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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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$_7$H$_9$N)$_4$], $M_r = 603{\cdot}47$, monoclinic, $P2_1/n$, $a = 10{\cdot}919\ (2)$, $b = 7{\cdot}835\ (1)$, $c = 35{\cdot}72\ (1)$ Å, $\beta = 93{\cdot}1\ (2)°$, $U = 3051\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}314$, $D_m = 1{\cdot}32$ Mg m^{-3}, μ(Mo $K\alpha$) = 0·75 mm^{-1}, $F(000) = 1272$, $T = 294$ K, final $R = 0{\cdot}057$ ($wR = 0{\cdot}052$) for 2806 independent reflections; (2): $\frac{1}{2}${[Ni(NCS)$_2$(C$_7$H$_9$N)$_3$(*R*)-(C$_8$H$_{11}$N)].[Ni(NCS)$_2$(C$_7$H$_9$N)$_3$(*S*)-(C$_8$H$_{11}$N)]}, $M_r = 617{\cdot}49$, monoclinic, $P2_1/c$, $a = 7{\cdot}710\ (3)$, $b = 14{\cdot}535\ (8)$, $c = 28{\cdot}75\ (1)$ Å, $\beta = 88{\cdot}52\ (3)°$, $U = 3221\ (2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}273$, $D_m = 1{\cdot}26$ Mg m^{-3}, μ(Mo $K\alpha$) = 0·71 mm^{-1}, $F(000) = 1304$, $T = 294$ K, final $R = 0{\cdot}085$ ($wR = 0{\cdot}076$) for 2735 independent reflections; (3): [Ni(NCS)$_2$(C$_7$H$_9$N)$_4$(*S*)-(C$_8$H$_{11}$N)], $M_r = 617{\cdot}49$, orthorhombic, $P2_12_12$, $a = 7{\cdot}66\ (2)$, $b = 14{\cdot}660\ (4)$, $c = 29{\cdot}009\ (9)$ Å, $U = 3258\ (9)$ Å^3, $Z = 4$, $D_x = 1{\cdot}259$, $D_m = 1{\cdot}26$ Mg m^{-3}, μ(Mo $K\alpha$) = 0·70 mm^{-1}, $F(000) = 1304$, $T = 294$ K, final $R = 0{\cdot}104$ ($wR = 0{\cdot}077$) for 1342 independent reflections. λ(Mo $K\alpha$) = 0·71069 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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Introduction. The formation of inclusion compounds of the type $[Ni(NCS)_2(\alpha\text{-arylalkylamine})_4]$ (host) with aromatic species (guest) was reviewed recently (Hanotier & de Radzitzky, 1984). Using 1-phenyl-1-ethylamine as the α-arylalkylamine ligand, clathrates of the tetraamine Ni^{II} isothiocyanates were found to form only from the racemic amine. The first complete crystal structures of the host complexes obtained from enantiomerically pure and racemic 1-phenyl-1-ethylamine (Nassimbeni, Niven & Zemke, 1985) showed that the coordinated —NCS groups were *cis* to each other. In the clathrates with *sec*-butylbenzene and *o*-xylene, however, the —NCS groups of the nickel complex were *trans* to each other.

Chiral discrimination by enclathration using such tetraamine nickel(II) isothiocyanates has been envisaged, making use of chiral α-arylalkylamine ligands. The nature of the Ni^{II} coordination and the possibility of preparing mixed-ligand complexes to enhance host–complex chirality are presently being investigated, and we report here the structure of $Ni^{II}NCS_2(\text{benzylamine})_4$, (1), and those obtained from racemic (2) and enantiomerically pure (3) 1-phenyl-1-ethylamine of the form $Ni^{II}(NCS)_2(\text{benzylamine})_3(\text{1-phenyl-1-ethylamine})$. These complexes are variations of the host molecules previously used to enclathrate aromatic guest species (Hanotier & de Radzitzky, 1984).

