Acta Cryst. (1968). B**24**, 367

The Crystal and Molecular Structure of Monothiosemicarbazidesilver(I) chloride

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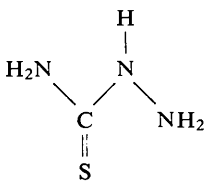
(*Received* 5 *July* 1967)

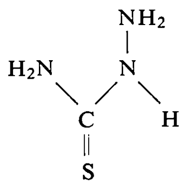
The crystal structure of monothiosemicarbazidesilver(I) chloride has been determined by three-dimensional X-ray analysis. There are eight formula units, $Ag[SC(NH_2)NHNH_2]Cl$, in an orthorhombic unit cell, $a=11{\cdot}93\pm0{\cdot}01$, $b=24{\cdot}86\pm0{\cdot}05$, $c=4{\cdot}078\pm0{\cdot}005$ Å, space group $P2_12_12_1$. Two kinds of silver atom are present, both with a tetrahedral environment: Ag(1) coordinates to two Cl's (Ag–Cl, $2{\cdot}65\pm0{\cdot}01$; $2{\cdot}75\pm0{\cdot}01$ Å) and to two S's (Ag–S, $2{\cdot}50\pm0{\cdot}01$, $2{\cdot}51\pm0{\cdot}01$ Å), Ag(2) is bonded to one Cl(Ag–Cl$=2{\cdot}66\pm0{\cdot}01$ Å) and to three S's (Ag–S, $2{\cdot}51\pm0{\cdot}01$, $2{\cdot}48\pm0{\cdot}01$, $2{\cdot}77\pm0.01$ Å). The complex is polymeric, with the coordination polyhedra linked in helical chains around a 2_1 axis parallel to the c direction. There is a particularly short translation period along c.

Introduction

Thiosemicarbazide is a well known chelating agent, a property connected with the molecular conformation, in which sulphur and the hydrazinic NH_2 group are *cis* with respect to the C–NH bond (I).



(I)



(II)

With this conformation of the ligand, five-membered chelation rings can be formed, as found in monomeric complex compounds (Cavalca, Nardelli & Branchi, 1960; Cavalca, Nardelli & Fava, 1962; Grønbæk & Rasmussen, 1962; Grønbæk, 1963; Grønbæk Hazell, 1966). On the other hand, the high value of the dipole moment (5·36 D) (Mautner & Kumler, 1956), particularly compared with those of semicarbazide (3·77 D) (Mautner & Kumler, 1956), thiourea and urea (4·89 and 4·56 D respectively) (Kumler & Fohlen, 1942), suggests that the *trans* (II) conformation is possible too, at least in dioxane solution. This conformation has also been found recently for thiosemicarbazide in the crystalline state (Domiano, Fava Gasparri & Nardelli, 1966), which in this respect is similar to biuret (Nardelli, Fava & Giraldi, 1963), dimethylglyoxime (Merritt & Lanterman, 1952; Godycki & Rundle, 1953) and glyoxime (Calleri, Ferraris & Viterbo, 1966*a*,*b*). It is probable that the energy barrier to hindered rotation of the C–N bond is of the same magnitude as that found in other thioamides (24–36 kcal.mole^{-1}) (Loewenstein, Melera, Rigny & Walter, 1964) and it can be overcome at the expense of chelation or of reticular packing energy.

An interesting point is whether thiosemicarbazide can behave as a monodentate ligand assuming the *trans* conformation (II), in the same way as biuret. The crystal structure of monothiosemicarbazidesilver(I) chloride was studied as, judging from the crystal data and particularly from the low value of the cell constant c (Nardelli, Fava Gasparri & Chierici, 1965), it was possible to foresee a polymeric structure in which the sharing of the anions and the low coordination number of the metal atom make it unlikely that two adjacent positions for chelation will be found. These views were confirmed by the crystal structure analysis reported in the present paper.

Experimental

Monothiosemicarbazidesilver chloride gives very slender colourless orthorhombic needles elongated along [001]. Cell constants, determined from Weissenberg and rotation photographs taken around the elongation axis (Ni-filtered Cu radiation, $\lambda=1{\cdot}5418$ Å) are as follows:

$Ag[SC(NH_2)NHNH_2]Cl$, $M=234{\cdot}5$

$a=11{\cdot}93\pm0{\cdot}01$, $b=24{\cdot}86\pm0{\cdot}05$, $c=4{\cdot}078\pm0{\cdot}005$ Å

$V=1209$ Å^3, $Z=8$, $D_x=2{\cdot}57$, $D_m=2{\cdot}69$ g.cm^{-3} (flotation)

$\mu=338{\cdot}4$ cm^{-1} (Cu $K\alpha$), $F(000)=896$

Space group: $P2_12_12_1$ (from systematic absences and structure analysis).

The intensity data were determined photometrically on the integrated and non-integrated equi-inclination Weissenberg photographs taken around [001] up to the third layer (multiple film technique, Cu $K\alpha$). Of the 1524 possible independent reflexions within the Cu $K\alpha$ sphere, 1379 were recorded; of these, 485 had intensities which were too weak to be estimated. Correction for absorption was applied assuming a cylindrical shape with a mean radius of $8{\cdot}10^{-4}$ cm, and the shape of the spots of non-equatorial layers was taken into account following Phillips (1956). The structure amplitudes were derived by means of the usual formulae, the absolute scale being established for each layer separately first by Wilson's method, then by correlation with the calculated values.

Structure analysis and refinement

At the beginning of the analysis there was no information concerning the extinctions of $00l$ reflexions, which could not be registered on the photographs around [001]; both the $P2_12_12$ and $P2_12_12_1$ space groups were therefore considered. There are four equivalent points in the general position of these two groups, and so there must be at least two non-equivalent silver atoms in the unit cell. Bearing this in mind, it was possible to find Ag–Ag vectors in the $P(U,V)$ Patterson projection for the two non-equivalent sets of metal atoms. Their contributions to the structure factors were sufficient to allow the use of the heavy atom method in the succeeding analysis. The projections on (001) are equal for the two possible space groups, and it was unnecessary to distinguish between them at the two-dimensional stage. The presence of the screw diad along [001] came from a three-dimensional Patterson calculation which was used to get the z coordinates for the heavier atoms. The analysis was then carried out with three-dimensional Fourier syntheses which gave the coordinates for all the atoms (except, of course, hydrogen).

The refinement was accomplished with several cycles of Booth's differential synthesis using anisotropic thermal parameters. These parameters were derived from the second derivatives of the electron density from differential synthesis, following the method of Nardelli & Fava (1960). At the end of this refinement, the ratios $r(x)=\sigma(x)/\varepsilon(x)$ between the e.s.d.'s and the shifts of the coordinates were as shown in Table 1 and the agreement indices (R, for observed reflexions only, R' assuming $F_o=\frac{1}{2}F_{\min}$ when $F_c>F_{\min}$ for unobserved reflexions; multiplicities not considered) were $R=10{\cdot}5\%$, $R'=15{\cdot}9\%$.

The final coordinates with e.s.d.'s (Cruickshank, 1949) are given in Table 1 and the comparison between observed and calculated peak shapes is shown in Table 2. As a consequence of the scaling by layers, the thermal parameters, B_{ij}, listed in Table 1 must be considered simply as additional parameters introduced to reduce the residuals. The F_c values reported in Table 3 are calculated with the final parameters of Table 1 using the scattering factors of Thomas & Umeda (1957) for Ag^+, of Dawson (1960) for S and of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for Cl^-, N and C. The standard deviations, quoted in the next section, are calculated from the formulae of Ahmed & Cruickshank (1953) for bond lengths and of Darlow (1960) for angles.

All the calculations were performed on the Olivetti Elea 6001/S computer of the Centro di Calcolo Elettronico della Università di Parma, with the programs of Nardelli, Musatti, Domiano & Andreetti (1964, 1965).

Discussion

Coordination around each silver atom is tetrahedral, and there are two kinds of tetrahedron which are equivalent from neither crystallographic nor chemical points of view: Ag(1) coordinates to two Cl's [Cl(2) and Cl(2^{i})] and two S's [S(1) and S(2^{ii})], while Ag(2) coordinates to one Cl [Cl(1)] and to three S's [S(1), S(1^{iii}) and S(2)] as shown in Fig. 1. From the values of distances and angles in the coordination polyhedra quoted in Table 4, it can be deduced that in both tetrahedra there are two shorter distances in the range 2·48–2·51 Å, one medium (2·65 and 2·66 Å) and one longer (2·75 and 2·77 Å). S(1) is bonded to three silver atoms: two bond lengths (2·50 and 2·51 Å) are near to the sum of covalent radii (2·57 Å), while the third (2·77 Å) is intermediate between the sums of covalent and ionic (3·07 Å) radii. S(2) is bonded to two silver atoms at distances which are not significantly different (2·48 and 2·51 Å) and are near to the sum of the covalent radii. These Ag–S distances are in good agreement with those found in other compounds as shown in Table 5.

