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Hydrogen Bonding in Thiosemicarbazide

BY GIOVANNI DARIO ANDREETTI, PAOLO DOMIANO, GIOVANNA FAVA GASPARRI,
MARIO NARDELLI AND PAOLO SGARABOTTO

Istituti di Chimica Generale e Strutturistica Chimica della Università, Parma, Italy

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The crystal structure of thiosemicarbazide, previously determined from photographic data, has been redetermined using intensity data collected by means of a four-circle Picker diffractometer at room temperature. The analysis has been valuable in giving a direct determination of the hydrogen atom positions. The final $R=2{\cdot}4\%$. Earlier hypotheses on the hydrogen bonding are confirmed.

Introduction

In a recent paper (Domiano, Fava Gasparri, Nardelli & Sgarabotto, 1969, DGNS) the crystal-structure analysis of thiosemicarbazide, carried out by photographic methods, was reported (final $R=9{\cdot}9\%$). The positions of the hydrogen atoms were postulated from consideration of the hybridization of the nitrogen atoms and the packing interactions, no direct determination of position being possible from the final difference synthesis. In order to obtain a more accurate structure analysis, suitable for the location of the hydrogen atoms, new data have been collected at room temperature with an automated Picker diffractometer.

Experimental and refinement

Cell parameters,* as determined by least-squares methods from 19 2θ values in the range $16{\cdot}2° \le 2\theta \le 42{\cdot}7°$ using Zr-filtered Mo $K\alpha_1$ radiation, are (at 24·0°C):

$$a = 4{\cdot}934 \pm 1,\ b = 7{\cdot}330 \pm 1,\ c = 8{\cdot}396 \pm 1\ \text{Å}$$
$$\alpha = 44{\cdot}39 \pm 1,\ \beta = 83{\cdot}23 \pm 1,\ \gamma = 76{\cdot}99 \pm 1\ °,$$
$$Z = 2,\ F(000) = 96,\ V = 206{\cdot}5\ \text{Å}^3,$$
$$D_x = 1{\cdot}49\ \text{g.cm}^{-3};\ \mu = 1{\cdot}44\ \text{cm}^{-1}\ (\text{Mo}\ K\alpha)$$

Space group: $P\bar{1}$ (from structure determination).

It must be pointed out that these improved cell parameters show some relevant differences with respect to those given by DGNS, particularly for the b constant.

A very well developed crystal approximately $0{\cdot}20 \times 0{\cdot}10 \times 0{\cdot}10$ mm^3 was mounted on a Picker diffractometer equipped with a pulse-height analyser and the intensity data were collected using Mo $K\alpha$ ($\lambda = 0{\cdot}71069$ Å) radiation and the ω–2θ scan method. Of a total of 936 measured reflexions ($2\theta_{\max} = 55°$), 100 were coded as 'unobserved', having $I_{hkl} < 2\sigma_I$ (σ_I from counting statistics) and were not used in the structure refinement. No corrections were made for absorption or for anomalous dispersion.

Starting with the DGNS set of parameters for non-hydrogen atoms, the structure was refined first by means of three cycles of full-matrix least squares (all unit weights) with isotropic temperature factors to $R=10{\cdot}7\%$, then by means of two cycles with anisotropic thermal parameters to $R=5{\cdot}7\%$. Then from a ΔF map five well defined peaks with heights between 0·69 and 0·54 e.Å^{-3} over a maximum background of 0·3 e.Å^{-3}, were found, giving good values for the N–H bond distances. The refinement was then carried out with all the atoms but refining only the heavy ones, and after two cycles the R index was 3·7%. A further improvement was obtained after two more cycles using weights ($w = 1/\sigma_F^2$) and refining also the hydrogen atoms isotropically; the final R value achieved in this way was 2·4% ($R_w = 2{\cdot}3\%$). The function minimized was $\sum w\Delta F^2$ and the expressions used for R and R_w were: $R = \sum |\Delta F| / \sum |F_o|$ and $R_w = \{\sum w\Delta F^2 / \sum wF_o^2\}^{1/2}$. The average shift/error ratio in the final cycle was 0·10 and the maximum value 0·54; the final value of the goodness of fit was 0·79. In Table 1 the final atomic parameters obtained in the present refinement are compared with those reported by DGNS. Table 2 gives the root-mean-square displacements and their orientations with respect to an orthogonal cartesian system of axes referred to the crystal axes by the matrix (0·2027, −0·0468, −0·0132/0, 0·1400, −0·1416/0,0, 0·1706). The thermal ellipsoids in the plane of the molecule are shown in Fig. 1. It must be pointed out that, in spite of the different methods followed in data collection and refinement, the agreement between corresponding parameters is quite good, as is also shown in Table 1. In Table 3 observed and calculated structure factors are compared. The atomic scattering factors used are those of Cromer & Waber (1965) for S, N, C, and those of Stewart, Davidson & Simpson (1965) for hydrogen. All the calculations were carried out on an IBM 7094-7040 direct coupled system, using the programs written or adapted by Stewart (1964).

Discussion

As shown in Table 4, bond distances and angles in the molecule agree quite well with those of the previous

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* The same orientation used in the previous paper (DGNS) is maintained. The transformation matrix from our cell to the Buerger's all acute 'reduced cell' is $\bar{1}00/0\bar{1}1/0\bar{1}0$.

analysis (DGNS), the maximum differences being 4σ for the C–S bond and 7σ for the S–C–N(1) angle. These differences are mainly due to the unit-cell parameters used in the analyses, since the fractional coordinates do not differ significantly.

The assumptions about the positions of the hydrogen atoms made by DGNS are confirmed. In Table 5, N–H bond distances and angles involving hydrogen atoms are listed together with N–H values corrected for thermal motion following Busing & Levy (1964); these last values agree quite well with those listed by Hamilton & Ibers (1968). As postulated by DGNS, H(3,1) and H(3,2) are practically symmetrical with respect to the mean molecular plane, so that the lone

