the photometer recordings. On the equator the reflexion with $Q=613$, *vvs*, has been omitted because of its very high intensity, being about ten times that with $Q=473$. For the 6th layer line the nearly meridional reflexions are smeared out along the layer line, which fact may be responsible for the high intensities observed. There is a good agreement between the observed and calculated intensities.

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Refinement of the Structure of Bisthiourea-nickel (II) Thiocyanate.

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The crystal structure of $Ni[SC(NH_2)_2]_2(NCS)_2$ has been refined by means of three-dimensional differential syntheses. Each nickel atom lies on a symmetry centre (space group $P\bar{1}$) and is octahedrally surrounded by four sulphur atoms from thiourea molecules (Ni–S $2{\cdot}53\pm0{\cdot}01$ and $2{\cdot}56\pm0{\cdot}01$ Å) and two nitrogen atoms from thiocyanate groups (Ni–N $1{\cdot}99\pm0{\cdot}01$ Å). The octahedra are linked in chains with each thionilic S atom shared by two adjacent nickel atoms. Bond distances and angles are discussed.

The structure of bisthiourea-nickel(II) thiocyanate, $Ni[SC(NH_2)_2]_2(NCS)_2$, was determined several years ago by Nardelli, Braibanti & Fava (1957) (hereafter called NBF) by means of generalized Fourier syntheses using an incomplete set of three-dimensional data (482 independent reflexions out of 1222 possible with Cu $K\alpha$ radiation). Because this compound is interesting from the point of view of the coordination of the thionilic sulphur and the thiocyanate group, the refinement of its structure was undertaken to improve the reliability of bond distances and angles with the use of all the observable data with Cu $K\alpha$ radiation.

Cell constants, remeasured, from rotation and Weissenberg photographs, are as follows:

$Ni[SC(NH_2)_2]_2(NCS)_2$. $M=327.1$; $a=3{\cdot}80\pm0{\cdot}01$, $b=7{\cdot}58\pm0{\cdot}01$, $c=10{\cdot}11\pm0{\cdot}01$ Å; $\alpha=92{\cdot}3\pm0{\cdot}2$, $\beta=98{\cdot}1\pm0{\cdot}2$, $\gamma=104{\cdot}1\pm0{\cdot}1°$, $V=278{\cdot}7$ Å^3, $Z=1$, $D_x=1{\cdot}95$, $D_m=1{\cdot}88$ g.cm^{-3} (flotation), $\mu=90{\cdot}2$ cm^{-1} (Cu $K\alpha$). Space group: $P\bar{1}$.

New three-dimensional intensity data were collected from integrated and non-integrated multiple film Weissenberg photographs taken around [100] with a sample of nearly square cross-section (edge 0·008 cm) and around [010] using a fragment with a mean radius of 0·008 cm. All the crystals were parallel twins with twinning axis [100], and yet the reflexions due to the two individuals were well separated in all the photographs (excepting of course the $0kl$ reflexions which were always superimposed). The complete indexing and intensity measurement (photometrically) of the spots from both individuals were straightforward in the series of photographs taken around [100]. In the series taken around [010], only the reflexions due to the individual rotating around that axis were considered. Four levels about the *a* axis and seven levels about the *b* axis were measured, giving a total of 1010 observed independent reflexions. The absorption correction for cylindrical samples was applied to [100] – and for spherical samples to [010] – data. Upper-level Weissenberg spot shape effects were considered following Phillips (1956) for the first series and following Scouloudi (1953) for the second series of data. The structure amplitudes (with the exception of $0kl$) were put on the same relative scale by the least-squares cross-correl-

ation method of Rollett & Sparks (1960). The absolute scale factors for $0kl$ and for hkl were determined separately by comparing F_o and F_c.

The refinement, starting from the NBF atomic coordinates ($R=21{\cdot}3\%$), was carried out with eleven cycles of Booth's differential synthesis, the first three with isotropic, the remainder with anisotropic thermal parameters. These parameters were derived from the second derivatives of the electron-density, following the method of Nardelli & Fava (1960). At the end of the refinement the agreement indices (R, for observed reflexions only; R', assuming $F_o=\frac{1}{2}F_{\min}$ when $F_c \geq F_{\min}$ for unobserved reflexions; multiplicities not considered) were: $R=10{\cdot}3\%$, $R'=12{\cdot}2\%$.

The final coordinates with their e.s.d.'s (Cruickshank, 1949) are given in Table 1. Observed and calculated peak heights and curvatures are compared in Table 2, in which the e.s.d.'s of electron-density and second derivatives are quoted too.

In view of the uncertainty in deriving the correct scale factors, the anisotropic thermal parameters, given in Table 3, do not have strict physical meaning. Rescaling by layers along [100] improves the R and R' indices to 9·4% and 11·4% respectively. The low value of the R index, obtained in this way, indicates also that further refinement of the structure in the $P1$ space group would not be worth while.

Table 1. *Final fractional coordinates* ($\times 10^4$) *and their e.s.d.'s* (in 10^{-3} Å)

	x/a (σ)	y/b (σ)	z/c (σ)
Ni	0	0	0
S(1)	116 (4)	2950 (4)	4231 (3)
S(2)	3983 (3)	−2228 (3)	333 (2)
N(1)	869 (12)	1050 (10)	1892 (6)
N(2)	4453 (15)	−1916 (16)	3003 (9)
N(3)	5851 (14)	−4444 (14)	2096 (10)
C(1)	614 (13)	1847 (15)	2874 (11)
C(2)	4816 (15)	−2931 (13)	1959 (10)

Table 2. *Atomic peak heights* (e.Å^{-3}), *curvatures* (e.Å^{-5}) *and e.s.d.'s*

		ϱ	$-A_{hh}$	$-A_{kk}$	$-A_{ll}$	A_{kl}	A_{hl}	A_{hk}
Ni	obs.	68·3	670	697	746	41	68	181
	calc.	70·5	663	699	745	41	66	178
S(1)	obs.	38·1	390	368	423	−7	42	123
	calc.	38·5	387	367	423	−7	42	123
S(2)	obs.	46·8	478	528	538	51	39	137
	calc.	47·1	474	526	538	51	38	137
N(1)	obs.	14·6	133	141	180	−16	26	45
	calc.	14·7	131	142	179	−16	25	43
N(2)	obs.	11·9	95	97	133	12	4	29
	calc.	12·0	94	99	133	12	4	28
N(3)	obs.	12·6	106	133	129	24	2	36
	calc.	12·6	103	133	128	23	3	34
C(1)	obs.	11·3	103	108	114	23	−5	26
	calc.	11·2	101	104	113	24	−6	26
C(2)	obs.	11·1	98	121	116	17	3	31
	calc.	11·3	96	122	115	16	3	30
e.s.d.		0·3	5	4	4	2	2	3

Table 3. *Thermal parameters with e.s.d.'s* ($\times 10$ Å^2)

	$B_{11}(\sigma)$	$B_{22}(\sigma)$	$B_{33}(\sigma)$	$B_{23}(\sigma)$	$B_{13}(\sigma)$	$B_{12}(\sigma)$
Ni	28 (2)	27 (1)	21 (1)	2 (1)	0 (1)	9 (2)
S(1)	25 (2)	34 (1)	13 (0)	−4 (1)	−3 (1)	14 (2)
S(2)	18 (1)	14 (1)	9 (0)	4 (1)	−2 (1)	7 (1)
N(1)	21 (5)	19 (3)	11 (1)	0 (3)	−2 (4)	9 (5)
N(2)	39 (7)	29 (5)	10 (1)	2 (4)	−5 (5)	15 (7)
N(3)	37 (7)	21 (4)	21 (2)	6 (5)	−2 (5)	12 (7)
C(1)	20 (5)	21 (4)	12 (2)	5 (4)	−3 (4)	7 (6)
C(2)	20 (5)	13 (3)	17 (2)	2 (4)	−2 (4)	1 (5)

Introducing the H atoms at the calculated positions did not improve the R index. An F_o-F_c synthesis, calculated without the hydrogen atom contributions, gave positive values of the electron density of about 1 e.Å^{-3} in the regions in which these atoms must be present.

