Tableau 4 (*suite*)

h	*k*	*l*	F_{obs}	F_{calc}
15	6	0	30,0	+26,3
16	3	0	5,0	−7,5
12	11	0	20,0	+19,1
13	10	0	25,0	+26,9
16	4	0	5,0	+8,7
15	7	0	20,0	+22,6
14	9	0	20,0	−22,1
16	5	0	10,0	−9,1
12	12	0	49,5	+41,7
1	1	1	54,1	−33,1
2	2	1	147,2	−175,1
3	3	1	163,8	+145,7
6	6	1	103,6	−118,2
1	2	1	38,3	+35,0
1	3	1	44,3	+65,5
1	4	1	26,5	−12,2
1	6	1	41,0	+38,3
1	7	1	129,7	−172,7
2	3	1	155,7	+140,9
2	5	1	35,8	−11,3

Tableau 4 (*suite*)

h	*k*	*l*	F_{obs}	F_{calc}
2	6	1	41,8	+64,0
2	7	1	93,5	+100,5
3	4	1	142,6	+99,3
3	7	1	62,0	−63,5
3	8	1	74,8	+75,7
4	5	1	77,9	−85,3
4	8	1	77,2	+74,6
3	0	1	25,5	−2,6
7	0	1	110,6	+132,7
9	0	1	54,6	−72,4

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The Crystal and Molecular Structure of Tetra(thiourea)nickel(II) Thiosulphate Monohydrate

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Tetra(thiourea)nickel(II) thiosulphate monohydrate is orthorhombic, $P2_12_12_1$: $a=19{\cdot}07(2)$, $b=10{\cdot}42(1)$, $c=8{\cdot}91(1)$ Å, $Z=4$. The crystal structure has been determined at room temperature from three-dimensional X-ray photographic data and refined by differential methods using anisotropic thermal parameters; final $R=8{\cdot}9\%$. Ni coordinates to four sulphur atoms from four thiourea molecules, and to a sulphur and to an oxygen atom from a thiosulphate group; this represents a distorted octahedral environment, with the $S_2O_3^{2-}$ group behaving as a bidentate ligand. Coordination does not seem to influence the shape and dimensions of thiourea ligands. The water molecule is hydrogen-bonded to two oxygen atoms from two thiosulphate groups (OH···O = 2·82 and 2·89 Å) and to two nitrogen atoms from a thiourea molecule (NH···O = 2·97 and 2·95 Å).

Introduction

Thiourea and ethylenethiourea adducts with thiosulphates of divalent metals have been prepared and described by Nardelli & Chierici (1958). An X-ray study of these compounds should give the type of metal coordination and, in particular, should define the structural situation of the $S_2O_3^{2-}$ group, which can coordinate as a mono or bidentate ligand or can form bridges between different metal atoms. Also it should be possible to examine whether coordination influences the bond distances and angles in the molecule. In the present paper the results obtained from the crystal structure analysis of tetra(thiourea)nickel(II) thiosulphate monohydrate, $Nitu_4S_2O_3.H_2O$ (*tu* = thiourea) are reported and discussed. As already published in a short account of this study (Fava Gasparri, Musatti & Nardelli, 1966), the thiosulphate group behaves as a bidentate ligand; this is the first direct confirmation of the chelating properties of this ligand. The infrared spectrum, observed by Newman (1967), can be interpreted satisfactorily on the basis of this evidence. The anhydrous formula previously assigned to this compound is incorrect: the water molecule, which was overlooked in the chemical analysis, was found from the electron-density distribution, determined in the present study.

Experimental

Cell constants, refined by a least-squares analysis of powder diffractometer data, are the same as those reported by Nardelli & Chierici (1958) (standard deviations are given in parentheses):

$Ni[SC(NH_2)_2]_4S_2O_3.H_2O$. $M=493{\cdot}35$

$a=19{\cdot}07(2)$, $b=10{\cdot}42(1)$, $c=8{\cdot}98(1)$ Å

$V=1784$ Å^3, $Z=4$, $D_m=1{\cdot}77$, $D_x=1{\cdot}82$ g.cm^{-3}

$\mu=82$ cm^{-1} (Cu $K\alpha$),

$F(000)=1016$.

Space group: $P2_12_12_1$ (D_2^4) (from systematic X-ray extinctions).

Two series of equi-inclination Weissenberg photographs (Cu $K\alpha$ radiation, multiple film technique) were taken at room temperature about [010] up to $k=8$ and about [001] up to $l=7$. 1996 independent reflexions were observed out of a possible 2283. The intensities were measured photometrically and corrected for Lorentz, polarization and spot-shape effects (Phillips, 1956). For the photographs taken around [010] the sample used was a nearly spherical fragment with a mean radius of 0·02 cm and was considered to be a sphere in calculating the absorption correction; the photographs around [001] were taken using a prism elongated along this axis, and for this set of data the cylindrical absorption correction was used, treating the sample as a cylinder with a mean radius of 0·014 cm. The data of both series were correlated and put on a common scale using the least-squares procedure of Rollett & Sparks (1960); the absolute scale determined by Wilson's (1942) method remained unchanged throughout the analysis.

Structure determination and refinement

The coordinates of Ni, S(2) and S(5) were found from a three-dimensional Patterson synthesis. A three-dimensional Fourier synthesis calculated using the phases of the contribution of these atoms to the structure factors ($R=49\%$) was used to determine the coordinates of the other sulphur atoms; the coordinates of the complete set of atoms were obtained after three further cycles of three-dimensional Fourier synthesis ($R=15{\cdot}9\%$). At this stage of the analysis, the electron-density distribution showed an additional peak; from its height and environment, this peak could be interpreted as representing a water molecule. By including this molecule when calculating the structure factors the R value fell to 14%. Refinement was then carried out by means of four cycles of Booth's differential synthesis, two calculated with isotropic, and two with anisotropic, thermal parameters. The final residual error indices were: $R=8{\cdot}9\%$, $R'=9{\cdot}8\%$ (R, for observed reflexions only; R', including $F_o=\frac{1}{2}F_{\min}$ when $F_c \geq F_{\min}$ for unobserved reflexions; multiplicities not considered). In Table 1 the final parameters with their estimated standard deviations (Cruickshank, 1949, 1956) and the ratios between the e.s.d.'s and the shifts of the coordinates are quoted. The B_{ij}'s were determined by the method of Nardelli & Fava (1960) using the second derivatives of the electron density from differential synthesis.

An F_o-F_c synthesis, carried out after the refinement, showed that the positions of the H's, determined from the assumptions of bond distances N–H = 1·03; O–H = 0·95 Å, planarity for thiourea, and tetrahedral environment for H_2O, were essentially correct. The hydrogen coordinates are reported in Table 2 with the observed electron density.

In Table 3 the observed atomic peak shapes for non-hydrogen atoms are compared with the calculated values. Observed and calculated (not including H atoms) structure factors are reported in Table 4. The atomic scattering factors used are those of Thomas & Umeda (1957) for Ni^{2+}, Dawson (1960) for S and Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for O, N and C.

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 (1964, 1965).

Table 1. *Final atomic fractional coordinates* ($\times 10^4$), *thermal parameters* ($\times 10$ Å^2) *with estimated standard deviations and ratios* (*e.s.d.*)/(*coordinate shift*)

	$x(\sigma)$	$y(\sigma)$	$z(\sigma)$	$B_{11}(\sigma)$	$B_{22}(\sigma)$	$B_{33}(\sigma)$	$B_{12}(\sigma)$	$B_{13}(\sigma)$	$B_{23}(\sigma)$	$\lvert r(x)\rvert$	$\lvert r(y)\rvert$	$\lvert r(z)\rvert$
Ni	964 (1)	783 (2)	636 (2)	30 (1)	29 (1)	30 (1)	0 (1)	0 (1)	0 (1)	6	5	2
S(1)	−151 (2)	367 (3)	1774 (4)	22 (1)	23 (1)	25 (2)	0 (2)	2 (2)	3 (2)	4	3	10
S(2)	1050 (2)	−1484 (3)	208 (4)	27 (1)	20 (1)	23 (2)	0 (2)	2 (2)	1 (2)	5	3	2
S(3)	616 (2)	1457 (3)	−1862 (4)	25 (1)	21 (1)	22 (1)	−1 (2)	−1 (2)	0 (2)	3	7	9
S(4)	1573 (2)	168 (3)	2955 (4)	23 (1)	20 (1)	21 (1)	0 (2)	0 (2)	0 (2)	2	15	∞
S(5)	2184 (2)	1843 (3)	−322 (4)	23 (1)	20 (1)	23 (1)	1 (2)	−1 (2)	0 (2)	9	5	18
S(6)	1653 (2)	3330 (3)	550 (3)	18 (1)	17 (1)	18 (1)	0 (1)	−1 (2)	0 (2)	∞	6	16
O(1)	990 (6)	2756 (9)	1144 (9)	21 (4)	18 (3)	18 (4)	2 (4)	0 (5)	−2 (5)	∞	4	4
O(2)	1497 (6)	4245 (11)	−638 (13)	30 (5)	22 (4)	28 (5)	2 (6)	0 (7)	3 (7)	8	3	32
O(3)	2046 (7)	3914 (10)	1721 (16)	26 (4)	25 (3)	28 (5)	−2 (5)	0 (6)	−1 (6)	5	5	4
O(4)	2992 (9)	6068 (12)	1437 (14)	44 (7)	34 (4)	33 (6)	−3 (8)	2 (9)	−3 (8)	12	5	5
N(1)	−1423 (7)	1323 (16)	1655 (16)	28 (5)	33 (6)	30 (7)	2 (8)	0 (8)	3 (9)	3	6	3
N(2)	−560 (9)	2693 (13)	918 (21)	32 (5)	29 (6)	40 (9)	4 (9)	1 (10)	4 (11)	4	7	2
N(3)	1662 (8)	−1039 (12)	−2391 (16)	33 (6)	32 (5)	28 (7)	−3 (8)	5 (9)	2 (9)	10	6	5
N(4)	1758 (9)	−3063 (12)	−1532 (18)	36 (7)	27 (4)	34 (8)	−2 (8)	5 (10)	1 (9)	3	∞	3
N(5)	−74 (10)	555 (13)	−4142 (12)	39 (7)	37 (5)	36 (7)	−2 (9)	−3 (9)	−4 (9)	3	3	4
N(6)	−317 (10)	−440 (12)	−1917 (15)	43 (7)	40 (6)	34 (8)	−10 (9)	2 (10)	3 (11)	8	7	2
N(7)	2065 (8)	1227 (12)	5400 (17)	29 (5)	38 (6)	30 (6)	1 (8)	−2 (8)	−2 (9)	8	60	4
N(8)	1206 (8)	2375 (12)	4288 (14)	30 (6)	25 (5)	28 (6)	3 (8)	1 (8)	0 (8)	8	∞	11
C(1)	−745 (7)	1555 (12)	1433 (13)	23 (5)	22 (4)	19 (5)	−2 (7)	0 (7)	−1 (7)	3	3	7
C(2)	1514 (8)	−1894 (11)	−1351 (16)	29 (6)	21 (4)	26 (7)	0 (8)	3 (9)	0 (9)	5	6	16
C(3)	22 (8)	479 (13)	−2693 (15)	27 (6)	29 (5)	28 (7)	−1 (8)	1 (9)	−6 (8)	19	8	76
C(4)	1609 (7)	1333 (12)	4283 (15)	22 (5)	22 (4)	21 (6)	−2 (7)	1 (7)	1 (8)	3	7	12

Table 2. *Calculated fractional coordinates and corresponding ϱ_{obs} values for hydrogen atoms*

	x	y	z	ϱ_{obs}
H(1)	−0·158	0·044	0·205	1·0(e.Å^{-3})
H(2)	−0·178	0·204	0·145	0·9
H(3)	−0·004	0·290	0·073	0·8
H(4)	−0·093	0·339	0·072	0·7
H(5)	0·149	−0·011	−0·229	0·8
H(6)	0·194	−0·131	−0·332	0·7
H(7)	0·165	−0·377	−0·075	0·5
H(8)	0·203	−0·330	−0·248	0·7
H(9)	−0·043	−0·004	−0·465	0·9
H(10)	0·019	0·125	−0·475	0·6
H(11)	−0·024	−0·052	−0·078	0·8
H(12)	−0·067	−0·103	−0·244	0·9
H(13)	0·209	0·193	0·621	0·5
H(14)	0·239	0·044	0·546	0·7
H(15)	0·125	0·305	0·513	0·5
H(16)	0·084	0·252	0·345	0·7
H(17)	0·271	0·533	0·132	0·7
H(18)	0·331	0·587	0·220	0·6

Discussion

The structure consists of complex molecules in which a nickel atom is octahedrally surrounded by four sulphur atoms from thiourea molecules and by one sulphur and one oxygen atom from a thiosulphate group. The H_2O molecule acts as a bridge connecting three different complexes as shown in Fig. 1.