Experimental. (1) was prepared according to the previously reported method (de Radzitzky & Hanotier, 1962) using benzylamine instead of the α-arylalkylamine, and crystal grown from methanol. (2) and (3) were prepared by dissolving 1·43 g $NiCl_2.6H_2O$ (0·006 mol) and 1·17 g KSCN (0·012 mol) in 6 ml water, and adding dropwise with stirring 1·28 g $NH_2CH_2C_6H_5$ (0·012 mol) dissolved in 10 ml *n*-hexane. When the mixture had taken on a uniform appearance, 1·45 g 1-phenyl-1-ethylamine (0·012 mol) [for (2): racemic mixture; for (3): enantiomerically pure (*S*)-(−) isomer (Aldrich)] dissolved in 10 ml *n*-hexane were added dropwise with stirring. The blue precipitate was washed twice with *n*-hexane and dried, crystals obtained from methanol. All crystal densities were measured in linear density columns containing water and KI solution (Oster & Yamamoto, 1963).

Intensity data collection: CAD-4 diffractometer, graphite-monochromated Mo *Kα* radiation; cell dimensions from 2θ angles for 24 reflections with $16 \leq 2\theta \leq 17°$ for (1), 25 reflections with $16 \leq 2\theta \leq 17°$ for (2), 24 reflections with $9 \leq 2\theta \leq 10°$ for (3); ω–2θ scans, final acceptance limit 20σ at 20° min^{-1} in ω and maximum recording time 40 s; intensities of three standard reflections checked every hour and recentring every 100 measured reflections. Intensities corrected for Lorentz and polarization factors [(1), (2) and (3)]; corrections for absorption [(1) and (2)] applied empirically on basis of ψ scans of three low-angle reflections. Structures solved by heavy-atom method and subsequent difference Fourier synthesis, with LS refinement of *F* magnitudes using *SHELX*76 (Sheldrick, 1978); owing to large number of atoms final refinement with only Ni and S atoms anisotropic, all other isotropic. Amino H atoms located only in difference Fourier map of (1), refined with constrained N—H bond lengths = 1·00 (3) Å and independent $U_{iso}(H_i)$. All other H atoms in calculated positions (C—H = 1·00 Å), for (1) and (2) chemically equivalent H atoms assigned common $U_{iso}(H_i)$, for (3) all $U_{iso}(H_i)$

Table 1. *Experimental data and structure refinement parameters*

	(1)	(2)	(3)
Data collection			
Crystal dimensions (mm)	0·33 × 0·20 × 0·13	0·28 × 0·23 × 0·10	0·40 × 0·13 × 0·10
Range scanned (θ) (°)	1–25	1–25	1–18
Range of indices *h,k,l*	−13→13, 0→9, 0→42	−9→9, 0→17, 0→34	−6→6, 0→12, 0→25
Transmission factors for experimental absorption correction based on ψ scan	0·967–0·999	0·945–0·999	(Lp absorption correction only)
Standard reflections (*hkl*)	$\bar{1}63, \bar{5}54, \bar{6},\bar{1},21$	$\bar{3},4,19, 2,9,12, 3,6,16$	$1,2,12, 0,\bar{3},12, 247$
and stability (%)	1·5	1·3	4·0
Scan width in ω (°)	(0·44 + 0·35tanθ)	(1·04 + 0·35tanθ)	(0·74 + 0·35tanθ)
Vertical aperture length (mm)	3	4	4
Aperture width (mm)	(1·10 + 1·05tanθ)	(1·36 + 1·05tanθ)	(1·27 + 1·05tanθ)
Number of reflections measured	5871	6031	2452
Number of unique reflections	4138	4336	1827
R_{int}	0·030	0·033	0·124
Criterion for recognizing unobserved reflections	$F_o < 3\sigma(F)[4\sigma(F)]$*	$F_o < 3\sigma(F)\ [4\sigma(F)]$*	$F_o < 1{\cdot}5\sigma(F)$
Number of unobserved reflections	1332 (1422)*	1601 (1726)*	485
Final refinement			
Maximum LS shift to e.s.d.	0·04	0·009	0·16
Maximum and minimum height in final difference Fourier synthesis (e $Å^{-3}$)	0·48; −0·41	0·64; −0·55	0·67; −0·71
Number of parameters	199	184	179
R	0·057 (0·054)*	0·085 (0·081)*	0·104
wR	0·052 (0·051)*	0·076 (0·074)*	0·077
w	$(\sigma^2F)^{-1}$	$(\sigma^2F)^{-1}$	$(\sigma^2F)^{-1}$
S	(2·38)*	(4·24)*	1·82

* For (1) and (2) the values appearing in parentheses relate to the LS refinement which was based on observed reflections with $F_o > 4\sigma(F)$. For publication purposes *R* and *wR* values were calculated also for all reflections with $F_o > 3\sigma(F)$.

