (7)	4·9 (1)
N(2)	4495 (4)	3783 (4)	3666 (7)	5·2 (1)
O(1)	3993 (3)	4216 (2)	−501 (5)	4·1 (1)
O(2)	2158 (3)	4257 (2)	−719 (5)	4·2 (1)
O(3)	1428 (3)	3658 (3)	2250 (6)	5·1 (1)
O(4)	2603 (3)	2920 (3)	4633 (6)	6·0 (1)
O(5)	2802 (3)	5002 (3)	3169 (7)	4·9 (1)

Table 2. *Atomic coordinates* (×10^4) *and equivalent isotropic temperature factors* (Å^2) *for* (2)

$$B_{\rm eq} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
Sr	2940 (2)	2619 (1)	3525 (1)	2·16 (2)
S(1)	436 (5)	6228 (3)	3931 (2)	4·00 (8)
S(2)	−7 (5)	4333 (3)	976 (2)	4·31 (9)
N(1)	1848 (16)	4326 (9)	3950 (7)	4·3 (3)
N(2)	1731 (16)	3336 (9)	2204 (7)	4·1 (3)
O(1)	5831 (11)	3665 (7)	3337 (5)	3·9 (2)
O(2)	5287 (12)	1888 (7)	2602 (5)	3·5 (2)
O(3)	4657 (11)	967 (6)	3969 (5)	3·0 (2)
O(4)	4677 (11)	2613 (7)	4878 (4)	3·5 (2)
O(5)	540 (11)	2305 (7)	4546 (5)	4·2 (2)
O(6)	1008 (11)	1241 (7)	2926 (5)	4·1 (2)
C(1)	6511 (20)	4360 (12)	3916 (9)	5·9 (4)
C(2)	7192 (17)	3269 (10)	2912 (8)	4·0 (3)
C(3)	6342 (18)	2655 (12)	2287 (8)	4·3 (3)
C(4)	6207 (17)	995 (10)	2786 (8)	3·6 (3)
C(5)	5136 (17)	403 (10)	3314 (7)	3·1 (3)
C(6)	6115 (19)	1141 (11)	4522 (8)	4·1 (3)
C(7)	5416 (20)	1715 (11)	5178 (7)	4·2 (3)
C(8)	4214 (20)	3232 (12)	5491 (8)	5·6 (4)
C(9)	1283 (17)	5108 (11)	3952 (7)	3·0 (3)
C(10)	1027 (17)	3732 (9)	1691 (8)	2·7 (3)

Table 3. *Bond lengths and coordination distances* (Å)

	(1) M = Ca^{2+}	(2) M = Sr^{2+}
O(1)–C(1)	1·428 (6)	1·454 (15)
O(1)–C(2)	1·424 (6)	1·416 (14)
C(2)–C(3)	1·485 (8)	1·487 (18)
C(3)–O(2)	1·437 (7)	1·445 (15)
O(2)–C(4)	1·418 (6)	1·429 (14)
C(4)–C(5)	1·482 (8)	1·499 (16)
C(5)–O(3)	1·435 (7)	1·439 (13)
O(3)–C(6)	1·419 (7)	1·443 (14)
C(6)–C(7)	1·366 (10)	1·506 (17)
C(7)–O(4)	1·437 (9)	1·430 (15)
O(4)–C(8)	1·387 (8)	1·419 (14)
S(1)–C(9)	1·641 (6)	1·655 (15)
S(2)–C(10)	1·640 (6)	1·648 (14)
N(1)–C(9)	1·139 (6)	1·148 (15)
N(2)–C(10)	1·146 (6)	1·147 (15)
$M\cdots$O(1)	2·446 (4)	2·657 (8)
$M\cdots$O(2)	2·479 (4)	2·665 (9)
$M\cdots$O(3)	2·433 (4)	2·690 (8)
$M\cdots$O(4)	2·455 (4)	2·628 (8)
$M\cdots$N(1)	2·397 (5)	2·590 (12)
$M\cdots$N(2)	2·389 (5)	2·613 (12)
$M\cdots$O(5)	2·360 (5)	2·657 (8)
$M\cdots$O(6)	—	2·565 (9)

Table 4. *Bond angles* (°) *and torsion angles* (°) [$\sigma = 1°$ *for* (1) *and* 3° *for* (2)] *in the polyether chain*