Distances and angles in the coordination polyhedron are:

Ni–S(1) = 2·396 (4) Å Ni–S(5) = 2·718 (4) Å
Ni–S(2) = 2·399 (4) Ni–O(1) = 2·107 (9)
Ni–S(3) = 2·442 (4)
Ni–S(4) = 2·469 (4)

S(1)–Ni–S(2) = 87·2(0·1)° S(2)–Ni–S(5) = 106·9(0·1)°
S(1)–Ni–S(3) = 101·8(0·1) S(3)–Ni–S(5) = 79·9(0·1)
S(1)–Ni–S(4) = 90·6(0·1) S(3)–Ni–O(1) = 85·6(0·2)

Table 3. *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	64·8	644	641	690	3	−1	4
	calc	65·9	651	648	616	2	1	2
S(1)	obs	39·4	416	400	375	13	3	3
	calc	39·0	412	399	380	4	−4	3
S(2)	obs	38·1	374	395	376	13	20	−2
	calc	38·3	388	385	376	10	11	−1
S(3)	obs	39·6	396	422	387	3	−9	−11
	calc	39·1	402	416	382	2	−4	−6
S(4)	obs	40·4	420	426	400	7	5	0
	calc	40·2	424	420	399	6	7	−1
S(5)	obs	40·4	433	433	400	−7	6	8
	calc	40·4	433	427	400	−6	8	5
S(6)	obs	43·6	465	474	433	−10	0	2
	calc	43·2	462	465	431	−11	3	4
O(1)	obs	15·3	129	148	144	−7	−7	13
	calc	15·1	125	139	136	−4	−6	10
O(2)	obs	13·7	127	113	109	−8	−1	6
	calc	13·8	129	109	110	−10	−1	4
O(3)	obs	14·1	118	144	108	3	14	7
	calc	14·5	120	146	112	5	15	10
O(4)	obs	11·8	87	96	108	−7	4	−1
	calc	12·3	97	98	110	−4	1	1
N(1)	obs	11·0	108	79	92	−2	−3	4
	calc	11·0	108	82	94	−5	−3	2
N(2)	obs	11·6	106	124	85	5	7	13
	calc	11·6	108	124	95	2	7	9
N(3)	obs	11·5	114	98	107	−3	15	−3
	calc	11·6	117	102	106	−5	10	0
N(4)	obs	10·9	92	96	89	−5	6	2
	calc	11·0	99	95	95	−6	2	4
N(5)	obs	9·9	76	82	98	−9	−4	−3
	calc	10·0	77	81	97	−6	−2	−1
N(6)	obs	10·3	87	88	93	0	−6	−21
	calc	10·5	93	93	95	−2	−9	−14
N(7)	obs	11·1	98	97	90	−4	7	0
	calc	10·9	95	102	89	−3	9	−1
N(8)	obs	11·7	97	115	110	−2	2	7
	calc	11·8	100	113	112	−2	2	4
C(1)	obs	11·2	106	98	111	−3	3	−2
	calc	11·1	108	100	110	−2	2	−1
C(2)	obs	10·2	97	104	92	1	0	−1
	calc	10·3	99	100	93	1	−3	−1
C(3)	obs	9·7	90	86	84	−8	−4	0
	calc	9·7	91	87	85	−3	−5	0
C(4)	obs	10·7	108	99	102	6	3	−7
	calc	10·7	107	98	100	5	4	−5
	e.s.d.	0·4	7	6	7	4	4	4

Table 4. *Observed and calculated structure factors*

A minus sign for F_o means 'less than'.

h	k	l	$10F_o$	$10F_c$	$\alpha°$	h	k	l	$10F_o$	$10F_c$	$\alpha°$	h	k	l	$10F_o$	$10F_c$	$\alpha°$	h	k	l	$10F_o$	$10F_c$	$\alpha°$	h	k	l	$10F_o$	$10F_c$	$\alpha°$
0	0	2	716	766	0	0	10	2	50-	65	180	1	7	0	135	110	90	2	3	11	37-	10	213	3	1	2	619	607	33	3	10	5	206	223	132
0	0	4	47-	23	180	0	10	3	223	209	0	1	7	1	164	126	323	2	4	0	634	637	0	3	1	3	1058	1046	225	3	10	6	173	180	107
0	0	6	128	166	0	0	10	4	44	77	180	1	7	2	321	278	233	2	4	1	766	822	258	3	1	4	349	319	169	3	10	7	189	125	170
0	0	8	902	959	180	0	10	5	51-	4	180	1	7	3	252	227	178	2	4	2	846	916	274	3	1	5	783	771	316	3	11	0	128	82	270
0	0	10	172	181	180	0	10	6	141	129	180	1	7	4	204	187	39	2	4	3	280	265	324	3	1	6	164	173	233	3	11	1	52-	79	219
0	1	1	944	1009	90	0	10	7	225	237	180	1	7	5	208	229	34	2	4	4	1037	1072	260	3	1	7	399	363	156	3	11	2	166	152	221
0	1	2	353	293	90	0	11	1	300	264	270	1	7	6	214	196	90	2	4	5	684	652	309	3	1	8	259	283	292	3	11	3	126	122	48
0	1	3	261	229	270	0	11	2	45-	17	270	1	7	7	294	300	8	2	4	6	598	552	266	3	1	9	237	226	200	3	11	4	214	200	267
0	1	4	1011	1077	90	0	11	3	145	118	90	1	7	8	154-	90	130	2	4	7	443	443	102	3	1	10	107	86	176	3	11	5	80	81	120
0	1	5	55-	40	270	0	11	4	86	51	90	1	7	9	103	139	39	2	4	8	225	216	271	3	1	11	120	129	22	3	11	6	223	243	210
0	1	6	61-	8	270	0	11	5	40-	38	90	1	8	0	319	263	90	2	4	9	280	286	16	3	2	0	845	974	90	3	12	0	122	171	270
0	1	7	357	358	270	0	11	6	177	175	270	1	8	1	437	398	10	2	4	10	154	154	148	3	2	1	472	432	282	3	12	1	80	139	293
0	1	8	223	195	90	0	12	0	258	217	0	1	8	2	783	732	273	2	4	11	29-	70	309	3	2	2	345	333	232	3	12	2	133	111	187
0	1	9	414	386	270	0	12	1	301	263	180	1	8	3	175	155	349	2	5	0	667	674	180	3	2	3	506	459	40	3	12	3	164	126	220
0	1	10	149	154	270	0	12	2	300	359	0	1	8	4	259	258	264	2	5	1	366	340	108	3	2	4	240	248	184	3	12	4	194	190	231
0	1	11	36-	18	270	0	12	3	202	192	0	1	8	5	107	86	320	2	5	2	228	323	194	3	2	5	110	127	344	3	12	5	17-	89	315
0	2	0	353	327	0	0	12	4	99	94	0	1	8	6	261	256	66	2	5	3	944	1014	14	3	2	6	116	69	157	3	13	0	347	353	90
0	2	1	393	319	180	0	12	5	114	134	0	1	8	7	97	151	214	2	5	4	93	76	141	3	2	7	607	579	11	3	13	1	66	97	298
0	2	2	1093	1211	180	0	13	1	189	188	90	1	8	8	210	254	169	2	5	5	418	404	8	3	2	8	124	165	337	3	13	2	218	195	85
0	2	3	99	64	180	0	13	2	22-	11	270	1	8	9	90	109	139	2	5	6	342	318	64	3	2	9	244	246	2	4	0	0	979	1438	180
0	2	4	294	250	0	1	0	1	495	533	90	1	9	0	51-	27	90	2	5	7	143-	46	354	3	2	10	240	227	341	4	0	1	34-	84	90
0	2	5	292	215	0	1	0	2	256	216	0	1	9	1	298	262	163	2	5	8	229	214	140	3	2	11	162	203	237	4	0	2	854	1003	180
0	2	6	99	41	180	1	0	3	1055	1285	90	1	9	2	271	223	349	2	5	9	168	147	310	3	3	0	424	344	270	4	0	3	146	73	270
0	2	7	531	480	0	1	0	4	735	802	180	1	9	3	229	210	25	2	5	10	62-	95	112	3	3	1	240	187	97	4	0	4	26-	0	0
0	2	8	378	401	180	1	0	5	357	438	90	1	9	4	233	207	20	2	6	0	387	380	160	3	3	2	370	342	112	4	0	5	217	162	90
0	2	9	261	220	0	1	0	6	60-	71	0	1	9	5	210	195	214	2	6	1	355	323	175	3	3	3	678	669	229	4	0	6	516	505	0
0	2	10	183	148	0	1	0	7	263	310	90	1	9	6	366	320	321	2	6	2	540	523	198	3	3	4	238	211	350	4	0	7	273	253	270
0	2	11	292	306	0	1	0	8	95	147	180	1	9	7	91-	38	138	2	6	3	298	281	60	3	3	5	775	748	321	4	0	8	150	162	0
0	3	1	484	409	90	1	0	9	61-	48	270	1	10	0	50-	51	90	2	6	4	194	174	213	3	3	6	336	299	258	4	0	9	100	136	90
0	3	2	818	894	90	1	0	10	51-	71	0	1	10	1	275	248	9	2	6	5	242	227	56	3	3	7	206	198	29	4	0	10	118	88	0
0	3	3	288	201	270	1	0	11	36-	78	270	1	10	2	280	256	274	2	6	6	303	284	358	3	3	8	170	152	43	4	0	11	147	176	90
0	3	4	504	477	90	1	1	0	628	846	90	1	10	3	202	187	266	2	6	7	475	485	143	3	3	9	240	222	22	4	1	0	623	620	0
0	3	5	76-	69	270	1	1	1	842	884	337	1	10	4	177	151	19	2	6	8	181	194	308	3	3	10	170	157	278	4	1	1	317	322	27
0	3	6	265	238	90	1	1	2	904	928	163	1	10	5	164	143	345	2	6	9	111-	34	312	3	3	11	147	191	63	4	1	2	1007	1100	358
0	3	7	313	286	270	1	1	3	535	464	210	1	10	6	221	228	44	2	6	10	71-	99	43	3	4	0	368	310	90	4	1	3	701	716	158
0	3	8	143	144	90	1	1	4	779	730	108	1	10	7	86	87	220	2	7	0	137-	36	0	3	4	1	978	1176	181	4	1	4	825	823	291
0	3	9	487	510	270	1	1	5	441	382	352	1	11	0	45-	66	90	2	7	1	487	456	305	3	4	2	359	306	225	4	1	5	869	837	150
0	3	10	279	265	270	1	1	6	342	320	324	1	11	1	141	135	270	2	7	2	131	143	329	3	4	3	705	704	149	4	1	6	107	46	265
0	3	11	38-	97	90	1	1	7	323	304	71	1	11	2	45-	21	302	2	7	3	724	730	308	3	4	4	693	686	354	4	1	7	284	250	101
0	4	0	886	1112	180	1	1	8	196	140	298	1	11	3	334	317	52	2	7	4	338	328	177	3	4	5	177	181	315	4	1	8	267	247	272