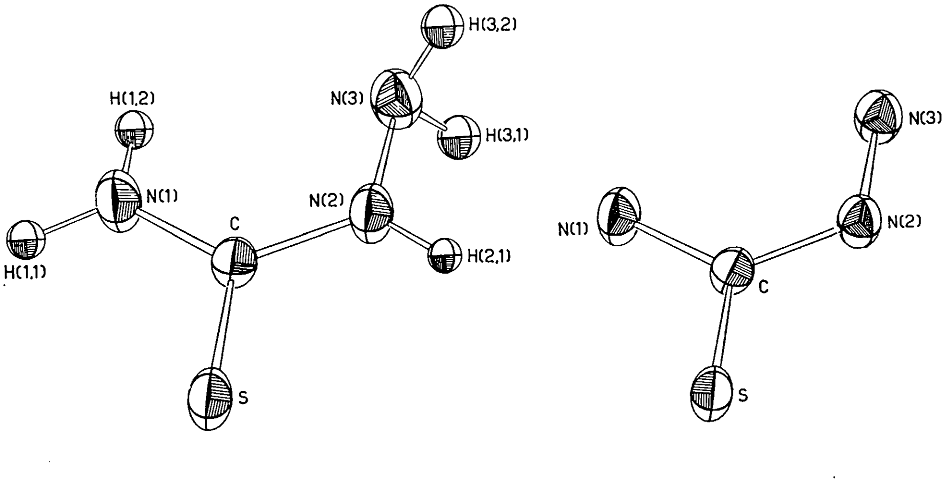
Table 1. *Final atomic fractional coordinates*

	Present work						Previous work (DGNS)					
	x/a	(σ)	y/b	(σ)	z/c	(σ)	x/a	(σ)	y/b	(σ)	z/c	(σ)
S	−0·00142	(8)	−0·12576	(8)	0·82102	(6)	−0·0013	(3)	−0·1260	(1)	0·8212	(1)
N(1)	0·25952	(27)	0·21177	(27)	0·73844	(21)	0·2607	(10)	0·2119	(2)	0·7391	(4)
N(2)	0·26454	(24)	0·22024	(23)	0·46188	(18)	0·2646	(10)	0·2203	(2)	0·4604	(5)
N(3)	0·42506	(30)	0·41955	(27)	0·32852	(21)	0·4259	(11)	0·4193	(3)	0·3283	(4)
C	0·18558	(25)	0·12088	(25)	0·66364	(20)	0·1836	(10)	0·1211	(3)	0·6616	(4)
H(1,1)	0·1943	(30)	0·1578	(28)	0·8659	(26)						
H(1,2)	0·3609	(31)	0·3293	(29)	0·6581	(24)						
H(2,1)	0·2090	(28)	0·1735	(27)	0·4082	(23)						
H(3,1)	0·3315	(34)	0·5584	(35)	0·1938	(30)						
H(3,2)	0·5742	(37)	0·3539	(34)	0·3072	(28)						

Table 2. *Root-mean-square displacements and direction cosines of the principal axes of the vibration ellipsoids*

Data given in parentheses refer to the analysis by DGNS.

	Axis	U_i	g_i^a	g_i^b	g_i^c
S	1	0·1595 (0·1614) Å	0·9848 (−0·9916)	−0·1727 (−0·0385)	0·0185 (0·1237)
	2	0·1846 (0·1789)	−0·1417 (0·1116)	0·7372 (−0·7390)	0·6607 (0·6644)
	3	0·2715 (0·2679)	0·1004 (0·0658)	0·6532 (0·6726)	0·7505 (0·7370)
N(1)	1	0·1617 (0·1565)	0·9828 (0·9332)	−0·1259 (−0·4490)	−0·1353 (−0·0263)
	2	0·1900 (0·1999)	−0·0222 (0·3257)	0·8073 (−0·6860)	0·5897 (0·6506)
	3	0·2793 (0·2757)	0·1835 (0·3102)	0·5765 (0·5726)	0·7962 (0·7589)
N(2)	1	0·1578 (0·1674)	0·9700 (−0·8311)	−0·2412 (0·5342)	−0·0301 (0·1550)
	2	0·1908 (0·1807)	−0·1476 (−0·2800)	−0·6829 (−0·6425)	0·7155 (0·7133)
	3	0·2416 (0·2422)	0·1931 (0·4806)	0·6896 (0·5494)	0·6980 (0·6835)
N(3)	1	0·1835 (0·1752)	−0·8224 (−0·7515)	0·5657 (0·6196)	−0·0604 (0·2265)
	2	0·2051 (0·1964)	−0·3999 (−0·2908)	−0·4992 (−0·6193)	0·7687 (0·7293)
	3	0·2362 (0·2408)	0·4047 (0·5921)	0·6563 (0·4823)	0·6368 (0·6456)
C	1	0·1659 (0·1677)	0·9845 (0·8801)	0·0904 (−0·3390)	−0·1505 (0·3324)
	2	0·1832 (0·1823)	−0·1459 (−0·4692)	0·8980 (−0·5139)	−0·4152 (0·7182)
	3	0·2013 (0·2029)	0·0976 (0·0726)	0·4307 (0·7881)	0·8972 (0·6113)



(a) (b)

Fig. 1. Thermal ellipsoids as seen in the plane of the molecule: (*a*) present work, (*b*) determination by DGNS.

Table 3. *Observed and calculated structure factors*

A minus sign for F_o means 'less than'.