In Table 4 observed and calculated (with contributions of H atoms) structure factors are compared. The scattering factors used are those of Thomas & Umeda (1957) for Ni^{2+}, of Dawson (1960) for S and of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for N and C, and McWeeny (1951) for H.

Discussion

The structure previously determined by NBF is confirmed: the coordination around Ni^{2+} is octahedral and concerns four S atoms from thiourea molecules and two N atoms from thiocyanate groups; the octahedra are linked in chains running along [100] with each thionilic S atom coordinated by two adjacent metal atoms. The new values of distances and angles are shown in Fig. 1.

The two Ni–S distances are just significantly different [$t_0 = (l_1-l_2)(\sigma_1^2+\sigma_2^2)^{-\frac{1}{2}} = 3{\cdot}06$; significance test of Cruickshank & Robertson, 1953]; both are greater than either the sum of Pauling's covalent radii (2·43 Å) or the value (2·46$_2$ Å) found by Lopez-Castro & Truter (1963) in $Ni[SC(NH_2)_2]_4Cl_2$, but lie within the range 2·4–2·6 Å which is usually observed for octahedral Ni^{2+} complexes. The Ni–N bond distance is consistent with the sum of Pauling's covalent radii (2·09 Å) and with the distances generally found in octahedral Ni^{2+} complexes [*e.g.* 2·02 and 1·98 Å in $Ni(NH_3)_3(NCS)_2$ (Poraj-Košic, 1956), 2·12$_0$ Å in $Ni[NH_2(CH_2)_2NH_2]_3(NO_3)_2$ (Swink & Atoji, 1960)].

The thiourea molecule is planar, the least-squares plane being: $0{\cdot}9574X+0{\cdot}2771Y+0{\cdot}0812Z=0{\cdot}8072$*; the largest distance from this plane is 0·007 Å for C(2).

* The X, Y, Z coordinates are in Å and referred to orthogonal axes. They can be obtained from those of Table 1 using the matrix:

$$\begin{pmatrix} a\sin\gamma & 0 & -c\sin\alpha\cos\beta^* \\ a\cos\gamma & b & c\cos\alpha \\ 0 & 0 & c\sin\alpha\sin\beta^* \end{pmatrix}$$

Table 4. *Observed and calculated structure factors*

A minus sign for F_o means 'less than'.

h	k	l	$\|10F_o\|$	$10F_c$
0	1	0	257	246
0	2	0	339	-323
0	3	0	48	68
0	4	0	277	332
0	5	0	107	114
0	6	0	61	44
0	7	0	67	61
0	8	0	19-	-10
0	9	0	110	101
0	0	1	257	325
0	1	1	322	357
0	1	$\bar{1}$	288	308
0	2	1	118	108
0	2	$\bar{1}$	365	335
0	3	1	61	-44
0	3	$\bar{1}$	27	29
0	4	1	59	79
0	4	$\bar{1}$	303	353
0	5	1	325	344
0	5	$\bar{1}$	172	205
0	6	1	27	36
0	6	$\bar{1}$	45	-31
0	7	1	40	-35
0	7	$\bar{1}$	75	-81
0	8	1	96	87
0	8	$\bar{1}$	126	117
0	9	1	142	137
0	9	$\bar{1}$	121	130
0	0	2	185	187
0	1	2	422	518
0	1	$\bar{2}$	121	-97
0	2	2	257	-226
0	2	$\bar{2}$	166	173
0	3	2	86	89
0	3	$\bar{2}$	500	500
0	4	2	295	354
0	4	$\bar{2}$	239	261
0	5	2	193	215
0	5	$\bar{2}$	70	72
0	6	2	19-	-14
0	6	$\bar{2}$	86	85
0	7	2	40	36
0	7	$\bar{2}$	32	-41
0	8	2	113	110
0	8	$\bar{2}$	75	84
0	9	2	67	54
0	9	$\bar{2}$	104	116
0	0	3	298	303
0	1	3	175	189
0	1	$\bar{3}$	387	441
0	2	3	183	202
0	2	$\bar{3}$	344	-299
0	3	3	27	43
0	3	$\bar{3}$	16-	-3
0	4	3	70	88
0	4	$\bar{3}$	360	408
0	5	3	266	294
0	5	$\bar{3}$	295	328
0	6	3	228	248
0	6	$\bar{3}$	70	-70
0	7	3	118	-117
0	7	$\bar{3}$	24-	4
0	8	3	32	-35
0	8	$\bar{3}$	142	153
0	9	$\bar{3}$	48	52
0	0	4	373	395
0	1	4	462	546
0	1	$\bar{4}$	139	-107
0	2	4	196	191
0	2	$\bar{4}$	53	-27
0	3	4	175	-156
0	3	$\bar{4}$	185	180
0	4	4	206	235
0	4	$\bar{4}$	132	150
0	5	4	239	252
0	5	$\bar{4}$	180	193
0	6	4	53	63
0	6	$\bar{4}$	16	8
0	7	4	48	-49
0	7	$\bar{4}$	42	46
0	8	4	16-	-5
0	8	$\bar{4}$	113	113
0	9	$\bar{4}$	110	106
0	0	5	451	462
0	1	5	121	118
0	1	$\bar{5}$	37	43
0	2	5	134	159
0	2	$\bar{5}$	70	-34
0	3	5	252	222
0	3	$\bar{5}$	298	270
0	4	5	45	46
0	4	$\bar{5}$	314	341
0	5	5	35	21
0	5	$\bar{5}$	107	-108
0	6	5	104	110
0	6	$\bar{5}$	126	-121
0	7	5	42	48
0	7	$\bar{5}$	158	161
0	8	5	42	-52
0	8	$\bar{5}$	142	152