0	4	1	1146	1267	180	1	1	9	370	376	145	1	11	4	219	211	9	2	7	5	223	226	45	3	4	6	275	261	29	4	1	9	231	229	155
0	4	2	647	689	180	1	1	10	149	140	69	1	11	5	374	374	155	2	7	6	150	143	299	3	4	7	323	321	72	4	1	10	202	171	191
0	4	3	892	926	180	1	1	11	273	264	59	1	11	6	58-	60	7	2	7	7	181	175	33	3	4	8	149	100	97	4	1	11	110	122	26
0	4	4	282	227	180	1	2	0	830	993	90	1	12	0	37-	23	90	2	7	8	105	120	357	3	4	9	147	109	350	4	2	0	624	591	0
0	4	5	756	786	180	1	2	1	619	568	20	1	12	1	160	172	168	2	7	9	145	169	188	3	4	10	62-	63	7	4	2	1	917	953	53
0	4	6	181	178	180	1	2	2	607	506	236	1	12	2	150	148	133	2	8	0	121-	26	180	3	5	0	783	856	270	4	2	2	527	517	234
0	4	7	781	809	180	1	2	3	561	426	279	1	12	3	338	303	149	2	8	1	688	626	265	3	5	1	561	529	288	4	2	3	589	530	25
0	4	8	139	135	0	1	2	4	498	457	223	1	12	4	204	186	151	2	8	2	674	608	63	3	5	2	900	935	204	4	2	4	827	812	241
0	4	9	84-	73	0	1	2	5	344	237	297	1	12	5	191	213	77	2	8	3	137	135	211	3	5	3	563	565	214	4	2	5	443	393	346
0	4	10	254	262	0	1	2	6	164	105	357	1	13	0	25-	47	90	2	8	4	246	217	69	3	5	4	418	405	1	4	2	6	233	236	325
0	4	11	32-	19	180	1	2	7	428	408	96	1	13	1	185	169	295	2	8	5	158	164	55	3	5	5	227	208	331	4	2	7	273	251	257
0	5	1	361	336	90	1	2	8	531	518	243	1	13	2	51	84	238	2	8	6	602	592	80	3	5	6	393	366	176	4	2	8	91	108	320
0	5	2	684	677	270	1	2	9	225	164	264	1	13	3	10-	111	17	2	8	7	259	277	94	3	5	7	282	274	225	4	2	9	133	112	220
0	5	3	579	550	90	1	2	10	42-	36	24	2	0	0	349	281	0	2	8	8	103	101	57	3	5	8	112	157	75	4	2	10	40-	73	91
0	5	4	936	996	270	1	2	11	28-	45	200	2	0	1	1443	1881	90	2	8	9	172	187	109	3	5	9	111-	65	121	4	2	11	189	211	228
0	5	5	301	310	270	1	3	0	36-	2	90	2	0	2	716	775	180	2	9	0	260	223	0	3	5	10	193	233	24	4	3	0	63	19	0
0	5	6	644	631	270	1	3	1	229	183	182	2	0	3	961	1011	90	2	9	1	282	234	125	3	6	0	114	112	270	4	3	1	825	879	276
0	5	7	225	209	90	1	3	2	915	950	233	2	0	4	334	338	180	2	9	2	649	633	346	3	6	1	313	289	287	4	3	2	458	413	114
0	5	8	134-	41	90	1	3	3	225	153	65	2	0	5	122	135	270	2	9	3	200	184	263	3	6	2	344	341	13	4	3	3	523	470	305
0	5	9	114-	37	90	1	3	4	485	484	245	2	0	6	60-	18	0	2	9	4	351	327	290	3	6	3	275	260	165	4	3	4	653	579	241
0	5	10	83-	87	270	1	3	5	621	606	222	2	0	7	170	159	270	2	9	5	275	276	99	3	6	4	225	214	245	4	3	5	810	770	29
0	6	0	391	291	0	1	3	6	387	350	202	2	0	8	137	191	180	2	9	6	168	138	238	3	6	5	345	306	298	4	3	6	516	466	48
0	6	1	372	351	180	1	3	7	147	127	199	2	0	9	296	317	270	2	9	7	122	119	196	3	6	6	158-	77	280	4	3	7	479	439	52
0	6	2	156	182	180	1	3	8	217	209	261	2	0	10	210	233	0	2	10	0	596	579	180	3	6	7	172	144	245	4	3	8	280	263	49
0	6	3	303	279	0	1	3	9	240	238	156	2	0	11	110	106	270	2	10	1	166	139	313	3	6	8	162	173	188	4	3	9	292	265	47
0	6	4	422	421	0	1	3	10	191	196	352	2	1	0	819	1125	180	2	10	2	424	396	61	3	6	9	107	116	115	4	3	10	152	121	73
0	6	5	72	85	0	1	3	11	38-	53	111	2	1	1	267	258	184	2	10	3	246	221	332	3	6	10	200	214	194	4	3	11	124	141	49
0	6	6	600	658	0	1	4	0	894	1079	90	2	1	2	1022	1212	124	2	10	4	38	63	247	3	7	0	126	65	90	4	4	0	556	541	0
0	6	7	483	511	180	1	4	1	707	625	120	2	1	3	1076	1079	232	2	10	5	376	350	317	3	7	1	284	279	30	4	4	1	540	524	294
0	6	8	138-	25	180	1	4	2	712	724	49	2	1	4	128	97	25	2	10	6	258	236	157	3	7	2	349	346	217	4	4	2	393	346	339
0	6	9	227	257	0	1	4	3	391	394	241	2	1	5	674	644	194	2	10	7	66	49	26	3	7	3	160	134	145	4	4	3	554	536	358
0	6	10	73-	57	0	1	4	4	187	179	7	2	1	6	563	545	31	2	11	0	131	140	0	3	7	4	592	582	21	4	4	4	243	269	53
0	7	1	313	272	90	1	4	5	567	545	323	2	1	7	129	75	179	2	11	1	219	178	152	3	7	5	112	104	313	4	4	5	644	632	355
0	7	2	466	458	270	1	4	6	506	476	324	2	1	8	592	581	7	2	11	2	225	204	38	3	7	6	397	436	118	4	4	6	154	154	63
0	7	3	340	323	90	1	4	7	139	116	9	2	1	9	181	170	3	2	11	3	84	80	351	3	7	7	265	294	187	4	4	7	141	128	206
0	7	4	120	42	270	1	4	8	200	214	292	2	1	10	107	96	338	2	11	4	198	167	126	3	7	8	105	110	101	4	4	8	173	167	119
0	7	5	192-	76	90	1	4	9	290	289	294	2	1	11	112	118	113	2	11	5	152	159	13	3	7	9	122	79	233	4	4	9	185	157	121
0	7	6	491	497	270	1	4	10	64-	68	311	2	2	0	835	976	0	2	11	6	217	228	147	3	8	0	319	290	90	4	5	0	365	397	180
0	7	7	179-	21	90	1	5	0	265	218	90	2	2	1	166	137	296	2	12	0	141	149	180	3	8	1	498	481	326	4	5	1	651	641	309
0	7	8	212	245	90	1	5	1	661	574	215	2	2	2	810	774	76	2	12	1	386	342	112	3	8	2	525	500	34	4	5	2	1037	1146	144
0	7	9	210	266	270	1	5	2	733	755	199	2	2	3	806	760	128	2	12	2	35-	74	345	3	8	3	372	243	8	4	5	3	663	615	248
0	8	0	256	245	180	1	5	3	112-	51	260	2	2	4	703	696	281	2	12	3	217	198	118	3	8	4	196	186	0	4	5	4	217	164	248
0	8	1	121-	47	180	1	5	4	173	126	9	2	2	5	693	646	244	2	12	4	29-	36	200	3	8	5	150	155	78	4	5	5	154	142	109
0	8	2	581	523	180	1	5	5	120	130	340	2	2	6	280	224	59	2	12	5	20-	49	235	3	8	6	290	300	9	4	5	6	271	277	29
0	8	3	506	482	0	1	5	6	502	493	136	2	2	7	386	377	204	2	13	0	208	212	180	3	8	7	154	177	268	4	5	7	142-	25	216
0	8	4	151-	39	180	1	5	7	370	351	325	2	2	8	382	392	120	2	13	1	105	94	245	3	8	8	156	176	294	4	5	8	93	114	236
0	8	5	651	677	0	1	5	8	134-	66	122	2	2	9	235	206	235	2	13	2	108	140	234	3	8	9	62-	72	155	4	5	9	191	193	222
0	8	6	200	199	0	1	5	9	113-	62	287	2	2	10	124	159	151	3	0	1	575	602	270	3	9	0	51-	19	270	4	5	10	237	290	331
0	8	7	172	164	0	1	5	10	177	160	47	2	2	11	28-	53	18	3	0	2	404	263	0	3	9	1	386	374	98	4	6	0	97	126	0
0	8	8	108-	86	0	1	6	0	357	336	90	2	3	0	816	1029	180	3	0	3	147	113	270	3	9	2	374	315	196	4	6	1	508	510	253
0	8	9	112	112	0	1	6	1	424	393	191	2	3	1	999	1126	166	3	0	4	1185	1412	180	3	9	3	321	301	85	4	6	2	324	325	351
0	9	1	626	580	270	1	6	2	456	436	4	2	3	2	466	418	151	3	0	5	598	558	270	3	9	4	217	197	347	4	6	3	445	426	295
0	9	2	271	290	270	1	6	3	477	484	218	2	3	3	95	79	73	3	0	6	416	387	180	3	9	5	237	218	75	4	6	4	133	128	212
0	9	3	745	770	270	1	6	4	437	420	143	2	3	4	770	756	283	3	0	7	196	159	270	3	9	6	87	82	167	4	6	5	600	601	44
0	9	4	233	201	90	1	6	5	154-	78	194	2	3	5	271	221	112	3	0	8	135	139	160	3	9	7	344	328	84	4	6	6	422	421	230
0	9	5	58-	27	90	1	6	6	158-	82	43	2	3	6	261	238	350	3	0	9	349	361	90	3	10	0	91	182	270	4	6	7	151-	29	169
0	9	6	103	95	90	1	6	7	154-	86	159	2	3	7	292	261	193	3	0	10	210	257	160	3	10	1	183	173	39	4	6	8	134-	117	202
0	9	7	330	303	90	1	6	8	138-	37	284	2	3	8	519	525	333	3	0	11	34-	28	270	3	10	2	167	177	203	4	6	9	217	265	61
0	10	0	374	332	180	1	6	9	175	187	54	2	3	9	286	258	317	3	1	0	726	995	90	3	10	3	271	202	98	4	6	10	150	137	220
0	10	1	59-	154	180	1	6	10	72-	33	315	2	3	10	235	231	291	3	1	1	581	532	26	3	10	4	97	80	335	4	7	0	428	385	0

