A Reinterpretation of the Crystal Structures of the *p*-, *m*-, and *o*-Xylene and Carbon Disulphide Clathrates of Tetrakis(4-ethylpyridine)di-isothiocyanato-nickel(II)†

Madeleine H. Moore, Luigi R. Nassimbeni,* and Margaret L. Niven
Department of Chemistry, University of Cape Town, Rondebosch 7700, South Africa

The crystal structures of the clathrates of $[Ni(NCS)_2(4Et\text{-}py)_4]$ (4Et-py = 4-ethylpyridine) with *p*-, *m*-, and *o*-xylene and carbon disulphide, reported previously in space group $P\bar{1}$, have been shown to be more appropriately refined in the tetragonal space group $I4_1/a$ (no. 88). Chemically there is no change in the structures.

The structures and properties of Werner clathrates, of the form $MX_2L_4\cdot G$ [M = Ni^{II}, X = NCS^-, L = substituted pyridine, G = guest molecule(s), such as *p*-, *m*-, *o*-xylene, chloroform, dimethyl sulphoxide] have recently been reviewed by Lipkowski[1] and have attracted much of our attention.[2] We previously reported[3] the structures of the clathrates of $[Ni(NCS)_2(4Et\text{-}py)_4]$ (4Et-py = 4-ethylpyridine) with *p*-, *m*-, and *o*-xylene and CS_2. The space group given was $P\bar{1}$, with $a \approx b \approx c \approx 17$ Å, $\alpha \approx \beta \approx \gamma \approx 60°$, and noted then that the cell transformation U (0, 1, −1; 1, 0, 0; 1, −1, −1) yields a pseudo-tetragonal cell in each case. Lipkowski[4] has pointed out that the similarity of *a*, *b*, and *c* and α, β, and γ in these structures allows two alternative cell transformations of interest: V (1, 0, −1; 0, 1, 0; 1, −1, 1) and W (1, −1 0; 0, 0, 1; 1, 1, −1). For the four structures, V again yields a pseudo-tetragonal cell but W gives equivalences and systematic absences consistent with space group $I4_1/a$ (no. 88).

We have carried out the transformation W on the $P\bar{1}$ unit cells of the four structures and now report the re-refinement of the structures with tetragonal symmetry.

† *Supplementary data available:* see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1990, Issue 1, pp. xix—xxii.

Experimental

Experimental details of the crystal preparation, *X*-ray data collection, and solution of the structures were as previously reported.[3]

Structure Refinement.—Refinement of the structures was carried out in the space group $I4_1/a$ (origin choice 2, origin at $\bar{1}$) having transformed the cell *via* W. The concomitant co-ordinate transformation is (0.5, −0.5, 0; 0.5, 0.5, 1; 0.5, 0.5, 0) followed by (0, 0.5, 0).

$[Ni(NCS)_2(4Et\text{-}py)_4]\cdot p\text{-}C_6H_4Me_2$. Host: non-hydrogens anisotropic. Guest: no disorder, carbons anisotropic. All H of CH and CH_2 in calculated positions with a single isotropic thermal parameter; methyl hydrogens as rigid groups with a single thermal parameter.

$[Ni(NCS)_2(4Et\text{-}py)_4]\cdot m\text{-}C_6H_4Me_2$. Host: non-hydrogens anisotropic; H of CH and CH_2 in calculated positions; methyl H as rigid groups; all H with single isotropic thermal parameter. Guest: disorder modelled with site occupancy 0.5 for some of the atoms; carbons treated isotropically, no hydrogens placed.