There are two kinds of chlorine atom: Cl(1) is bonded to Ag(2) at 2·66 Å and Cl(2) is shared by two adjacent Ag(1) atoms at 2·65 and 2·75 Å. The shorter values of these Ag–Cl distances are intermediate between the

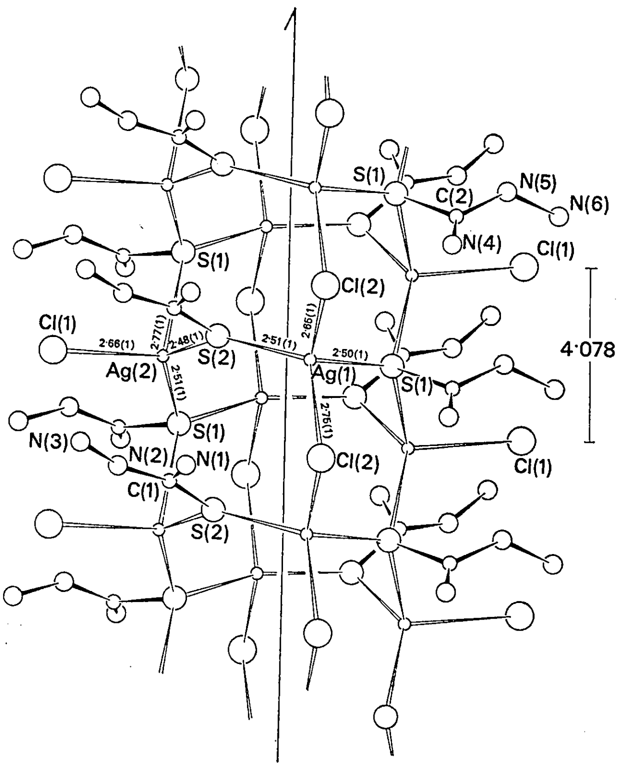


Fig. 1. $Ag[SC(NH_2)NHNH_2]Cl$: clinographic projection of a chain of coordination tetrahedra.

sum of covalent radii (2·52 Å) and the same distance (2·77 Å) in AgCl (Wilman, 1940); the longer value is equal to the distance in AgCl.

The coordination polyhedra are linked in chains around a screw diad running parallel to [001], in such a way that the Ag and S atoms form a kind of tube having a distorted octagonal cross-section. The internal channel of the tube is about 1·5 Å in diameter, and is determined by the S–S contacts (Fig. 2): the chlorine atoms are on the external part of this chain.

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

	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}	$\|r(x)\|$	$\|r(y)\|$	$\|r(z)\|$
Ag(1)	475 ± 2	4174 ± 1	927 ± 9	34 ± 1	30 ± 1	48 ± 3	−1 ± 2	6 ± 3	3 ± 2	3	∞	22
Ag(2)	3748 ± 2	3985 ± 1	4596 ± 7	35 ± 1	32 ± 1	40 ± 2	−4 ± 2	−7 ± 2	−2 ± 1	10	∞	7
Cl(1)	5153 ± 6	3157 ± 3	4981 ± 22	28 ± 3	24 ± 3	27 ± 6	−1 ± 7	−1 ± 7	8 ± 5	10	∞	11
Cl(2)	−654 ± 5	3711 ± 3	5756 ± 24	25 ± 3	25 ± 3	24 ± 6	0 ± 6	−1 ± 6	3 ± 5	27	∞	13
S(1)	2401 ± 6	3805 ± 2	12 ± 22	19 ± 2	21 ± 2	30 ± 6	−7 ± 5	1 ± 6	−2 ± 4	6	∞	110
S(2)	4604 ± 5	4824 ± 3	6697 ± 21	23 ± 2	24 ± 3	32 ± 7	1 ± 6	−4 ± 6	−1 ± 4	26	∞	15
N(1)	6462 ± 18	5029 ± 11	10145 ± 81	19 ± 7	27 ± 14	24 ± 21	11 ± 25	−8 ± 21	4 ± 17	23	54	57
N(2)	6041 ± 18	4166 ± 9	9346 ± 72	23 ± 8	21 ± 8	33 ± 23	−4 ± 20	0 ± 22	−2 ± 15	11	43	13
N(3)	7028 ± 28	4017 ± 14	10979 ± 68	62 ± 19	24 ± 14	57 ± 31	13 ± 33	−19 ± 36	2 ± 27	8	∞	9
N(4)	3036 ± 35	2761 ± 22	543 ± 86	57 ± 27	61 ± 32	50 ± 33	6 ± 51	4 ± 50	8 ± 45	9	14	4
N(5)	3098 ± 21	2238 ± 12	−579 ± 98	39 ± 14	27 ± 14	75 ± 49	16 ± 40	2 ± 41	2 ± 25	8	63	20
N(6)	1386 ± 23	2893 ± 11	−1885 ± 63	51 ± 12	34 ± 19	73 ± 26	4 ± 32	−40 ± 27	10 ± 25	39	14	5
C(1)	5810 ± 32	4658 ± 12	8678 ± 127	21 ± 16	22 ± 12	36 ± 33	−1 ± 31	15 ± 35	11 ± 23	27	30	33
C(2)	2286 ± 18	3117 ± 12	−765 ± 47	30 ± 5	45 ± 9	17 ± 30	−44 ± 1	−23 ± 1	13 ± 0	9	27	16

Table 2. *Atomic peak heights* (e.$Å^{-3}$) *and curvatures* (e.$Å^{-5}$)

		ϱ	$-A_{hh}$	$-A_{kk}$	$-A_{ll}$	A_{kl}	A_{hl}	A_{hk}
Ag(1)	obs.	88·5	839	848	496	−10	41	30
	calc.	90·2	838	845	507	−10	37	29
Ag(2)	obs.	90·2	836	867	520	−27	−38	−23
	calc.	91·9	835	866	528	−25	−35	−23
Cl(1)	obs.	29·9	284	297	169	−11	−15	31
	calc.	30·5	284	296	171	−11	−15	30
Cl(2)	obs.	31·8	321	313	181	1	5	4
	calc.	32·3	320	314	184	1	6	4
S(1)	obs.	30·7	296	317	181	−11	−2	−5
	calc.	31·4	295	316	186	−8	−2	−5
S(2)	obs.	29·7	301	297	182	−3	−27	12
	calc.	30·5	300	297	186	−3	−27	12
N(1)	obs.	10·2	107	95	57	6	1	12
	calc.	10·3	107	96	57	5	2	12
N(2)	obs.	10·7	103	97	58	−4	3	5
	calc.	10·9	103	96	58	−4	3	5
N(3)	obs.	8·6	64	71	51	−3	−5	10
	calc.	8·6	65	71	52	−3	−3	10
N(4)	obs.	7·5	66	52	43	−1	−4	16
	calc.	7·3	64	52	43	−2	−4	14
N(5)	obs.	8·1	74	74	43	4	−5	2
	calc.	8·5	73	73	45	3	−5	2
N(6)	obs.	8·4	64	82	44	−5	−16	8
	calc.	8·4	64	82	45	−5	−15	8
C(1)	obs.	10·0	97	105	54	5	20	16
	calc.	10·0	96	106	55	5	20	14
C(2)	obs.	8·4	95	60	52	−20	−6	5
	calc.	8·5	95	60	51	−17	−4	5
	e.s.d.	0·7	9	10	6	5	5	6

Both non-equivalent thiosemicarbazide molecules are in a *trans* (II) conformational state and behave as monodentate ligands. This behaviour can be explained by the consideration that in the coordination polyhedra chain, which must be a particularly stable configuration of this silver compound, there are no sites near enough to each other to be occupied by two atoms of the same organic molecule. The molecule therefore assumes the *trans* conformation as a result of packing requirements.