S(1)–Ni–O(1) = 96·1(0·3) S(4)–Ni–S(5) = 88·2(0·1)
S(2)–Ni–S(3) = 98·9(0·1) S(4)–Ni–O(1) = 93·3(0·3)
S(2)–Ni–S(4) = 81·2(0·1) S(5)–Ni–O(1) = 69·4(0·3)

The four Ni–S(*tu*) bond distances are within ±0·04 Å of the value (2·43 Å) corresponding to the sum of Pauling's covalent radii, and agree with the value

Table 4 (*cont.*)

h	k	l	$10F_o$	$10F_c$	$\alpha°$	h	k	l	$10F_o$	$10F_c$	$\alpha°$	h	k	l	$10F_o$	$10F_c$	$\alpha°$	h	k	l	$10F_o$	$10F_c$	$\alpha°$	h	k	l	$10F_o$	$10F_c$	$\alpha°$
4	7	1	540	519	36	5	4	5	191	195	67	6	1	9	122	97	225	6	12	2	116	104	62	7	9	1	240	225	9	8	6	9	231	261	252
4	7	2	326	291	34	5	4	6	416	364	167	6	1	10	145	89	276	6	12	3	84	64	207	7	9	2	269	240	117	8	7	0	280	252	180
4	7	3	181	159	256	5	4	7	294	254	82	6	1	11	27-	35	330	6	12	4	129	162	72	7	9	3	296	276	207	8	7	1	129	168	210
4	7	4	430	391	38	5	4	8	92-	57	324	6	2	0	896	955	0	6	13	0	15-	59	0	7	9	4	225	192	27	8	7	2	202	179	64
4	7	5	298	278	91	5	4	9	177	236	139	6	2	1	621	606	229	6	13	1	14-	79	52	7	9	5	202	229	261	8	7	3	193	191	284
4	7	6	258	276	110	5	4	10	99	99	107	6	2	2	1003	1050	134	7	0	1	516	484	90	7	9	6	83-	85	270	8	7	4	202	183	256
4	7	7	152	157	43	5	5	0	126	132	270	6	2	3	712	657	354	7	0	2	648	679	0	7	9	7	74-	38	206	8	7	5	129	113	142
4	7	8	135	154	215	5	5	1	762	802	356	6	2	4	357	273	294	7	0	3	371	313	270	7	10	0	657	686	90	8	7	6	256	237	41
4	7	9	139	226	186	5	5	2	433	407	62	6	2	5	500	422	337	7	0	4	103	75	0	7	10	1	368	382	200	8	7	7	156-	44	21
4	8	0	221	211	180	5	5	3	481	464	305	6	2	6	328	308	30	7	0	5	59-	39	270	7	10	2	168	122	95	8	7	8	99	152	40
4	8	1	424	403	114	5	5	4	581	525	12	6	2	7	191	166	142	7	0	6	296	279	0	7	10	3	256	226	175	8	7	9	180	192	350
4	8	2	323	298	62	5	5	5	246	238	264	6	2	8	105	85	87	7	0	7	774	749	90	7	10	4	55	56	9	8	8	0	156	202	180
4	8	3	372	340	194	5	5	6	286	250	332	6	2	9	259	247	94	7	0	8	63-	58	180	7	10	5	225	230	25	8	8	1	223	202	156
4	8	4	321	294	230	5	5	7	246	225	219	6	2	10	338	325	329	7	0	9	172	122	270	7	10	6	63-	39	194	8	8	2	326	309	59
4	8	5	382	379	132	5	5	8	131	122	49	6	3	0	464	371	180	7	0	10	44-	28	180	7	10	7	156	161	220	8	8	3	203	203	64
4	8	6	131	142	194	5	5	9	154	183	190	6	3	1	298	295	266	7	1	0	932	1146	270	7	11	0	100	123	270	8	8	4	286	232	270
4	8	7	269	287	179	5	5	10	129	168	296	6	3	2	344	319	25	7	1	1	711	816	171	7	11	1	261	249	205	8	8	5	160	150	352
4	8	8	120	137	106	5	6	0	605	595	90	6	3	3	252	239	224	7	1	2	701	812	271	7	11	2	125	127	284	8	8	6	130-	59	98
4	8	9	56-	40	270	5	6	1	410	415	101	6	3	4	670	627	72	7	1	3	282	274	143	7	11	3	152	132	152	8	8	7	152	156	181
4	9	0	248	258	0	5	6	2	659	615	299	6	3	5	443	390	167	7	1	4	311	273	16	7	11	4	198	177	142	8	8	8	120	124	306
4	9	1	366	379	86	5	6	3	481	464	118	6	3	6	219	212	185	7	1	5	225	164	279	7	11	5	31-	41	180	8	9	0	575	639	180
4	9	2	100-	112	17	5	6	4	495	485	328	6	3	7	653	584	129	7	1	6	366	331	182	7	12	0	93	89	90	8	9	1	286	289	218
4	9	3	259	247	4	5	6	5	269	276	73	6	3	8	303	261	119	7	1	7	489	478	336	7	12	1	219	166	344	8	9	2	349	318	200
4	9	4	143	116	101	5	6	6	237	332	35	6	3	9	179	171	152	7	1	8	226	241	126	7	12	2	279	268	261	8	9	3	179	165	187
4	9	5	275	273	18	5	6	7	108	93	313	6	3	10	120	64	197	7	1	9	282	251	268	7	12	3	40	47	229	8	9	4	166	224	202
4	9	6	164	142	150	5	6	8	240	228	345	6	4	0	491	502	0	7	1	10	177	191	104	7	12	4	16-	54	292	8	9	5	135	125	195
4	9	7	225	233	289	5	6	9	273	318	247	6	4	1	254	267	110	7	2	0	768	660	270	8	0	0	233	187	180	8	9	6	198	196	287
4	10	0	49-	19	0	5	6	10	54-	80	164	6	4	2	529	493	128	7	2	1	460	487	27	8	0	1	875	1116	270	8	9	7	118	119	164
4	10	1	112	111	225	5	7	0	101	84	270	6	4	3	789	776	55	7	2	2	751	723	291	8	0	2	240	219	0	8	10	0	116	91	180
4	10	2	460	425	24	5	7	1	510	475	108	6	4	4	324	281	37	7	2	3	525	530	296	8	0	3	121	80	90	8	10	1	72	77	199
4	10	3	256	243	178	5	7	2	229	216	219	6	4	5	319	314	341	7	2	4	445	417	312	8	0	4	191	172	0	8	10	2	45-	61	225
4	10	4	372	346	68	5	7	3	154	99	82	6	4	6	407	384	341	7	2	5	724	679	242	8	0	5	634	580	270	8	10	3	41	72	328
4	10	5	156	168	259	5	7	4	87	61	306	6	4	7	129	124	197	7	2	6	550	494	16	8	0	6	187	136	180	8	10	4	347	349	260
4	10	6	206	193	72	5	7	5	326	324	179	6	4	8	214	197	207	7	2	7	581	541	187	8	0	7	254	237	90	8	10	5	145	138	24
4	10	7	93	109	233	5	7	6	139	136	100	6	4	9	76-	81	55	7	2	8	242	216	99	8	0	8	62-	89	0	8	10	6	58-	59	97
4	11	0	110	121	0	5	7	7	372	407	124	6	4	10	214	239	320	7	2	9	151	339	201	8	0	9	54-	12	270	8	11	0	155	160	0
4	11	1	296	263	132	5	7	8	142-	100	316	6	5	0	1177	1313	0	7	2	10	237	219	137	8	0	10	41-	39	0	8	11	1	250	220	94
4	11	2	290	269	221	5	7	9	102-	131	185	6	5	1	560	567	262	7	3	0	347	339	90	8	1	0	712	753	0	8	11	2	221	188	321
4	11	3	212	203	226	5	8	0	621	604	90	6	5	2	441	423	35	7	3	1	517	512	21	8	1	1	449	448	272	8	11	3	183	181	333
4	11	4	91	111	264	5	8	1	275	262	135	6	5	3	634	580	248	7	3	2	586	538	72	8	1	2	741	770	95	8	11	4	189	190	90
4	11	5	116	98	172	5	8	2	206	182	111	6	5	4	330	321	74	7	3	3	342	332	61	8	1	3	454	493	356	8	11	5	97	102	68
4	11	6	47-	86	324	5	8	3	215	177	60	6	5	5	179	160	100	7	3	4	649	594	42	8	1	4	362	379	224	8	12	0	93-	105	180
4	12	0	405	383	180	5	8	4	240	221	117	6	5	6	240	206	342	7	3	5	273	226	21	8	1	5	286	256	329	8	12	1	68	57	15
4	12	1	99	84	311	5	8	5	330	329	148	6	5	7	279	280	68	7	3	6	158	156	14	8	1	6	395	341	87	8	12	2	34	58	20
4	12	2	221	211	252	5	8	6	231	234	159	6	5	8	261	246	157	7	3	7	321	280	74	8	1	7	170	150	56	8	12	3	103	109	242
4	12	3	97	109	250	5	8	7	189	199	26	6	5	9	126	115	333	7	3	8	172	124	6	8	1	8	62-	68	45	9	0	1	407	399	90
4	12	4	154	166	103	5	8	8	97-	97	266	6	5	10	67-	62	295	7	3	9	282	291	198	8	1	9	170	110	34	9	0	2	957	1071	0
4	13	0	225	254	0	5	8	9	44-	72	321	6	6	0	517	531	0	7	3	10	97	83	302	8	1	10	143	134	254	9	0	3	131	29	270
4	13	1	25-	58	5	5	9	0	267	352	270	6	6	1	391	366	99	7	4	0	806	776	90	8	2	0	598	576	0	9	0	4	232	293	0
5	0	1	707	735	270	5	9	1	449	427	53	6	6	2	275	230	11	7	4	1	569	612	4	8	2	1	510	504	209	9	0	5	418	390	270
5	0	2	154	113	180	5	9	2	368	371	191	6	6	3	103	96	325	7	4	2	313	272	296	8	2	2	429	412	116	9	0	6	269	264	0
5	0	3	1343	1516	270	5	9	3	139	124	323	6	6	4	323	273	122	7	4	3	296	279	240	8	2	3	703	592	93	9	0	7	143	100	90
5	0	4	351	297	0	5	9	4	342	345	209	6	6	5	242	218	6	7	4	4	397	348	166	8	2	4	365	331	105	9	0	8	61-	42	0
5	0	5	974	948	270	5	9	5	193	180	180	6	6	6	420	405	149	7	4	5	118	85	2	8	2	5	351	299	293	9	0	9	124	107	270
5	0	6	114	103	0	5	9	6	363	394	167	6	6	7	217	212	254	7	4	6	422	408	115	8	2	6	323	300	78	9	0	10	147	142	180
5	0	7	65-	25	90	5	9	7	122	125	108	6	6	8	382	405	149	7	4	7	114	89	189	8	2	7	540	525	134	9	1	0	745	770	270
5	0	8	347	331	0	5	10	0	366	367	90	6	6	9	98	97	304	7	4	8	154	112	232	8	2	8	250	246	228	9	1	1	795	877	6
5	0	9	414	399	270	5	10	1	200	194	335	6	7	0	147	104	180	7	4	9	73-	60	233	8	2	9	326	314	39	9	1	2	491	464	276
5	0	10	48-	57	180	5	10	2	328	293	159	6	7	1	342	314	49	7	4	10	51-	5	315	8	2	10	245	259	267	9	1	3	387	343	5
5	0	11	30-	60	270	5	10	3	261	263	356	6	7	2	441	432	194	7	5	0	280	312	90	8	3	0	328	334	0	9	1	4	624	563	351
5	1	0	254	204	270	5	10	4	240	220	339	6	7	3	261	232	134	7	5	1	472	444	97	8	3	1	289	340	136	9	1	5	164	152	351
5	1	1	1402	1534	138	5	10	5	118	110	159	6	7	4	187	196	195	7	5	2	349	340	18	8	3	2	112	84	297	9	1	6	225	209	15
5	1	2	846	848	103	5	10	6	160	180	230	6	7	5	202	196	213	7	5	3	774	740	100	8	3	3	215	195	14	9	1	7	296	273	187
5	1	3	749	718	144	5	10	7	63	68	293	6	7	6	149	158	339	7	5	4	321	282	331	8	3	4	76	65	225	9	1	8	451	444	45
5	1	4	445	438	349	5	11	0	82	77	90	6	7	7	135	122	308	7	5	5	646	610	131	8	3	5	244	207	218	9	1	9	217	211	210
5	1	5	277	263	52	5	11	1	108	86	88	6	7	8	138-	33	168	7	5	6	401	366	338	8	3	6	284	283	244	9	1	10	273	241	14
5	1	6	202	175	209	5	11	2	311	297	113	6	7	9	86	192	274	7	5	7	267	258	83	8	3	7	124	85	242	9	2	0	431	431	90
5	1	7	588	488	35	5	11	3	240	233	332	6	8	0	140-	30	180	7	5	8	122-	57	289	8	3	8	286	253	196	9	2	1	489	499	342
5	1	8	166	137	217	5	11	4	93	70	42	6	8	1	315	293	189	7	5	9	120	87	113	8	3	9	128	124	318	9	2	2	722	732	56
5	1	9	126	113	346	5	11	5	152	144	356	6	8	2	105	64	52	7	5	10	59-	88	106	8	3	10	152	158	172	9	2	3	500	476	111
5	1	10	208	179	285	5	11	6	42-	99	323	6	8	3	368	372	292	7	6	0	300	271	90	8	4	0	636	624	0	9	2	4	212	179	105
5	1	11	30-	88	347	5	12	0	34-	68	90	6	8	4	282	263	293	7	6	1	603	622	75	8	4	1	191	194	11	9	2	5	338	322	106
5	2	0	189	268	270	5	12	1	95	99	289	6	8	5	91	65	354	7	6	2	519	489	244	8	4	2	647	696	120	9	2	6	663	640	297
5	2	1	1041	1232	167	5	12	2	55	64	329	6	8	6	139	130	213	7	6	3	305	268	81	8	4	3	521	478	87	9	2	7	374	336	134
5	2	2	732	610	133	5	12	3	101	100	246	6	8	7	361	374	208	7	6	4	697	676	179	8	4	4	279	220	127	9	2	8	177	188	283
5	2	3	95	68	297	5	12	4	114	124	358	6	8	8	92-	61	220	7	6	5	546	552	53	8	4	5	150	140	302	9	2	9	237	218	159
5	2	4	552	525	99	5	13	0	112	152	90	6	9	0	204	104	180	7	6	6	155-	106	168	8	4	6	359	331	67	9	2	10	292	306	247
5	2	5	496	448	242	5	13	1	243	318	161	6	9	1	340	336	106	7	6	7	397	369	86	8	4	7	170	157	116	9	3	0	145	104	270
5	2	6	682	615	150	5	13	2	7-	52	267	6	9	2	89	110	124	7	6	8	168	153	287	8	4	8	248	221	156	9	3	1	667	643	151
5	2	7	481	424	26	6	0	0	766	780	0	6	9	3	277	262	122	7	6	9	137	167	251	8	4	9	133	62	236	9	3	2	842	856	172
5	2	8	387	371	73	6	0	1	510	582	270	6	9	4	416	402	96	7	7	0	321	299	90	8	4	10	70-	85	102	9	3	3	1083	1117	166
5	2	9	244	241	25	6	0	2	1005	1171	180	6	9	5	86	66	246	7	7	1	176-	114	311	8	5	0	628	605	0	9	3	4	504	473	123
5	2	10	193	177	111	6	0	3	279	270	270	6	9	6	86-	43	270	7	7	2	581	568	196	8	5	1	479	483	147	9	3	5	311	317	46
5	2	11	135	167	85	6	0	4	53-	4	0	6	9	7	162	129	124	7	7	3	626	585	312	8	5	2	609	619	339	9	3	6	451	404	181
5	3	0	1198	1389	270	6	0	5	410	371	90	6	10	0	48-	98	0	7	7	4	296	271	201	8	5	3	252	232	245	9	3	7	279	232	112
5	3	1	514	448	24	6	0	6	95	5	0	6	10	1	464	457	163	7	7	5	273	370	229	8	5	4	105	108	136	9	3	8	76-	27	285
5	3	2	466	420	132	6	0	7	65-	51	270	6	10	2	229	243	237	7	7	6	282	272	214	8	5	5	517	485	125	9	3	9	172	161	305
5	3	3	760	722	252	6	0	8	196	204	180	6	10	3	282	273	145	7	7	7	162-	50	270	8	5	6	164	161	154	9	3	10	210	228	23
5	3	4	791	781	345	6	0	9	57-	1	270	6	10	4	334	314	275	7	7	8	326	380	235	8	5	7	99	86	154	9	4	0	449	438	90
5	3	5	437	405	339	6	0	10	46-	47	180	6	10	5	219	215	137	7	7	9	85-	88	215	8	5	8	107	142	185	9	4	1	558	528	319
5	3	6	334	298	292	6	0	11	28-	61	90	6	10	6	206	218	321	7	8	0	146-	51	90	8	5	9	139	143	266	9	4	2	292	246	255
5	3	7	141	144	24	6	1	0	668	609	180	6	10	7	156	180	159	7	8	1	301	302	108	8	6	0	334	294	180	9	4	3	387	355	79
5	3	8	535	532	105	6	1	1	619	705	323	6	11	0	28	19	180	7	8	2	149	127	229	8	6	1	202	182	86	9	4	4	330	320	206
5	3	9	215	209	205	6	1	2	214	282	271	6	11	1	42	101	251	7	8	3	282	262	181	8	6	2	139	139	90	9	4	5	126	120	235
5	3	10	187	187	205	6	1	3	720	714	64	6	11	2	338	319	248	7	8	4	145	162	127	8	6	3	156	131	184	9	4	6	98-	11	135
5	4	0	754	765	270	6	1	4	775	747	234	6	11	3	168	166	314	7	8	5	162	122	208	8	6	4	479	432	278	9	4	7	193	197	218
5	4	1	435	412	19	6	1	5	462	451	23	6	11	4	114	127	50	7	8	6	141	133	328	8	6	5	496	495	274	9	4	8	154	129	203
5	4	2	848	872	257	6	1	6	758	712	212	6	11	5	154	147	325	7	8	7	115-	70	216	8	6	6	191	183	65	9	4	9	168	125	140
5	4	3	231	191	36	6	1	7	556	471	5	6	12	0	131	138	180	7	8	8	85-	33	270	8	6	7	193	183	261	9	4	10	90	90	85
5	4	4	405	357	336	6	1	8	65-	93	107	6	12	1	80	89	197	7	9	0	542	533	90	8	6	8	202	207	356	9	5	0	435	408	90