Table 2. *Fractional atomic coordinates* ($\times 10^4$) *and isotropic or equivalent isotropic thermal parameters* ($Å^2 \times 10^3$)

	x	y	z	U_{iso}/U_{eq}
Structure (1)				
Ni(1)	429 (1)	711 (1)	1576·8 (2)	30·9 (2)*
N(1)	−260 (4)	2745 (6)	1280 (1)	34 (1)
C(1)	−628 (5)	4010 (8)	1150 (2)	33 (2)
S(1)	−1156 (2)	5777 (3)	967 (1)	62 (1)*
N(2)	1165 (4)	−1321 (7)	1891 (1)	38 (1)
C(2)	1610 (5)	−2364 (8)	2085 (2)	34 (2)
S(2)	2226 (1)	−3857 (2)	2355·9 (4)	42 (1)*
N(3)	−4 (5)	1838 (7)	2107 (1)	36 (1)
C(31)	−542 (5)	3554 (8)	2148 (2)	40 (2)
C(32)	−1918 (5)	3554 (8)	2148 (2)	32 (2)
C(33)	−2492 (6)	2774 (8)	2441 (2)	36 (2)
C(34)	−3763 (6)	2814 (8)	2457 (2)	44 (2)
C(35)	−4461 (6)	3641 (9)	2183 (2)	52 (2)
C(36)	−3909 (6)	4410 (9)	1891 (2)	49 (2)
C(37)	−2635 (5)	4371 (9)	1872 (2)	42 (2)
N(4)	−1319 (5)	−565 (7)	1550 (1)	39 (1)
C(41)	−2348 (6)	177 (9)	1325 (2)	46 (2)
C(42)	−3577 (5)	−696 (9)	1362 (2)	40 (2)
C(43)	−4187 (6)	−544 (9)	1689 (2)	48 (2)
C(44)	−5339 (6)	−1304 (9)	1722 (2)	57 (2)
C(45)	−5845 (7)	−2188 (10)	1424 (2)	59 (2)
C(46)	−5280 (7)	−2381 (9)	1102 (2)	59 (2)
C(47)	−4112 (6)	−1622 (9)	1065 (2)	50 (2)
N(5)	964 (5)	−613 (8)	1090 (2)	49 (1)
C(51)	557 (6)	−20 (10)	719 (2)	58 (2)
C(52)	1129 (6)	−930 (9)	393 (2)	51 (2)
C(53)	470 (7)	−2109 (9)	183 (2)	56 (2)
C(54)	979 (7)	−2864 (10)	−129 (2)	61 (2)
C(55)	2108 (7)	−2435 (10)	−228 (2)	66 (2)
C(56)	2788 (8)	−1260 (11)	−20 (2)	79 (3)
C(57)	2305 (7)	−511 (11)	299 (2)	70 (2)
N(6)	2230 (5)	1876 (7)	1645 (1)	40 (1)
C(61)	2527 (6)	3579 (8)	1488 (2)	45 (2)
C(62)	3033 (6)	3487 (8)	1108 (2)	41 (2)
C(63)	2453 (6)	4214 (10)	795 (2)	57 (2)
C(64)	2987 (7)	4224 (11)	452 (2)	73 (2)
C(65)	4110 (8)	3473 (11)	416 (2)	79 (3)
C(66)	4715 (8)	2706 (11)	719 (2)	74 (3)
C(67)	4177 (6)	2719 (9)	1068 (2)	53 (2)
Structure (2)				
Ni(1)	7516 (2)	−701 (1)	2358 (1)	38 (1)*
N(1)	9704 (12)	−1483 (7)	2380 (3)	55 (3)
C(1)	10911 (14)	−1920 (7)	2472 (4)	41 (3)
S(1)	12602 (4)	−2528 (2)	2608 (1)	58 (1)*
N(2)	5353 (11)	106 (6)	2327 (3)	48 (3)
C(2)	4212 (13)	596 (7)	2261 (4)	41 (3)
S(2)	2587 (4)	1289 (2)	2159 (1)	64 (1)*
N(3)	6289 (10)	−1879 (6)	2041 (3)	41 (2)
C(31)	6496 (15)	−2155 (7)	1553 (4)	45 (3)
C(32)	5846 (16)	−3186 (8)	1506 (4)	66 (4)
C(33)	5503 (13)	−1571 (7)	1208 (4)	41 (3)
C(34)	4115 (14)	−1029 (8)	1360 (4)	57 (4)
C(35)	3212 (15)	−504 (8)	1025 (4)	61 (4)
C(36)	3714 (16)	−567 (9)	561 (5)	75 (4)
C(37)	5087 (17)	−1105 (9)	415 (5)	80 (4)
C(38)	6025 (16)	−1603 (8)	751 (4)	63 (4)
N(4)	8676 (11)	−135 (6)	1737 (3)	45 (3)
C(41)	7798 (13)	594 (8)	1477 (4)	54 (3)
C(42)	8878 (13)	1039 (7)	1096 (4)	44 (3)
C(43)	9936 (15)	1785 (8)	1212 (4)	61 (4)
C(44)	10995 (17)	2181 (9)	855 (5)	71 (4)
C(45)	10976 (16)	1848 (9)	413 (5)	69 (4)
C(46)	9886 (16)	1134 (9)	287 (5)	71 (4)
C(47)	8883 (14)	755 (8)	642 (4)	58 (3)
N(5)	6596 (10)	−1287 (6)	3019 (3)	42 (2)
C(51)	5328 (14)	−783 (9)	3297 (4)	62 (3)
C(52)	5166 (14)	−1060 (8)	3793 (4)	48 (3)
C(53)	4165 (17)	−1824 (9)	3928 (5)	75 (4)
C(54)	4011 (18)	−2076 (10)	4393 (5)	85 (5)
C(55)	4813 (18)	−1627 (10)	4737 (5)	83 (5)
C(56)	5796 (16)	−869 (10)	4610 (5)	80 (4)
C(57)	5951 (15)	−590 (9)	4144 (4)	66 (4)
N(6)	8736 (11)	356 (6)	2773 (3)	51 (3)
C(61)	10068 (16)	92 (9)	3114 (4)	71 (4)
C(62)	9939 (15)	587 (9)	3572 (4)	63 (4)
C(63)	9243 (15)	1446 (9)	3622 (5)	73 (4)
C(64)	9106 (20)	1875 (12)	4068 (6)	107 (6)
C(65)	9743 (20)	1380 (12)	4455 (6)	114 (6)
C(66)	10472 (19)	562 (12)	4394 (6)	106 (6)
C(67)	10593 (17)	163 (10)	3950 (5)	87 (5)