In Table 6 distances and angles in the thiosemicarbazide ligands, as found in the silver complex, are compared with those found in uncoordinated thiosemicarbazide and in other complexes. Owing to the presence of the heavy silver atoms, the accuracy of the coordi-

Table 3. *Observed and calculated structure factors*

A minus sign for F_o means 'less than'.

h	k	l	$\|10F_o\|$	$10F_c$	α°
0	1	1	65-	20	90
0	2	0	1782	1399	0
0	2	1	831	645	0
0	2	2	181	120	0
0	2	3	672	695	0
0	3	1	87-	1	270
0	3	2	220	191	270
0	3	3	326	323	270
0	4	0	2409	2421	180
0	4	1	993	809	0
0	4	2	1530	1526	180
0	4	3	765	742	0
0	5	1	1520	1298	90
0	5	2	293	306	90
0	5	3	195-	106	90
0	6	0	2258	2306	180
0	6	1	600	477	180
0	6	2	875	1004	180
0	7	1	121-	116	270
0	7	2	278	193	90
0	7	3	270	192	90
0	8	0	697	502	0
0	8	1	1394	1341	180
0	8	2	399	293	0
0	8	3	1071	968	180
0	9	1	750	628	270
0	9	2	152-	56	270
0	9	3	675	652	90
0	10	0	2923	2969	0
0	10	1	1035	1001	180
0	10	2	1204	1195	0
0	10	3	557	519	180
0	11	1	1275	1148	270
0	11	2	167-	104	270
0	11	3	249-	4	270
0	12	0	843	919	0
0	12	1	161-	69	180
0	12	2	174-	212	0
0	12	3	303	290	0
0	13	1	826	665	270
0	13	2	674	762	270
0	13	3	371	357	270
0	14	0	494	464	180
0	14	1	649	509	0
0	14	2	587	591	180
0	14	3	785	714	0
0	15	1	1155	996	90
0	15	2	470	496	270
0	15	3	265-	167	90
0	16	0	571	577	180
0	16	1	193-	56	180
0	16	2	199-	68	0
0	16	3	265-	52	0
0	17	1	679	527	90
0	17	2	348	288	90
0	17	3	283	298	90
0	18	0	431	543	180
0	18	1	410	408	180
0	18	2	207	91	0
0	18	3	317	367	180
0	19	1	212-	57	90
0	19	2	760	754	90
0	19	3	320	367	90
0	20	0	411	331	180
0	20	1	375	362	180
0	20	2	208-	74	0
0	20	3	243-	107	180
0	21	1	878	803	270
0	21	2	303	315	90
0	21	3	232-	52	90
0	22	0	533	470	0
0	22	1	220-	105	180
0	22	2	323	309	0
0	22	3	217-	53	0
0	23	1	927	974	270
0	23	2	365	376	270
0	23	3	200-	276	270
0	24	0	225-	63	0
0	24	1	215-	8	180
0	24	2	186-	69	180
0	24	3	180-	64	0
0	25	1	208-	108	90
0	25	2	263	291	270
0	25	3	155-	77	90
0	26	0	213-	19	180
0	26	1	197-	29	0
0	26	2	161-	147	0
0	26	3	116-	29	180
0	27	1	723	751	90
0	27	2	145-	174	90
0	28	0	188-	129	180
0	28	1	171-	96	0
0	28	2	126-	79	0
0	29	1	214	263	90
0	29	2	267	351	90
0	30	0	151-	241	180
0	30	1	130-	18	0
0	32	0	82-	17	0
1	0	1	622	723	90
1	0	2	216	270	0

h	k	l	$\|10F_o\|$	$10F_c$	α°
1	0	3	161-	40	90
1	1	0	1185	1333	270
1	1	1	1544	1710	183
1	1	2	802	1028	215
1	1	3	309	317	262
1	2	0	68-	68	270
1	2	1	1014	972	97
1	2	2	418	436	120
1	2	3	259	255	118
1	3	0	1002	930	90
1	3	1	990	846	311
1	3	2	1480	1550	162
1	3	3	806	747	313
1	4	0	716	588	270
1	4	1	949	800	149
1	4	2	558	508	224
1	4	3	285	258	132
1	5	0	1325	1362	90
1	5	1	1175	1046	358
1	5	2	1043	1019	101
1	5	3	225	246	343
1	6	0	418	382	90
1	6	1	492	340	307
1	6	2	132-	104	261
1	6	3	300	381	281
1	7	0	741	687	90
1	7	1	1607	1521	15
1	7	2	991	972	11
1	7	3	503	454	64
1	8	0	710	636	90
1	8	1	772	710	326
1	8	2	506	498	302
1	8	3	814	685	299
1	9	0	982	985	270
1	9	1	478	374	158
1	9	2	1055	1040	329
1	9	3	236-	164	83
1	10	0	831	666	90
1	10	1	147-	161	59
1	10	2	275	359	327
1	10	3	244-	221	350
1	11	0	704	728	270
1	11	1	1866	1794	177
1	11	2	377	415	318
1	11	3	250-	230	204
1	12	0	1757	1723	90
1	12	1	1035	953	132
1	12	2	551	688	108
1	12	3	578	546	103
1	13	0	583	447	90
1	13	1	199	236	213
1	13	2	565	494	119
1	13	3	446	437	338
1	14	0	173-	60	90
1	14	1	674	732	156
1	14	2	399	328	195
1	14	3	426	445	124
1	15	0	482	533	90
1	15	1	679	706	356
1	15	2	429	521	131
1	15	3	305	301	329
1	16	0	1204	1159	270
1	16	1	597	546	334
1	16	2	733	643	268
1	16	3	265-	207	302
1	17	0	199-	104	90
1	17	1	400	471	329
1	17	2	204-	109	211
1	17	3	263-	163	173
1	18	0	342	361	270
1	18	1	910	946	328
1	18	2	424	442	305
1	18	3	443	470	310
1	19	0	425	279	270
1	19	1	213-	206	41
1	19	2	448	447	330
1	19	3	251-	143	142
1	20	0	602	536	90
1	20	1	342	367	255
1	20	2	328	270	2
1	20	3	242-	247	282
1	21	0	225-	36	90
1	21	1	268	297	154
1	21	2	503	556	358
1	21	3	231-	230	163
1	22	0	1218	1187	90
1	22	1	663	629	183
1	22	2	365	445	80
1	22	3	216-	183	176
1	23	0	399	245	90
1	23	1	219-	106	20
1	23	2	323	265	47
1	23	3	199-	79	354
1	24	0	349	197	90
1	24	1	263	279	148
1	24	2	185-	225	156
1	24	3	178-	287	112
1	25	0	220-	53	90
1	25	1	255	204	353
1	25	2	174-	155	177

h	k	l	$\|10F_o\|$	$10F_c$	α°
1	25	3	152-	172	319
1	26	0	729	748	270
1	26	1	462	419	19
1	26	2	461	433	248
1	27	0	201-	3	270
1	27	1	225	183	192
1	27	2	144-	166	191
1	28	0	392	323	270
1	28	1	361	375	338
1	28	2	124-	123	306
1	29	0	170-	31	90
1	29	1	151-	136	12
1	29	2	97-	26	190
1	30	0	149-	46	90
1	30	1	331	298	252
1	31	0	184	111	90
2	0	0	1363	1704	0
2	0	1	2725	2991	90
2	0	2	109	139	0
2	0	3	242	305	90
2	1	0	533	549	0
2	1	1	1022	1004	286
2	1	2	861	1044	302
2	1	3	378	407	266
2	2	0	1128	1242	0
2	2	1	1463	1459	79
2	2	2	536	584	104
2	2	3	242	269	25
2	3	0	399	464	0
2	3	1	862	832	71
2	3	2	546	507	354
2	3	3	197-	66	292
2	4	0	380	312	180
2	4	1	1881	1783	279
2	4	2	679	764	108
2	4	3	638	610	309
2	5	0	767	742	180
2	5	1	1160	1055	102
2	5	2	454	500	113
2	5	3	330	257	77
2	6	0	392	248	180
2	6	1	1527	1490	267
2	6	2	457	394	258
2	6	3	296	348	297
2	7	0	1160	1210	180
2	7	1	439	388	303
2	7	2	950	876	166
2	7	3	227-	133	56
2	8	0	945	962	180
2	8	1	1003	864	139
2	8	2	1161	1127	230
2	8	3	452	436	138
2	9	0	869	831	0
2	9	1	916	889	257
2	9	2	159-	68	212
2	9	3	401	334	140
2	10	0	292	366	0
2	10	1	1770	1773	121
2	10	2	664	704	273
2	10	3	707	671	148
2	11	0	1395	1320	0
2	11	1	1181	1098	278
2	11	2	832	784	321
2	11	3	254-	188	199
2	12	0	1192	1156	0
2	12	1	514	613	106
2	12	2	520	513	37
2	12	3	259-	104	7
2	13	0	431	494	0
2	13	1	427	421	40
2	13	2	404	461	305
2	13	3	274	268	24
2	14	0	342	343	0
2	14	1	514	433	290
2	14	2	415	484	81
2	14	3	386	431	325
2	15	0	633	571	180
2	15	1	1058	1106	73
2	15	2	362	293	224
2	15	3	520	606	41
2	16	0	194-	117	180
2	16	1	437	542	282
2	16	2	201-	97	300
2	16	3	265-	229	344
2	17	0	843	825	180
2	17	1	203-	99	239
2	17	2	652	679	155
2	17	3	261-	49	265
2	18	0	1065	1135	180
2	18	1	209-	212	225
2	18	2	493	548	224
2	18	3	256-	250	97
2	19	0	323	150	0
2	19	1	661	697	232
2	19	2	466	530	103
2	19	3	350	393	181
2	20	0	482	487	180
2	20	1	467	458	130
2	20	2	207-	180	246
2	20	3	300	339	116