(2·46 Å) found in tetra(thiourea)nickel(II) chloride (Lopez-Castro & Truter, 1963). They are a little shorter than the distances found in bis(thiourea)nickel(II) thiocyanate [2·531(6) and 2·564(9) Å, Nardelli, Fava Gasparri, Giraldi Battistini & Domiano (1966)], in bis(2-thioimidazolidine)nickel(II) thiocyanate [2·507(8)

Table 4 (*cont.*)

h	k	l	$10F_o$	$10F_c$	$\alpha°$	h	k	l	$10F_o$	$10F_c$	$\alpha°$	h	k	l	$10F_o$	$10F_c$	$\alpha°$	h	k	l	$10F_o$	$10F_c$	$\alpha°$	h	k	l	$10F_o$	$10F_c$	$\alpha°$
9	5	1	430	414	66	10	3	5	85-	51	240	11	2	4	380	360	226	12	1	7	231	193	229	13	1	5	324	307	254	14	1	6	181	140	321
9	5	2	504	474	156	10	3	6	86-	50	324	11	2	5	458	431	357	12	1	8	97	136	133	13	1	6	305	300	274	14	1	9	196	190	356
9	5	3	317	301	111	10	3	7	389	365	296	11	2	6	573	522	219	12	1	9	206	186	230	13	1	7	309	314	324	14	2	0	55-	65	0
9	5	4	470	428	249	10	3	8	323	266	220	11	2	7	242	232	13	12	2	0	529	502	0	13	1	8	23-	85	124	14	2	1	217	270	292
9	5	5	472	469	11	10	3	9	256	236	228	11	2	8	194	172	309	12	2	1	261	235	162	13	1	9	35-	75	160	14	2	2	185	158	206
9	5	6	458	428	210	10	3	10	26-	13	339	11	2	9	200	200	301	12	2	2	242	207	260	13	2	0	458	438	90	14	2	3	335	314	97
9	5	7	248	221	98	10	4	0	274	283	180	11	2	10	23-	66	225	12	2	3	223	229	112	13	2	1	370	334	61	14	2	4	193	142	107
9	5	8	196	205	211	10	4	1	88-	8	325	11	3	0	795	766	270	12	2	4	246	210	344	13	2	2	421	374	343	14	2	5	347	322	307
9	5	9	85-	21	188	10	4	2	454	487	123	11	3	1	460	388	26	12	2	5	152	134	161	13	2	3	120	117	277	14	2	6	122	81	108
9	6	0	676	706	90	10	4	3	460	432	185	11	3	2	554	512	223	12	2	6	301	265	254	13	2	4	217	174	168	14	2	7	110	82	47
9	6	1	319	319	311	10	4	4	347	309	218	11	3	3	225	222	219	12	2	7	296	261	243	13	2	5	452	393	352	14	2	8	38-	57	62
9	6	2	428	440	152	10	4	5	172	125	123	11	3	4	449	435	234	12	2	8	156	146	161	13	2	6	313	297	62	14	2	9	128	127	182
9	6	3	435	436	272	10	4	6	97-	63	156	11	3	5	129	115	248	12	2	9	34-	26	220	13	2	7	217	182	64	14	3	0	85-	2	0
9	6	4	395	369	171	10	4	7	200	204	241	11	3	6	298	264	212	12	3	0	80-	71	0	13	2	8	108	92	5	14	3	1	120	132	247
9	6	5	602	591	287	10	4	8	79-	40	198	11	3	7	80-	85	215	12	3	1	730	705	21	13	2	9	87	90	301	14	3	2	351	342	155
9	6	6	198	188	110	10	4	9	152	137	173	11	3	8	208	182	54	12	3	2	238	223	161	13	3	0	368	360	90	14	3	3	359	322	227
9	6	7	89	105	264	10	5	0	133-	69	180	11	3	9	227	203	177	12	3	3	225	156	109	13	3	1	89	89	239	14	3	4	86-	83	207
9	6	8	158	173	257	10	5	1	644	644	71	11	4	0	943	1037	90	12	3	4	386	350	101	13	3	2	265	240	55	14	3	5	414	363	159
9	6	9	76	69	165	10	5	2	136	160	142	11	4	1	91-	71	245	12	3	5	76	63	215	13	3	3	510	480	326	14	3	6	366	349	34
9	7	0	158	178	90	10	5	3	229	224	208	11	4	2	646	649	54	12	3	6	198	168	25	13	3	4	70	71	94	14	3	7	168	153	64
9	7	1	187-	11	5	10	5	4	619	575	262	11	4	3	282	263	283	12	3	7	525	509	25	13	3	5	430	406	244	14	3	8	223	215	49
9	7	2	189	187	35	10	5	5	420	386	97	11	4	4	279	234	105	12	3	8	65-	42	355	13	3	6	160	147	288	14	3	9	97	83	141
9	7	3	225	197	332	10	5	6	175	133	336	11	4	5	292	308	268	12	3	9	143	133	287	13	3	7	73-	45	354	14	4	0	98-	13	180
9	7	4	571	530	28	10	5	7	124	134	322	11	4	6	145	146	295	12	4	0	93-	77	0	13	3	8	258	189	256	14	4	1	227	235	317
9	7	5	139	113	294	10	5	8	170	126	352	11	4	7	185	169	314	12	4	1	292	274	219	13	3	9	162	138	139	14	4	2	286	274	322
9	7	6	237	234	299	10	5	9	149	152	294	11	4	8	393	390	310	12	4	2	349	333	141	13	4	0	135	125	90	14	4	3	250	197	350
9	7	7	149-	50	48	10	6	0	680	666	180	11	4	9	170	149	173	12	4	3	365	341	238	13	4	1	793	810	167	14	4	4	714	727	290
9	7	8	280	319	329	10	6	1	179	152	44	11	5	0	78	70	270	12	4	4	386	355	270	13	4	2	409	363	312	14	4	5	217	182	283
9	8	0	305	284	270	10	6	2	315	300	63	11	5	1	638	624	153	12	4	5	168	167	181	13	4	3	603	600	211	14	4	6	200	176	223
9	8	1	133	124	197	10	6	3	231	215	133	11	5	2	273	282	153	12	4	6	286	296	15	13	4	4	177	152	221	14	4	7	145	124	130
9	8	2	153-	13	243	10	6	4	238	206	358	11	5	3	800	794	252	12	4	7	198	187	230	13	4	5	96-	97	0	14	4	8	56-	72	297
9	8	3	292	293	175	10	6	5	147	102	149	11	5	4	137	160	162	12	4	8	99	114	74	13	4	6	334	326	351	14	5	0	175	146	180
9	8	4	235	222	227	10	6	6	193	231	41	11	5	5	649	658	278	12	4	9	185	183	32	13	4	7	286	294	32	14	5	1	374	376	288
9	8	5	233	238	37	10	6	7	131	137	159	11	5	6	135-	62	163	12	5	0	475	470	180	13	4	8	173	161	322	14	5	2	89	119	18
9	8	6	125-	36	274	10	6	8	351	366	12	11	5	7	122	124	31	12	5	1	296	281	9	13	5	0	812	830	270	14	5	3	502	514	359
9	8	7	120	127	95	10	6	9	137	121	111	11	5	8	99-	70	53	12	5	2	474	464	205	13	5	1	374	322	66	14	5	4	138-	46	117
9	8	8	87-	54	39	10	7	0	223	224	0	11	5	9	212	244	294	12	5	3	144-	48	295	13	5	2	359	321	264	14	5	5	256	268	318
9	9	0	359	356	90	10	7	1	149	134	189	11	6	0	389	400	90	12	5	4	217	203	179	13	5	3	208	247	305	14	5	6	61	62	247
9	9	1	416	383	192	10	7	2	206	173	149	11	6	1	215	181	174	12	5	5	140-	1	180	13	5	4	105	89	45	14	5	7	129	155	338
9	9	2	84	155	182	10	7	3	290	265	238	11	6	2	246	202	300	12	5	6	99	91	306	13	5	5	260	251	221	14	5	8	68	88	234
9	9	3	351	362	209	10	7	4	219	206	265	11	6	3	202	186	218	12	5	7	63	50	78	13	5	6	108	87	41	14	6	0	215	217	0
9	9	4	430	432	29	10	7	5	183	183	108	11	6	4	502	493	358	12	5	8	91-	105	69	13	5	7	342	331	65	14	6	1	269	203	246
9	9	5	229	243	272	10	7	6	212	182	105	11	6	5	282	276	185	12	5	9	122	161	191	13	5	8	183	166	60	14	6	2	313	291	105
9	9	6	114	116	166	10	7	7	183	173	159	11	6	6	82	101	66	12	6	0	93	131	0	13	6	0	93	63	90	14	6	3	290	276	66
9	9	7	47	36	290	10	7	8	149	185	162	11	6	7	166	155	62	12	6	1	261	242	273	13	6	1	244	249	78	14	6	4	422	407	262
9	10	0	527	545	270	10	8	0	298	296	180	11	6	8	120	130	319	12	6	2	351	353	114	13	6	2	129	160	288	14	6	5	208	184	356
9	10	1	214	209	210	10	8	1	153-	16	225	11	7	0	141	142	270	12	6	3	72	42	55	13	6	3	129	107	164	14	6	6	126	106	97
9	10	2	282	272	287	10	8	2	156	164	306	11	7	1	466	446	113	12	6	4	393	342	255	13	6	4	198	168	47	14	6	7	162	179	78
9	10	3	256	234	157	10	8	3	168	164	313	11	7	2	357	311	117	12	6	5	145	143	75	13	6	5	105	93	68	14	6	8	43-	75	168
9	10	4	145	120	237	10	8	4	143	135	243	11	7	3	198	161	82	12	6	6	129	130	290	13	6	6	158	156	199	14	7	0	126	121	180
9	10	5	162	154	8	10	8	5	271	249	352	11	7	4	70	30	15	12	6	7	149	168	127	13	6	7	103-	36	0	14	7	1	227	231	136
9	10	6	68	101	97	10	8	6	145	142	306	11	7	5	147	154	48	12	6	8	83-	93	196	13	6	8	166	199	192	14	7	2	95	78	182
9	11	0	80	85	90	10	8	7	72	82	263	11	7	6	149	159	169	12	7	0	603	597	0	13	7	0	147	129	270	14	7	3	280	264	23
9	11	1	231	230	1	10	9	0	387	383	0	11	7	7	376	396	52	12	7	1	192-	99	221	13	7	1	227	196	149	14	7	4	244	233	142
9	11	2	208	207	248	10	9	1	368	367	283	11	7	8	200	268	117	12	7	2	309	314	279	13	7	2	154	117	199	14	7	5	181	198	27
9	11	3	31-	53	284	10	9	2	280	278	250	11	8	0	275	272	270	12	7	3	122	103	326	13	7	3	126	136	217	14	7	6	129	120	322
9	11	4	29-	13	193	10	9	3	250	252	292	11	8	1	143	120	278	12	7	4	265	258	280	13	7	4	95	73	173	14	7	7	59	87	102
9	11	5	91	134	283	10	9	4	105	98	330	11	8	2	397	397	233	12	7	5	145	177	351	13	7	5	185	186	243	14	8	0	140-	20	0
9	12	0	193	186	90	10	9	5	46-	34	10	11	8	3	261	255	264	12	7	6	238	333	294	13	7	6	215	153	60	14	8	1	269	269	262
9	12	1	74	91	307	10	9	6	187	210	353	11	8	4	126	137	306	12	7	7	120	100	196	13	7	7	76	101	302	14	8	2	317	331	118
9	12	2	126	136	92	10	9	7	27	60	324	11	8	5	259	284	305	12	7	8	103	146	167	13	8	0	309	324	90	14	8	3	156	131	188
9	12	3	14-	49	156	10	10	0	42-	25	180	11	8	6	263	269	171	12	8	0	208	189	180	13	8	1	487	486	14	14	8	4	158	155	142
10	0	0	600	651	0	10	10	1	208	209	39	11	8	7	172	188	312	12	8	1	103	128	104	13	8	2	166	172	312	14	8	5	131	137	194
10	0	1	246	248	270	10	10	2	42-	54	93	11	9	0	395	386	270	12	8	2	95	121	214	13	8	3	136-	17	66	14	8	6	179	237	88
10	0	2	791	864	0	10	10	3	152	157	329	11	9	1	133	100	67	12	8	3	181	173	7	13	8	4	340	346	19	14	9	0	240	249	0
10	0	3	475	504	90	10	10	4	150	138	193	11	9	2	237	240	310	12	8	4	242	227	56	13	8	5	246	251	8	14	9	1	154	150	119
10	0	4	74	65	0	10	10	5	267	244	38	11	9	3	87	46	56	12	8	5	267	283	31	13	8	6	196	226	22	14	9	2	158	144	61
10	0	5	133	79	270	10	10	6	39-	66	246	11	9	4	361	371	346	12	8	6	99	106	46	13	9	0	252	222	90	14	9	3	34-	72	278
10	0	6	65-	2	0	10	11	0	120	124	180	11	9	5	137	141	74	12	8	7	70-	56	99	13	9	1	131	127	28	14	9	4	114	93	88
10	0	7	229	218	90	10	11	1	214	163	77	11	9	6	164	178	21	12	9	0	470	459	0	13	9	2	84	98	256	14	9	5	84	84	148
10	0	8	143	141	0	10	11	2	108	152	182	11	10	0	110	106	270	12	9	1	131	157	4	13	9	3	298	297	60	14	10	0	31-	48	0
10	0	9	271	290	90	10	11	3	105	115	284	11	10	1	378	371	348	12	9	2	44-	74	190	13	9	4	212	185	166	14	10	1	36-	40	154
10	0	10	110	114	180	10	11	4	114	106	129	11	10	2	57	74	198	12	9	3	143	122	3	13	9	5	204	224	109	14	10	2	267	270	37
10	1	0	70	37	180	10	12	0	22-	46	180	11	10	3	145	133	31	12	9	4	149	136	71	13	9	6	34-	77	89	14	10	3	131	151	245
10	1	1	263	229	314	10	12	1	147	158	341	11	10	4	97	103	73	12	9	5	91	99	27	13	10	0	263	264	90	14	11	1	16-	54	191
10	1	2	714	707	95	11	0	1	363	349	90	11	10	5	108	109	126	12	9	6	107	108	344	13	10	1	78	86	7	15	0	1	439	418	270
10	1	3	105	115	283	11	0	2	739	755	180	11	11	0	227	218	90	12	10	0	110	113	180	13	10	2	156	144	317	15	0	2	177	221	0
10	1	4	164	164	95	11	0	3	682	710	90	11	11	1	238	236	104	12	10	1	120	126	221	13	10	3	212	203	200	15	0	3	519	544	270
10	1	5	275	249	256	11	0	4	435	416	0	11	11	2	99	121	18	12	10	2	156	169	126	13	10	4	82	78	190	15	0	4	166	166	180
10	1	6	682	655	93	11	0	5	489	475	90	11	11	3	126	144	219	12	10	3	300	321	284	13	11	0	170	112	90	15	0	5	571	551	270
10	1	7	177	143	345	11	0	6	150	162	180	11	11	4	152	135	342	12	10	4	193	205	88	13	11	1	82	118	76	15	0	6	231	211	0
10	1	8	290	261	203	11	0	7	542	489	90	11	12	0	15-	9	270	12	10	5	80	93	97	13	11	2	206	228	305	15	0	7	272	257	270
10	1	9	107	62	141	11	0	8	105	102	0	12	0	0	705	717	0	12	11	0	28-	39	0	14	0	0	233	195	0	15	0	8	147	151	0
10	1	10	112	92	293	11	0	9	46-	27	90	12	0	1	374	380	90	12	11	1	183	218	262	14	0	1	646	672	90	15	1	0	120	80	270
10	2	0	231	275	0	11	0	10	30-	70	180	12	0	2	107	113	180	12	11	2	64	65	186	14	0	2	437	404	180	15	1	1	237	224	182
10	2	1	103	107	273	11	1	0	426	374	270	12	0	3	389	365	90	12	11	3	137	155	187	14	0	3	791	820	90	15	1	2	271	241	26
10	2	2	135	147	339	11	1	1	334	345	352	12	0	4	64-	2	0	13	0	1	198	162	90	14	0	4	65-	51	180	15	1	3	376	367	175
10	2	3	173	168	190	11	1	2	1110	1222	116	12	0	5	326	294	270	13	0	2	519	544	180	14	0	5	611	627	270	15	1	4	252	212	53
10	2	4	237	226	162	11	1	3	275	224	40	12	0	6	261	355	180	13	0	3	217	190	270	14	0	6	168	242	0	15	1	5	135	140	20
10	2	5	193	142	268	11	1	4	382	344	148	12	0	7	145	99	270	13	0	4	825	850	180	14	0	7	159	104	270	15	1	6	279	228	301
10	2	6	154	125	150	11	1	5	399	389	101	12	0	8	229	49	180	13	0	5	242	207	270	14	0	8	116	71	180	15	1	7	101	100	78
10	2	7	177	150	249	11	1	6	430	399	230	12	0	9	162	150	90	13	0	6	393	449	180	14	0	9	55-	107	270	15	1	8	141	99	146
10	2	8	217	198	161	11	1	7	267	243	126	12	0	10	24-	53	180	13	0	7	59-	62	90	14	1	0	173	158	180	15	2	0	323	301	90
10	2	9	286	264	10	11	1	8	189	163	112	12	1	0	107	165	0	13	0	8	168	132	180	14	1	1	242	216	126	15	2	1	103	105	255
10	2	10	172	188	150	11	1	9	298	272	110	12	1	1	227	239	103	13	0	9	191	178	270	14	1	2	495	472	192	15	2	2	252	252	349
10	3	0	586	549	0	11	1	10	229	253	278	12	1	2	292	303	72	13	1	0	120	107	90	14	1	3	78	125	179	15	2	3	512	473	183
10	3	1	464	431	19	11	2	0	49-	29	90	12	1	3	498	472	163	13	1	1	588	574	245	14	1	4	132	114	249	15	2	4	433	390	147
10	3	2	210	217	24	11	2	1	258	293	167	12	1	4	447	428	2	13	1	2	277	264	100	14	1	5	352	440	122	15	2	5	107	115	285
10	3	3	145	97	169	11	2	2	126	52	177	12	1	5	366	319	217	13	1	3	529	515	262	14	1	6	251	258	235	15	2	6	214	195	68
10	3	4	512	448	259	11	2	3	279	242	155	12	1	6	332	324	347	13	1	4	160	136	142	14	1	7	141	161	111	15	2	7	42-	32	90