h	k	l	$100F_o$	$100F_c$
0	0	1	1340	-1329
0	0	2	2561	-2539
0	0	3	799	-815
0	0	4	80-	-67
0	0	5	273	273
0	0	6	839	840
0	0	7	100-	-54
0	1	1	46-	23
0	1	2	1854	-1777
0	1	3	2817	-2784
0	1	4	1753	1688
0	1	5	1180	1138
0	1	6	345	370
0	1	7	791	-790
0	1	8	256	-252
0	$\bar{1}$	0	2055	1945
0	$\bar{1}$	1	1287	1244
0	$\bar{1}$	2	103	115
0	$\bar{1}$	3	573	-597
0	$\bar{1}$	4	1000	-994
0	$\bar{1}$	5	185	190
0	$\bar{1}$	6	258	276
0	2	1	485	497
0	2	2	3411	-3390
0	2	3	666	657
0	2	4	2052	2113
0	2	5	440	426
0	2	6	139	-108
0	2	7	897	-889
0	2	8	522	-517
0	2	9	337	345
0	$\bar{2}$	0	1069	-1093
0	$\bar{2}$	1	738	755
0	$\bar{2}$	2	1200	1186
0	$\bar{2}$	3	269	281
0	$\bar{2}$	4	1188	-1180
0	$\bar{2}$	5	86-	-33
0	3	2	145	144
0	3	3	2720	2733
0	3	4	78	94
0	3	5	215	217
0	3	6	1253	-1270
0	3	7	401	-387
0	3	8	71-	-3
0	3	9	582	592
0	$\bar{3}$	0	1770	-1756
0	$\bar{3}$	1	832	838
0	$\bar{3}$	2	786	782
0	$\bar{3}$	3	108-	35
0	$\bar{3}$	4	92-	-91
0	4	9	255	272
0	4	10	88	-72
0	$\bar{4}$	0	878	-877
0	$\bar{4}$	1	363	-360
0	$\bar{4}$	$\bar{1}$	341	-343
0	$\bar{4}$	2	558	575
0	$\bar{4}$	3	74-	12
0	5	2	1081	1072
0	5	3	110	91
0	5	4	200	-230
0	5	5	1108	-1107
0	5	6	397	-384
0	5	7	444	430
0	5	8	1305	1304
0	5	9	82	-44
0	5	10	778	-765
0	$\bar{5}$	0	116-	-77
0	$\bar{5}$	1	317	-314
0	$\bar{5}$	$\bar{1}$	134	-144
0	$\bar{5}$	2	139	-131
0	6	2	611	609
0	6	3	629	-618
0	6	4	657	-634
0	6	5	69-	-54
0	6	6	252	254
0	6	7	288	286
0	6	8	974	961
0	6	9	702	-689
0	6	10	563	-556
0	$\bar{6}$	0	147	-144
0	$\bar{6}$	$\bar{1}$	424	413
0	7	2	86-	-41
0	7	3	75-	-65
0	7	4	773	-761
0	7	5	209	203
0	7	6	874	871
0	7	7	209	190
0	7	8	424	-420
0	7	9	350	-350
0	7	10	178	-182
0	$\bar{8}$	$\bar{1}$	173	178
0	8	3	90	-63
0	8	4	269	-271
0	8	5	364	372
0	8	6	342	342
0	8	7	72-	33
0	8	8	559	-575
0	8	9	188	-197
0	8	10	99-	-20
0	9	6	116-	119
0	9	7	100-	-59
0	9	8	440	-440
0	9	9	115-	61
1	0	0	1711	1628
1	0	1	2515	2533
1	0	$\bar{1}$	432	410
1	0	2	779	-787
1	0	$\bar{2}$	996	-965
1	0	3	2498	-2523
1	0	$\bar{3}$	1007	1033
1	0	4	350	335
1	0	$\bar{4}$	879	-865
1	0	5	560	568
1	0	$\bar{5}$	1032	1036
1	0	6	554	549
1	0	$\bar{6}$	403	401
1	0	7	161	-150
1	0	$\bar{7}$	211	205
1	1	0	63-	-98
1	1	1	2028	2056
1	1	$\bar{1}$	1524	1488
1	1	2	3608	-3648
1	1	$\bar{2}$	699	719
1	1	3	585	-604
1	1	$\bar{3}$	556	-561
1	1	4	819	825
1	1	$\bar{4}$	1647	-1662
1	1	5	766	766
1	1	$\bar{5}$	424	425
1	1	6	449	435
1	1	$\bar{6}$	599	602
1	1	7	298	-298
1	1	8	573	-569
1	$\bar{1}$	0	5238	5419
1	$\bar{1}$	1	548	554
1	$\bar{1}$	$\bar{1}$	3306	-3263
1	$\bar{1}$	2	373	-364
1	$\bar{1}$	$\bar{2}$	3214	-3229
1	$\bar{1}$	3	537	-537
1	$\bar{1}$	$\bar{3}$	307	271
1	$\bar{1}$	4	436	-434
1	$\bar{1}$	$\bar{4}$	880	666
1	$\bar{1}$	5	260	-253
1	$\bar{1}$	$\bar{5}$	681	688
1	$\bar{1}$	6	519	525
1	$\bar{1}$	$\bar{6}$	502	492
1	$\bar{1}$	$\bar{7}$	468	-483
1	$\bar{1}$	$\bar{8}$	571	-549
1	2	0	1475	-1418
1	2	1	2876	-2822
1	2	$\bar{1}$	2621	2575
1	2	2	1118	-1072
1	2	$\bar{2}$	1071	1080
1	2	3	1660	1670
1	2	$\bar{3}$	789	-801
1	2	4	523	533
1	2	$\bar{4}$	328	-332
1	2	5	1250	1277
1	2	$\bar{5}$	302	-309
1	2	6	764	-754
1	2	7	489	-491
1	2	8	361	-375
1	$\bar{2}$	0	654	666
1	$\bar{2}$	1	640	633
1	$\bar{2}$	$\bar{1}$	1580	-1580
1	$\bar{2}$	2	503	510
1	$\bar{2}$	$\bar{2}$	990	-975
1	$\bar{2}$	3	89	54
1	$\bar{2}$	$\bar{3}$	926	-915
1	$\bar{2}$	4	507	-502
1	$\bar{2}$	$\bar{4}$	2368	2253
1	$\bar{2}$	5	193	-175
1	$\bar{2}$	$\bar{5}$	702	742
1	$\bar{2}$	$\bar{6}$	428	-415
1	$\bar{2}$	$\bar{7}$	1225	-1252
1	$\bar{2}$	$\bar{8}$	80-	42
1	3	0	790	-793
1	3	1	1625	-1618
1	3	$\bar{1}$	144	135
1	3	2	511	-491
1	3	$\bar{2}$	1214	1231
1	3	3	919	927
1	3	$\bar{3}$	107	-121
1	3	4	2902	2885
1	3	$\bar{4}$	80-	-24
1	3	5	490	-492
1	3	6	1956	-1982
1	3	7	64	69
1	3	8	227	228
1	3	9	336	334
1	$\bar{3}$	0	960	-958
1	$\bar{3}$	1	156	-141
1	$\bar{3}$	$\bar{1}$	69-	-29
1	$\bar{3}$	2	662	654
1	$\bar{3}$	$\bar{2}$	966	-929
1	$\bar{3}$	3	514	510
1	$\bar{3}$	$\bar{3}$	1321	1335
1	$\bar{3}$	4	468	-465
1	$\bar{3}$	$\bar{4}$	1569	1594
1	$\bar{3}$	$\bar{5}$	673	-681
1	$\bar{3}$	$\bar{6}$	591	-559
1	$\bar{3}$	$\bar{7}$	841	-852
1	$\bar{3}$	$\bar{8}$	73	60
1	$\bar{3}$	$\bar{9}$	671	687
1	4	0	368	-378
1	4	1	1191	-1179
1	4	$\bar{1}$	322	-318
1	4	2	1376	1384
1	4	$\bar{2}$	67-	38
1	4	3	934	922
1	4	$\bar{3}$	478	473
1	4	4	709	645
1	4	5	1392	-1107
1	4	6	1034	-1029
1	4	7	532	-540
1	4	8	1357	1359
1	4	9	488	467
1	4	10	382	-357
1	$\bar{4}$	0	965	-956
1	$\bar{4}$	1	81-	-22
1	$\bar{4}$	$\bar{1}$	133	-144
1	$\bar{4}$	2	224	228
1	$\bar{4}$	$\bar{2}$	568	574
1	$\bar{4}$	3	149	150
1	$\bar{4}$	$\bar{3}$	1556	1569
1	$\bar{4}$	$\bar{4}$	573	-586
1	$\bar{4}$	$\bar{5}$	603	-687
1	$\bar{4}$	$\bar{6}$	821	-822
1	$\bar{4}$	$\bar{7}$	155	169
1	$\bar{4}$	$\bar{8}$	412	386
1	$\bar{4}$	$\bar{9}$	534	547
1	5	0	689	-709
1	5	1	521	515
1	5	$\bar{1}$	266	-255
1	5	2	1255	1283
1	5	$\bar{2}$	116	-121
1	5	3	118	-128
1	5	4	832	-822
1	5	5	523	-503
1	5	6	383	-364
1	5	7	79	71
1	5	8	1435	1428
1	5	9	111	-103
1	5	10	490	-478
1	$\bar{5}$	0	327	-351
1	$\bar{5}$	1	311	-298
1	$\bar{5}$	$\bar{1}$	322	323
1	$\bar{5}$	$\bar{2}$	460	439
1	$\bar{5}$	$\bar{3}$	212	226
1	$\bar{5}$	$\bar{4}$	76-	-9
1	$\bar{5}$	$\bar{5}$	1117	-1124
1	$\bar{5}$	$\bar{6}$	822	-812
1	$\bar{5}$	$\bar{7}$	1525	1523
1	$\bar{5}$	$\bar{8}$	319	321
1	$\bar{5}$	$\bar{9}$	161	-168
1	$\bar{5}$	$\bar{10}$	296	-304
1	6	0	92-	36
1	6	1	437	438
1	6	2	278	298
1	6	3	383	385