h	k	l	$\|10F_o\|$	$10F_c$	α°
2	21	0	697	777	0
2	21	1	220-	218	234
2	21	2	327	333	19
2	21	3	250	285	131
2	22	0	443	352	0
2	22	1	217	250	146
2	22	2	382	346	20
2	22	3	211-	56	171
2	23	0	621	743	0
2	23	1	307	310	292
2	23	2	287	347	331
2	23	3	193-	238	19
2	24	0	317	193	0
2	24	1	212-	102	178
2	24	2	182-	52	7
2	24	3	171-	127	232
2	25	0	219-	64	180
2	25	1	204-	217	45
2	25	2	141-	158	256
2	25	3	144-	265	23
2	26	0	209-	30	180
2	26	1	194-	156	297
2	26	2	156-	34	253
2	27	0	474	504	180
2	27	1	181-	138	129
2	27	2	332	312	163
2	28	0	183-	133	180
2	28	1	165-	159	13
2	28	2	119-	105	73
2	29	0	335	326	180
2	29	1	206	204	152
2	29	2	186	262	123
2	30	0	266	288	180
2	30	1	152	175	66
2	31	0	278	263	0
3	0	1	1089	1120	90
3	0	2	474	463	180
3	0	3	540	589	90
3	1	0	1058	1142	270
3	1	1	723	724	243
3	1	2	107	254	210
3	1	3	677	730	298
3	2	0	1426	1604	270
3	2	1	500	543	118
3	2	2	825	866	232
3	2	3	566	552	151
3	3	0	576	649	270
3	3	1	1224	1179	249
3	3	2	317	243	209
3	3	3	878	965	278
3	4	0	1877	2099	270
3	4	1	125-	98	209
3	4	2	841	938	251
3	4	3	399	404	234
3	5	0	1237	1349	90
3	5	1	957	867	186
3	5	2	634	682	98
3	5	3	516	549	190
3	6	0	1477	1501	90
3	6	1	706	608	317
3	6	2	551	571	54
3	6	3	780	717	310
3	7	0	1490	1526	90
3	7	1	853	741	62
3	7	2	622	629	76
3	7	3	589	552	86
3	8	0	1795	1859	90
3	8	1	147-	169	334
3	8	2	998	963	47
3	8	3	611	587	348
3	9	0	425	524	270
3	9	1	1052	1013	41
3	9	2	503	437	352
3	9	3	643	604	46
3	10	0	526	615	90
3	10	1	998	981	145
3	10	2	283	200	236
3	10	3	476	425	96
3	11	0	1521	1607	270
3	11	1	598	603	160
3	11	2	647	678	307
3	11	3	258-	97	295
3	12	0	311	186	270
3	12	1	1001	1012	157
3	12	2	711	749	199
3	12	3	369	424	109
3	13	0	862	885	270
3	13	1	816	715	196
3	13	2	339	310	312
3	13	3	278	301	265
3	14	0	684	814	270
3	14	1	206	166	169
3	14	2	490	584	241
3	14	3	265-	104	310
3	15	0	831	884	90
3	15	1	492	540	195
3	15	2	580	579	106
3	15	3	283	371	218
3	16	0	199-	41	90
3	16	1	949	924	339

h	k	l	$\|10F_o\|$	$10F_c$	α°
3	16	2	305	235	350
3	16	3	283	346	307
3	17	0	843	977	90
3	17	1	206	166	357
3	17	2	397	437	121
3	17	3	259-	187	213
3	18	0	672	777	90
3	18	1	396	452	331
3	18	2	621	637	40
3	18	3	252-	214	326
3	19	0	392	304	270
3	19	1	717	851	21
3	19	2	415	379	293
3	19	3	243-	249	42
3	20	0	317	326	90
3	20	1	514	558	209
3	20	2	339	338	338
3	20	3	232-	143	120
3	21	0	342	303	270
3	21	1	348	386	79
3	21	2	436	468	325
3	21	3	219-	229	106
3	22	0	228-	91	90
3	22	1	516	599	151
3	22	2	160-	116	330
3	22	3	203-	88	88
3	23	0	354	171	90
3	23	1	339	329	162
3	23	2	292	279	25
3	23	3	164-	159	183
3	24	0	222-	35	270
3	24	1	209-	110	356
3	24	2	177-	181	200
3	24	3	160-	98	351
3	25	0	304	288	90
3	25	1	245	283	191
3	25	2	203	210	112
3	26	0	205-	136	270
3	26	1	299	311	345
3	26	2	272	241	188
3	27	0	192-	38	90
3	27	1	175-	152	210
3	27	2	226	197	199
3	28	0	177-	188	90
3	28	1	192	175	335
3	28	2	109-	200	73
3	29	0	158-	71	270
3	29	1	356	319	358
3	30	0	135-	53	270
3	30	1	113-	97	265
3	31	0	278	192	90
4	0	0	241	119	180
4	0	1	1379	1404	90
4	0	2	147-	208	180
4	0	3	450	415	270
4	1	0	519	566	180
4	1	1	1150	1206	46
4	1	2	745	838	254
4	1	3	549	541	28
4	2	0	297	461	180
4	2	1	478	494	93
4	2	2	629	672	151
4	2	3	223	295	237
4	3	0	431	557	180
4	3	1	1780	1798	91
4	3	2	151-	74	169
4	3	3	690	637	110
4	4	0	335	461	0
4	4	1	238	349	264
4	4	2	437	451	63
4	4	3	236-	84	294
4	5	0	1065	1172	180
4	5	1	223	204	203
4	5	2	672	756	127
4	5	3	384	410	176
4	6	0	849	903	0
4	6	1	968	1024	280
4	6	2	448	497	21
4	6	3	244-	228	329
4	7	0	234	275	0
4	7	1	1846	1891	262
4	7	2	354	395	109
4	7	3	632	671	247
4	8	0	640	662	180
4	8	1	157-	148	193
4	8	2	894	959	239
4	8	3	252-	177	355
4	9	0	1502	1577	0
4	9	1	415	523	296
4	9	2	579	601	315
4	9	3	256-	144	64
4	10	0	571	626	189
4	10	1	353	197	158
4	10	2	867	939	243
4	10	3	270	229	238
4	11	0	956	1046	0
4	11	1	674	704	66
4	11	2	491	495	291
4	11	3	469	509	78
4	12	0	177-	38	180

h	k	l	$\|10F_o\|$	$10F_c$	α°
4	12	1	540	514	135
4	12	2	340	326	212
4	12	3	303	343	199
4	13	0	380	437	180
4	13	1	777	719	53
4	13	2	379	379	198
4	13	3	281	325	33
4	14	0	374	368	0
4	14	1	609	636	96
4	14	2	491	488	62
4	14	3	265-	76	79
4	15	0	779	803	180
4	15	1	245	205	355
4	15	2	524	525	142
4	15	3	263-	209	6
4	16	0	760	834	0
4	16	1	356	366	303
4	16	2	580	615	20
4	16	3	290	320	6
4	17	0	212-	18	0
4	17	1	590	710	249
4	17	2	351	362	103
4	17	3	253-	288	241
4	18	0	627	568	180
4	18	1	555	589	271
4	18	2	404	308	208
4	18	3	245-	231	20
4	19	0	507	406	0
4	19	1	437	440	199
4	19	2	207-	32	272
4	19	3	350	399	165
4	20	0	875	953	180
4	20	1	459	549	254
4	20	2	605	570	203
4	20	3	223-	139	155
4	21	0	602	679	0
4	21	1	428	335	119
4	21	2	198-	116	327
4	21	3	270	301	103
4	22	0	227-	5	180
4	22	1	266	322	154
4	22	2	233	206	273
4	22	3	220	291	175
4	23	0	292	221	0
4	23	1	212-	180	16
4	23	2	181-	85	195
4	23	3	250	265	350
4	24	0	462	319	0
4	24	1	324	396	100
4	24	2	287	277	340
4	24	3	141-	56	147
4	25	0	209-	78	0
4	25	1	193-	84	18
4	25	2	136-	75	34
4	26	0	545	354	0
4	26	1	181-	141	31
4	26	2	261	290	12
4	27	0	304	272	180
4	27	1	166-	126	209
4	27	2	120-	124	116
4	28	0	330	331	180
4	28	1	258	227	293
4	28	2	230	289	119
4	29	0	259	333	180
4	29	1	268	234	205
4	30	0	494	558	180
4	31	0	72-	68	0
5	0	1	233	255	90
5	0	2	859	1044	180
5	0	3	431	307	90
5	1	0	678	806	270
5	1	1	296	356	276
5	1	2	454	358	22
5	1	3	245-	193	300
5	2	0	488	646	270
5	2	1	2146	2286	1
5	2	2	736	818	211
5	2	3	638	542	8
5	3	0	906	968	270
5	3	1	486	423	286
5	3	2	537	555	342
5	3	3	412	439	298
5	4	0	812	971	270
5	4	1	870	941	353
5	4	2	533	616	281
5	4	3	305	323	286
5	5	0	151-	14	90
5	5	1	750	845	189
5	5	2	171-	115	274
5	5	3	232	271	205
5	6	0	595	586	90
5	6	1	944	1002	182
5	6	2	748	856	27
5	6	3	272	252	225
5	7	0	425	435	90
5	7	1	285	394	114
5	7	2	411	321	147
5	7	3	258-	144	57
5	8	0	767	881	90
5	8	1	1352	1467	172