Table 2 (*cont.*)

	x	y	z	U_{iso}/U_{eq}
Structure (3)				
Ni(1)	2479 (8)	1937 (2)	7333 (1)	57 (1)*
N(1)	332 (39)	2759 (18)	7376 (9)	99 (10)
C(1)	−830 (42)	3127 (22)	7456 (9)	60 (10)
S(1)	−2518 (17)	3748 (4)	7596 (3)	88 (4)*
N(2)	4601 (31)	1080 (16)	7295 (8)	67 (8)
C(2)	5763 (37)	630 (18)	7248 (9)	53 (9)
S(2)	7525 (18)	−74 (5)	7182 (2)	86 (4)*
N(3)	3416 (26)	2574 (16)	7950 (7)	70 (9)
C(31)	4538 (43)	1995 (26)	8220 (11)	116 (12)
C(32)	4879 (38)	2268 (23)	8722 (11)	74 (11)
C(33)	4206 (45)	1746 (24)	9079 (13)	102 (13)
C(34)	4455 (43)	2054 (27)	9542 (12)	96 (12)
C(35)	5257 (45)	2795 (26)	9647 (12)	98 (13)
C(36)	5835 (51)	3351 (29)	9311 (17)	141 (17)
C(37)	5568 (52)	3088 (30)	8866 (15)	137 (15)
N(4)	3769 (27)	3083 (16)	7034 (8)	57 (7)
C(41)	3571 (45)	3404 (20)	6557 (12)	98 (14)
C(42)	4326 (42)	4390 (20)	6478 (10)	95 (12)
C(43)	4562 (33)	2825 (19)	6193 (9)	44 (9)
C(44)	4130 (44)	2880 (22)	5726 (10)	81 (11)
C(45)	4964 (47)	2417 (24)	5385 (12)	112 (14)
C(46)	6296 (43)	1810 (23)	5504 (12)	98 (12)
C(47)	6757 (42)	1730 (20)	5978 (11)	104 (14)
C(48)	5850 (40)	2258 (19)	6310 (10)	66 (10)
N(5)	1404 (26)	1425 (14)	6714 (8)	52 (7)
C(51)	2049 (33)	627 (14)	6482 (7)	46 (9)
C(52)	1211 (38)	242 (23)	6070 (10)	66 (10)
C(53)	−36 (60)	−446 (33)	6130 (16)	177 (19)
C(54)	−1185 (56)	−696 (28)	5739 (15)	154 (18)
C(55)	−920 (47)	−258 (26)	5339 (13)	123 (14)
C(56)	519 (52)	370 (25)	5255 (13)	133 (14)
C(57)	1392 (34)	703 (19)	5659 (10)	60 (10)
N(6)	1276 (24)	883 (13)	7735 (7)	47 (7)
C(61)	−106 (48)	1203 (27)	8081 (13)	141 (15)
C(62)	117 (60)	619 (34)	8533 (17)	160 (19)
C(63)	−809 (62)	1076 (34)	8906 (19)	187 (21)
C(64)	−653 (52)	470 (29)	9358 (16)	153 (17)
C(65)	372 (56)	−362 (32)	9346 (17)	168 (18)
C(66)	998 (50)	−836 (27)	8959 (15)	135 (16)
C(67)	855 (48)	−317 (29)	8562 (13)	112 (13)