	(1)	(2)
C(1)–O(1)–C(2)	110·5 (4)	111·7 (10)
O(1)–C(2)–C(3)	107·9 (5)	107·2 (10)
C(2)–C(3)–O(2)	107·5 (5)	110·9 (11)
C(3)–O(2)–C(4)	112·7 (5)	115·0 (10)
O(2)–C(4)–C(5)	107·3 (5)	108·5 (10)
C(4)–C(5)–O(3)	108·0 (5)	111·8 (11)
C(5)–O(3)–C(6)	115·7 (5)	113·2 (10)
O(3)–C(6)–C(7)	111·8 (7)	107·3 (11)
C(6)–C(7)–O(4)	114·9 (7)	108·4 (10)
C(7)–O(4)–C(8)	118·0 (6)	110·0 (10)
N(1)–C(9)–S(1)	179·1 (6)	178·3 (14)
N(2)–C(10)–S(2)	179·0 (6)	177·9 (13)
C(1)–O(1)–C(2)–C(3)	−176	−174
O(1)–C(2)–C(3)–O(2)	−57	−58
C(2)–C(3)–O(2)–C(4)	−173	−88
C(3)–O(2)–C(4)–C(5)	−177	163
O(2)–C(4)–C(5)–O(3)	54	−52
C(4)–C(5)–O(3)–C(6)	172	−73
C(5)–O(3)–C(6)–C(7)	−177	−179
O(3)–C(6)–C(7)–O(4)	−42	−59
C(6)–C(7)–O(4)–C(8)	−133	−172

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters, more complete lists of bond lengths and angles, and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43743 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

As can be seen from the stereoscopic view (*PLUTO*; Motherwell & Clegg, 1978) of each compound (Figs. 1 and 2), the two complexes are very different. In (1) the Ca^{2+} cation is sevenfold coordinated with an approximate pentagonal-bipyramid geometry, the four O atoms of the triglyme chain and one thiocyanate anion, coordinated through the N atom, being in equatorial positions; the other anion and one water molecule are in apical positions. The wrapping mode of the polyether chain is similar to that observed in the complexes of tetraglyme with Ca, Sr and Ba thiocyanates (Wei *et al.*, 1987*a*) and with HgCl$_2$ (Iwamoto, 1973). The ether O atoms and the Ca^{2+} cation are coplanar within ~0·1 Å.* The four O atoms are located at adjacent corners of a nearly regular hexagon, two adjacent corners being empty and the cation being located at the centre. The torsion angles (Table 4) in the triglyme chain along O(1) to O(4) indicate the sequence $ag^{\mp}a$ $ag^{\pm}a$ $ag^{\mp}a$, *i.e.* with alternating signs. This sequence is typical for cyclic polyethers when equatorially coordinated to a fitting cation (Dale, 1980). The coordination distances are shorter than those observed in the tetraglyme calcium thiocyanate complex: the mean values of Ca···O(glyme) and Ca···N are 2·453 (5) and 2·393 (5) Å in (1) compared with 2·486 (3) and 2·466 (5) Å respectively in the tetraglyme complex. This may be a consequence of the different coordination number, seven for the present complex against eight for the tetraglyme complex (Shannon, 1976).

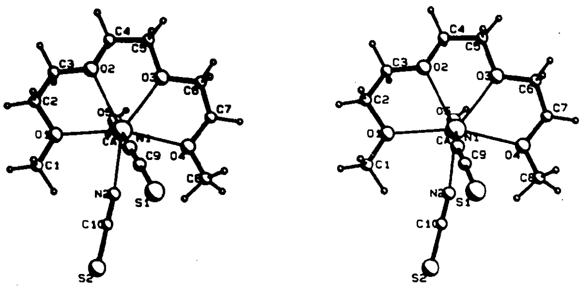

Fig. 1. Stereoscopic view of the complex (1) with atom numbering.

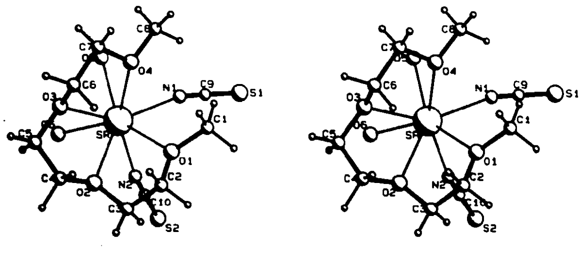

Fig. 2. Stereoscopic view of the complex (2) with atom numbering.