Table 3 (*cont.*)

h	k	l	$\|10F_o\|$	$10F_c$	α°
5	8	2	510	612	60
5	8	3	260-	233	113
5	9	0	335	237	90
5	9	1	750	760	41
5	9	2	283	179	100
5	9	3	263-	237	22
5	10	0	176-	45	270
5	10	1	402	474	158
5	10	2	543	579	195
5	10	3	390	411	30
5	11	0	507	571	270
5	11	1	320	442	64
5	11	2	339	332	35
5	11	3	390	269	96
5	12	0	437	403	270
5	12	1	573	545	357
5	12	2	711	791	199
5	12	3	344	447	359
5	13	0	1077	1121	270
5	13	1	277	299	93
5	13	2	402	438	311
5	13	3	260-	100	88
5	14	0	361	431	270
5	14	1	203-	218	354
5	14	2	320	344	246
5	14	3	262-	92	251
5	15	0	621	525	270
5	15	1	484	621	199
5	15	2	442	420	269
5	15	3	350	362	230
5	16	0	482	343	90
5	16	1	213-	53	245
5	16	2	473	539	14
5	16	3	251-	170	206
5	17	0	462	336	90
5	17	1	217	204	226
5	17	2	208-	56	183
5	17	3	400	419	257
5	18	0	722	609	90
5	18	1	219-	66	55
5	18	2	421	485	41
5	18	3	234-	109	35
5	19	0	779	769	90
5	19	1	693	751	16
5	19	2	245	206	112
5	19	3	223-	223	359
5	20	0	228-	129	270
5	20	1	348	406	174
5	20	2	224	188	287
5	20	3	208-	185	30
5	21	0	551	486	90
5	21	1	457	492	71
5	21	2	201	257	81
5	21	3	300	399	92
5	22	0	278	149	270
5	22	1	302	291	174
5	22	2	183-	129	256
5	22	3	172-	129	21
5	23	0	330	234	270
5	23	1	489	408	128
5	23	2	290	218	303
5	23	3	250	284	113
5	24	0	211-	84	90
5	24	1	196-	68	298
5	24	2	159-	98	160
5	25	0	627	659	270
5	25	1	225	253	204
5	25	2	554	498	269
5	26	0	188-	64	90
5	26	1	170-	161	315
5	26	2	126-	28	194
5	27	0	172-	58	270
5	27	1	217	187	246
5	27	2	102-	151	234
5	28	0	153-	92	90
5	28	1	208	126	346
5	29	0	526	568	90
5	29	1	315	317	350
5	30	0	97-	64	270
6	0	0	380	493	180
6	0	1	165-	92	270
6	0	2	177-	104	180
6	0	3	694	645	270
6	1	0	989	1069	180
6	1	1	1090	1101	341
6	1	2	599	653	175
6	1	3	839	780	344
6	2	0	425	456	180
6	2	1	439	410	85
6	2	2	179-	132	139
6	2	3	291	312	292
6	3	0	1845	2108	180
6	3	1	351	347	283
6	3	2	875	916	187
6	3	3	259-	181	195
6	4	0	817	854	0
6	4	1	170-	107	160
6	4	2	510	539	42
6	4	3	260-	159	149
6	5	0	167-	133	180
6	5	1	528	581	182
6	5	2	211	246	255
6	5	3	928	903	168
6	6	0	411	481	0
6	6	1	176-	77	224
6	6	2	322	280	36
6	6	3	263-	108	115
6	7	0	1218	1366	0
6	7	1	334	349	208
6	7	2	364	480	6
6	7	3	324	358	174
6	8	0	368	436	180
6	8	1	386	354	298
6	8	2	570	535	227
6	8	3	265-	112	302
6	9	0	1579	1841	0
6	9	1	560	555	48
6	9	2	516	597	354
6	9	3	240	256	19
6	10	0	197-	168	0
6	10	1	467	533	290
6	10	2	637	764	277
6	10	3	353	445	292
6	11	0	195-	107	0
6	11	1	418	575	350
6	11	2	460	415	188
6	11	3	353	501	338
6	12	0	311	291	0
6	12	1	247	195	91
6	12	2	498	502	312
6	12	3	262-	282	263
6	13	0	1027	1155	180
6	13	1	386	423	312
6	13	2	654	732	172
6	13	3	259-	156	348
6	14	0	211-	79	180
6	14	1	813	810	99
6	14	2	231	237	112
6	14	3	270	298	119
6	15	0	216-	56	180
6	15	1	215-	208	119
6	15	2	263	248	97
6	15	3	300	301	123
6	16	0	221-	128	180
6	16	1	307	296	109
6	16	2	394	412	101
6	16	3	240-	178	95
6	17	0	462	431	0
6	17	1	472	429	207
6	17	2	205-	163	41
6	17	3	231-	294	192
6	18	0	216	157	0
6	18	1	826	744	273
6	18	2	202-	39	218
6	18	3	219-	51	337
6	19	0	519	648	0
6	19	1	219-	233	187
6	19	2	197-	187	299
6	19	3	205-	153	206
6	20	0	317	336	0
6	20	1	612	699	265
6	20	2	339	346	296
6	20	3	189-	117	237
6	21	0	224-	139	0
6	21	1	212-	94	115
6	21	2	236	205	194
6	21	3	169-	84	338
6	22	0	197-	49	0
6	22	1	205-	87	252
6	22	2	373	446	297
6	22	3	145-	113	234
6	23	0	211-	120	180
6	23	1	196-	112	40
6	23	2	159-	242	148
6	24	0	475	382	180
6	24	1	573	608	87
6	24	2	261	262	214
6	25	0	189-	107	0
6	25	1	171-	141	96
6	25	2	129-	74	15
6	26	0	212-	76	0
6	26	1	247	239	97
6	26	2	234	220	96
6	27	0	156-	17	0
6	27	1	215	205	224
6	28	0	135-	134	0
6	28	1	437	364	264
6	29	0	259	171	180
7	0	1	448	400	270
7	0	2	727	879	180
7	0	3	265-	155	270
7	1	0	177-	204	270
7	1	1	285	332	192
7	1	2	399	436	68
7	1	3	326	238	195
7	2	0	1388	1534	90
7	2	1	1014	1076	348
7	2	2	881	954	116
7	2	3	265-	271	307
7	3	0	875	998	270
7	3	1	521	590	351
7	3	2	448	406	305
7	3	3	265-	216	342
7	4	0	361	426	90
7	4	1	693	739	257
7	4	2	276	227	21
7	4	3	265-	56	76
7	5	0	184-	182	270
7	5	1	324	333	257
7	5	2	196-	102	311
7	5	3	291	255	235
7	6	0	640	721	270
7	6	1	299	352	120
7	6	2	524	532	330
7	6	3	253	371	64
7	7	0	475	529	90
7	7	1	194-	178	121
7	7	2	415	335	94
7	7	3	265-	180	338
7	8	0	741	851	270
7	8	1	914	944	158
7	8	2	256	414	286
7	8	3	265-	182	85
7	9	0	514	581	90
7	9	1	494	498	132
7	9	2	389	490	88
7	9	3	263-	176	110
7	10	0	361	289	270
7	10	1	762	858	195
7	10	2	537	517	174
7	10	3	261-	203	260
7	11	0	311	286	270
7	11	1	329	425	100
7	11	2	207-	179	154
7	11	3	330	383	143
7	12	0	437	533	90
7	12	1	366	438	326
7	12	2	588	686	136
7	12	3	400	443	313
7	13	0	1071	1133	270
7	13	1	609	625	17
7	13	2	624	616	244
7	13	3	248-	109	50
7	14	0	221-	11	90
7	14	1	467	588	355
7	14	2	207-	71	263
7	14	3	241-	227	10
7	15	0	349	333	270
7	15	1	381	385	353
7	15	2	276	242	311
7	15	3	270	271	312
7	16	0	227-	47	270
7	16	1	217	274	66
7	16	2	288	305	337
7	16	3	223	247	87
7	17	0	824	775	90
7	17	1	220-	102	245
7	17	2	436	452	49
7	17	3	270	291	278
7	18	0	227-	120	90
7	18	1	307	325	161
7	18	2	193-	220	3
7	18	3	197-	25	106
7	19	0	545	655	90
7	19	1	263	217	142
7	19	2	179	181	87
7	19	3	181-	39	349
7	20	0	368	302	270
7	20	1	492	452	199
7	20	2	178-	74	278
7	20	3	161-	98	287
7	21	0	266	209	90
7	21	1	476	439	135
7	21	2	301	310	179
7	21	3	135-	256	123
7	22	0	209-	12	270
7	22	1	193-	19	135
7	22	2	155-	106	115
7	23	0	380	244	270
7	23	1	258	216	63
7	23	2	373	337	217
7	24	0	187-	85	90
7	24	1	169-	152	342
7	24	2	124-	50	74
7	25	0	406	337	270
7	25	1	394	407	355
7	25	2	227	220	292
7	26	0	155-	28	270
7	26	1	135-	70	216
7	27	0	134-	147	90
7	27	1	176	175	302
7	28	0	105-	56	90
8	0	0	241	222	0
8	0	1	197-	178	90
8	0	2	668	636	0
8	0	3	264-	234	270
8	1	0	253	312	180
8	1	1	712	739	312
8	1	2	708	782	96
8	1	3	373	341	332