h	k	l	$\|10F_o\|$	$10F_c$
0	0	6	137	113
0	1	6	432	423
0	1	$\bar{6}$	32-	21
0	2	6	137	134
0	2	$\bar{6}$	288	251
0	3	6	185	-150
0	3	$\bar{6}$	183	159
0	4	6	24-	4
0	4	$\bar{6}$	175	171
0	5	6	255	264
0	5	$\bar{6}$	52	54
0	6	6	64	61
0	6	$\bar{6}$	21-	-3
0	7	6	16-	-11
0	7	$\bar{6}$	19-	-19
0	8	$\bar{6}$	104	121
0	0	7	311	271
0	1	7	368	336
0	1	$\bar{7}$	78	-53
0	2	7	81	-77
0	2	$\bar{7}$	61	-54
0	3	7	48	-36
0	3	$\bar{7}$	347	320
0	4	7	81	73
0	4	$\bar{7}$	220	225
0	5	7	121	127
0	5	$\bar{7}$	27-	-3
0	6	7	158	156
0	6	$\bar{7}$	48	34
0	7	7	64	63
0	7	$\bar{7}$	32	40
0	8	$\bar{7}$	56	69
0	0	8	153	-124
0	1	8	150	126
0	1	$\bar{8}$	91	79
0	2	8	339	316
0	2	$\bar{8}$	121	115
0	3	8	24-	2
0	3	$\bar{8}$	188	165
0	4	8	56	-58
0	4	$\bar{8}$	161	157
0	5	8	172	171
0	5	$\bar{8}$	83	83
0	6	8	180	175
0	6	$\bar{8}$	81	-76
0	7	8	2-	-52
0	7	$\bar{8}$	75	83
0	0	9	126	73
0	1	9	252	219
0	1	$\bar{9}$	185	-152
0	2	9	139	135
0	2	$\bar{9}$	115	97
0	3	9	32	26
0	3	$\bar{9}$	247	267
0	4	9	104	101
0	4	$\bar{9}$	51	54
0	5	9	42	44
0	5	$\bar{9}$	40	-51
0	6	9	48	45
0	6	$\bar{9}$	13-	7
0	7	$\bar{9}$	129	142
0	0	10	169	146
0	1	10	104	96
0	1	$\bar{10}$	64	46
0	2	10	212	182
0	2	$\bar{10}$	24-	-16
0	3	10	72	68
0	3	$\bar{10}$	67	68
0	4	10	118	-112
0	4	$\bar{10}$	166	166
0	5	10	40	37
0	5	$\bar{10}$	35	-33
0	6	$\bar{10}$	29	-29
0	0	11	27-	-22
0	1	11	279	248
0	1	$\bar{11}$	81	-71
0	2	11	118	105
0	2	$\bar{11}$	223	193
0	3	11	64	-64
0	3	$\bar{11}$	164	170
0	4	11	10-	-14
0	4	$\bar{11}$	21	-11
0	5	$\bar{11}$	64	-67
0	0	12	21-	-14
0	1	12	21	17
0	1	$\bar{12}$	24-	26
0	2	12	75	68
0	2	$\bar{12}$	113	99
0	3	$\bar{12}$	204	208
0	4	$\bar{12}$	8-	38
1	0	0	131	150
1	1	0	324	359
1	$\bar{1}$	0	215	207
1	2	0	226	239
1	$\bar{2}$	0	181	198
1	3	0	162	160
1	$\bar{3}$	0	520	565
1	4	0	44	-26
1	$\bar{4}$	0	64	81
1	5	0	41	-28

h	k	l	$\|10F_o\|$	$10F_c$
1	$\bar{5}$	0	146	-146
1	6	0	207	198
1	$\bar{6}$	0	53	62
1	7	0	113	116
1	$\bar{7}$	0	233	216
1	8	0	86-	2
1	$\bar{8}$	0	86	92
1	$\bar{9}$	0	37	-42
1	0	1	302	-298
1	0	$\bar{1}$	124	-85
1	1	1	139	-103
1	$\bar{1}$	1	472	509
1	1	$\bar{1}$	755	949
1	$\bar{1}$	$\bar{1}$	218	-181
1	2	1	448	519
1	$\bar{2}$	1	559	682
1	2	$\bar{1}$	404	442
1	$\bar{2}$	$\bar{1}$	328	367
1	3	1	64	77
1	$\bar{3}$	1	200	226
1	3	$\bar{1}$	101	-84
1	$\bar{3}$	$\bar{1}$	248	263
1	4	1	41	-44
1	$\bar{4}$	1	26	-7
1	4	$\bar{1}$	116	-90
1	$\bar{4}$	$\bar{1}$	26	38
1	5	1	116	113
1	$\bar{5}$	1	120	127
1	5	$\bar{1}$	185	178
1	$\bar{5}$	$\bar{1}$	48	60
1	6	1	196	198
1	$\bar{6}$	1	11	8
1	6	$\bar{1}$	146	116
1	$\bar{6}$	$\bar{1}$	120	137
1	7	1	53	38
1	$\bar{7}$	1	200	177
1	7	$\bar{1}$	72	69
1	$\bar{7}$	$\bar{1}$	37-	26
1	8	1	53	22
1	$\bar{8}$	1	131	130
1	8	$\bar{1}$	44	27
1	$\bar{8}$	$\bar{1}$	139	135
1	9	1	79	-53
1	$\bar{9}$	$\bar{1}$	72	71
1	0	2	203	216
1	0	$\bar{2}$	260	279
1	1	2	177	189
1	$\bar{1}$	2	279	-268
1	1	$\bar{2}$	98	123
1	$\bar{1}$	$\bar{2}$	22-	13
1	2	2	203	209
1	$\bar{2}$	2	253	277
1	2	$\bar{2}$	272	290
1	$\bar{2}$	$\bar{2}$	68	69
1	3	2	173	176
1	$\bar{3}$	2	396	487
1	3	$\bar{2}$	302	328
1	$\bar{3}$	$\bar{2}$	215	244
1	4	2	98	99
1	$\bar{4}$	2	146	153
1	4	$\bar{2}$	29-	0
1	$\bar{4}$	$\bar{2}$	279	316
1	5	2	37-	-10
1	$\bar{5}$	2	33	-31
1	5	$\bar{2}$	79	75
1	$\bar{5}$	$\bar{2}$	120	-121
1	6	2	139	134
1	$\bar{6}$	2	146	181
1	6	$\bar{2}$	166	160
1	$\bar{6}$	$\bar{2}$	26-	33
1	7	2	207	205
1	$\bar{7}$	2	222	219
1	7	$\bar{2}$	146	85
1	$\bar{7}$	$\bar{2}$	268	273
1	8	2	26-	-15
1	$\bar{8}$	2	79	-32
1	8	$\bar{2}$	79	-77
1	$\bar{8}$	$\bar{2}$	139	143
1	9	2	37	-25
1	$\bar{9}$	$\bar{2}$	37	-38
1	0	3	44	-15
1	0	$\bar{3}$	98	107
1	1	3	86	89
1	$\bar{1}$	3	90	106
1	1	$\bar{3}$	222	272
1	$\bar{1}$	$\bar{3}$	305	-280
1	2	3	389	429
1	$\bar{2}$	3	162	190
1	2	$\bar{3}$	192	211
1	$\bar{2}$	$\bar{3}$	290	341
1	3	3	313	330
1	$\bar{3}$	3	64	97
1	3	$\bar{3}$	22-	27
1	$\bar{3}$	$\bar{3}$	392	467
1	4	3	120	-110
1	$\bar{4}$	3	222	244
1	4	$\bar{3}$	230	179
1	$\bar{4}$	$\bar{3}$	57	79
1	5	3	37-	18
1	$\bar{5}$	3	33-	22