$[Ni(NCS)_2(4Et\text{-}py)_4]\cdot o\text{-}C_6H_4Me_2$. Host: non-hydrogens anisotropic; H of CH and CH_2 in calculated positions; methyl H

Table 1. Crystal data, experimental and refinement parameters for structures $[Ni(NCS)_2(4Et\text{-}py)_4]\cdot G$[a]

Host	$[Ni(NCS)_2(Et\text{-}py)_4]$			
Guest,G	$p\text{-}C_6H_4Me_2$	$m\text{-}C_6H_4Me_2$	$o\text{-}C_6H_4Me_2$	CS_2
Host:Guest	1:1	1:1	1:1	1:2
a/Å	17.710(3)	17.750(3)	17.664(3)	17.472(1)
c/Å	24.253(9)	24.752(4)	24.857(4)	24.401(6)
U/Å^3	7 606(3)	7 798(2)	7 756(2)	7 449(2)
Observed reflections,[b] N_o	1 422	1 501	1 870	2 363
No. of variables, N_v	224	208	200	212
R[c]	0.037	0.072	0.086	0.083
R'[d]	0.032	0.072	0.086	0.083
w	$(\sigma^2F)^{-1}$	Unity	Unity	Unity
S[e]	5.42	5.22	6.48	6.33
U_{iso} (H of CH, CH_2)	0.098(5)	0.17(1)	0.13(1)	0.088(9)
(H of CH_3)	0.25(1)	0.17(1)	0.13(1)	0.19(3)
(guest non-H)		0.091(6)—0.219(8)	0.196(5)	
Max., min. residual electron density/e Å^{-3}	0.24, −0.24	0.59, −0.29	1.16, −0.49	0.69, −1.62

[a] Details in common: space group $I4_1/a$; $Z = 8$. [b] $I_{rel} > 2\sigma I_{rel}$. [c] $R = (\Sigma||F_o|-|F_c||)/\Sigma|F_o|$. [d] $R' = (\Sigma w^{\frac{1}{2}}||F_o|-|F_c||)/\Sigma w^{\frac{1}{2}}|F_o|$. [e] $S = (\Sigma w||F_o|-|F_c||)^2/(N_o-N_v)^{\frac{1}{2}}$.

Table 2. Fractional atomic co-ordinates ($\times 10^4$) with estimated standard deviations (e.s.d.s) in parentheses for $[Ni(NCS)_2(4Et\text{-}py)_4]\cdot G$