8	2	0	215	208	180
8	2	1	396	449	95
8	2	2	325	327	60
8	2	3	264-	146	5
8	3	0	197-	161	180
8	3	1	609	708	288
8	3	2	227	262	259
8	3	3	264-	32	135
8	4	0	494	502	180
8	4	1	199	172	118
8	4	2	528	447	152
8	4	3	263-	65	181
8	5	0	201-	44	180
8	5	1	405	474	259
8	5	2	641	626	262
8	5	3	262-	175	182
8	6	0	204-	50	0
8	6	1	381	430	240
8	6	2	205-	112	189
8	6	3	371	387	202
8	7	0	266	204	0
8	7	1	386	453	101
8	7	2	449	398	241
8	7	3	259-	281	184
8	8	0	374	285	0
8	8	1	255	283	0
8	8	2	445	374	303
8	8	3	256-	35	21
8	9	0	595	735	0
8	9	1	949	1056	85
8	9	2	437	372	72
8	9	3	253-	179	82
8	10	0	590	712	0
8	10	1	427	430	19
8	10	2	647	692	329
8	10	3	300	334	346
8	11	0	450	510	0
8	11	1	304	291	9
8	11	2	466	404	69
8	11	3	250	280	328
8	12	0	272	234	180
8	12	1	489	510	66
8	12	2	207-	153	268
8	12	3	237-	159	16
8	13	0	225-	17	0
8	13	1	750	844	281
8	13	2	276	249	136
8	13	3	230-	290	296
8	14	0	919	952	180
8	14	1	410	428	112
8	14	2	528	569	172
8	14	3	221-	217	156
8	15	0	228-	136	180
8	15	1	268	320	253
8	15	2	199-	140	208
8	15	3	211-	53	152
8	16	0	228-	5	0
8	16	1	344	347	238
8	16	2	194-	123	104
8	16	3	199-	249	202
8	17	0	226-	59	180
8	17	1	459	454	112
8	17	2	292	279	231
8	17	3	184-	202	138
8	18	0	591	581	0
8	18	1	211	191	270
8	18	2	292	211	347
8	18	3	167-	107	148
8	19	0	219-	246	0
8	19	1	468	440	117
8	19	2	166	168	265
8	19	3	108-	140	181
8	20	0	580	618	0
8	20	1	197-	106	3
8	20	2	482	404	313
8	21	0	431	401	0
8	21	1	188-	91	231
8	21	2	148-	152	6
8	22	0	194-	95	180
8	22	1	177-	182	8
8	22	2	290	253	259
8	23	0	181-	35	0
8	23	1	402	353	269
8	23	2	188	161	120
8	24	0	596	486	180
8	24	1	148-	166	66
8	24	2	216	260	189
8	25	0	150-	60	180
8	25	1	304	194	91
8	26	0	129-	87	180
8	26	1	104-	54	197
8	27	0	98-	44	180
9	0	1	448	523	270
9	0	2	231	160	180
9	0	3	313	365	270
9	1	0	1008	1088	270
9	1	1	211-	107	183
9	1	2	446	422	299
9	1	3	354	360	193
9	2	0	1103	1205	90
9	2	1	334	227	281
9	2	2	473	529	105
9	2	3	253-	130	255
9	3	0	212-	149	90
9	3	1	568	569	345
9	3	2	315	316	44
9	3	3	276	227	297
9	4	0	551	675	90
9	4	1	213-	164	157
9	4	2	292	257	84
9	4	3	251-	196	125
9	5	0	855	998	90
9	5	1	215-	19	34
9	5	2	788	844	85
9	5	3	249-	149	311
9	6	0	386	261	270
9	6	1	462	407	50
9	6	2	208-	75	265
9	6	3	305	404	76
9	7	0	220-	169	90
9	7	1	520	533	149
9	7	2	208-	143	123
9	7	3	242-	194	96
9	8	0	836	974	270
9	8	1	218-	139	64
9	8	2	207-	261	253
9	8	3	305	302	44
9	9	0	741	748	270
9	9	1	489	547	146
9	9	2	490	450	238
9	9	3	310	322	130
9	10	0	545	580	270
9	10	1	440	419	224
9	10	2	205-	99	277
9	10	3	280	318	276
9	11	0	278	215	270
9	11	1	220-	218	52
9	11	2	275	276	187
9	11	3	221-	210	178
9	12	0	228-	224	90
9	12	1	348	388	249
9	12	2	320	291	107
9	12	3	260	215	272
9	13	0	228-	101	90
9	13	1	780	788	356
9	13	2	253	201	147
9	13	3	204-	224	304
9	14	0	368	377	90
9	14	1	217-	130	232
9	14	2	401	280	127
9	14	3	192-	96	148
9	15	0	317	323	90
9	15	1	215-	96	0
9	15	2	290	222	50
9	15	3	180-	152	275
9	16	0	223-	186	90
9	16	1	258	246	61
9	16	2	179-	66	138
9	16	3	164-	239	64
9	17	0	311	310	90
9	17	1	425	452	163
9	17	2	256	281	13
9	17	3	144-	103	116
9	18	0	213-	138	270
9	18	1	242	322	25
9	18	2	161-	152	318
9	19	0	205-	6	90
9	19	1	402	349	156
9	19	2	151-	115	245
9	20	0	418	447	270
9	20	1	217	181	174
9	20	2	242	216	225
9	21	0	165-	101	90
9	21	1	166	170	166
9	21	2	411	356	179
9	22	0	172-	120	270
9	22	1	294	250	184
9	22	2	103-	91	104
9	23	0	158-	10	270
9	23	1	277	186	352
9	24	0	140-	102	90
9	24	1	118-	117	174
9	25	0	117-	10	90
9	26	0	76-	33	90
10	0	0	755	875	180
10	0	1	472	597	90
10	0	2	205-	161	180
10	0	3	151-	103	90
10	1	0	224-	137	180
10	1	1	217	254	265
10	1	2	505	556	101
10	1	3	233-	181	190
10	2	0	526	595	180
10	2	1	310	321	114
10	2	2	402	348	120
10	2	3	232-	174	139
10	3	0	437	409	0
10	3	1	381	461	301
10	3	2	231	190	25
10	3	3	231-	189	1
10	4	0	342	370	0
10	4	1	348	440	235
10	4	2	290	362	26
10	4	3	336	361	228
10	5	0	247	300	0
10	5	1	220-	247	351
10	5	2	390	366	284
10	5	3	226-	220	16
10	6	0	354	376	0
10	6	1	543	590	275
10	6	2	279	359	209
10	6	3	336	347	250
10	7	0	278	298	180
10	7	1	220-	171	70
10	7	2	287	368	205
10	7	3	217-	102	250
10	8	0	228-	196	180
10	8	1	381	380	46
10	8	2	256	287	227
10	8	3	212-	226	45
10	9	0	228-	47	180
10	9	1	467	497	99
10	9	2	197-	140	142
10	9	3	206-	215	219
10	10	0	228-	282	180
10	10	1	712	729	56
10	10	2	194-	70	120
10	10	3	250	313	29
10	11	0	227-	240	0
10	11	1	217-	162	98
10	11	2	347	272	78
10	11	3	189-	165	253
10	12	0	278	292	180
10	12	1	214	209	48
10	12	2	165-	50	110
10	12	3	179-	55	315
10	13	0	533	504	0
10	13	1	211-	107	301
10	13	2	281	293	52
10	13	3	166-	139	355
10	14	0	368	302	180
10	14	1	252	273	245
10	14	2	173-	82	186
10	14	3	151-	236	207
10	15	0	216-	193	0
10	15	1	201-	8	151
10	15	2	166-	72	37
10	16	0	210-	25	180
10	16	1	391	405	254
10	16	2	157-	135	233
10	17	0	533	467	180
10	17	1	228	186	255
10	17	2	505	450	208
10	18	0	330	252	0
10	18	1	176	207	73
10	18	2	205	151	258
10	19	0	185-	168	180
10	19	1	167-	89	131
10	19	2	284	314	243
10	20	0	399	298	0
10	20	1	410	373	54
10	20	2	104-	134	300
10	21	0	596	419	0
10	21	1	280	194	110
10	22	0	144-	31	0
10	22	1	247	210	327
10	23	0	266	180	0
10	24	0	482	365	180
11	0	1	219-	222	270
11	0	2	195-	61	0
11	0	3	201-	18	270
11	1	0	228-	238	270
11	1	1	712	719	1
11	1	2	168	182	331
11	1	3	201-	17	111
11	2	0	242	354	90
11	2	1	582	597	204
11	2	2	195-	99	58
11	2	3	200-	94	215
11	3	0	228-	109	270
11	3	1	219-	80	140
11	3	2	382	381	356
11	3	3	198-	162	198
11	4	0	227-	109	90
11	4	1	344	384	183
11	4	2	192-	65	195
11	4	3	195-	126	163
11	5	0	227-	232	90
11	5	1	737	721	178
11	5	2	233	221	73
11	5	3	300	339	190
11	6	0	227-	100	90
11	6	1	459	530	10
11	6	2	281	267	149
11	6	3	220	281	36
11	7	0	247	200	270
11	7	1	339	356	167
11	7	2	351	350	178
11	7	3	180-	99	117
11	8	0	225-	245	270