h	k	l	$\|10F_o\|$	$10F_c$
1	5	$\bar{3}$	139	154
1	$\bar{5}$	$\bar{3}$	33-	-19
1	6	3	173	170
1	$\bar{6}$	3	131	125
1	6	$\bar{3}$	90	116
1	$\bar{6}$	$\bar{3}$	94	107
1	7	3	128	130
1	$\bar{7}$	3	200	199
1	7	$\bar{3}$	79	73
1	$\bar{7}$	$\bar{3}$	161	161
1	8	3	18-	-29
1	$\bar{8}$	3	86	72
1	8	$\bar{3}$	44	-42
1	$\bar{8}$	$\bar{3}$	79	71
1	9	3	72	-60
1	$\bar{9}$	$\bar{3}$	26-	20
1	0	4	113	-111
1	0	$\bar{4}$	64	80
1	1	4	309	328
1	$\bar{1}$	4	86	105
1	1	$\bar{4}$	215	197
1	$\bar{1}$	$\bar{4}$	173	194
1	2	4	181	177
1	$\bar{2}$	4	426	528
1	2	$\bar{4}$	351	388
1	$\bar{2}$	$\bar{4}$	18-	1
1	3	4	162	167
1	$\bar{3}$	4	188	222
1	3	$\bar{4}$	90	-69
1	$\bar{3}$	$\bar{4}$	170	210
1	4	4	162	152
1	$\bar{4}$	4	268	-280
1	4	$\bar{4}$	135	-130
1	$\bar{4}$	$\bar{4}$	287	375
1	5	4	37-	27
1	$\bar{5}$	4	46	-44
1	5	$\bar{4}$	170	178
1	$\bar{5}$	$\bar{4}$	151	176
1	6	4	53	-41
1	$\bar{6}$	4	339	349
1	6	$\bar{4}$	317	314
1	$\bar{6}$	$\bar{4}$	146	-163
1	7	4	79	89
1	$\bar{7}$	4	146	150
1	7	$\bar{4}$	37-	6
1	$\bar{7}$	$\bar{4}$	72	66
1	$\bar{8}$	4	29-	24
1	8	$\bar{4}$	79	-57
1	$\bar{8}$	$\bar{4}$	162	161
1	9	4	26-	-15
1	$\bar{9}$	$\bar{4}$	18-	-1
1	0	5	79	98
1	0	$\bar{5}$	245	267
1	1	5	44	-21
1	$\bar{1}$	5	359	395
1	1	$\bar{5}$	377	448
1	$\bar{1}$	$\bar{5}$	37-	-12
1	2	5	222	211
1	$\bar{2}$	5	188	183
1	2	$\bar{5}$	26-	16
1	$\bar{2}$	$\bar{5}$	26-	41
1	3	5	268	269
1	$\bar{3}$	5	173	165
1	3	$\bar{5}$	79	-54
1	$\bar{3}$	$\bar{5}$	302	406
1	4	5	33-	-4
1	$\bar{4}$	5	57	-12
1	4	$\bar{5}$	53	58
1	$\bar{4}$	$\bar{5}$	135	156
1	5	5	113	-99
1	$\bar{5}$	5	37-	-1
1	5	$\bar{5}$	124	117
1	$\bar{5}$	$\bar{5}$	53	-49
1	6	5	120	106
1	$\bar{6}$	5	124	108
1	6	$\bar{5}$	146	136
1	$\bar{6}$	$\bar{5}$	29-	22
1	7	5	113	97
1	$\bar{7}$	5	173	173
1	7	$\bar{5}$	29-	31
1	$\bar{7}$	$\bar{5}$	64	56
1	$\bar{8}$	5	64	28
1	8	$\bar{5}$	26-	11
1	$\bar{8}$	$\bar{5}$	86	77
1	$\bar{9}$	5	18-	-48
1	0	6	116	-108
1	0	$\bar{6}$	37-	7
1	1	6	98	100
1	$\bar{1}$	6	192	180
1	1	$\bar{6}$	294	333
1	$\bar{1}$	$\bar{6}$	83	122
1	2	6	324	332
1	$\bar{2}$	6	385	388
1	2	$\bar{6}$	181	201
1	$\bar{2}$	$\bar{6}$	211	261
1	3	6	33-	16
1	$\bar{3}$	6	90	89
1	3	$\bar{6}$	41	-27
1	$\bar{3}$	$\bar{6}$	57	71
1	4	6	33-	-30

h	k	l	$\|10F_o\|$	$10F_c$
1	$\bar{4}$	6	53	-39
1	4	$\bar{6}$	33-	16
1	$\bar{4}$	$\bar{6}$	83	79
1	5	6	37-	35
1	$\bar{5}$	6	222	209
1	5	$\bar{6}$	218	227
1	$\bar{5}$	$\bar{6}$	57	54
1	6	6	79	80
1	$\bar{6}$	6	90	93
1	6	$\bar{6}$	33	36
1	$\bar{6}$	$\bar{6}$	29-	-13
1	$\bar{7}$	6	37-	18
1	7	$\bar{6}$	57	-59
1	$\bar{7}$	$\bar{6}$	29-	11
1	$\bar{8}$	6	26-	-19
1	8	$\bar{6}$	18-	22
1	$\bar{8}$	$\bar{6}$	188	178
1	0	7	155	157
1	0	$\bar{7}$	351	381
1	1	7	139	-134
1	$\bar{1}$	7	131	137
1	1	$\bar{7}$	230	226
1	$\bar{1}$	$\bar{7}$	68	-84
1	2	7	33-	14
1	$\bar{2}$	7	200	193
1	2	$\bar{7}$	29	32
1	$\bar{2}$	$\bar{7}$	128	-149
1	3	7	290	281
1	$\bar{3}$	7	170	154
1	3	$\bar{7}$	83	75
1	$\bar{3}$	$\bar{7}$	166	190
1	4	7	131	126
1	$\bar{4}$	7	60	38
1	4	$\bar{7}$	79	81
1	$\bar{4}$	$\bar{7}$	233	277
1	5	7	79	-80
1	$\bar{5}$	7	37-	-6
1	5	$\bar{7}$	113	113
1	$\bar{5}$	$\bar{7}$	41	-50
1	6	7	44	42
1	$\bar{6}$	7	207	199
1	6	$\bar{7}$	90	88
1	$\bar{6}$	$\bar{7}$	29-	-13
1	$\bar{7}$	7	173	173
1	7	$\bar{7}$	26-	25
1	$\bar{7}$	$\bar{7}$	128	123
1	$\bar{8}$	7	79	-89
1	0	8	75	-66
1	0	$\bar{8}$	159	158
1	1	8	105	-104
1	$\bar{1}$	8	302	285
1	1	$\bar{8}$	290	284
1	$\bar{1}$	$\bar{8}$	90	-103
1	2	8	166	150
1	$\bar{2}$	8	268	247
1	2	$\bar{8}$	90	90
1	$\bar{2}$	$\bar{8}$	33-	14
1	3	8	226	223
1	$\bar{3}$	8	116	-105
1	3	$\bar{8}$	128	-124
1	$\bar{3}$	$\bar{8}$	90	106
1	4	8	60	58
1	$\bar{4}$	8	33-	-12
1	4	$\bar{8}$	116	114
1	$\bar{4}$	$\bar{8}$	113	139
1	5	8	53	44
1	$\bar{5}$	8	116	119
1	5	$\bar{8}$	192	196
1	$\bar{5}$	$\bar{8}$	155	171
1	6	8	7-	8
1	$\bar{6}$	8	207	202
1	6	$\bar{8}$	83	81
1	$\bar{6}$	$\bar{8}$	26-	18
1	$\bar{7}$	8	94	82
1	7	$\bar{8}$	22-	-17
1	$\bar{7}$	$\bar{8}$	26	-25
1	0	9	86	78
1	0	$\bar{9}$	155	147
1	1	9	166	157
1	$\bar{1}$	9	46-	29
1	1	$\bar{9}$	53	-24
1	$\bar{1}$	$\bar{9}$	155	181
1	2	9	33-	10
1	$\bar{2}$	9	120	101
1	2	$\bar{9}$	33-	38
1	$\bar{2}$	$\bar{9}$	68	-76
1	3	9	131	131
1	$\bar{3}$	9	105	97
1	3	$\bar{9}$	26	63
1	$\bar{3}$	$\bar{9}$	72	80
1	4	9	146	145
1	$\bar{4}$	9	79	-69
1	4	$\bar{9}$	53	76
1	$\bar{4}$	$\bar{9}$	215	246
1	5	9	18-	-45
1	$\bar{5}$	9	72	66
1	5	$\bar{9}$	109	107
1	$\bar{5}$	$\bar{9}$	68	75
1	$\bar{6}$	9	230	221
1	6	$\bar{9}$	113	105