1	6	4	1576	1592
1	6	5	501	-500
1	6	6	928	962
1	6	7	821	811
1	6	8	78	-95
1	6	9	155	-159
1	6	10	501	-524
1	$\bar{6}$	0	112	83
1	$\bar{6}$	$\bar{1}$	95	82
1	$\bar{6}$	$\bar{2}$	386	397
1	$\bar{6}$	$\bar{3}$	318	-289
1	$\bar{6}$	$\bar{4}$	302	-302
1	$\bar{6}$	$\bar{5}$	405	-396
1	$\bar{6}$	$\bar{6}$	186	187
1	$\bar{6}$	$\bar{7}$	617	615
1	$\bar{6}$	$\bar{8}$	499	505
1	$\bar{6}$	$\bar{9}$	464	-471
1	$\bar{6}$	$\bar{10}$	688	-675
1	7	1	108	115
1	7	2	90	74
1	7	3	166	-180
1	7	4	744	-750
1	7	5	164	164
1	7	6	668	675
1	7	7	689	711
1	7	8	535	-559
1	7	9	514	-514
1	7	10	480	-453
1	$\bar{7}$	$\bar{2}$	145	157
1	$\bar{7}$	$\bar{3}$	518	-485
1	$\bar{7}$	$\bar{4}$	154	-153
1	$\bar{7}$	$\bar{5}$	276	255
1	$\bar{7}$	$\bar{6}$	265	254
1	$\bar{7}$	$\bar{7}$	70-	53
1	$\bar{7}$	$\bar{8}$	333	319
1	$\bar{7}$	$\bar{9}$	727	-711
1	$\bar{7}$	$\bar{10}$	215	-212
1	8	3	228	-233
1	8	4	263	218
1	8	5	76-	-25
1	8	6	465	478
1	8	7	213	207
1	8	8	677	-695
1	8	9	352	-369
1	8	10	95	98
1	$\bar{8}$	$\bar{4}$	169	-167
1	$\bar{8}$	$\bar{5}$	273	260
1	$\bar{8}$	$\bar{6}$	369	353
1	$\bar{8}$	$\bar{7}$	201	-194
1	$\bar{8}$	$\bar{8}$	335	-303
1	$\bar{8}$	$\bar{9}$	143	-148
1	9	6	151	152
1	9	7	440	-422
1	9	8	173	-189
1	9	9	81-	-31
2	0	0	191	175
2	0	1	2190	2122
2	0	$\bar{1}$	761	775
2	0	2	1408	-1401
2	0	$\bar{2}$	409	-394
2	0	3	717	-713
2	0	$\bar{3}$	1070	-1092
2	0	4	172	-174
2	0	$\bar{4}$	1025	-1041
2	0	5	329	336
2	0	$\bar{5}$	1079	1087
2	0	6	394	374
2	0	$\bar{6}$	775	780
2	0	7	156	124
2	0	$\bar{7}$	273	-240
2	1	0	340	355
2	1	1	1575	-1570
2	1	$\bar{1}$	3082	3017
2	1	2	1101	-1130
2	1	$\bar{2}$	291	288
2	1	3	284	-267
2	1	$\bar{3}$	1932	-1941
2	1	4	110	92
2	1	$\bar{4}$	160	-156
2	1	5	1188	1199
2	1	$\bar{5}$	73-	9
2	1	6	96	-96
2	1	$\bar{6}$	413	416
2	1	7	132	-144
2	$\bar{1}$	0	85	92
2	$\bar{1}$	1	2023	2071
2	$\bar{1}$	$\bar{1}$	668	-682
2	$\bar{1}$	2	208	205
2	$\bar{1}$	$\bar{2}$	1210	-1236
2	$\bar{1}$	3	1114	-1121
2	$\bar{1}$	$\bar{3}$	371	-401
2	$\bar{1}$	4	160	-172
2	$\bar{1}$	$\bar{4}$	79-	-42
2	$\bar{1}$	5	78-	13
2	$\bar{1}$	$\bar{5}$	1453	1433
2	$\bar{1}$	6	254	243
2	$\bar{1}$	$\bar{6}$	67	-4
2	$\bar{1}$	$\bar{7}$	226	-200
2	$\bar{1}$	$\bar{8}$	468	-488
2	2	0	596	563
2	2	1	1920	-1879
2	2	$\bar{1}$	851	851
2	2	2	1544	[illegible]
2	2	$\bar{2}$	1278	1263
2	2	3	366	-336
2	2	$\bar{3}$	664	-666
2	2	4	2338	2362
2	2	$\bar{4}$	335	-332
2	2	5	483	485
2	2	$\bar{5}$	279	-285
2	2	6	876	-872
2	2	7	288	-302
2	2	8	105	-115
2	$\bar{2}$	0	1313	1252
2	$\bar{2}$	1	451	461
2	$\bar{2}$	$\bar{1}$	2327	-2361
2	$\bar{2}$	2	257	242
2	$\bar{2}$	$\bar{2}$	1251	-1277
2	$\bar{2}$	3	88	-10
2	$\bar{2}$	$\bar{3}$	1294	1297
2	$\bar{2}$	4	155	-177
2	$\bar{2}$	$\bar{4}$	880	900
2	$\bar{2}$	5	317	-311
2	$\bar{2}$	$\bar{5}$	345	338
2	$\bar{2}$	$\bar{6}$	65-	-277
2	$\bar{2}$	$\bar{7}$	548	-634
2	$\bar{2}$	$\bar{8}$	367	-337
2	3	0	158	-182
2	3	1	2364	-2333
2	3	$\bar{1}$	332	343
2	3	2	457	462
2	3	$\bar{2}$	329	317
2	3	3	856	895
2	3	$\bar{3}$	662	666
2	3	4	1323	1306
2	3	$\bar{4}$	142	-143
2	3	5	61-	2
2	3	6	765	-752
2	3	7	1175	1176
2	3	8	645	652
2	3	9	473	451
2	$\bar{3}$	0	76-	-40
2	$\bar{3}$	1	74-	41
2	$\bar{3}$	$\bar{1}$	1130	-1139
2	$\bar{3}$	2	261	261
2	$\bar{3}$	$\bar{2}$	125	134
2	$\bar{3}$	3	236	209
2	$\bar{3}$	$\bar{3}$	304	295
2	$\bar{3}$	$\bar{4}$	1328	1345
2	$\bar{3}$	$\bar{5}$	153	-139
2	$\bar{3}$	$\bar{6}$	846	847
2	$\bar{3}$	$\bar{7}$	662	-684
2	$\bar{3}$	$\bar{8}$	544	567
2	$\bar{3}$	$\bar{9}$	513	505
2	4	0	1456	-1485
2	4	1	64	61
2	4	$\bar{1}$	183	-153
2	4	2	1217	1233
2	4	$\bar{2}$	243	261
2	4	3	742	736
2	4	$\bar{3}$	517	515
2	4	4	207	-187
2	4	5	350	-370
2	4	6	906	-902
2	4	7	486	-492
2	4	8	1038	1029
2	4	9	362	366
2	$\bar{4}$	0	355	-338
2	$\bar{4}$	1	365	-340
2	$\bar{4}$	$\bar{1}$	139	129
2	$\bar{4}$	2	123	135
2	$\bar{4}$	$\bar{2}$	77-	21
2	$\bar{4}$	$\bar{3}$	772	774
2	$\bar{4}$	$\bar{4}$	393	407
2	$\bar{4}$	$\bar{5}$	1060	-1055
2	$\bar{4}$	$\bar{6}$	250	-252
2	$\bar{4}$	$\bar{7}$	231	-246
2	$\bar{4}$	$\bar{8}$	456	463
2	$\bar{4}$	$\bar{9}$	537	539
2	5	0	364	-382
2	5	1	285	269
2	5	$\bar{1}$	489	-485
2	5	2	650	666
2	5	$\bar{2}$	102	-98
2	5	3	1570	1589
2	5	4	1620	-1644
2	5	5	1307	-1317
2	5	6	210	232
2	5	7	812	800
2	5	8	223	230
2	5	9	369	377
2	5	10	408	-419
2	$\bar{5}$	0	276	-271
2	$\bar{5}$	1	139	-110
2	$\bar{5}$	$\bar{1}$	241	212
2	$\bar{5}$	$\bar{2}$	305	301
2	$\bar{5}$	$\bar{3}$	476	480
2	$\bar{5}$	$\bar{4}$	657	-643
2	$\bar{5}$	$\bar{5}$	476	-463
2	$\bar{5}$	$\bar{6}$	70-	-33
2	$\bar{5}$	$\bar{7}$	399	399
2	$\bar{5}$	$\bar{8}$	285	284
2	$\bar{5}$	$\bar{9}$	166	156
2	6	0	131	110
2	6	1	1270	259
2	6	2	557	571
2	6	3	65-	53
2	6	4	1095	-1101
2	6	5	633	-625
2	6	6	388	397
2	6	7	1245	1272
2	6	8	77-	24
2	6	9	371	-361
2	6	10	739	-718
2	$\bar{6}$	$\bar{1}$	292	280
2	$\bar{6}$	$\bar{2}$	95-	55
2	$\bar{6}$	$\bar{3}$	102	-81
2	$\bar{6}$	$\bar{4}$	162	-149
2	$\bar{6}$	$\bar{5}$	363	-380
2	$\bar{6}$	$\bar{6}$	66	-58
2	$\bar{6}$	$\bar{7}$	1057	1034
2	$\bar{6}$	$\bar{8}$	162	-164
2	$\bar{6}$	$\bar{9}$	416	-358
2	7	1	353	328
2	7	2	163	-171
2	7	3	524	-522
2	7	4	66	62
2	7	5	446	-445
2	7	6	734	744
2	7	7	785	793
2	7	8	393	-399
2	7	9	703	-709
2	7	10	273	-273
2	$\bar{7}$	$\bar{3}$	202	-200
2	$\bar{7}$	$\bar{4}$	78	-56
2	$\bar{7}$	$\bar{5}$	73-	-14
2	$\bar{7}$	$\bar{6}$	182	189
2	$\bar{7}$	$\bar{7}$	287	293
2	$\bar{7}$	$\bar{8}$	80-	-67
2	$\bar{7}$	$\bar{9}$	370	-374