The structure of the Sr^{2+} complex (2) is more unusual. The coordination around the Sr^{2+} cation is eightfold and has an approximately square antiprismatic geometry with the triglyme chain tightly folded to offer its four oxygens in a square arrangement from one side, while the thiocyanate anions and both water molecules coordinate in a second square arrangement from the opposite side. More specifically, the four O atoms of the triglyme chain are coplanar within 0·08 Å, with the Sr^{2+} cation 1·717 (5) Å out of this plane.* The four other coordinated atoms, N(1), N(2) of the anions and O(5), O(6) of the water molecules, lie in a second plane parallel to the first one and with the Sr^{2+} cation 1·289 (5) Å out of it (maximum deviation from the mean plane = 0·12 Å). The angle between the two mean planes is only 2°.

The structure of this Sr^{2+} complex (2) bears a striking resemblance to the structures of the complexes of Sr(SCN)$_2$.2H$_2$O and Ca(SCN)$_2$.2H$_2$O with 12-crown-4 (Wei, Tinant, Declercq, Van Meerssche & Dale, 1987*b*), and also to that of the complex between CaCl$_2$.8H$_2$O and 12-crown-4 (North, Steiner, van Reemoortere & Boer, 1976). In all three cases, the four oxygens of the ring form the coordination square on one side. Actually, the tightly coiled conformation of the triglyme chain can be derived directly from the cyclic conformation $ag^{\pm}g^{\pm}$ $ag^{\pm}g^{\pm}$ $ag^{\pm}g^{\pm}$ $ag^{\pm}g^{\pm}$ of the crown ether by cutting it open at one CC bond and turning the 'side CH$_2$' (now CH$_3$) out of the ring, leaving the 'corner CH$_2$' (now CH$_3$) in position. The resulting sequence of torsion angles (Table 4) $ag^{\pm}g^{\pm}$ $ag^{\pm}g^{\pm}$ $ag^{\pm}a$ thus retains two genuine corners $g^{\pm}g^{\pm}$ (Dale, 1980), and all *gauche* angles are of the same sign.

Such genuine corners are relatively frequent in crown-ether complexes, but more rare in open-chain ligands. In the 'type II' HgCl$_2$ complex of poly(ethylene oxide), $ag^{\pm}g^{\pm}$ units alternate in sign, resulting in a zigzag conformation (Yokoyama, Ishihara, Iwamoto & Tadokoro, 1969). In the RbI complex of tetraethyleneglycol bis(8-oxyquinoline)ether there is only one such unit at one end (Saenger, Brand, Vögtle & Weber, 1977), and this is also the case in the Sr(SCN)$_2$ complex of hexaethylene glycol (Yamaguchi, Miki, Yasuoka & Kasai, 1982) as well as the Ba(SCN)$_2$ complex of hexaglyme (Weber, Hirayama, Saenger & Sheldrick, 1984). In the Sr(SCN)$_2$ complex with the longer heptaethylene glycol chain, there is an additional sequence of two $ag^{\pm}g^{\pm}$ units of the same sign at the other end, producing a coordination square of the same type as observed in the present work (Ohmoto,

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* See deposition footnote.

* See deposition footnote.

Kai, Yasuoka, Kasai, Yanagida & Okakara, 1979). Finally, there is an unusual dimeric complex between triglyme and $Co^{II}Cl.SbCl_6$, where it is the middle unit which forms a genuine corner (Kinneging, Vermin & Gorter, 1982).

In all observed cases, cyclic or acyclic, the *gauche* CO bond has a greater value of the torsion angle than the adjacent *gauche* CC bond of the same unit. This is also observed for the present Sr^{2+} complex (2); 88 (3)° and 73 (3)° for the CO bonds compared with the more normal values of 58 (3)° and 52 (3)° for the CC bonds (Table 4).

There are no obvious linkages between the complex units in any of the two structures.

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References

Dale, J. (1980). *Isr. J. Chem.* **20**, 3–11.

Dale, J. Krane, J. & Thomassen, T. (1987). In preparation.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press (Present distributor D. Reidel, Dordrecht.)

Iwamoto, R. (1973). *Bull. Chem. Soc. Jpn*, **46**, 1114–1127.

Kinneging, A. J., Vermin, W. J. & Gorter, S. (1982). *Acta Cryst.* B**38**, 1824–1826.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

North, P. P., Steiner, E. C., van Remoortere, F. P. & Boer, F. P. (1976). *Acta Cryst.* B**32**, 370–376.