h	k	l	$\|10F_o\|$	$10F_c$
1	$\bar{6}$	$\bar{9}$	101	-103
1	$\bar{7}$	9	18-	9
1	0	10	64	58
1	0	$\bar{10}$	143	140
1	1	10	72	-73
1	$\bar{1}$	10	298	283
1	1	$\bar{10}$	215	205
1	$\bar{1}$	$\bar{10}$	48-	7
1	2	10	128	122
1	$\bar{2}$	10	64	65
1	2	$\bar{10}$	79	-77
1	$\bar{2}$	$\bar{10}$	48	54
1	3	10	155	150
1	$\bar{3}$	10	60	-64
1	3	$\bar{10}$	57	-61
1	$\bar{3}$	$\bar{10}$	128	146
1	4	10	14-	4
1	$\bar{4}$	10	26-	6
1	4	$\bar{10}$	166	127
1	$\bar{4}$	$\bar{10}$	53	52
1	$\bar{5}$	10	143	133
1	5	$\bar{10}$	128	133
1	$\bar{5}$	$\bar{10}$	37	39
1	$\bar{6}$	10	64	62
1	6	$\bar{10}$	7-	4
1	0	11	44	43
1	0	$\bar{11}$	151	146
1	1	11	29-	-16
1	$\bar{1}$	11	222	178
1	1	$\bar{11}$	151	136
1	$\bar{1}$	$\bar{11}$	101	124
1	2	11	22-	-1
1	$\bar{2}$	11	226	217
1	2	$\bar{11}$	41	33
1	$\bar{2}$	$\bar{11}$	22-	-30
1	$\bar{3}$	11	44	-43
1	3	$\bar{11}$	37	-47
1	$\bar{3}$	$\bar{11}$	64	-79
1	4	11	113	-112
1	4	$\bar{11}$	22-	8
1	$\bar{4}$	$\bar{11}$	146	146
1	$\bar{5}$	11	120	135
1	5	$\bar{11}$	124	142
1	0	$\bar{12}$	237	230
1	$\bar{1}$	12	143	147
1	1	$\bar{12}$	90	83
1	$\bar{1}$	$\bar{12}$	33-	-11
1	$\bar{2}$	12	14-	-7
1	2	$\bar{12}$	83	-78
1	$\bar{2}$	$\bar{12}$	64	-69
1	$\bar{3}$	12	11-	-17
1	3	$\bar{12}$	37	44
1	$\bar{3}$	$\bar{12}$	18-	58
1	0	$\bar{13}$	11-	35
2	0	0	509	528
2	1	0	135	-118
2	$\bar{1}$	0	433	413
2	2	0	211	-188
2	$\bar{2}$	0	60	77
2	3	0	241	252
2	$\bar{3}$	0	18-	5
2	4	0	192	191
2	$\bar{4}$	0	165	160
2	5	0	33-	-11
2	$\bar{5}$	0	72	61
2	6	0	44	-12
2	$\bar{6}$	0	200	202
2	7	0	90	89
2	$\bar{7}$	0	86	86
2	$\bar{8}$	0	101	-105
2	$\bar{9}$	0	22-	11
2	0	1	26-	12
2	0	$\bar{1}$	128	138
2	1	1	53	42
2	$\bar{1}$	1	343	352
2	1	$\bar{1}$	37	63
2	$\bar{1}$	$\bar{1}$	302	295
2	2	1	22-	44
2	$\bar{2}$	1	237	248
2	2	$\bar{1}$	196	196
2	$\bar{2}$	$\bar{1}$	381	381
2	3	1	131	130
2	$\bar{3}$	1	48	-43
2	3	$\bar{1}$	113	106
2	$\bar{3}$	$\bar{1}$	105	-83
2	4	1	135	135
2	$\bar{4}$	1	162	161
2	4	$\bar{1}$	131	124
2	$\bar{4}$	$\bar{1}$	98	-87
2	5	1	101	110
2	$\bar{5}$	1	298	305
2	5	$\bar{1}$	33-	25
2	$\bar{5}$	$\bar{1}$	268	256
2	6	1	22-	-24
2	$\bar{6}$	1	124	134
2	6	$\bar{1}$	44	-40
2	$\bar{6}$	$\bar{1}$	173	160
2	7	1	14-	23
2	$\bar{7}$	1	113	-114
2	7	$\bar{1}$	57	51

h	k	l	$\|10F_o\|$	$10F_c$
2	$\bar{7}$	$\bar{1}$	26-	-21
2	$\bar{8}$	1	37	29
2	$\bar{8}$	$\bar{1}$	26-	-19
2	$\bar{9}$	1	66	84
2	$\bar{9}$	$\bar{1}$	113	117
2	0	2	135	166
2	0	$\bar{2}$	98	115
2	1	2	151	158
2	$\bar{1}$	2	60	52
2	1	$\bar{2}$	113	-91
2	$\bar{1}$	$\bar{2}$	359	370
2	2	2	57	-60
2	$\bar{2}$	2	18-	31
2	2	$\bar{2}$	283	268
2	$\bar{2}$	$\bar{2}$	18-	21
2	3	2	159	151
2	$\bar{3}$	2	241	246
2	3	$\bar{2}$	317	306
2	$\bar{3}$	$\bar{2}$	18-	-4
2	4	2	264	275
2	$\bar{4}$	2	72	80
2	4	$\bar{2}$	68	75
2	$\bar{4}$	$\bar{2}$	128	127
2	5	2	37-	26
2	$\bar{5}$	2	181	188
2	5	$\bar{2}$	72	-71
2	$\bar{5}$	$\bar{2}$	181	174
2	6	2	33	-52
2	$\bar{6}$	2	207	207
2	6	$\bar{2}$	37	33
2	$\bar{6}$	$\bar{2}$	41	42
2	7	2	37	44
2	$\bar{7}$	2	44	-26
2	7	$\bar{2}$	44	48
2	$\bar{7}$	$\bar{2}$	109	113
2	$\bar{8}$	2	48	-37
2	$\bar{8}$	$\bar{2}$	44	45
2	$\bar{9}$	2	113	114
2	$\bar{9}$	$\bar{2}$	14-	26
2	0	3	226	236
2	0	$\bar{3}$	26-	22
2	1	3	68	-65
2	$\bar{1}$	3	407	447
2	1	$\bar{3}$	139	143
2	$\bar{1}$	$\bar{3}$	181	165
2	2	3	120	117
2	$\bar{2}$	3	48	-44
2	2	$\bar{3}$	22-	10
2	$\bar{2}$	$\bar{3}$	260	263
2	3	3	98	117
2	$\bar{3}$	3	215	-236
2	3	$\bar{3}$	143	152
2	$\bar{3}$	$\bar{3}$	94	95
2	4	3	131	128
2	$\bar{4}$	3	146	147
2	4	$\bar{3}$	245	246
2	$\bar{4}$	$\bar{3}$	188	-175
2	5	3	120	109
2	$\bar{5}$	3	343	377
2	5	$\bar{3}$	41	43
2	$\bar{5}$	$\bar{3}$	192	186
2	6	3	18-	30
2	$\bar{6}$	3	33	40
2	6	$\bar{3}$	37	-46
2	$\bar{6}$	$\bar{3}$	370	364
2	$\bar{7}$	3	26-	11
2	7	$\bar{3}$	48	49
2	$\bar{7}$	$\bar{3}$	26-	29
2	$\bar{8}$	3	44	56
2	$\bar{8}$	$\bar{3}$	86	-79
2	9	3	79	80
2	$\bar{9}$	$\bar{3}$	37	34
2	0	4	279	326
2	0	$\bar{4}$	48	-34
2	1	4	166	170
2	$\bar{1}$	4	131	131
2	1	$\bar{4}$	109	-97
2	$\bar{1}$	$\bar{4}$	359	372
2	2	4	37	-26
2	$\bar{2}$	4	101	112
2	2	$\bar{4}$	109	112
2	$\bar{2}$	$\bar{4}$	268	266
2	3	4	29-	0
2	$\bar{3}$	4	41	-43
2	3	$\bar{4}$	226	231
2	$\bar{3}$	$\bar{4}$	72	-61
2	4	4	200	199
2	$\bar{4}$	4	37	42
2	4	$\bar{4}$	53	54
2	$\bar{4}$	$\bar{4}$	72	77
2	5	4	109	103
2	$\bar{5}$	4	200	221
2	5	$\bar{4}$	41	36
2	$\bar{5}$	$\bar{4}$	170	169
2	6	4	60	-60
2	$\bar{6}$	4	101	116
2	6	$\bar{4}$	33	40
2	$\bar{6}$	$\bar{4}$	173	170
2	$\bar{7}$	4	44	-47
2	7	$\bar{4}$	79	83