2	8	3	427	-425
2	8	4	92-	70
2	8	5	403	390
2	8	6	529	539
2	8	7	339	-328
2	8	8	311	-290
2	8	9	449	-478
2	8	10	200	201
2	$\bar{8}$	$\bar{6}$	94-	27
2	$\bar{8}$	$\bar{7}$	135	-64
2	9	6	88-	-38
2	9	7	205	230
2	9	8	408	-407
2	9	9	171	-150
3	0	0	873	894
3	0	1	134	-137
3	0	$\bar{1}$	1385	1375
3	0	2	185	-206
3	0	$\bar{2}$	745	-746
3	0	3	617	-612
3	0	$\bar{3}$	1865	-1878
3	0	4	308	-303
3	0	$\bar{4}$	560	573
3	0	5	530	539
3	0	$\bar{5}$	405	415
3	0	6	103-	59
3	0	$\bar{6}$	409	411
3	1	0	1430	1440
3	1	1	750	-744
3	1	$\bar{1}$	802	794
3	1	2	994	-1023
3	1	$\bar{2}$	333	322
3	1	3	906	-907
3	1	$\bar{3}$	1078	-1072
3	1	4	567	883
3	1	$\bar{4}$	316	-311
3	1	5	547	536
3	1	$\bar{5}$	85	23
3	1	6	77	72
3	1	$\bar{6}$	252	258
3	1	7	136	-149
3	$\bar{1}$	0	123	-134
3	$\bar{1}$	1	1199	1212
3	$\bar{1}$	$\bar{1}$	108	-118
3	$\bar{1}$	2	153	-264
3	$\bar{1}$	$\bar{2}$	551	-580
3	$\bar{1}$	3	288	-292
3	$\bar{1}$	$\bar{3}$	647	-641
3	$\bar{1}$	4	290	-264
3	$\bar{1}$	$\bar{4}$	63-	82
3	$\bar{1}$	5	75-	13
3	$\bar{1}$	$\bar{5}$	1096	1098
3	$\bar{1}$	$\bar{6}$	373	378
3	$\bar{1}$	$\bar{7}$	649	-632
3	2	0	618	600
3	2	1	1790	-1786
3	2	$\bar{1}$	966	999
3	2	2	552	-550
3	2	$\bar{2}$	140	132
3	2	3	56-	14
3	2	$\bar{3}$	362	371
3	2	4	792	794
3	2	$\bar{4}$	573	-571
3	2	5	615	623
3	2	$\bar{5}$	496	-495
3	2	6	69	-32
3	2	7	899	-679
3	2	8	107	105
3	$\bar{2}$	0	569	-569
3	$\bar{2}$	1	873	894
3	$\bar{2}$	$\bar{1}$	563	-576
3	$\bar{2}$	2	349	333
3	$\bar{2}$	$\bar{2}$	423	-437
3	$\bar{2}$	3	270	-276
3	$\bar{2}$	$\bar{3}$	262	245
3	$\bar{2}$	4	82	-70
3	$\bar{2}$	$\bar{4}$	371	374
3	$\bar{2}$	$\bar{5}$	878	874
3	$\bar{2}$	$\bar{6}$	527	-528
3	$\bar{2}$	$\bar{7}$	371	-356
3	$\bar{2}$	$\bar{8}$	203	-228
3	3	0	1432	-1339
3	3	1	499	-531
3	3	$\bar{1}$	401	450
3	3	2	104	116
3	3	$\bar{2}$	723	723
3	3	3	904	893
3	3	$\bar{3}$	485	461
3	3	4	420	440
3	3	$\bar{4}$	407	-394
3	3	5	183	190
3	3	6	609	-618
3	3	7	664	-663
3	3	8	293	260
3	$\bar{3}$	0	97	131
3	$\bar{3}$	1	96-	93
3	$\bar{3}$	$\bar{1}$	972	-990
3	$\bar{3}$	2	112	99
3	$\bar{3}$	$\bar{2}$	66-	-73
3	$\bar{3}$	3	105	101
3	$\bar{3}$	$\bar{3}$	1048	1050
3	$\bar{3}$	$\bar{4}$	240	233
3	$\bar{3}$	$\bar{5}$	160	-157
3	$\bar{3}$	$\bar{6}$	531	-523
3	$\bar{3}$	$\bar{7}$	256	-266
3	$\bar{3}$	$\bar{8}$	76	55
3	4	0	734	-724
3	4	1	381	-512
3	4	$\bar{1}$	378	-376
3	4	2	316	314
3	4	$\bar{2}$	321	320
3	4	3	2262	2266
3	4	$\bar{3}$	479	468
3	4	4	578	-576
3	4	5	1101	-1099
3	4	6	415	-389
3	4	7	227	203
3	4	8	91	81
3	4	9	578	577
3	$\bar{4}$	0	74	52
3	$\bar{4}$	1	80	-83
3	$\bar{4}$	$\bar{1}$	356	-368
3	$\bar{4}$	$\bar{2}$	435	408
3	$\bar{4}$	$\bar{3}$	262	275
3	$\bar{4}$	$\bar{4}$	242	221
3	$\bar{4}$	$\bar{5}$	372	-371
3	$\bar{4}$	$\bar{6}$	514	-499
3	$\bar{4}$	$\bar{7}$	230	-219
3	$\bar{4}$	$\bar{8}$	669	679
3	5	0	276	-258
3	5	1	66-	2
3	5	$\bar{1}$	462	-459
3	5	2	972	962
3	5	3	634	647
3	5	4	541	-545
3	5	5	1173	-1178
3	5	6	398	-399
3	5	7	946	959
3	5	8	450	436
3	5	9	92-	79
3	$\bar{5}$	0	88-	5
3	$\bar{5}$	$\bar{1}$	154	126
3	$\bar{5}$	$\bar{2}$	64	56
3	$\bar{5}$	$\bar{3}$	179	211
3	$\bar{5}$	$\bar{4}$	72	-62
3	$\bar{5}$	$\bar{5}$	615	-603
3	$\bar{5}$	$\bar{6}$	168	155
3	$\bar{5}$	$\bar{7}$	94	77
3	$\bar{5}$	$\bar{8}$	328	324
3	6	0	193	204
3	6	1	607	597
3	6	2	205	208
3	6	3	410	-408
3	6	4	64-	-1
3	6	5	1102	-1107
3	6	6	385	384
3	6	7	1010	1011
3	6	8	218	245
3	6	9	465	-485
3	6	10	391	-389
3	$\bar{6}$	$\bar{2}$	78-	27
3	$\bar{6}$	$\bar{3}$	85	84
3	$\bar{6}$	$\bar{4}$	357	-327
3	$\bar{6}$	$\bar{5}$	139	-131
3	$\bar{6}$	$\bar{6}$	285	258
3	$\bar{6}$	$\bar{7}$	195	188
3	$\bar{6}$	$\bar{8}$	77-	2
3	7	2	78	63
3	7	3	678	-678
3	7	4	420	-414
3	7	5	265	266
3	7	6	742	740
3	7	7	142	113
3	7	8	69-	27
3	7	9	661	-685
3	7	10	174	-142
3	$\bar{7}$	$\bar{5}$	96-	-55
3	$\bar{7}$	$\bar{6}$	83-	61
3	$\bar{7}$	$\bar{7}$	418	398
3	8	3	414	-409
3	8	4	118	-121
3	8	5	609	616
3	8	6	279	292
3	8	7	105	70
3	8	8	575	-581
3	8	9	551	-530
3	9	6	110	80
3	9	7	163	-201
3	9	8	551	-564
4	0	0	997	1011
4	0	1	83	57
4	0	$\bar{1}$	150	136
4	0	2	240	-230
4	0	$\bar{2}$	420	-415
4	0	3	600	-597
4	0	$\bar{3}$	726	-727
4	0	4	104	88
4	0	$\bar{4}$	123-	56
4	0	5	183	194
4	0	$\bar{5}$	329	320
4	0	$\bar{6}$	242	244
4	1	0	878	898
4	1	1	547	-553
4	1	$\bar{1}$	728	750
4	1	2	519	-508
4	1	$\bar{2}$	384	-403
4	1	3	294	-287
4	1	$\bar{3}$	65-	18
4	1	4	87-	76
4	1	$\bar{4}$	585	-583
4	1	5	407	404
4	1	$\bar{5}$	158	-173
4	1	$\bar{6}$	277	257
4	$\bar{1}$	0	367	354
4	$\bar{1}$	1	99	92
4	$\bar{1}$	$\bar{1}$	223	217
4	$\bar{1}$	2	265	263
4	$\bar{1}$	$\bar{2}$	748	-748
4	$\bar{1}$	3	258	-260
4	$\bar{1}$	$\bar{3}$	847	-852
4	$\bar{1}$	4	259	-250
4	$\bar{1}$	$\bar{4}$	951	955
4	$\bar{1}$	5	330	329
4	$\bar{1}$	$\bar{6}$	81	43
4	2	0	540	-534
4	2	1	436	-429
4	2	$\bar{1}$	812	839
4	2	2	605	-619
4	2	$\bar{2}$	656	630
4	2	3	332	317
4	2	$\bar{3}$	69-	8
4	2	4	320	327
4	2	$\bar{4}$	682	-683
4	2	5	389	393
4	2	6	100-	-96
4	2	7	373	-360
4	$\bar{2}$	0	265	-271
4	$\bar{2}$	1	421	424
4	$\bar{2}$	$\bar{1}$	245	-241
4	$\bar{2}$	2	81	101
4	$\bar{2}$	$\bar{2}$	189	-204
4	$\bar{2}$	3	82-	-31
4	$\bar{2}$	$\bar{3}$	80	-76
4	$\bar{2}$	$\bar{4}$	374	383
4	$\bar{2}$	$\bar{5}$	476	476
4	$\bar{2}$	$\bar{6}$	96-	-84
4	$\bar{2}$	$\bar{7}$	609	-593
4	3	0	420	-396
4	3	1	887	-915
4	3	$\bar{1}$	115	107
4	3	2	427	-435
4	3	$\bar{2}$	647	639
4	3	3	1509	1505
4	3	$\bar{3}$	293	277
4	3	4	247	293
4	3	5	366	-343