	G = *p*-$C_6H_4Me_2$			*m*-$C_6H_4Me_2$			*o*-$C_6H_4Me_2$			$2CS_2$		
	X/*a*	*Y*/*b*	*Z*/*c*	*X*/*a*	*Y*/*b*	*Z*/*c*	*X*/*a*	*Y*/*b*	*Z*/*c*	*X*/*a*	*Y*/*b*	*Z*/*c*
Ni	5 000	2 500	5 552(0)	5 000	2 500	5 5121	5 000	2 500	5 502(1)	5 000	2 500	5 535(1)
S	2 604(1)	3 781(1)	5 518(1)	2 615(2)	3 759(2)	5 489(2)	2 604(2)	1 227(2)	5 454(2)	2 545(1)	3 685(2)	5 578(1)
N(1)	4 041(2)	3 170(2)	5 525(1)	4 057(5)	3 181(5)	5 490(3)	4 050(5)	1 819(5)	5 477(4)	4 050(4)	3 205(4)	5 515(3)
C(1)	3 451(3)	3 420(2)	5 522(2)	3 480(6)	3 418(5)	5 489(4)	3 449(6)	1 580(5)	5 467(4)	3 440(4)	3 407(4)	5 543(3)
N(11)	5 449(2)	3 207(2)	6 186(1)	5 472(5)	3 196(4)	6 133(3)	5 471(5)	1 795(5)	6 120(3)	5 489(4)	3 200(4)	6 160(2)
C(12)	4 992(3)	3 590(3)	6 520(2)	5 028(6)	3 570(7)	6 472(4)	5 020(7)	1 427(8)	6 451(5)	5 034(5)	3 572(6)	6 513(4)
C(13)	5 240(3)	4 037(3)	6 947(2)	5 291(7)	4 001(7)	6 891(4)	5 271(8)	1 003(8)	6 882(5)	5 300(6)	4 012(6)	6 942(4)
C(14)	6 004(3)	4 118(3)	7 044(2)	6 061(7)	4 068(6)	6 973(4)	6 032(7)	930(7)	6 972(5)	6 085(6)	4 103(5)	7 014(4)
C(15)	6 468(3)	3 722(3)	6 697(2)	6 506(6)	3 687(6)	6 621(5)	6 494(6)	1 312(6)	6 626(5)	6 551(5)	3 729(5)	6 647(4)
C(16)	6 182(3)	3 286(2)	6 283(2)	6 206(6)	3 258(5)	6 213(4)	6 203(6)	1 738(6)	6 212(5)	6 252(5)	3 293(5)	6 234(4)
C(17)	6 309(3)	4 611(3)	7 507(2)	6 392(8)	4 543(7)	7 426(5)	6 353(9)	468(8)	7 428(5)	6 396(7)	4 596(6)	7 474(4)
C(18)	5 988(4)	4 437(4)	8 042(2)	6 026(10)	4 472(11)	7 929(7)	5 974(10)	591(11)	7 939(7)	6 177(9)	4 336(8)	8 021(5)
N(21)	4 484(2)	1 821(2)	4 924(1)	4 479(4)	1 831(4)	4 893(3)	4 477(4)	3 176(5)	4 891(3)	4 464(3)	1 830(3)	4 910(2)
C(22)	4 136(2)	2 151(2)	4 499(2)	4 120(5)	2 150(5)	4 481(4)	4 122(5)	2 851(6)	4 481(4)	4 107(4)	2 157(4)	4 492(3)
C(23)	3 804(2)	1 758(3)	4 076(2)	3 792(5)	1 760(6)	4 067(4)	3 790(6)	3 247(6)	4 063(4)	3 770(4)	1 767(4)	4 061(3)
C(24)	3 814(2)	979(3)	4 068(2)	3 810(5)	977(6)	4 065(4)	3 803(6)	4 034(6)	4 063(4)	3 786(4)	968(4)	4 063(3)
C(25)	4 169(2)	639(2)	4 512(2)	4 169(5)	636(5)	4 501(4)	4 158(6)	4 365(6)	4 497(5)	4 147(4)	621(4)	4 501(3)
C(26)	4 497(2)	1 068(3)	4 921(2)	4 499(5)	1 069(6)	4 897(4)	4 481(6)	3 935(6)	4 891(4)	4 483(4)	1 058(4)	4 910(3)
C(27)	3 478(3)	495(3)	3 619(2)	3 473(7)	490(7)	3 627(4)	3 436(9)	4 503(7)	3 627(5)	3 429(6)	493(5)	3 608(4)
C(28)	2 906(3)	884(4)	3 280(3)	2 917(8)	870(9)	3 291(5)	2 884(9)	4 114(10)	3 280(6)	2 934(6)	917(7)	3 214(4)
C(1G)	325(4)	5 181(6)	4 505(3)	1 829(13)*	2 307(13)	7 198(10)	106(14)	536(14)	5 345(11)	3 531(11)	3 070(9)	7 616(7)
C(2G)	510(3)	4 528(5)	4 772(4)	2 229(11)	2 758(11)	6 987(7)	430(14)	402(14)	4 833(11)			
C(3G)	204(4)	4 325(4)	5 273(4)	1 593(12)	1 813(12)	7 568(9)	451(13)	−174(13)	4 336(9)			
C(31G)	405(4)	3 597(4)	5 558(3)				888(26)*	707(26)	4 580(19)			
C(4G)				2 080(17)*	1 771(15)	8 020(12)						
C(5G)				2 274(16)*	2 292(15)	7 577(13)						
C(6G)				1 200(20)*	2 152(19)	6 858(14)						
C(21G)							411(25)*	1 174(27)	5 535(18)			
S(1G)										3 093(3)	3 308(3)	7 097(3)
S(2G)										3 957(5)	2 912(4)	8 130(4)

* Atom at site occupancy 0.5 for disorder model.