Table 4 (*cont.*)

h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c
2 $\bar{7}$ $\bar{4}$	57	54	2 3 $\bar{7}$	192	198	2 $\bar{4}$ $\bar{10}$	215	216	3 3 $\bar{2}$	96	81	3 $\bar{1}$ 5	56	59	3 $\bar{2}$ $\bar{8}$	162	159	4 $\bar{5}$ 2	29	34
2 $\bar{8}$ $\bar{4}$	22-	-4	2 3 7	75	74	2 4 $\bar{10}$	44	37	3 3 2	113	86	3 1 5	113	116	3 2 8	22-	-16	4 5 2	33-	1
2 8 4	22-	-1	2 4 7	173	183	2 4 10	53	-53	3 4 2	26	30	3 $\bar{1}$ 5	44	33	3 2 8	26	23	4 6 2	143	170
2 0 5	290	324	2 4 7	173	177	2 5 $\bar{10}$	11-	43	3 4 2	222	197	3 2 5	162	134	3 3 8	60	53	4 6 2	18-	-7
2 0 5	143	151	2 4 $\bar{7}$	41	43	2 5 10	3-	6	3 4 2	29	24	3 3 5	90	74	3 3 8	14-	7	4 0 3	26-	17
2 1 5	41-	-10	2 4 $\bar{7}$	29-	-29	2 5 10	60	-59	3 4 2	339	285	3 2 5	75	-74	3 3 8	53	-56	4 0 3	44-	-27
2 $\bar{1}$ 5	188	208	2 5 7	135	145	2 0 11	29-	-33	3 5 2	68	59	3 2 5	64	-53	3 4 8	26	-23	4 1 3	37-	-68
2 1 5	37	23	2 5 7	53	-56	2 1 11	14-	-60	3 5 2	64	-54	3 3 5	44	37	3 4 8	109	100	4 $\bar{1}$ 3	159	216
2 $\bar{1}$ 5	37-	29	2 5 $\bar{7}$	33-	-24	2 1 $\bar{11}$	33-	18	3 5 2	57	55	3 3 5	203	209	3 4 8	26-	26	4 1 3	94	90
2 2 5	29-	-30	2 6 7	72	67	2 1 $\bar{11}$	98	101	3 5 2	29-	-17	3 3 5	26	-23	3 5 8	26	-27	4 $\bar{1}$ 3	53-	-25
2 2 5	72	-75	2 6 $\bar{7}$	75	66	2 2 11	11-	45	3 6 2	72	70	3 3 5	181	165	3 5 8	170	160	4 2 3	94	125
2 2 5	57	54	2 6 7	116	118	2 2 $\bar{11}$	181	174	3 6 2	105	-81	3 4 5	44	-48	3 6 8	105	94	4 2 3	83	81
2 2 5	207	223	2 7 7	22-	5	2 2 11	109	124	3 7 2	143	131	3 4 5	124	127	3 0 9	79	79	4 2 3	159	159
2 3 5	173	174	2 7 $\bar{7}$	185	188	2 3 $\bar{11}$	33	30	3 7 2	113	88	3 4 5	128	119	3 1 9	11-	-43	4 3 3	94	-120
2 $\bar{3}$ 5	60	73	2 0 8	44	44	2 $\bar{3}$ 11	33	32	3 8 2	75	69	3 4 5	159	144	3 1 9	94	-76	4 $\bar{3}$ 3	14-	42
2 3 5	226	223	2 0 $\bar{8}$	146	-151	2 4 $\bar{11}$	22	28	3 $\bar{8}$ 2	181	164	3 5 5	44	-54	3 1 9	143	147	4 3 3	151	144
2 $\bar{3}$ 5	257	270	2 1 8	86	91	2 0 12	22	28	3 0 3	75	71	3 5 5	86	76	3 2 9	33	32	4 4 3	29	-30
2 4 5	60	60	2 $\bar{1}$ 8	94	83	2 1 $\bar{12}$	113	111	3 0 3	196	176	3 5 5	109	90	3 3 9	26	-33	4 4 3	139	-124
2 4 5	328	362	2 1 $\bar{8}$	173	176	2 $\bar{1}$ $\bar{12}$	44	-49	3 1 3	192	158	3 6 5	53	-61	3 3 9	113	91	4 5 3	94	116
2 4 5	64	68	2 $\bar{1}$ 8	44-	-14	2 2 12	14-	17	3 $\bar{1}$ 3	83	66	3 6 5	37	34	3 5 9	44	-49	4 5 3	33	-28
2 4 5	44	56	2 2 8	155	152	3 0 0	257	203	3 1 3	196	183	3 7 5	173	158	3 4 9	143	136	4 6 3	72	75
2 5 5	26-	6	2 2 $\bar{8}$	26-	2	3 1 0	139	130	3 $\bar{1}$ 3	120	-89	3 7 5	22-	-10	3 5 9	135	119	4 6 3	166	159
2 5 5	48	38	2 2 8	203	198	3 $\bar{1}$ 0	11-	15	3 2 3	192	155	3 8 5	11-	-10	3 0 10	75	63	4 0 4	29-	42
2 5 5	90	-99	2 2 8	181	253	3 2 0	26-	18	3 2 3	33	-17	3 0 6	86	-74	3 1 10	26-	20	4 0 4	41	-65
2 5 5	64	-71	2 3 8	18-	-20	3 2 0	44	-34	3 2 3	26-	-1	3 0 6	44	45	3 1 10	48	50	4 1 4	26-	2
2 6 5	57	21	2 3 8	29-	-21	3 3 0	33	-21	3 3 3	37	33	3 1 6	162	139	3 2 10	48	-46	4 $\bar{1}$ 4	90	99
2 6 5	72	-78	2 3 8	79	72	3 3 0	359	290	3 3 3	33	38	3 $\bar{1}$ 6	44	-42	3 3 10	57	55	4 1 4	44-	24
2 6 5	48	16	2 3 8	98	116	3 4 0	44	45	3 $\bar{3}$ 3	101	79	3 1 6	113	111	3 $\bar{3}$ 10	98	61	4 $\bar{1}$ 4	53	72
2 6 5	159	165	2 4 8	37	36	3 4 0	268	223	3 3 3	26-	17	3 $\bar{1}$ 6	143	136	3 4 10	14-	-5	4 2 4	128	162
2 7 5	26-	-1	2 4 8	177	180	3 5 0	90	77	3 $\bar{3}$ 3	185	154	3 2 6	196	169	3 0 11	57	48	4 2 4	105	108
2 7 5	124	177	2 4 $\bar{8}$	26-	16	3 $\bar{5}$ 0	44	-17	3 4 3	64	-63	3 2 6	203	203	3 1 11	14-	-8	4 2 4	101	105
2 7 5	113	108	2 4 8	94	-105	3 6 0	90	-73	3 4 3	218	208	3 2 6	26-	17	3 1 11	173	158	4 3 4	22-	2
2 8 5	79	81	2 5 8	196	210	3 7 0	139	108	3 4 3	135	120	3 2 6	120	115	3 2 11	48	41	4 3 4	48	45
2 8 5	86	-84	2 5 8	26-	17	3 8 0	98	90	3 4 3	155	124	3 3 6	57	-54	4 0 0	44	49	4 4 4	60	-72
2 0 6	75	102	2 5 8	57	69	3 9 0	3-	30	3 5 3	64	61	3 3 6	105	105	4 1 0	48-	-72	4 4 4	29-	21
2 0 6	37	-36	2 6 8	101	-101	3 0 1	79	-56	3 5 3	29-	-1	3 3 6	22-	-17	4 $\bar{1}$ 0	120	127	4 5 4	64	58
2 1 6	188	185	2 6 $\bar{8}$	33	35	3 0 $\bar{1}$	139	127	3 $\bar{5}$ 3	94	95	3 $\bar{3}$ 6	26-	14	4 2 0	18-	2	4 5 4	33	29
2 $\bar{1}$ 6	109	128	2 6 8	162	169	3 1 1	94	94	3 5 3	101	35	3 4 6	44	40	4 3 0	75	90	4 6 4	57	52