and 2·544(14) Å, Nardelli, Fava Gasparri, Musatti & Manfredotti (1966)] and in bis(thioacetamide)nickel(II) thiocyanate [2·446(10) and 2·546(10) Å, Capacchi, Fava Gasparri, Nardelli & Pelizzi (1968)] and lie in the lower limit of the range 2·4–2·6 Å expected for the Ni–S distances in octahedral nickel complexes.

Table 4 (*cont.*)

h	k	l	$10F_o$	$10F_c$	$\alpha°$	h	k	l	$10F_o$	$10F_c$	$\alpha°$	h	k	l	$10F_o$	$10F_c$	$\alpha°$	h	k	l	$10F_o$	$10F_c$	$\alpha°$	h	k	l	$10F_o$	$10F_c$	$\alpha°$
15	2	8	145	133	170	16	2		402	360	274	17	2	5	170	194	261	18	4	0	101	111	180	19	6	4	269	291	148	21	3	2	60-	51	211
15	3	0	86-	11	270	16	2	5	101	65	246	17	2	6	173	169	88	18	4	1	191	157	160	19	6	5	149	168	38	21	3	3	55-	22	180
15	3	1	227	166	174	16	2	6	191	181	289	17	2	7	34-	36	329	18	4	2	279	245	123	19	7	0	120	117	90	21	3	4	114	104	147
15	3	2	252	210	268	16	2	7	39-	22	170	17	2	8	120	128	3	18	4	3	223	208	142	19	7	1	147	105	105	21	3	5	26-	44	328
15	3	3	258	222	333	16	2	8	219	212	344	17	3	0	116	110	270	18	4	4	342	347	110	19	7	2	139	126	330	21	4	0	223	210	90
15	3	4	267	225	7	16	3	0	86-	14	0	17	3	1	315	287	82	18	4	5	200	178	162	19	7	3	66	83	298	21	4	1	141	129	225
15	3	5	273	249	314	16	3	1	196	165	303	17	3	2	435	391	59	18	4	6	196	177	101	19	7	4	64	102	196	21	4	2	172	160	41
15	3	6	141	150	342	16	3	2	86-	52	180	17	3	3	89	97	52	18	4	7	230	218	191	19	8	0	72-	4	270	21	4	3	261	256	162
15	3	7	139	127	355	16	3	3	122	150	249	17	3	4	110	126	83	18	5	0	445	428	0	19	8	1	116	76	65	21	4	4	102	53	139
15	3	8	47-	54	228	16	3	4	581	541	261	17	3	5	162	155	119	18	5	1	145	133	284	19	8	2	158	229	268	21	5	0	88-	26	90
15	4	0	598	588	270	16	3	5	252	233	80	17	3	6	101	111	42	18	5	2	156	143	244	20	0	0	402	385	0	21	5	1	168	156	194
15	4	1	106	149	19	16	3	6	120	97	76	17	3	7	147	149	83	18	5	3	367	325	172	20	0	1	112	145	90	21	5	2	189	190	229
15	4	2	416	368	288	16	3	7	118	125	74	17	4	0	95-	69	270	18	5	4	118	93	277	20	0	2	112	70	0	21	5	3	152	86	143
15	4	3	384	356	72	16	3	8	200	209	238	17	4	1	298	291	25	18	5	5	166	160	200	20	0	3	206	192	1	21	5	4	141	212	190
15	4	4	370	324	179	16	4	0	97-	47	0	17	4	2	68	110	148	18	5	6	145	167	306	20	0	4	48-	7	180	21	6	0	63	73	270
15	4	5	382	360	41	16	4	1	355	345	44	17	4	3	90-	10	221	18	6	0	410	417	0	20	0	5	42-	66	90	21	6	1	139	129	293
15	4	6	223	162	22	16	4	2	584	573	35	17	4	4	154	114	126	18	6	1	175	140	88	20	0	6	74	87	180	21	6	2	74	76	22
15	4	7	250	240	90	16	4	3	437	439	27	17	4	5	110	97	242	18	6	2	221	222	26	20	1	0	264	260	180	21	6	3	252	284	212
15	4	8	179	203	101	16	4	4	93	71	41	17	4	6	233	224	164	18	6	3	120	133	302	20	1	1	210	192	339	21	7	1	41-	31	22
15	5	0	256	216	90	16	4	5	495	499	2	17	4	7	122	127	158	18	6	4	139	147	2	20	1	2	126	88	140	22	0	0	284	295	180
15	5	1	384	358	18	16	4	6	275	268	142	17	5	0	134-	6	90	18	6	5	99	91	345	20	1	3	181	179	300	22	0	1	46-	53	90
15	5	2	141-	32	313	16	4	7	166	165	359	17	5	1	139	135	112	18	6	6	41-	62	329	20	1	4	386	373	97	22	0	2	168	152	0
15	5	3	87	50	281	16	4	8	33-	110	80	17	5	2	172	171	117	18	7	0	124	138	0	20	1	5	118	144	280	22	0	3	40-	29	90
15	5	4	298	263	23	16	5	0	185	166	0	17	5	3	99	55	269	18	7	1	223	223	54	20	1	6	170	164	102	22	0	4	47-	28	180
15	5	5	91	84	279	16	5	1	191	214	289	17	5	4	280	271	340	18	7	2	168	166	225	20	2	0	252	237	0	22	0	5	74	98	270
15	5	6	166	144	136	16	5	2	431	394	89	17	5	5	102-	21	137	18	7	3	200	207	135	20	2	1	254	251	264	22	1	0	147	164	0
15	5	7	231	237	150	16	5	3	145	135	161	17	5	6	202	208	257	18	7	4	156	162	77	20	2	2	374	351	77	22	1	1	86	84	58
15	5	8	110	101	8	16	5	4	206	178	60	17	5	7	120	142	118	18	7	5	116	151	176	20	2	3	225	214	158	22	1	2	321	315	36
15	6	0	194	179	270	16	5	5	168	143	158	17	6	0	138-	13	270	18	8	0	74	54	180	20	2	4	189	154	44	22	1	3	212	245	156
15	6	1	141	125	253	16	5	6	300	325	62	17	6	1	217	235	344	18	8	1	214	224	57	20	2	5	277	270	235	22	1	4	152	141	356
15	6	2	233	218	349	16	5	7	55	55	156	17	6	2	63	73	121	18	8	2	114	126	174	20	2	6	271	307	107	22	1	5	204	272	190
15	6	3	292	266	144	16	6	0	52	78	180	17	6	3	200	196	64	18	8	3	66	70	339	20	3	0	116	104	0	22	2	0	22-	47	180
15	6	4	175	165	295	16	6	1	256	247	280	17	6	4	78	41	168	18	9	0	19-	89	180	20	3	1	182	171	148	22	2	1	103	98	116
15	6	5	135	120	321	16	6	2	84	84	157	17	6	5	227	219	46	19	0	1	100	120	90	20	3	2	114	92	353	22	2	2	189	177	342
15	6	6	194	196	325	16	6	3	91	80	235	17	6	6	68	84	237	19	0	2	454	452	0	20	3	3	124	102	116	22	2	3	185	248	118
15	6	7	131	161	270	16	6	4	185	174	164	17	7	0	154-	3	90	19	0	3	177	155	90	20	3	4	57-	82	292	22	2	4	286	297	268
15	7	0	116	100	270	16	6	5	111-	56	282	17	7	1	223	256	40	19	0	4	56	87	180	20	3	5	172	185	189	22	3	0	70	114	180
15	7	1	258	262	17	16	6	6	116	130	118	17	7	2	145	159	146	19	0	5	163	194	90	20	3	6	194	238	263	22	3	1	59	79	70
15	7	2	78	99	304	16	6	7	54-	56	193	17	7	3	143	126	251	19	0	6	172	172	0	20	4	0	77-	108	180	22	3	2	124	123	216
15	7	3	162	177	161	16	7	0	332	326	0	17	7	4	76	69	210	19	0	7	42	51	90	20	4	1	215	193	268	22	3	3	162	165	34
15	7	4	84	66	0	16	7	1	97	88	289	17	7	5	118	139	315	19	1	0	223	194	270	20	4	2	145	156	179	22	3	4	35-	33	199
15	7	5	132-	22	319	16	7	2	139	134	126	17	8	0	108-	8	90	19	1	1	154	122	89	20	4	3	214	231	121	22	4	0	56-	63	0
15	7	6	114	109	270	16	7	3	250	248	271	17	8	1	215	231	200	19	1	2	126	150	253	20	4	4	191	174	263	22	4	1	124	132	310
15	8	0	464	488	90	16	7	4	256	258	100	17	8	2	74	101	219	19	1	3	244	239	287	20	4	5	193	235	254	22	4	2	97	114	41
15	8	1	130-	80	342	16	7	5	103	38	117	17	8	3	242	252	158	19	1	4	107	135	156	20	5	0	426	413	0	22	4	3	112	130	157
15	8	2	231	227	109	16	7	6	84	124	60	17	8	4	72-	11	135	19	1	5	112	91	218	20	5	1	301	310	100	22	4	4	22-	19	242
15	8	3	294	307	150	16	8	0	242	247	0	17	9	0	26-	25	90	19	1	6	120	90	39	20	5	2	191	185	278	22	5	0	108	111	180
15	8	4	112	111	153	16	8	1	156	133	13	17	9	1	150	179	63	19	1	7	139	152	31	20	5	3	227	240	62	22	5	1	72	90	52
15	8	5	187	189	45	16	8	2	263	261	347	17	9	2	23-	67	56	19	2	0	68	31	270	20	5	4	63	69	180	22	5	2	246	305	189
15	8	6	49-	74	116	16	8	3	244	262	189	18	0	0	674	682	0	19	2	1	156	130	47	20	5	5	32	73	282	22	5	3	35-	46	290
15	9	0	68	82	90	16	8	4	189	203	274	18	0	1	525	505	270	19	2	2	145	130	332	20	6	0	194	203	180	23	0	1	103	97	270
15	9	1	41-	22	225	16	8	5	160	202	180	18	0	2	280	255	0	19	2	3	170	159	316	20	6	1	256	296	94	23	0	2	105	58	0
15	9	2	206	184	217	16	9	0	31-	67	0	18	0	3	202	204	270	19	2	4	78	43	140	20	6	2	177	182	162	23	0	3	31-	35	90
15	9	3	30-	18	186	16	9	1	324	359	93	18	0	4	301	305	0	19	2	5	162	159	179	20	6	3	154	169	101	23	0	4	105	129	180
15	9	4	172	180	178	16	9	2	181	191	267	18	0	5	149	141	90	19	2	6	62	100	309	20	6	4	63	82	332	23	1	0	39-	62	90
15	10	0	63	46	90	16	9	3	78	98	8	18	0	6	70	67	180	19	2	7	135	169	148	20	7	0	68	117	180	23	1	1	99	96	102
15	10	1	150	183	28	16	9	4	116	171	229	18	0	7	14-	35	270	19	3	0	263	250	270	20	7	1	198	226	271	23	1	2	229	210	99
15	10	2	23-	40	82	16	10	0	145	197	180	18	1	0	323	280	0	19	3	1	194	174	130	20	7	2	135	151	213	23	1	3	70	66	12
15	10	3	16-	60	162	16	10	1	107	155	65	18	1	1	508	465	24	19	3	2	170	146	74	20	7	3	55-	9	180	23	1	4	20-	34	5
16	0	0	795	851	180	17	0	1	345	343	270	18	1	2	261	226	304	19	3	3	99	80	34	21	0	1	52-	11	270	23	2	0	61	68	90
16	0	1	542	519	270	17	0	2	430	395	0	18	1	3	105	76	58	19	3	4	77	101	183	21	0	2	156	133	0	23	2	1	277	271	209
16	0	2	581	568	180	17	0	3	204	194	90	18	1	4	91	92	189	19	3	5	311	314	175	21	0	3	97	77	90	23	2	2	167	202	96
16	0	3	30-	35	90	17	0	4	248	213	0	18	1	5	76	97	11	19	3	6	145	161	257	21	0	4	376	363	180	23	2	3	120	137	114
16	0	4	231	235	180	17	0	5	168	141	90	18	1	6	139	181	239	19	4	0	296	274	90	21	0	5	87	73	90	23	3	0	280	292	270
16	0	5	212	216	270	17	0	6	89	82	0	18	1	7	217	225	337	19	4	1	280	264	21	21	0	6	26-	60	0	23	3	1	66	87	52
16	0	6	122	170	0	17	0	7	42-	54	270	18	2	0	307	273	180	19	4	2	81-	74	82	21	1	0	118	117	90	23	3	2	87	61	172
16	0	7	47-	55	270	17	0	8	99	137	0	18	2	1	284	260	358	19	4	3	210	202	235	21	1	1	225	189	18	23	3	3	149	195	243
16	0	8	149	148	0	17	1	0	739	724	270	18	2	2	391	358	142	19	4	4	64	49	350	21	1	2	99	98	200	23	4	0	89	90	270
16	1	0	53-	76	0	17	1	1	326	294	115	18	2	3	288	247	7	19	4	5	183	183	318	21	1	3	221	132	334	23	4	1	133	153	274
16	1	1	460	425	309	17	1	2	223	202	2	18	2	4	101	51	90	19	4	6	105	102	246	21	1	4	135	128	111	23	4	2	32-	71	222
16	1	2	42-	35	268	17	1	3	158	155	139	18	2	5	139	129	6	19	5	0	115-	5	270	21	1	5	63	79	205	24	0	0	133	95	180
16	1	3	271	320	267	17	1	4	149	126	209	18	2	6	76	65	121	19	5	1	107	99	122	21	1	6	18-	12	228	24	0	1	194	221	90
16	1	4	317	305	239	17	1	5	219	199	347	18	2	7	267	283	2	19	5	2	244	243	112	21	2	0	139	83	90	24	0	2	31-	50	0
16	1	5	219	220	122	17	1	6	87	119	286	18	3	0	452	454	0	19	5	3	150	154	54	21	2	1	212	212	342	24	1	0	95	73	180
16	1	6	194	117	153	17	1	7	42-	57	270	18	3	1	349	299	327	19	5	4	133	122	141	21	2	1	194	177	16	24	1	1	129	136	14
16	1	7	252	246	27	17	1	8	122	131	72	18	3	2	300	266	64	19	5	5	70	78	100	21	2	3	173	187	42	24	1	2	147	157	229
16	1	8	122	160	211	17	2	0	56-	19	270	18	3	3	77-	57	264	19	6	0	124	114	90	21	2	4	103	110	182	24	2	0	22-	46	180
16	2	0	181	198	180	17	2	1	168	141	247	18	3	4	206	187	105	19	6	1	91	31	48	21	2	5	103	119	58	24	2	1	21-	19	330
16	2	1	342	335	8	17	2	2	382	358	279	18	3	5	64-	8	284	19	6	2	129	133	185	21	3	0	160	143	270	24	2	2	15-	84	218
16	2	2	244	212	17	17	2	3	193	165	233	18	3	6	179	192	121	19	6	3	206	191	198	21	3	1	78	93	149	24	3	0	24-	43	0
16	2	3	208	155	99	17	2	4	152	148	297	18	3	7	93	101	22																				