Table 3 (*cont.*)

h	k	l	$100F_o$	$100F_c$	h	k	l	$100F_o$	$100F_c$	h	k	l	$100F_o$	$100F_c$	h	k	l	$100F_o$	$100F_c$
4	3	6	304	-296	4	$\bar{4}$	$\bar{4}$	123	-140	4	6	7	322	325	5	1	$\bar{1}$	520	524
4	$\bar{3}$	7	97	-102	4	$\bar{4}$	$\bar{5}$	205	-192	4	6	8	412	435	5	1	2	462	-483
4	$\bar{3}$	0	466	-461	4	$\bar{4}$	$\bar{6}$	282	-273	4	6	9	398	-403	5	1	$\bar{2}$	188	177
4	$\bar{3}$	1	230	247	4	$\bar{4}$	$\bar{7}$	101	82	4	7	2	88	54	5	1	3	78-	-29
4	$\bar{3}$	$\bar{1}$	164	-148	4	5	0	199	-186	4	7	3	473	-470	5	1	$\bar{3}$	311	317
4	$\bar{3}$	$\bar{2}$	89	94	4	5	1	440	446	4	7	4	581	-589	5	1	4	86-	37
4	$\bar{3}$	$\bar{3}$	241	223	4	5	$\bar{1}$	142	-632	4	7	5	363	372	5	1	$\bar{4}$	543	-520
4	$\bar{3}$	$\bar{4}$	170	173	4	5	2	448	439	4	7	6	388	397	5	1	5	215	189
4	$\bar{3}$	$\bar{5}$	228	229	4	5	3	88-	53	4	7	7	535	521	5	$\bar{1}$	0	370	372
4	$\bar{3}$	$\bar{6}$	565	-555	4	5	4	296	302	4	7	8	296	-291	5	$\bar{1}$	1	153	114
4	$\bar{3}$	$\bar{7}$	114	-110	4	5	5	1152	-1161	4	7	9	621	-581	5	$\bar{1}$	$\bar{1}$	194	-196
4	4	0	531	-493	4	5	6	225	-228	4	8	5	401	408	5	$\bar{1}$	2	90	69
4	4	1	502	-532	4	5	7	562	553	4	8	6	443	428	5	$\bar{1}$	$\bar{2}$	188	-189
4	4	$\bar{1}$	293	-304	4	5	8	461	474	4	8	7	94-	66	5	$\bar{1}$	$\bar{3}$	89	-125
4	4	2	603	595	4	$\bar{5}$	$\bar{2}$	234	229	4	8	8	565	-593	5	$\bar{1}$	$\bar{4}$	215	226
4	4	$\bar{2}$	353	334	4	$\bar{5}$	$\bar{3}$	75	40	5	0	0	519	547	5	$\bar{1}$	$\bar{5}$	254	245
4	4	3	804	807	4	$\bar{5}$	$\bar{4}$	124	126	5	0	1	69-	-2	5	2	0	147	159
4	4	4	392	394	4	$\bar{5}$	$\bar{5}$	127	134	5	0	$\bar{1}$	189	186	5	2	1	554	-536
4	4	5	808	-799	4	$\bar{5}$	$\bar{6}$	105	98	5	0	2	146	-151	5	2	$\bar{1}$	308	314
4	4	6	706	-694	4	$\bar{5}$	$\bar{7}$	88-	42	5	0	$\bar{2}$	540	-553	5	2	2	642	-632
4	4	7	269	270	4	6	1	538	560	5	0	3	125	-116	5	2	$\bar{2}$	475	462
4	4	8	246	280	4	6	2	539	516	5	0	$\bar{3}$	80-	24	5	2	3	521	496
4	$\bar{4}$	0	44-	-28	4	6	3	438	-427	5	0	4	159	-150	5	2	$\bar{3}$	114	-114
4	$\bar{4}$	$\bar{1}$	241	-255	4	6	4	715	-714	5	0	$\bar{4}$	269	-244	5	2	4	283	282
4	$\bar{4}$	$\bar{2}$	143	164	4	6	5	114	-130	5	1	0	82-	74	5	2	5	72-	-6
4	$\bar{4}$	$\bar{3}$	427	421	4	6	6	142	341	5	1	1	71-	25	5	$\bar{2}$	0	91	25