2 1 6	41-	-3	2 7 8	14-	-23	3 $\bar{1}$ 1	33	28	3 6 3	53	-44	3 4 6	90	81	4 3 0	83	96	4 0 5	37-	34
2 $\bar{1}$ 6	230	247	2 $\bar{7}$ 8	14-	30	3 1 $\bar{1}$	366	346	3 6 3	7-	-1	3 4 6	64	61	4 4 0	33	31	4 $\bar{1}$ 5	124	150
2 2 6	44	-43	2 0 9	185	198	3 $\bar{1}$ 1	60-	-4	3 6 3	68	63	3 5 6	98	91	4 5 0	44	-58	4 1 5	68	67
2 2 6	94	106	2 0 9	48	-45	3 2 1	207	202	3 7 3	131	117	3 5 6	124	84	4 6 0	124	132	4 1 5	53	-58
2 2 6	245	263	2 1 9	173	177	3 2 1	222	190	3 $\bar{7}$ 3	41	37	3 $\bar{5}$ 6	155	147	4 0 1	116	-87	4 2 5	18-	-3
2 2 6	275	323	2 $\bar{1}$ 9	75	-78	3 2 $\bar{1}$	37	36	3 8 3	196	181	3 6 6	44	47	4 0 $\bar{1}$	41-	-42	4 2 5	18-	43
2 3 6	68	-61	2 1 9	41-	-3	3 2 $\bar{1}$	83	75	3 8 3	101	78	3 6 6	22-	-13	4 1 1	44-	1	4 2 5	60	41
2 3 6	37	34	2 $\bar{1}$ 9	57	63	3 3 1	44	-29	3 0 4	33-	-4	3 7 6	44	43	4 $\bar{1}$ 1	79	103	4 3 5	14-	3
2 3 6	109	111	2 2 9	18-	-4	3 3 1	128	114	3 0 4	159	143	3 7 6	101	-93	4 1 $\bar{1}$	48-	45	4 3 5	181	192
2 3 6	26-	-28	2 2 9	26-	-21	3 3 $\bar{1}$	166	-145	3 1 4	283	247	3 0 7	44	36	4 $\bar{1}$ $\bar{1}$	60	64	4 4 5	131	124
2 4 6	131	126	2 2 9	166	156	3 3 $\bar{1}$	200	138	3 $\bar{1}$ 4	101	-79	3 0 $\bar{7}$	253	256	4 2 1	120	159	4 4 5	86	77
2 4 6	135	129	2 2 9	72	94	3 4 1	26-	-11	3 1 4	57	43	3 1 7	26-	20	4 2 $\bar{1}$	79	99	4 5 5	75	-81
2 4 6	29-	-8	2 3 9	11-	-7	3 4 $\bar{1}$	226	171	3 $\bar{1}$ 4	166	145	3 $\bar{1}$ 7	33	-28	4 3 1	211	234	4 6 5	68	68
2 4 6	68	-66	2 3 9	124	126	3 4 $\bar{1}$	37	43	3 2 4	75	86	3 1 $\bar{7}$	48	43	4 3 $\bar{1}$	26-	10	4 0 6	48	-52
2 5 6	143	156	2 3 9	155	154	3 4 $\bar{1}$	98	72	3 2 4	215	211	3 $\bar{1}$ $\bar{7}$	113	116	4 3 1	14-	36	4 1 6	57	54
2 5 6	207	227	2 3 9	86	103	3 5 1	131	130	3 2 4	79	74	3 2 7	86	66	4 3 $\bar{1}$	41	47	4 $\bar{1}$ 6	33-	20
2 5 6	33	-16	2 4 9	135	136	3 5 $\bar{1}$	131	121	3 2 4	64	-54	3 2 $\bar{7}$	86	63	4 4 1	29	-33	4 2 6	124	152
2 5 6	124	133	2 4 9	53	-45	3 5 $\bar{1}$	166	134	3 3 4	33	-32	3 2 $\bar{7}$	86	-79	4 4 $\bar{1}$	72	-70	4 3 6	60	64
2 6 6	68	70	2 4 9	116	124	3 $\bar{5}$ 1	166	139	3 3 4	302	308	3 2 7	101	-104	4 5 1	98	124	4 4 6	26-	21
2 6 6	41	41	2 5 9	22-	13	3 6 1	90	-90	3 3 4	33	-24	3 3 7	181	187	4 5 $\bar{1}$	44	42	4 5 6	33	25
2 6 6	59	69	2 5 9	18-	-13	3 8 1	33	25	3 3 4	105	-69	3 3 $\bar{7}$	75	75	4 6 1	105	121	4 6 6	7-	-5
2 7 6	120	-135	2 5 9	26-	-15	3 $\bar{7}$ 1	68	57	3 4 4	37	42	3 3 $\bar{7}$	26-	15	4 6 $\bar{1}$	120	118	4 0 7	57	43
2 $\bar{7}$ 6	48	48	2 6 9	22	-26	3 7 $\bar{1}$	101	-82	3 4 4	86	-78	3 4 $\bar{7}$	94	82	4 0 2	60	47	4 1 7	33-	27
2 8 6	48	49	2 6 9	60	28	3 8 1	203	185	3 4 4	41	41	3 4 $\bar{7}$	101	93	4 0 2	33	4	4 $\bar{1}$ 7	41-	34
2 8 6	37	35	2 0 10	173	190	3 8 $\bar{1}$	101	97	3 4 4	309	264	3 4 7	173	161	4 1 2	41-	40	4 2 7	18-	38
2 0 7	275	295	2 0 $\bar{10}$	44	34	3 0 2	188	179	3 5 4	101	-101	3 5 7	124	-128	4 $\bar{1}$ 2	48-	10	4 3 7	90	100
2 0 7	86	101	2 1 10	48	57	3 0 $\bar{2}$	241	218	3 5 4	143	141	3 5 $\bar{7}$	53	35	4 1 $\bar{2}$	48-	-4	4 4 7	48	43
2 1 7	109	104	2 $\bar{1}$ 10	124	130	3 1 2	230	215	3 5 4	287	254	3 5 $\bar{7}$	26-	19	4 $\bar{1}$ 2	90	99	4 5 7	68	-74
2 $\bar{1}$ 7	68	67	2 1 $\bar{10}$	79	66	3 $\bar{1}$ 2	181	-155	3 6 4	79	74	3 6 7	94	86	4 2 2	14-	39	4 0 8	26-	-11
2 1 7	44	-38	2 $\bar{1}$ 10	41-	-30	3 1 $\bar{2}$	135	118	3 6 4	101	-84	3 6 $\bar{7}$	14-	15	4 2 2	57	68	4 $\bar{1}$ 8	33-	-58
2 $\bar{1}$ 7	188	217	2 2 10	14	4	3 $\bar{1}$ 2	124	103	3 $\bar{7}$ 4	113	110	3 7 $\bar{7}$	57	35	4 2 2	146	152	4 2 8	64	72
2 2 7	155	-143	2 2 $\bar{10}$	86	-84	3 2 2	26-	44	3 7 4	22	-21	3 0 8	109	-106	4 2 2	90	84	4 3 8	72	93
2 $\bar{2}$ 7	151	-153	2 2 $\bar{10}$	64	35	3 2 $\bar{2}$	44	-28	3 $\bar{8}$ 4	113	106	3 0 8	146	139	4 3 2	79	98	4 4 8	14-	-28
2 2 $\bar{7}$	116	128	2 $\bar{2}$ 10	155	186	3 2 2	33	41	3 8 4	131	105	3 1 8	44	37	4 3 $\bar{2}$	53	44	4 $\bar{1}$ 9	14-	-10
2 2 7	60	76	2 3 10	18-	-18	3 2 $\bar{2}$	124	-107	3 0 5	44	44	3 $\bar{1}$ 8	83	75	4 4 2	46	-59	4 2 9	7-	-24
2 3 7	26-	17	2 3 $\bar{10}$	44	44	3 3 2	22-	27	3 0 5	222	220	3 1 8	79	87	4 4 2	64	53			
2 3 7	124	121	2 3 $\bar{10}$	159	174	3 3 2	272	244	3 1 5	68	43	3 $\bar{1}$ 8	64	46						