* $U_{eq} = \frac{1}{3}$(trace of the orthogonalized U_{ij} matrix).

fixed at $1{\cdot}2U_{iso}(C_i)$. Complex neutral-atom scattering factors for H were taken from Stewart, Davidson & Simpson (1965), for all other atoms from Cromer & Mann (1968), with dispersion corrections from Cromer & Liberman (1970). Molecular parameters were obtained from *PARST* (Nardelli, 1983), drawings from *PLUTO* (Motherwell, 1974). All computations carried out on a Sperry 1100 computer. All further experimental data and structure solution parameters are summarized in Table 1.

Discussion. Atomic parameters are given in Table 2, selected torsion angles in Table 3.* Views of the molecules and atomic numbering are shown in Figs. 1, 2 and 3.

The structures consist of discrete mononuclear molecules with each Ni atom coordinated to six N-donor ligands, the N atoms forming a distorted octahedron. The average Ni—N distances for the three

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* Lists of anisotropic thermal parameters, H-atom parameters, bond distances, bond and torsion angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43771 (55 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Selected torsion angles* (°)

Structure (1)			
N(1)—Ni(1)—N(3)—C(31)	−3·0 (7)	N(1)—Ni(1)—N(5)—C(51)	−10·9 (7)
Ni(1)—N(3)—C(31)—C(32)	92·9 (7)	Ni(1)—N(5)—C(51)—C(52)	172·5 (6)
N(3)—C(31)—C(32)—C(33)	65·9 (10)	N(5)—C(51)—C(52)—C(57)	−78·4 (10)
N(1)—Ni(1)—N(4)—C(41)	5·6 (6)	N(1)—Ni(1)—N(6)—C(61)	−0·4 (6)
Ni(1)—N(4)—C(41)—C(42)	−172·8 (5)	Ni(1)—N(6)—C(61)—C(62)	−92·8 (7)
N(4)—C(41)—C(42)—C(43)	70·9 (10)	N(6)—C(61)—C(62)—C(67)	−65·9 (10)
Structure (2)			
N(1)—Ni(1)—N(3)—C(31)	−78·6 (9)	N(1)—Ni(1)—N(5)—C(51)	158·0 (8)
Ni(1)—N(3)—C(31)—C(32)	163·6 (7)	Ni(1)—N(5)—C(51)—C(52)	−161·9 (8)
Ni(1)—N(3)—C(31)—C(33)	−75·7 (11)	N(5)—C(51)—C(52)—C(57)	99·0 (14)
N(3)—C(31)—C(33)—C(34)	−19·4 (15)	N(1)—Ni(1)—N(6)—C(61)	−29·8 (8)
N(1)—Ni(1)—N(4)—C(41)	−178·8 (8)	Ni(1)—N(6)—C(61)—C(62)	−138·6 (9)
Ni(1)—N(4)—C(41)—C(42)	170·2 (7)	N(6)—C(61)—C(62)—C(63)	−27·3 (17)
N(4)—C(41)—C(42)—C(43)	−86·8 (13)		
Structure (3)			
N(1)—Ni(1)—N(3)—C(31)	152·1 (21)	N(1)—Ni(1)—N(5)—C(51)	−168·2 (20)
Ni(1)—N(3)—C(31)—C(32)	−163·7 (22)	Ni(1)—N(5)—C(51)—C(52)	175·5 (19)
N(3)—C(31)—C(32)—C(33)	111·1 (36)	N(5)—C(51)—C(52)—C(53)	−92·8 (37)
N(1)—Ni(1)—N(4)—C(41)	−77·5 (24)	N(1)—Ni(1)—N(6)—C(61)	−27·8 (21)
Ni(1)—N(4)—C(41)—C(42)	165·9 (19)	Ni(1)—N(6)—C(61)—C(62)	−140·9 (25)
Ni(1)—N(4)—C(41)—C(43)	−76·6 (30)	N(6)—C(61)—C(62)—C(67)	−26·6 (54)
N(4)—C(41)—C(43)—C(48)	−17·9 (40)		