Ohmoto, H., Kai, Y., Yasuoka, N., Kasai, N., Yanagida, S. & Okakara, M. (1979). *Bull. Chem. Soc. Jpn*, **52**, 1209–1210.

Saenger, W., Brand, H., Vögtle, F. & Weber, W. (1977). *Metal/Ligand Interactions in Organic Chemistry and Biochemistry*, Part 1, edited by B. Pullman & N. Goldblum, p. 363. Dordrecht: Reidel.

Shanon, R. D. (1976). *Acta Cryst.* A**32**, 751–767.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1984). *SHELX84*. Personal communication.

Weber, G., Hirayama, F., Saenger, W. & Sheldrick, G. M. (1984). *Acta Cryst.* C**40**, 1570–1572.

Wei, Y. Y., Tinant, B., Declercq, J.-P., Van Meerssche, M. & Dale, J. (1987*a*). *Acta Cryst.* C**43**, 1076–1080.

Wei, Y. Y., Tinant, B., Declercq, J.-P., Van Meerssche, M. & Dale, J. (1987*b*). *Acta Cryst.* In the press.

Yamaguchi, I., Miki, K., Yasuoka, N. & Kasai, N. (1982). *Bull. Chem. Soc. Jpn*, **55**, 1372–1375.

Yokoyama, M. Ishihara, H., Iwamoto, R. & Tadokoro, H. (1969). *Macromolecules*, **2**, 184–192.

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Structures of Tetrakis(benzylamine)diisothiocyanatonickel(II) and Tris(benzylamine)-diisothiocyanato(1-phenyl-1-ethylamine)nickel(II)

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Abstract. (1): $[Ni(NCS)_2(C_7H_9N)_4]$, $M_r = 603\cdot47$, monoclinic, $P2_1/n$, $a = 10\cdot919$ (2), $b = 7\cdot835$ (1), $c = 35\cdot72$ (1) Å, $\beta = 93\cdot1$ (2)°, $U = 3051$ (1) Å^3, $Z = 4$, $D_x = 1\cdot314$, $D_m = 1\cdot32$ Mg m^{-3}, μ(Mo $K\alpha$) = $0\cdot75$ mm^{-1}, $F(000) = 1272$, $T = 294$ K, final $R = 0\cdot057$ ($wR = 0\cdot052$) for 2806 independent reflections; (2): $\frac{1}{2}\{[Ni(NCS)_2(C_7H_9N)_3(R)\text{-}(C_8H_{11}N)].[Ni(NCS)_2(C_7H_9N)_3(S)\text{-}(C_8H_{11}N)]\}$, $M_r = 617\cdot49$, monoclinic, $P2_1/c$, $a = 7\cdot710$ (3), $b = 14\cdot535$ (8), $c = 28\cdot75$ (1) Å, $\beta = 88\cdot52$ (3)°, $U = 3221$ (2) Å^3, $Z = 4$, $D_x = 1\cdot273$, $D_m = 1\cdot26$ Mg m^{-3}, μ(Mo $K\alpha$) = $0\cdot71$ mm^{-1}, $F(000) = 1304$, $T = 294$ K, final $R = 0\cdot085$ ($wR = 0\cdot076$) for 2735 independent reflections; (3): $[Ni(NCS)_2(C_7H_9N)_4(S)\text{-}(C_8H_{11}N)]$, $M_r = 617\cdot49$, orthorhombic, $P2_12_12$, $a = 7\cdot66$ (2), $b = 14\cdot660$ (4), $c = 29\cdot009$ (9) Å, $U = 3258$ (9) Å^3, $Z = 4$, $D_x = 1\cdot259$, $D_m = 1\cdot26$ Mg m^{-3}, μ(Mo $K\alpha$) = $0\cdot70$ mm^{-1}, $F(000) = 1304$, $T = 294$ K, final $R = 0\cdot104$ ($wR = 0\cdot077$) for 1342 independent reflections. λ(Mo $K\alpha$) = $0\cdot71069$ Å. The three structures consist of distorted octahedral nickel complexes with NiN_6 coordination spheres, the –NCS groups in *trans* positions in all three structures. The complex in (1) has approximate mirror symmetry; the conformations of enantiomerically equivalent molecules from (2) and (3) are similar.

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