The $S_2O_3^{2-}$ group, being coordinated through an oxygen atom O(1), and a sulphur atom S(5), is behaving as a chelating agent. The Ni–O (2·10 Å) distance corresponds with those usually observed in octahedral nickel complexes [*e.g.* 2·12 Å in nickel acetate tetrahydrate (van Niekerk & Schoening, 1953), 2·08 Å in nickel glycine dihydrate (Stosick, 1945) and 2·03 Å in diaquobis(salicylaldehydato)nickel (Stewart, Lingafelter & Breazeale, 1961)], whereas the Ni–S(thiosulphate) distance (2·71 Å) is significantly longer than the maximum value (2·6 Å) for octahedral Ni-complexes. Therefore, the coordination polyhedron could be considered also as a distorted square pyramid with an extra coordination site under the base.

The Ni–S and Ni–O distances in the octahedral Ni-complexes are longer by 0·2–0·3 Å than in the planar square complexes of the same metal for which the ranges Ni–S = 2·1–2·3, Ni–O = 1·83–1·84 Å are observed [*e.g.* 2·21–2·22 in nickel bis(dithiocarbamate) (Fava Gasparri, Nardelli & Villa, 1967), 2·20–2·21 in nickel bis(*N*,*N*′-diethyldithiocarbamate) (Bonamico, Dessy, Mariani, Vaciago & Zambonelli, 1965), 2·20–2·21 in nickel bis(*N*,*N*′-di-n-propyldithiocarbamate) (Peyronel & Pignedoli, 1966), 2·23–2·24 in nickel

xanthate (Franzini, 1963), 2·16 in bis(thiosemicarbazidato)nickel (Cavalca, Nardelli & Fava, 1962) and 2·15 Å in nickel bis(methylthiohydroxamate) (Sato, Nagata, Shiro & Koyama, 1966) for Ni–S; 1·83 in nickel salicylaldoxime (Merritt, Guare & Lessor, 1956) and 1·84 Å in bis(salicylaldoximate)nickel (Stewart & Lingafelter, 1959) for Ni–O].

In addition to the Ni–O(1) and Ni–S(5) interactions, the orientation of the $S_2O_3^{2-}$ group is determined by the hydrogen bonding involving the free thiosulphate

Table 5. *Bond lengths and angles in* $S_2O_3^{2-}$ *group*

	Ni*tu*$_4S_2O_3.H_2O$ (*)	$BaS_2O_3.H_2O$ (**)	$Na_2S_2O_3.5H_2O$ (***)	$Na_2S_2O_3$ (****)	$Mg(OH_2)_6S_2O_3$ (*****)
S(6)–S(5)	2·01 (1) Å	1·96 (1) Å	1·97 Å	2·01 (2) Å	2·02 (1) Å
S(6)–O(1)	1·50 (1)	1·57 (4)	1·59 $\sigma < 0{\cdot}06$ Å	1·52 (3)	1·48 (1)
S(6)–O(2)	1·46 (1)	1·52 (3)	1·46	1·46 (3)	1·48 (4)
S(6)–O(3)	1·43 (1)	1·44 (3)	1·40	1·40 (3)	
O(1)–S(6)–O(2)	110·7 (0·6)°	105·3 (2·3)°		109·4 (3·6)°	110·9 (1·1)°
O(1)–S(6)–O(3)	110·4 (0·7)	104·3 (1·9)		107·9 (3·6)	110·0 (1·1)
O(2)–S(6)–O(3)	111·6 (0·7)	116·3 (1·4)	104–115° $\sigma < 5°$	114·4 (3·6)	
S(5)–S(6)–O(1)	104·8 (0·4)	109·7 (1·3)		108·3 (2·4)	107·6 (0·8)
S(5)–S(6)–O(2)	108·6 (0·5)	112·0 (0·8)		109·3 (2·4)	108·5 (0·8)
S(5)–S(6)–O(3)	110·6 (0·5)	108·7 (1·3)		107·6 (2·4)	

(*) Present paper.
(**) Nardelli & Fava (1962).
(***) Taylor & Beevers (1952).
(****) Sándor & Csordás (1961).
(*****) Nardelli, Fava & Giraldi (1962).

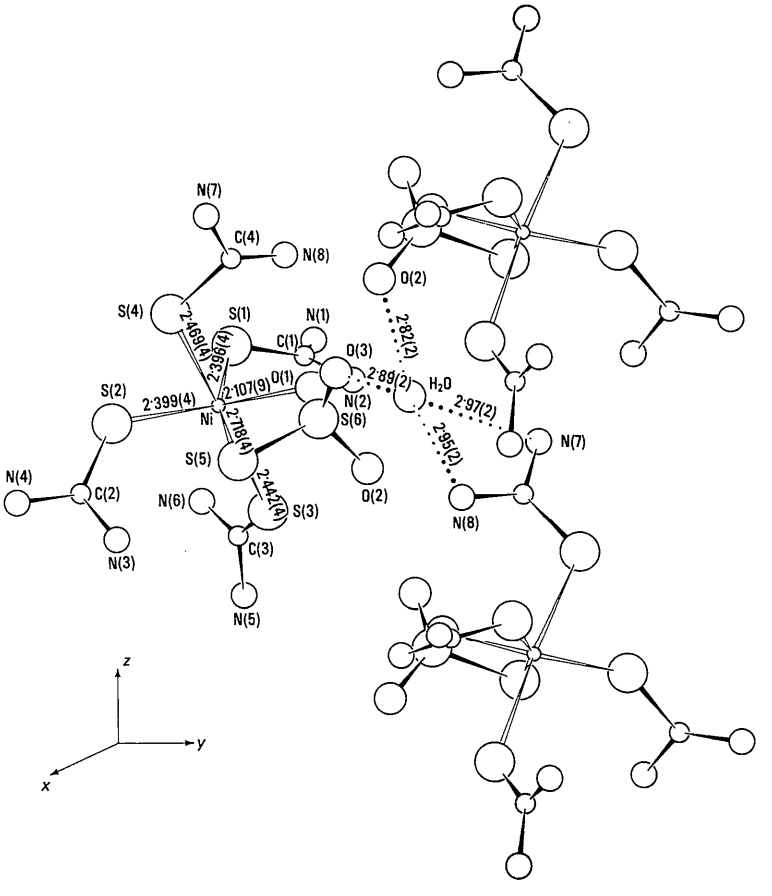

Fig. 1. Clinographic view of metal coordination and hydrogen bonding involving H_2O.

oxygen atoms, the water molecule and the $-NH_2$ groups from adjacent thiourea molecules. The distances and angles in the $S_2O_3^{2-}$ group agree fairly well with those generally observed in other thiosulphates, as shown in Table 5.