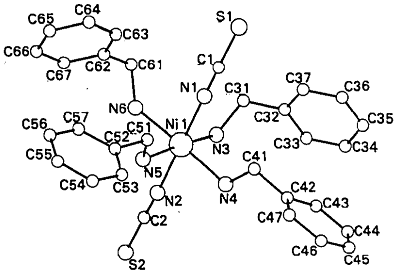

Fig. 1. Perspective view of (1) with atomic nomenclature.

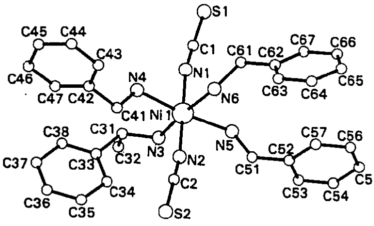

Fig. 2. Perspective view of one molecule from structure (2), containing one (*S*)-1-phenyl-1-ethylamine ligand. The enantiomeric molecule in the same structure is related by a centre of inversion.

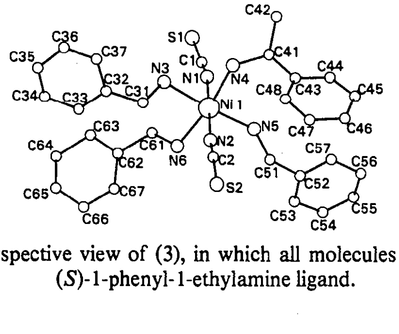

Fig. 3. Perspective view of (3), in which all molecules contain one (*S*)-1-phenyl-1-ethylamine ligand.

structures are 2·046 (6) Å for the nickel–isothiocyanate bonds and 2·151 (17) Å for the nickel–amine bonds (e.s.d.'s in parentheses). These values are in accordance with those obtained by the authors for related nickel complexes (Nassimbeni, Niven & Zemke, 1986), irrespective of *trans* or *cis* orientation of the two —NCS ligands. In the three structures reported here, the —NCS groups are near linear, average N—C—S for six values 178·7 (14)°, and Ni—N—CS angles 172·2 (28)°. The coordinated —NCS groups of the nickel complexes are *trans* to each other in these structures, in contrast to the *cis* coordination in the chemically very similar $[Ni(NCS)_2$(1-phenyl-1-ethylamine)$_4]$ complexes (Nassimbeni *et al.*, 1986).

In (1) all four benzylamine ligands lie on one side of their N—N—N—N plane, shown by the small N(1)—Ni(1)—N(*n*)—C(*n*1), (*n* = 3–6) torsion angles (Table 3). The two amine ligands with N(3) and N(6) have conformation-governing torsion angles equal but opposite in sign and are *cis* to each other. The conformations of the other two amine ligands [with N(4) and N(5)] are similarly related. The molecule thus has approximate mirror symmetry, the mirror plane containing the S(1)—Ni(1)—S(2) moiety and bisecting the N(3)—Ni(1)—N(6) angle.

In (2) the introduction of an α-methyl on one of the four amine ligands destroys this molecular near-symmetry; the conformations of these ligands are now different from those in (1) (*cf.* Table 3).