Table 2. Bond lengths (Å) with e.s.d.s in parentheses for $[Ni(NCS)_2(4Et\text{-}py)_4]\cdot G$

G = *p*-$C_6H_4Me_2$		*m*-$C_6H_4Me_2$		*o*-$C_6H_4Me_2$		$2CS_2$	
Ni–N(1)	2.074(4)	Ni–N(1)	2.064(9)	Ni–N(1)	2.066(9)	Ni–N(1)	2.067(7)
Ni–N(11)	2.136(3)	Ni–N(11)	2.142(8)	Ni–N(11)	2.145(8)	Ni–N(11)	2.133(6)
Ni–N(21)	2.145(3)	Ni–N(21)	2.148(8)	Ni–N(21)	2.143(8)	Ni–N(21)	2.139(5)
S–C(1)	1.631(5)	S–C(1)	1.649(11)	S–C(1)	1.619(11)	S–C(1)	1.639(7)
N(1)–C(1)	1.132(6)	N(1)–C(1)	1.109(14)	N(1)–C(1)	1.140(14)	N(1)–C(1)	1.127(10)
N(11)–C(12)	1.332(6)	N(11)–C(12)	1.329(13)	N(11)–C(12)	1.316(15)	N(11)–C(12)	1.339(11)
N(11)–C(16)	1.328(6)	N(11)–C(16)	1.323(14)	N(11)–C(16)	1.317(14)	N(11)–C(16)	1.357(11)
C(12)–C(13)	1.376(7)	C(12)–C(13)	1.370(16)	C(12)–C(13)	1.380(19)	C(12)–C(13)	1.378(14)
C(13)–C(14)	1.379(8)	C(13)–C(14)	1.387(18)	C(13)–C(14)	1.367(19)	C(13)–C(14)	1.394(15)
C(14)–C(15)	1.369(7)	C(14)–C(15)	1.356(16)	C(14)–C(15)	1.364(17)	C(14)–C(15)	1.377(13)
C(14)–C(17)	1.522(7)	C(14)–C(17)	1.520(17)	C(14)–C(17)	1.510(18)	C(14)–C(17)	1.513(14)
C(15)–C(16)	1.366(7)	C(15)–C(16)	1.371(15)	C(15)–C(16)	1.375(17)	C(15)–C(16)	1.366(13)
C(17)–C(18)	1.447(7)	C(17)–C(18)	1.410(22)	C(17)–C(18)	1.453(22)	C(17)–C(18)	1.463(16)
N(21)–C(22)	1.335(5)	N(21)–C(22)	1.327(12)	N(21)–C(22)	1.328(13)	N(21)–C(22)	1.325(9)
N(21)–C(26)	1.336(6)	N(21)–C(26)	1.353(13)	N(21)–C(26)	1.339(14)	N(21)–C(26)	1.349(9)
C(22)–C(23)	1.373(6)	C(22)–C(23)	1.370(14)	C(22)–C(23)	1.381(14)	C(22)–C(23)	1.383(10)
C(23)–C(24)	1.382(8)	C(23)–C(24)	1.388(15)	C(23)–C(24)	1.389(15)	C(23)–C(24)	1.398(10)
C(24)–C(25)	1.382(6)	C(24)–C(25)	1.392(14)	C(24)–C(25)	1.379(16)	C(24)–C(25)	1.382(10)
C(24)–C(27)	1.509(7)	C(24)–C(27)	1.510(15)	C(24)–C(27)	1.509(17)	C(24)–C(27)	1.517(12)
C(25)–C(26)	1.380(6)	C(25)–C(26)	1.376(14)	C(25)–C(26)	1.364(16)	C(25)–C(26)	1.387(10)
C(27)–C(28)	1.476(8)	C(27)–C(28)	1.455(18)	C(27)–C(28)	1.472(21)	C(27)–C(28)	1.492(15)
C(1G)–C(2G)	1.364(13)	C(1G)–C(6G)	1.424(42)	C(1G)–C(2G)	1.415(38)	S(1G)–C(1G)	1.538(19)
C(2G)–C(3G)	1.377(13)	C(1G)–C(2G)	1.192(30)	C(1G)–C(21G)	1.337(53)	S(2G)–C(1G)	1.482(20)
C(3G)–C(31G)	1.508(13)	C(1G)–C(3G)	1.335(32)	C(2G)–C(3G)	1.604(35)		
C(3G)–C(1G^{I})	1.389(13)	C(2G)–C(5G)	1.682(36)	C(2G)–C(31G)	1.158(53)		
		C(3G)–C(4G)	1.415(37)	C(1G)–C(3G^{II})	1.414(36)		
		C(3G)–C(5G)	1.478(35)				
		C(4G)–C(2G^{III})	1.480(40)				
		C(2G)–C(5G^{III})	1.398(42)				