Table 3 (*cont.*)

h	k	l	$\vert 10F_o \vert$	$10F_c$	$\alpha°$
11	8	1	261	304	6
11	8	2	238	162	131
11	8	3	173-	151	55
11	9	0	368	366	270
11	9	1	364	327	24
11	9	2	411	415	207
11	9	3	164-	187	56
11	10	0	411	447	270
11	10	1	358	351	193
11	10	2	175-	136	341
11	10	3	154-	214	173
11	11	0	217-	66	90
11	11	1	400	410	0
11	11	2	208	196	187
11	11	3	141-	34	221
11	12	0	221	226	270
11	12	1	315	278	235
11	12	2	163-	117	352
11	13	0	209-	59	90
11	13	1	193-	118	324
11	13	2	156-	104	317
11	14	0	203-	115	270
11	14	1	187-	127	267
11	14	2	148-	121	224
11	15	0	196-	164	90
11	15	1	220	254	168
11	15	2	138-	150	24
11	16	0	354	321	90
11	16	1	171-	54	63
11	16	2	356	283	126
11	17	0	179-	121	90
11	17	1	342	314	151
11	17	2	113-	110	90
11	18	0	266	215	90
11	18	1	236	173	90
11	18	2	278	288	77
11	19	0	330	226	270
11	19	1	136-	81	135
11	20	0	469	339	270
11	20	1	119-	176	139
11	21	0	123-	130	270
11	22	0	575	457	270
12	0	0	621	724	180
12	0	1	555	548	270
12	0	2	239	187	180
12	0	3	154-	175	270
12	1	0	221-	3	0
12	1	1	207-	151	161
12	1	2	251	233	113
12	1	3	153-	192	153
12	2	0	494	433	180
12	2	1	253	258	169
12	2	2	174-	86	164
12	2	3	330	322	175
12	3	0	220-	101	0
12	3	1	206-	195	75
12	3	2	224	192	90
12	3	3	148-	193	72
12	4	0	285	235	0
12	4	1	204	280	127
12	4	2	267	259	328
12	4	3	200	255	170
12	5	0	241	225	0
12	5	1	250	244	323
12	5	2	168-	62	45
12	5	3	138-	192	332
12	6	0	571	595	0
12	6	1	201-	118	350
12	6	2	415	392	340
12	7	0	213-	132	180
12	7	1	241	241	283
12	7	2	162-	148	220
12	8	0	210-	25	0
12	8	1	236	288	351
12	8	2	157-	117	52
12	9	0	207-	92	180
12	9	1	301	269	101
12	9	2	152-	92	313
12	10	0	576	559	180
12	10	1	263	252	326
12	10	2	309	266	139
12	11	0	197-	11	180
12	11	1	443	428	95
12	11	2	140-	129	84
12	12	0	507	537	180
12	12	1	175-	53	79
12	12	2	265	214	165
12	13	0	185-	5	180
12	13	1	165	196	118
12	13	2	234	199	110
12	14	0	177-	12	0
12	14	1	159-	58	173
12	14	2	276	203	293
12	15	0	168-	142	0
12	15	1	334	262	258
12	15	2	94-	34	90
12	16	0	614	467	0
12	16	1	256	225	274
12	17	0	145-	85	180
12	17	1	318	314	275
12	18	0	130-	110	0
12	18	1	107-	134	11
12	19	0	112-	52	0
12	20	0	80-	129	180
13	0	1	184-	189	270
13	0	2	144-	18	180
13	1	0	311	309	90
13	1	1	391	392	15
13	1	2	143-	149	42
13	2	0	304	219	270
13	2	1	183-	156	320
13	2	2	143-	155	289
13	3	0	198-	42	270
13	3	1	223	206	63
13	3	2	141-	192	336
13	4	0	197-	15	90
13	4	1	180-	130	203
13	4	2	138-	39	336
13	5	0	195-	162	270
13	5	1	217	251	176
13	5	2	135-	54	310
13	6	0	361	261	90
13	6	1	175-	118	196
13	6	2	322	274	114
13	7	0	488	464	270
13	7	1	171-	141	230
13	7	2	357	302	225
13	8	0	184-	65	270
13	8	1	166-	57	188
13	8	2	192	175	157
13	9	0	179-	46	270
13	9	1	255	195	328
13	9	2	243	360	190
13	10	0	590	431	270
13	10	1	155-	80	319
13	10	2	217	146	267
13	11	0	647	500	90
13	11	1	255	201	347
13	12	0	380	291	270
13	12	1	326	327	345
13	13	0	150-	154	90
13	13	1	129-	187	9
13	14	0	139-	84	90
13	14	1	117-	132	241
13	15	0	259	236	270
13	16	0	241	155	90
14	0	0	168-	173	180
14	0	1	400	412	270
14	0	2	93-	1	180
14	1	0	335	264	180
14	1	1	250	243	235
14	2	0	167-	165	180
14	2	1	209	137	225
14	3	0	165-	90	180
14	3	1	206	228	259
14	4	0	163-	27	0
14	4	1	304	260	92
14	5	0	285	235	0
14	5	1	140-	73	66
14	6	0	155-	150	0
14	6	1	287	260	71
14	7	0	151-	108	0
14	7	1	184	217	79
14	8	0	145-	1	0
14	8	1	124-	155	49
14	9	0	138-	124	180
14	9	1	116-	97	73
14	10	0	130-	107	180
14	10	1	250	265	277
14	11	0	519	424	180
14	12	0	106-	169	180
14	13	0	86-	133	180
15	1	0	557	473	90
15	2	0	115-	167	90
15	3	0	112-	45	270
15	4	0	108-	83	90
15	5	0	221	213	270
15	6	0	93-	119	270
15	7	0	78-	104	270

Table 4. *Bond distances and angles in the coordination tetrahedra*

Ag(1)–Cl(2)	2·65 ± 1 Å	Ag(2)–Cl(1)	2·66 ± 1 Å
Ag(1)–S(1)	2·50 ± 1	Ag(2)–S(1)	2·51 ± 1
Ag(1)–Cl(2^{i})	2·75 ± 1	Ag(2)–S(2)	2·48 ± 1
Ag(1)–S(2^{ii})	2·51 ± 1	Ag(2)–S(1^{iii})	2·77 ± 1
Cl(2)–Ag(1)–S(1)	114·7 ± 0·2°	Cl(1)–Ag(2)–S(1)	108·1 ± 0·3°
Cl(2)–Ag(1)–Cl(2^{i})	98·0 ± 0·3	Cl(1)–Ag(2)–S(2)	111·8 ± 0·2
Cl(2)–Ag(1)–S(2^{ii})	108·6 ± 0·3	Cl(1)–Ag(2)–S(1^{iii})	101·2 ± 0·3
S(1)—Ag(1)–Cl(2^{i})	100·5 ± 0·2	S(1)—Ag(2)–S(2)	132·3 ± 0·3
S(1)—Ag(1)–S(2^{ii})	114·6 ± 0·2	S(1)—Ag(2)–S(1^{iii})	101·2 ± 0·2
Cl(2′)–Ag(1)–S(2^{ii})	119·5 ± 0·3	S(2)—Ag(2)–S(1^{iii})	95·7 ± 0·2

Table 5. Ag–S *distances* (Å) *in* Ag(I) *complexes*

$Ag[SC(NH_2)NHNH_2]Cl$	2·48, 2·50, 2·51, 2·77	Present paper
AgSCN	2·43	Lindqvist (1957)
$NH_4Ag(SCN)_2$	2·63, 2·65, 2·74	Lindqvist & Strandberg (1957)
$Ag[SC(NH_2)_2]_2Cl$	2·43, 2·48, 2·49, 2·53, 2·54, 2·58	Vizzini & Amma (1966)
$Ag(CH_2S)_3ClO_4.H_2O$	2·48, 2·52, 2·66	Ashworth, Domenicano, Prout & Vaciago (1967)
$Ag(CH_2S)_3NO_3.H_2O$	2·55, 2·61, 2·69	
$Ag(CH_2S)_3NO_3$	2·61, 2·64, 2·69, 2·70, 2·73	
$Ag[(CH_2S)_3]_2NO_3$	2·46, 2·59, 2·60	Ashworth, Domenicano, Scaramuzza, Prout & Vaciago (1967)