h	k	l	$100F_o$	$100F_c$	h	k	l	$100F_o$	$100F_c$	h	k	l	$100F_o$	$100F_c$	h	k	l	$100F_o$	$100F_c$
5	$\bar{2}$	1	81-	-39	5	4	5	589	-576	6	0	$\bar{1}$	98	90					
5	$\bar{2}$	$\bar{1}$	91-	-16	5	4	6	397	-388	6	0	$\bar{2}$	77-	-33					
5	$\bar{2}$	$\bar{2}$	312	-315	5	4	7	76	70	6	1	0	279	285					
5	$\bar{2}$	$\bar{3}$	175	-159	5	$\bar{4}$	$\bar{3}$	83-	21	6	1	1	85	-88					
5	$\bar{2}$	$\bar{4}$	671	663	5	$\bar{4}$	$\bar{4}$	107-	9	6	1	$\bar{1}$	146	136					
5	$\bar{2}$	$\bar{5}$	76-	0	5	5	0	482	-475	6	1	2	348	-347					
5	3	0	285	-263	5	5	1	300	301	6	1	$\bar{2}$	135	127					
5	3	1	620	-626	5	5	2	686	659	6	$\bar{1}$	$\bar{1}$	79-	-58					
5	3	$\bar{1}$	101	88	5	5	3	90-	74	6	$\bar{1}$	$\bar{2}$	313	-306					
5	3	2	82-	-4	5	5	4	482	-456	6	2	0	88-	20					
5	3	$\bar{2}$	489	472	5	5	5	230	-225	6	2	1	283	-277					
5	3	3	328	324	5	5	6	94	-146	6	2	$\bar{1}$	239	225					
5	3	4	646	640	5	5	7	104	119	6	2	2	145	-157					
5	3	5	176	-182	5	6	2	382	375	6	2	3	108-	-83					
5	3	6	415	-403	5	6	3	139	-135	6	3	0	283	-270					
5	$\bar{3}$	$\bar{1}$	99-	-51	5	6	4	691	-694	6	3	1	126	-114					
5	$\bar{3}$	$\bar{2}$	81-	13	5	6	5	72	-55	6	3	2	93-	-60					
5	$\bar{3}$	$\bar{3}$	79-	47	5	6	6	92	100	6	3	3	91	50					
5	$\bar{3}$	$\bar{4}$	217	222	5	6	7	556	544	6	3	4	412	422					
5	$\bar{3}$	$\bar{5}$	77-	14	5	7	4	532	-509	6	4	1	86	-66					
5	4	0	422	-417	5	7	5	97	82	6	4	2	396	370					
5	4	1	95-	41	5	7	6	483	467	6	4	3	239	231					
5	4	$\bar{1}$	401	-399	5	7	7	321	322	6	4	4	124	-111					
5	4	2	233	218	6	0	0	133-	103	6	5	3	190	200					
5	4	3	218	224	6	0	1	171	172	6	5	4	390	-364					
5	4	4	549	559															