The four independent thiourea molecules are planar; their least-squares planes are quoted in Table 6, in which bond distances and angles are compared with corresponding values for the uncomplexed molecule (Truter, 1967). From this comparison, it appears that the coordination does not significantly influence the dimensions of the ligand as observed in other metal thiourea complexes [*e.g.*: bis(thiourea)zinc acetate (Cavalca, Fava Gasparri, Andreetti & Domiano, 1967) and tris(thiourea)zinc sulphate (Andreetti, Cavalca & Musatti, in press)]. The four thiourea molecules are tilted with respect to the Ni–S bonds by the angles Ni–S(1)–C(1) = 112·4(0·5)°, Ni–S(2)–C(2) = 114·3(0·4)°, Ni–S(3)–C(3) = 106·6(0·3)°, Ni–S(4)–C(4) = 115·2(0·5)°, and the tilting is of the same kind of that found in other thiourea complexes [*e.g.*: 100·6(0·3)° and 101·2(0·3)° in bis(thiourea)zinc acetate (Cavalca, Fava Gasparri, Andreetti & Domiano, 1967); 113° in bis(thiourea)cadmium chloride (Nardelli, Cavalca & Braibanti, 1957); 108·6° in bis(thiourea)zinc chloride (Kunchur & Truter, 1958); 105°, 108° and 113° in tris(thiourea)copper(I) chloride (Okaya & Knobler, 1964); 101·9(0·4)°, 107·7(0·4)° and 107·0(0·5)° in tris(thiourea)zinc sulphate (Andreetti, Cavalca & Musatti, 1968)].

The H_2O molecule forms four contacts, which can be considered as hydrogen bonds, with two oxygen atoms from two $S_2O_3^{2-}$ groups and two nitrogen atoms from the same *tu*(4) thiourea molecule in a distorted tetrahedral environment:

O(4)···O(3)	= 2·89(2) Å	O(4)···N(7^{v})	= 2·97(2) Å
O(4)···O(2^{viii})	= 2·82(2)	O(4)···N(8^{v})	= 2·95(2)

The water H(17) and H(18) hydrogen atoms are close to the lines connecting two oxygen atoms as indicated also by the value (92·5°) of the O(3)–O(4)–O(2^{viii}) angle.

Other contacts could be considered as hydrogen bonds, with the following distances and angles:

N(3)H(5)···S(3)	= 3·31(1) Å	H(5)N(3)S(3)	= 18·7°
N(6)H(11)··S(1)	= 3·43(1)	H(11)N(6)S(1)	= 19·3
N(2)H(4)···S(4^{i})	= 3·38(2)	H(4)N(2)S(4^{i})	= 28·1
N(2)H(3)···O(1)	= 2·96(2)	H(3)N(2)O(1)	= 17·3
N(8)H(16)··O(1)	= 2·88(2)	H(16)N(8)O(1)	= 34·1
N(4)H(7)···O(2^{ii})	= 2·96(2)	H(7)N(4)O(2^{ii})	= 27·3
N(5)H(9)···O(2^{iii})	= 3·05(2)	H(9)N(5)O(2^{iii})	= 27·4
N(4)H(8)···O(3^{iv})	= 2·91(2)	H(8)N(4)O(3^{iv})	= 22·9
N(1)H(1)···O(3^{vi})	= 3·14(2)	H(1)N(1)O(3^{vi})	= 9·8

Some of these contacts are indicated in Fig. 2, which shows a diagrammatic projection of the structure on (001).

The other distances less than 3·5 Å are:

N(6)–S(2)	= 3·40 Å	N(8)–O(3)	= 3·23 Å
N(1)–O(4^{vi})	= 3·46	N(3)–N(7^{iii})	= 3·18
N(3)–S(4^{iv})	= 3·50	N(3)–N(7^{iv})	= 3·49
N(6)–S(3^{iii})	= 3·46	N(4)–N(7^{iv})	= 3·43
N(6)–O(2^{iii})	= 3·17	N(5)–N(8^{vii})	= 3·40

i	$\bar{x}, y+\frac{1}{2}, \frac{1}{2}-z$	v	$\frac{1}{2}-x, 1-y, z-\frac{1}{2}$
ii	$x, y-1, z$	vi	$\bar{x}, y-\frac{1}{2}, \frac{1}{2}-z$
iii	$\bar{x}, y-\frac{1}{2}, \bar{z}-\frac{1}{2}$	vii	$x, y, z-1$
iv	$\frac{1}{2}-x, \bar{y}, z-\frac{1}{2}$	viii	$\frac{1}{2}-x, 1-y, z+\frac{1}{2}$

A recent study of the infrared spectrum of crystals of Ni*tu*$_4$S$_2$O$_3$.H$_2$O by Newman (1967) has shown that ν_4 (S–O asymmetric stretching vibration), which is doubly degenerate [1123 cm^{-1}, Siebert (1954)] in the $S_2O_3^{2-}$ ion owing to the C_{3v} symmetry of the $-SO_3$ group, is split into two peaks at 1153 and 1092 cm^{-1} in the nickel-thiourea complex. Thus the degeneracy is removed by chelation.

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Table 6. *Least-squares planes, bond distances and angles for the thiourea molecules*

X, Y, Z are the coordinates in Å.

tu(1): S(1)C(1)N(1)N(2)	$0{\cdot}0952X + 0{\cdot}3289Y + 0{\cdot}9395Z = 1{\cdot}5972$
tu(2): S(2)C(2)N(3)N(4)	$0{\cdot}8478X + 0{\cdot}2411Y + 0{\cdot}4723Z = 1{\cdot}4109$
tu(3): S(3)C(3)N(5)N(6)	$-0{\cdot}7320X + 0{\cdot}6585Y + 0{\cdot}1745Z = -0{\cdot}1508$
tu(4): S(4)C(4)N(7)N(8)	$0{\cdot}7861X + 0{\cdot}4891Y - 0{\cdot}5386Z = 0{\cdot}7148$

	S–C	C–N*	S–C–N*	N–C–N
tu(1)	1·71 (1) Å	1·33 (2) – 1·32 (2) Å	119·3 (1·1) – 122·3 (1·2)°	118·3 (1·4)°
tu(2)	1·71 (2)	1·32 (2) – 1·32 (2)	121·7 (1·0) – 121·2 (1·1)	117·1 (1·4)
tu(3)	1·69 (2)	1·32 (2) – 1·35 (2)	119·8 (1·2) – 121·1 (1·1)	119·0 (1·4)
tu(4)	1·70 (1)	1·33 (2) – 1·33 (2)	119·7 (1·0) – 124·0 (1·1)	116·3 (1·2)
tu†	1·720 (9)	1·340 (6)	120·5 (0·5)	119·0 (0·5)

* The first value gives the bond or the angle involving the N atom with the lower-numbered label.
† Uncomplexed (Truter, 1967).

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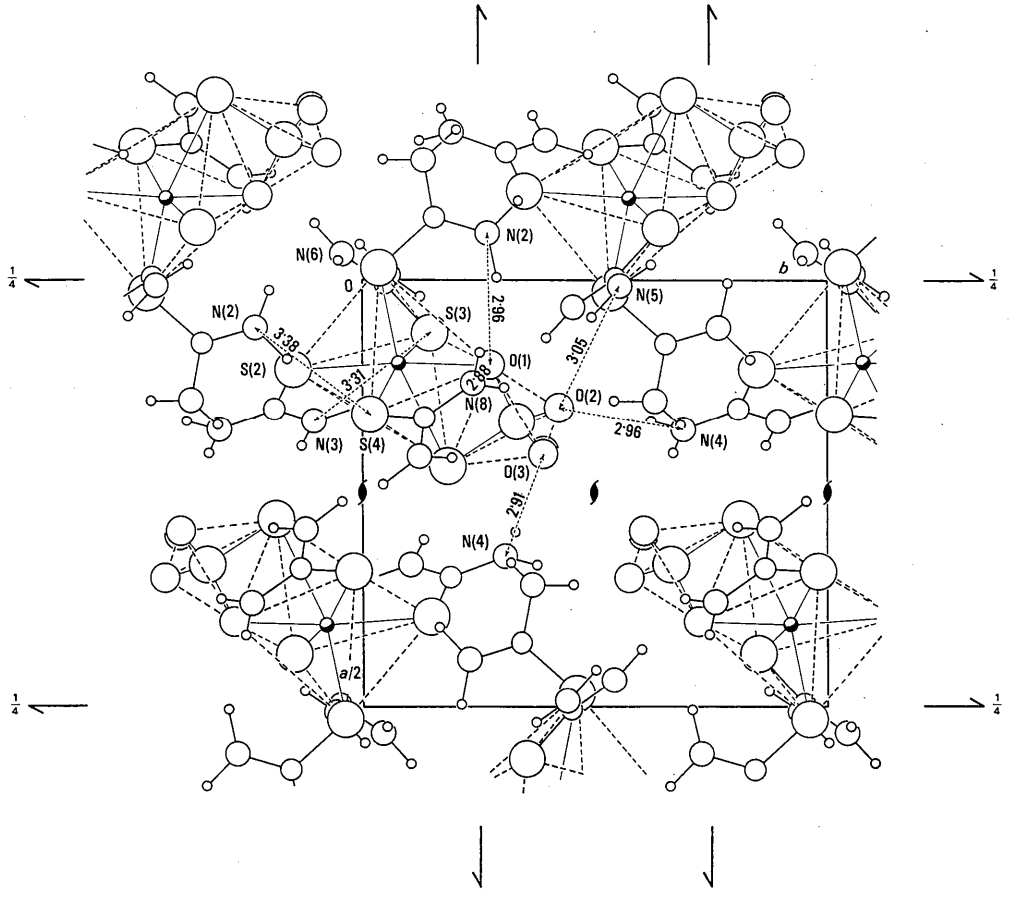

Fig.2. Diagrammatic projection of the structure of $Ni tu_4S_2O_3.H_2O$ on (001).

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The Crystal Structure of Ninhydrin

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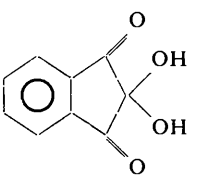
The crystal structure of ninhydrin, $C_9H_6O_4$, has been determined using filtered Cu $K\alpha$ radiation and a Nonius integrating Weissenberg camera. The crystals are monoclinic, $P2_1$, with $a=11{\cdot}24$, $b=6{\cdot}06$, $c=5{\cdot}77$ Å, $\beta=99{\cdot}1°$, and $Z=2$. The intensities of 849 independent reflections were measured with a densitometer. The structure was solved using Patterson and Fourier methods and refined by the anisotropic full-matrix least-squares method to an R value of 0·062. The molecules are connected in layers parallel to (100) by two types of hydrogen bonds. Each molecule is linked along **c** by two hydroxyl–carbonyl bonds and about a screw axis in the **b** direction by two hydroxyl–hydroxyl bonds. The oxygen–oxygen distance is 2·80 Å for both types. One carbonyl oxygen is not involved in the hydrogen bonding scheme.

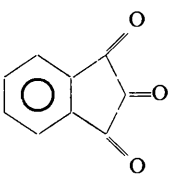
Introduction

Interest in ninhydrin, $C_9H_6O_4$, was originally stimulated by its rather high melting point and its resistance to dehydration in air which is unusual for a *gem*-dihydroxy compound; that is, a compound with two hydroxyl groups attached to the same carbon atom. It is a valuable reagent in the qualitative and quantitative determination of α-amino acids, proteins, and some of their degradation products because the reaction produces a deep blue color and also causes a stoichiometric amount of carbon dioxide to be evolved. Since this work began, reference has been made (Tollin & Cochran, 1964) to an unpublished thesis (Tollin, 1963) concerning this crystal structure. An account of the initial synthesis of ninhydrin by Ruhemann in 1910 and his subsequent study of its reactions, including the one cited above, has recently appeared (West, 1965).

Ninhydrin(I) is also referred to as triketoindane monohydrate or 1,2,3-indantrione monohydrate, which are, as are chloral hydrate and alloxan monohydrate, misnomers because there are no water molecules as such in any of the structures. The structure of the anhydrous form of ninhydrin, triketoindane(II), has also been reported (Bolton, 1965).



(I)



(II)

Other structure determinations of *gem*-dihydroxy compounds are chloral hydrate (Brown & Levy, 1962), alloxan monohydrate or 5,5-dihydroxybarbituric acid (Singh, 1965), alloxan tetrahydrate or 5,5-dihydroxybarbituric acid trihydrate (Mootz & Jeffrey, 1965), and 3,3,4,4-tetrahydrofurantetrol (Mighell & Jacobson, 1964). In these structures all groups of the type OH, NH, or CO, when present, form at least one hydrogen bond.

Experimental

Crystals of commercially obtained ninhydrin were grown by slow cooling of an aqueous solution which had been saturated at a temperature slightly above am-

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