Symmetry translations: I $-x, -y+1, -z+1$; II $-x, -y, 1-z$; III $\frac{1}{2}-x, \frac{1}{2}-y, \frac{3}{2}-z$.

as rigid groups; all H with single isotropic thermal parameter. Guest: disorder modelled with site occupancy 0.5 for the ring substituents; only carbons of guest placed and all modelled with a single isotropic thermal parameter.

$[Ni(NCS)_2(4Et\text{-}py)_4]\cdot 2CS_2$. Host: non-hydrogens anisotropic; H of CH and CH_2 in calculated positions with a single isotropic thermal parameter; methyl hydrogens as rigid groups

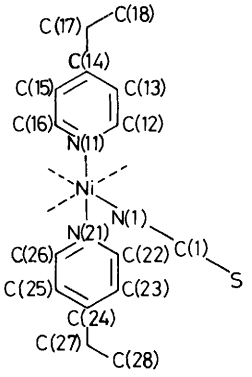

Figure. Schematic representation of the host molecule showing atomic numbering

Table 4. Bond angles (°) with e.s.d.s in parentheses for $[Ni(NCS)_2(4Et\text{-}py)_4]\cdot G$

G = p-$C_6H_4Me_2$

N(11)–Ni–N(21)	176.6(2)	C(22)–N(21)–C(26)	116.3(3)
N(1)–Ni–N(21)	87.1(1)	N(21)–C(22)–C(23)	123.6(4)
N(1)–Ni–N(11)	89.6(1)	C(22)–C(23)–C(24)	120.7(4)
Ni–N(1)–C(1)	167.8(4)	C(23)–C(24)–C(27)	124.9(4)
S–C(1)–N(1)	179.8(4)	C(23)–C(24)–C(25)	115.6(4)
Ni–N(11)–C(16)	123.6(3)	C(25)–C(24)–C(27)	119.5(4)
Ni–N(11)–C(12)	120.7(3)	C(24)–C(25)–C(26)	120.8(4)
C(12)–N(11)–C(16)	115.7(4)	N(21)–C(26)–C(25)	123.1(4)
N(11)–C(12)–C(13)	123.8(5)	C(24)–C(27)–C(28)	114.1(5)
C(12)–C(13)–C(14)	120.1(5)	C(1G)–C(2G)–C(3G)	122.9(7)
C(13)–C(14)–C(17)	122.1(5)	C(2G)–C(3G)–C(31G)	122.4(7)
C(13)–C(14)–C(15)	115.6(5)	C(2G)–C(3G)–C(1G^{I})	116.3(7)
C(15)–C(14)–C(17)	122.2(5)	C(2G)–C(1G)–C(3G^{I})	120.7(7)
C(14)–C(15)–C(16)	121.2(5)	C(31G)–C(3G)–C(1G^{I})	121.4(7)
N(11)–C(16)–C(15)	123.5(5)	N(11)–Ni–N(11^{IV})	87.9(4)
C(14)–C(17)–C(18)	113.6(5)	N(21)–Ni–N(21^{IV})	89.5(4)
Ni–N(21)–C(26)	123.7(3)	N(1)–Ni–N(1^{IV})	176.4(4)
Ni–N(21)–C(22)	120.0(3)		