Table 6. *Distances* (Å) *and angles*

$Ag[SC(NH_2)NHNH_2]Cl$ (*a*)				$SC(NH_2)NHNH_2$ (*b*)
S(2)–C(1)	1·70 ± 4	S(1)–C(2)	1·74 ± 3	1·685 ± 5
C(1)–N(1)	1·35 ± 4	C(2)–N(6)	1·29 ± 4	1·313 ± 6
C(1)–N(2)	1·28 ± 4	C(2)–N(4)	1·37 ± 5	1·337 ± 6
N(2)–N(3)	1·40 ± 4	N(4)–N(5)	1·38 ± 6	1·399 ± 6
N(1)–N(3)	2·63 ± 4	N(5)–N(6)	2·69 ± 4	2·720 ± 6
S(2) C(1) N(1)	122·2 ± 2·2	S(1) C(2) N(6)	123·7 ± 2·1	119·7 ± 0·3
S(2) C(1) N(2)	120·9 ± 2·5	S(1) C(2) N(4)	120·8 ± 2·4	121·5 ± 0·3
N(1)C(1) N(2)	115·8 ± 3·4	N(6)C(2) N(4)	113·7 ± 3·3	118·8 ± 0·4
C(1) N(2)N(3)	122·2 ± 2·7	C(2) N(4)N(5)	121·0 ± 3·5	122·5 ± 0·4

(*a*) Present paper; (*b*) Research in progress; (*c*) Cavalca, Nardelli & Branchi (1960);

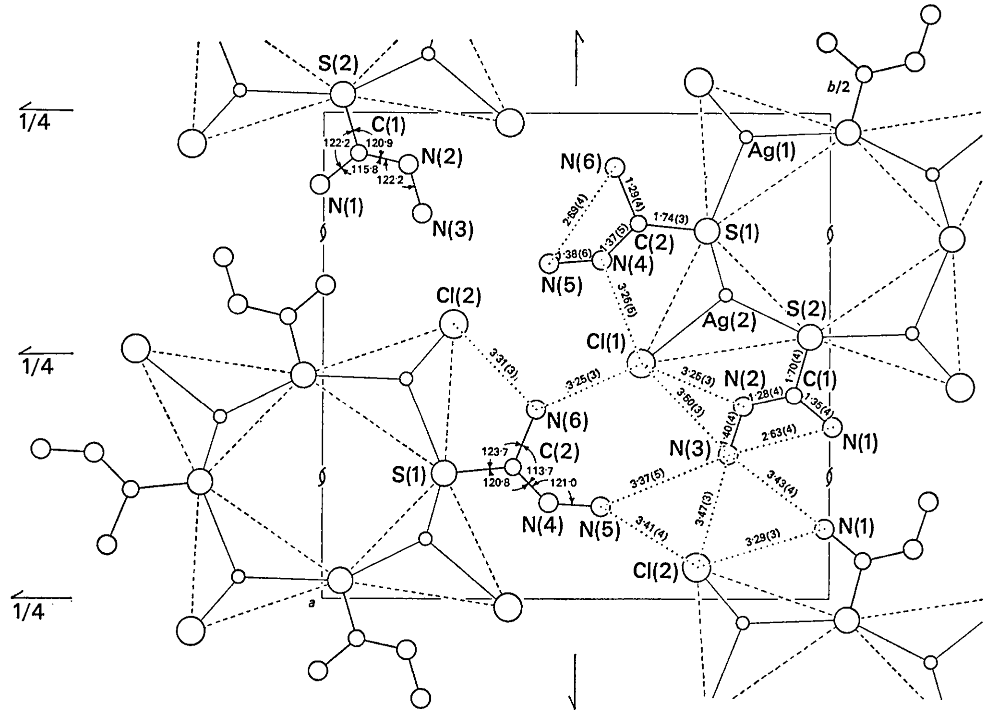


Fig. 2. Ag[SC(NH$_2$)NHNH$_2$]Cl: projection of the structure on (001).

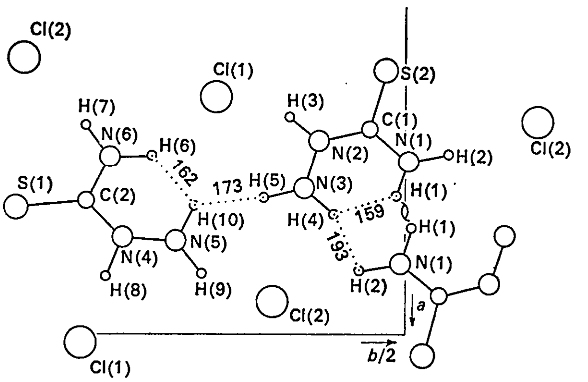


Fig. 3. Ag[SC(NH$_2$)NHNH$_2$]Cl: projection on (001), showing unacceptable contacts for hydrogen atoms in calculated positions.

nates of the lighter atoms is not good enough to warrant discussion of these distances.

The deviations from planarity in the S(2)C(1)N(1)-N(2)N(3) molecule are small enough in comparison with the e.s.d.'s to be insignificant. The same cannot be said for the S(1)C(2)N(4)N(5)N(6) molecule in which the displacement (0·3 Å) of N(4) out of the other atom plane is significant; nevertheless, it is not certain that this displacement is real.

A direct location of hydrogen atoms was not attempted. However, putting them in the positions calculated assuming complete planarity of the molecule and trigonal bonds around the nitrogen atoms, led to unacceptable contacts as shown in Fig. 3, in which the hydrogen atoms are in the calculated positions. This indicated that distortions from coplanarity affect some of the hydrogen atoms as could be expected for the

(°) *in thiosemicarbazide molecules*

Zn[SC(NH$_2$)NHNH$_2$]Cl$_2$ (*c*)	Ni[SC(NH$_2$)N$_2$H$_2$]$_2$ (*d*)	Ni[SC(NH$_2$)NHNH$_2$]$_2$SO$_4$.3H$_2$O (*e*)	Ni[SC(NH$_2$)NHNH$_2$]$_2$SO$_4$ (*f*)	
		α	*β cis*	*β trans*
1·73 ± 2	1·746 ± 13	1·75 ± 3	1·718 ± 2	1·720 ± 2
1·29 ± 3	1·436 ± 17	1·29 ± 5	1·332 ± 3	1·306 ± 3
1·28 ± 4	1·247 ± 15	1·33 ± 5	1·360 ± 3	1·340 ± 3
1·44 ± 3	1·537 ± 17	1·44 ± 4	1·376 ± 3	1·419 ± 3
—	—	—	—	—
122·9 ± 1·6	122·3 ± 0·9	121·18	121·85	121·40
118·3 ± 1·5	120·9 ± 1·0	118·07	117·74	119·23
118·8 ± 2·3	116·4 ± 1·1	120·56	120·41	119·36
131·4 ± 2·4	109·8 ± 1·0	119·42	119·58	120·36

(*d*) Cavalca, Nardelli & Fava (1962); (*e*) Grønbæk & Rasmussen (1962); (*f*) Grønbæk (1963).

Table 7. *Hydrogen bonding and contacts less than* 3·5 Å

Cl(1)–N(2)	3·25 ± 3 Å	Cl(1)–N(2)—C(1)	122·4°
Cl(1)–N(4)	3·26 ± 5	Cl(1)–N(4)—C(2)	121·5
Cl(1)–N(6^{iv})	3·25 ± 3	Cl(1)–N(6^{iv})—C(2^{iv})	148·3
Cl(2)–N(6^{iii})	3·31 ± 3	Cl(2)–N(6^{iii})—C(2^{iii})	117·5
Cl(2)–N(1^{v})	3·29 ± 3	Cl(2)–N(1^{v})—C(1^{v})	117·3
Cl(2)–N(5^{vii})	3·41 ± 4	Cl(2)–N(5^{vii})–N(4^{vii})	116·6
Cl(2)–N(3^{viii})	3·47 ± 3	Cl(2)–N(3^{viii})–N(2^{viii})	173·8
N(1)–N(3^{v})	3·43 ± 4	N(1)–N(3^{v})—N(2^{v})	119·6
N(3)–N(5^{ix})	3·37 ± 5	N(3)–N(5^{ix})—N(4^{ix})	151·4

i	$x, y, z-1$	vi	$x-\frac{1}{2}, \frac{1}{2}-y, \bar{z}$
ii	$\frac{1}{2}-x, 1-y, z-\frac{1}{2}$	vii	$x-\frac{1}{2}, \frac{1}{2}-y, 1-z$
iii	$x, y, z+1$	viii	$x-1, y, z-1$
iv	$x+\frac{1}{2}, \frac{1}{2}-y, \bar{z}$	ix	$x+\frac{1}{2}, \frac{1}{2}-y, 1-z$
v	$\frac{1}{2}-x, 1-y, \frac{1}{2}+z$	x	$\frac{3}{2}-x, 1-y, \frac{1}{2}+z$

hydrazinic part of the molecule. These distortions are frequent in other molecules of the same kind, as found recently in thiourea by Truter (1967) and in uncomplexed thiosemicarbazide (research in progress).

Some of the NH···Cl contacts shown in Table 7 are probably hydrogen bonds; in the same table, the other packing contacts less than 3·5 Å are also quoted.

The authors are indebted to the Consiglio Nazionale delle Ricerche (Roma) for financial support.

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