The C–S distance in a thiourea molecule is just significantly longer than in free thiourea ($1{\cdot}707 \pm 0{\cdot}012$ Å, Kunchur & Truter, 1958), so the previous conclusion of NBF that coordination causes an increasing of the single bond character for that bond, seems to remain valid. This view has been supported also by the study of the infrared spectrum of $Ni[SC(NH_2)_2]_2(NCS)_2$ (Yamaguchi, Penland, Mizushima, Lane, Curran & Quagliano, 1958). Considering the table of C–S and C–N distances in various thioamide-metal complexes, made by Lopez-Castro & Truter (1963) and the results of the refinement of the structure of $Cu[SC(NH_2)_2]_3Cl$ (Okaya & Knobler, 1964), it appears that, among these complexes, only in $Ni[SC(NH_2)_2]_2(NCS)_2$ and in $Zn[SC(NH_2)_2]_2Cl_2$ (Kunchur & Truter, 1958) is a small but significant increase of the C–S bond length observed.

The two C–N distances in the thiourea molecule are not significantly different and the same can be said of the two S–C–N angles. Bond angles around the S atom of thiourea show severe distortions from the tetrahedral values, and the orientation of the thiourea molecule is determined by the packing of the coordination polyhedra chains; the dihedral angle formed by the plane of thiourea and the plane of S(2) atoms in a chain is 79·8°.

Bond distances in NCS indicate a prevailing double-bond character both in C–S and in N–C bonds, in agreement with an isothiocyanate structure. The lack of linearity in the NCS group is not significant; the line through NCS makes an angle of 64·3° with the plane of S(2) atoms in a chain.

The present refinement confirms the previous (NBF) observation that the van der Waals radius of S (1·85 Å) proposed by Pauling is too long. The contacts S(2)–S(2″) = 3·396 ± 0·007 and S(1)–S(1^{vii}) = 3·446 ± 0·008 Å are not consistent with Pauling's value, while they are in good agreement with the value of 3·43 Å found by Ždanov & Zvonkova (1950) in $K_2Co(NCS)_4 . 4H_2O$, and of 3·47 Å found by van der Helm, Lessor & Merritt (1960) in rhodanine. The other contacts shorter than 3·5 Å are:

N(1)–N(2)	3·07 ± 0·02 Å
N(2)–N(1′)	3·25 ± 0·02
N(1)–N(3‴)	3·46 ± 0·02
N(2)–N(3^{iv})	3·34 ± 0·02
C(1)–N(2)	3·51 ± 0·02
N(2)–C(1′)	3·24 ± 0·02
C(1)–N(3‴)	3·22 ± 0·02
C(2)–N(3^{iv})	3·34 ± 0·02
N(1)–C(2^{iv})	3·33 ± 0·02
S(1)–N(2^{v})	3·47 ± 0·01
S(1)–N(3^{vi})	3·48 ± 0·01

′	$1+x, y, z$	v	$1-x, \bar{y}, 1-z$
″	$1-x, \bar{y}, \bar{z}$	vi	$x-1, 1+y, z$
‴	$x, 1+y, z$	vii	$\bar{x}, 1-y, 1-z$
iv	$x-1, y, z$		

All the calculations were performed on the Olivetti Elea 6001/S computer of the *Centro di Calcolo Elettronico della Università di Parma* using the programs of Nardelli, Musatti, Domiano & Andreetti (1965). The authors are indebted to the *Consiglio Nazionale delle Ricerche* for financial support.

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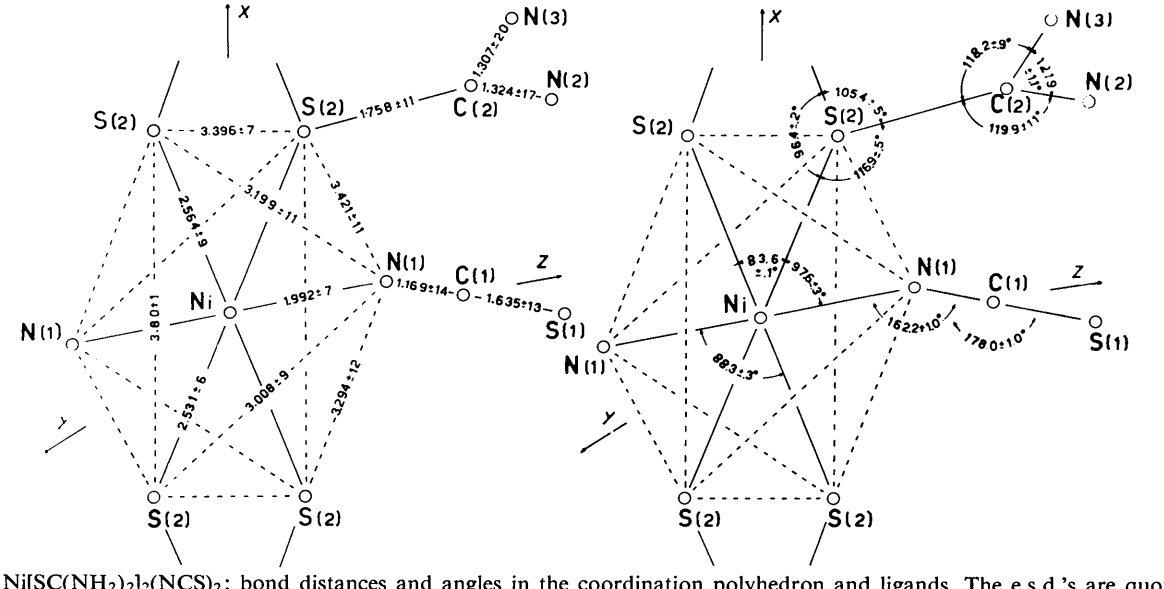

Fig. 1. $Ni[SC(NH_2)_2]_2(NCS)_2$: bond distances and angles in the coordination polyhedron and ligands. The e.s.d.'s are quoted in units of the last place.