G = m-$C_6H_4Me_2$

N(11)–Ni–N(21)	177.4(4)	C(23)–C(24)–C(25)	116.4(10)
N(1)–Ni–N(21)	87.4(4)	C(25)–C(24)–C(27)	119.3(10)
N(1)–Ni–N(11)	89.9(4)	C(24)–C(25)–C(26)	120.3(10)
Ni–N(1)–C(1)	166.4(9)	N(21)–C(26)–C(25)	122.8(10)
S–C(1)–N(1)	179.2(10)	C(24)–C(27)–C(28)	114.5(11)
Ni–N(11)–C(16)	122.8(7)	C(2G)–C(1G)–C(3G)	158.2(25)
Ni–N(11)–C(12)	120.7(7)	C(3G)–C(4G)–C(2G^{III})	117.8(28)
C(12)–N(11)–C(16)	116.5(9)	C(6G)–C(1G)–C(3G)	91.9(22)
N(11)–C(12)–C(13)	123.8(11)	C(6G)–C(1G)–C(2G)	109.8(24)
C(12)–C(13)–C(14)	119.5(10)	C(3G)–C(5G)–C(2G^{III})	119.5(15)
C(13)–C(14)–C(17)	122.4(11)	C(1G)–C(2G)–C(5G^{III})	89.7(17)
C(13)–C(14)–C(15)	116.0(11)	C(1G)–C(3G)–C(4G)	112.8(22)
C(15)–C(14)–C(17)	121.6(12)	C(2G)–C(5G)–C(3G)	103.3(21)
C(14)–C(15)–C(16)	121.5(11)	C(2G)–C(5G^{III})–C(2G^{III})	137.1(26)
N(11)–C(16)–C(15)	122.7(10)	C(4G)–C(2G^{III})–C(5G^{III})	104.4(19)
C(14)–C(17)–C(18)	115.1(13)	C(1G)–C(5G)–C(2G)	45.1(16)
Ni–N(21)–C(26)	122.3(6)	N(11)–Ni–N(11^{IV})	88.3(4)
Ni–N(21)–C(22)	121.2(6)	N(21)–Ni–N(21^{IV})	89.0(4)
C(22)–N(21)–C(26)	116.4(8)	N(1)–Ni–N(1^{IV})	177.0(4)
N(21)–C(22)–C(23)	124.3(9)		
C(22)–C(23)–C(24)	119.9(9)		
C(23)–C(24)–C(27)	124.4(10)		

Table 4 (*continued*)

G = o-$C_6H_4Me_2$

N(11)–Ni–N(21)	177.2(4)	C(23)–C(24)–C(27)	122.9(10)
N(1)–Ni–N(21)	87.4(4)	C(23)–C(24)–C(25)	115.5(10)
N(1)–Ni–N(11)	89.9(4)	C(25)–C(24)–C(27)	121.6(10)
Ni–N(1)–C(1)	166.0(9)	C(24)–C(25)–C(26)	121.1(10)
S–C(1)–N(1)	178.9(9)	N(21)–C(26)–C(25)	123.6(10)
Ni–N(11)–C(16)	123.4(7)	C(24)–C(27)–C(28)	116.7(12)
Ni–N(11)–C(12)	120.0(8)	C(2G)–C(1G)–C(21G)	107.1(28)
C(12)–N(11)–C(16)	116.4(10)	C(1G)–C(2G)–C(31G)	134.1(33)
N(11)–C(12)–C(13)	124.0(12)	C(1G)–C(2G)–C(3G)	144.0(23)
C(12)–C(13)–C(14)	119.5(12)	C(3G)–C(2G)–C(31G)	81.8(29)
C(13)–C(14)–C(17)	122.9(12)	C(2G)–C(1G)–C(3G^{II})	135.1(18)
C(13)–C(14)–C(15)	116.1(12)	C(2G)–C(3G)–C(1G^{II})	80.7(25)
C(15)–C(14)–C(17)	121.0(12)	C(21)G–C(1G)–C(3G)^{II}	117.6(30)
C(14)–C(15)–C(16)	121.1(11)	N(11)–Ni–N(11^{IV})	88.6(4)
N(11)–C(16)–C(15)	122.8(10)	N(21)–Ni–N(21^{IV})	89.7(4)
C(14)–C(17)–C(18)	113.8(13)	N(1)–Ni–N(1^{IV})	176.6(4)
Ni–N(21)–C(26)	123.7(6)		
Ni–N(21)–C(22)	120.4(7)		
C(22)–N(21)–C(26)	115.9(8)		
N(21)–C(22)–C(23)	123.8(10)		
C(22)–C(23)–C(24)	120.1(10)		