Crystals of (2) were obtained from a preparation with racemic 1-phenyl-1-ethylamine, and contain equal amounts of molecules with one (*R*)- or one (*S*)-1-phenyl-1-ethylamine in centrosymmetric $P2_1/c$. (3) represents the noncentrosymmetric version of (2), made from only (*S*)-(−)-1-phenyl-1-ethylamine. A comparison of torsion angles (Table 3) for the amine ligands in (2) and (3) shows that the complexes are similar in the two structures. The absolute structure of (3) is governed by the use of enantiomerically pure laevorotatory amine, the absolute configuration of which was known [*Aldrichimica Acta* (1980), **13**(1), 13]. A better refinement ($wR = 0·076$ for correct structure *vs* 0·083 for inverse) merely serves to substantiate the correct configuration.

The molecular packing efficiency decreases with decrease of molecular symmetry; values of volume occupied per non-H atom are 19·6 Å^3 for (1), 20·1 Å^3 for (2), 20·4 Å^3 for (3). This trend is also visible in the decrease of densities from (1) to (3).

The large *R* values obtained for (2) ($R = 0·085$) and (3) ($R = 0·104$) are ascribed mainly to some disorder of the phenyl moieties of the amine ligands, which is demonstrated also by the large $U_{\rm iso}$ values obtained for some of their C atoms. The growth of crystals (2) and (3) with 3:1 ratio of the two amine ligands which were added in equal amounts is an interesting aspect, and studies of comparative complex stabilities and complex formation processes are indicated. The enclathration of chiral aromatic species, *e.g.* *sec*-butylbenzene, by host complexes (2) and (3) is envisaged. We hope that the enantiomeric selectivity of inclusion will be enhanced by having host molecules with different ligands in uneven ratios, thereby reducing the overall symmetry of the host structure. Further work in this area is in progress.

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Potassium Naphthionate*

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Abstract. $C_{10}H_8NO_3S^-.K^+$, $M_r = 261{\cdot}3$, orthorhombic, $P2_1cn$ (non-standard setting of $Pna2_1$), $a = 34{\cdot}478\,(20)$, $b = 8{\cdot}512\,(8)$, $c = 7{\cdot}019\,(5)$ Å, $V = 2059{\cdot}9$ Å^3, $Z = 8$, $D_m = 1{\cdot}681\,(5)$, $D_x = 1{\cdot}685$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}69$ mm^{-1}, $F(000) = 1072$, room temperature, $R = 0{\cdot}043$ for 1752 reflections $[I > \sigma(I)]$. There are two non-equivalent molecules in each asymmetric unit. Both K^+ ions are seven-coordinated to pentagonal bipyramids of O atoms, with K—O distances of 2·747–2·900 (4) Å. The bond lengths and interbond angles retain standard values; the two non-equivalent naphthalene rings are inclined at 24·46 (5)°.

Introduction. All the naphthionate salts prepared by Corbridge, Brown & Wallwork (1966) resulted in hydrated crystals except those of potassium and thallium. From the crystal structure determinations of the sodium (Brown & Corbridge, 1966) and calcium (Brown, Ehrenberg & Yadav, 1984) salts, it is evident that the crystals retained water of crystallization primarily to complete the coordination sphere around the cation. The structure determination of the potassium salt was undertaken to ascertain how the cation coordination could be satisfied in the anhydrous state, and the suggestion that this would be possible because of the larger size of the K^+ ion is confirmed.

Experimental. Crystals, pink to brown in colour, by slow evaporation of a neutral solution of naphthionic acid in aqueous potassium hydroxide; density by flotation in a CCl_4/dibromobenzene mixture; lattice parameters by least-squares analysis of the separations of 46 $\alpha_1\alpha_2$ doublets (Main & Woolfson, 1963) in a 57·3 mm radius camera and refined on the diffractometer; crystal 0·22 × 0·17 × 0·21 mm mounted about **b** on a Stoe Stadi-2 diffractometer for intensity measurements, supplemented by a small number of visually estimated data from **c** Weissenberg photographs which were used for cross correlation of the layers and also for planes where $\theta < 5°$. 2179 reflections measured, 427 considered unobserved. Index range h 0–44, k 0–11, l 0–8, no correction for absorption or extinction. Three different direct-methods programs gave no solution to the structure which was solved using a special modification (Sheldrick, 1976*a*) of the *SHELX* program (Sheldrick, 1976*b*). Least-squares refinement on F using NRC programs (Ahmed,

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* Potassium 4-amino-1-naphthalenesulfonate. (Note that the atomic numbering used throughout corresponds to the alternative description 1-amino-4-naphthalenesulfonate.)

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