G = $2CS_2$

N(11)–Ni–N(21)	177.4(3)	C(14)–C(17)–C(18)	114.0(10)
N(1)–Ni–N(21)	87.6(3)	Ni–N(21)–C(26)	122.4(4)
N(1)–Ni–N(11)	89.8(3)	Ni–N(21)–C(22)	121.3(5)
Ni–N(1)–C(1)	161.0(7)	C(22)–N(21)–C(26)	116.3(6)
S–C(1)–N(1)	178.7(8)	N(21)–C(22)–C(23)	125.0(7)
Ni–N(11)–C(16)	123.9(5)	C(22)–C(23)–C(24)	118.8(7)
Ni–N(11)–C(12)	120.1(6)	C(23)–C(24)–C(27)	122.4(7)
C(12)–N(11)–C(16)	116.0(7)	C(23)–C(24)–C(25)	116.7(7)
N(11)–C(12)–C(13)	124.0(9)	C(25)–C(24)–C(27)	120.9(7)
C(12)–C(13)–C(14)	119.4(9)	C(24)–C(25)–C(26)	120.6(7)
C(13)–C(14)–C(17)	120.8(9)	N(21)–C(26)–C(25)	122.7(7)
C(13)–C(14)–C(15)	116.4(9)	C(24)–C(27)–C(28)	116.0(8)
C(15)–C(14)–C(17)	122.8(10)	S(1G)–C(1G)–S(2G)	174.9(12)
C(14)–C(15)–C(16)	121.3(9)	N(11)–Ni–N(11^{IV})	88.7(4)
N(11)–C(16)–C(15)	122.8(8)	N(21)–Ni–N(21^{IV})	89.0(4)
		N(1)–Ni–N(1^{IV})	177.3(4)

Symmetry translations: I—III as in Table 3; IV $1 - x, \frac{1}{2} - y, z$.

with a single isotropic thermal parameter. Guest: no disorder, C and S atoms anisotropic. Further details of the final refinements are given in Table 1.

Results and Discussion

The atom numbering scheme for the host molecule is shown in the Figure. Final fractional atomic co-ordinates are reported in Table 2, bond lengths and angles in Tables 3 and 4 respectively.

In the tetragonal space group the structures refine somewhat better than they did in $P\bar{1}$, as evidenced by generally smaller standard deviations in molecular parameters. It is interesting that no parameter correlation was observed in the previously reported triclinic structures. Chemically there is no change in the structures, although the host molecules are now required to have two-fold symmetry (Ni at Wyckoff site e) and hence no further discussion is required.

Acknowledgements

We sincerely thank Professor J. Lipkowski of the Institute of Physical Chemistry of the Polish Academy of Sciences for acquainting us with the alternative cell transformation leading to tetragonal symmetry. We also thank the University of Cape Town and the Foundation for Research and Development for financial support.

References

1 J. Lipkowski, in 'Inclusion Compounds,' eds. J. L. Atwood, J. E. D. Davies, and D. D. MacNicol, Academic Press, London, 1984, vol. 1, ch. 3.
2 L. R. Nassimbeni, M. L. Niven, and M. W. Taylor, *J. Coord. Chem.*, 1989, **19**, 339.
3 M. H. Moore, L. R. Nassimbeni, and M. L. Niven, *J. Chem. Soc, Dalton Trans.*, 1987, 2125.
4 J. Lipkowski, personal communication, 1988.

Received 21*st March* 1989; *Paper* 9/01220F