Table 4. *Bond distances and angles in the molecule of thiosemicarbazide*

	Present work	Previous work (DGNS)
S——C	1·707 (2) Å	1·685 (5) Å
C——N(1)	1·316 (3)	1·313 (6)
C——N(2)	1·326 (2)	1·337 (6)
N(2)–N(3)	1·411 (2)	1·399 (6)
S——C——N(1)	122·0 (0·1)°	119·7 (0·3)°
S——C——N(2)	119·2 (0·2)	121·5 (0·3)
N(1)–C——N(2)	118·8 (0·2)	118·8 (0·4)
C——N(2)–N(3)	121·1 (0·2)	122·5 (0·4)

pair of the N(3) atom must lie in it, and must interact with the H(1,2) and H(1,2^v) atoms in a double system of centrosymmetrical bifurcated hydrogen-bonds as shown in Fig. 2. This interpretation is supported also by a calculation carried out assuming the N(3) atom to have four s^np^m hybrid orbitals in such a way that three of them are directed towards N(2), H(3,1), H(3,2) and the fourth is determined by the orthogonality condition. The lone pair orbital is found to lie in the molecular plane making an angle of 109·7° with respect to the N(2)–N(3) direction so that it points to the centre of the dimer formed by two centrosymmetrical molecules, and overlaps, to the same extent the two H(1,2) and H(1,2^v) hydrogen atoms. [The authors are indebted to Professor L. Oleari (Istituto di Chimica Fisica, Università di Parma) who kindly performed the calculation]. This is indicated also by the values of the two distances N(3)···H(1,2) = 2·34 Å and N(3^v)···H(1,2) = 2·39 Å which are practically equal and significantly shorter than the sum of the van der Waals radii for H and N (2·70 Å). Fig. 3 shows the NH···S linkages:

N(1)···S^{iii}	3·409 Å	N(2)···S^i	3·359 Å
H(1,1)···S^{iii}	2·55	H(2,1)···S^i	2·54
S^{iii}–N(1)–H(1,1)	11·1°	S^i–N(2)–H(2,1)	8·4°

which form two-dimensional arrangements parallel to (1$\bar{1}$0). These arrangements are joined together by hydrogen bonds involving N(3):

N(3)——S^{ii}	3·816 Å	N(3)——S^{iv}	3·514 Å
H(3,2)–S^{ii}	2·97	H(3,1)–S^{iv}	2·65
H(3,2)–N(3)–S^{ii}	28·9°	H(3,1)–N(3)–S^{iv}	11·1°

Atoms belonging to different asymmetric units are labelled as follows:

Superscripts	Coordinates
none	x, y, z
i	$\bar{x}, \bar{y}, 1-z$
ii	$1-x, \bar{y}, 1-z$
iii	$\bar{x}, \bar{y}, 2-z$
iv	$x, 1+y, z-1$
v	$1-x, 1-y, 1-z$

Table 5. *Bond distances and angles involving* H *atoms in the molecule*

The corrected values are calculated following Busing & Levy (1964).

	Found	Riding model	Upper bound
N(1)—H(1,1)	0·88 Å	0·92 Å	0·97 Å
N(1)—H(1,2)	0·86	0·88	0·93
N(2)—H(2,1)	0·83	0·86	0·90
N(3)—H(3,1)	0·89	0·91	0·97
N(3)—H(3,2)	0·86	0·89	0·94
C——N(1)—H(1,1)	119·1 (1·5)°		
C——N(1)—H(1,2)	119·4 (1·4)		
H(1,1)–N(1)—H(1,2)	121·4 (2·1)		
C——N(2)—H(2,1)	119·8 (1·0)		
N(3)—N(2)—H(2,1)	119·0 (1·0)		
N(2)—N(3)—H(3,1)	107·5 (1·5)		
N(2)—N(3)—H(3,2)	105·3 (1·7)		
H(3,1)–N(3)—H(3,2)	108·1 (1·7)		

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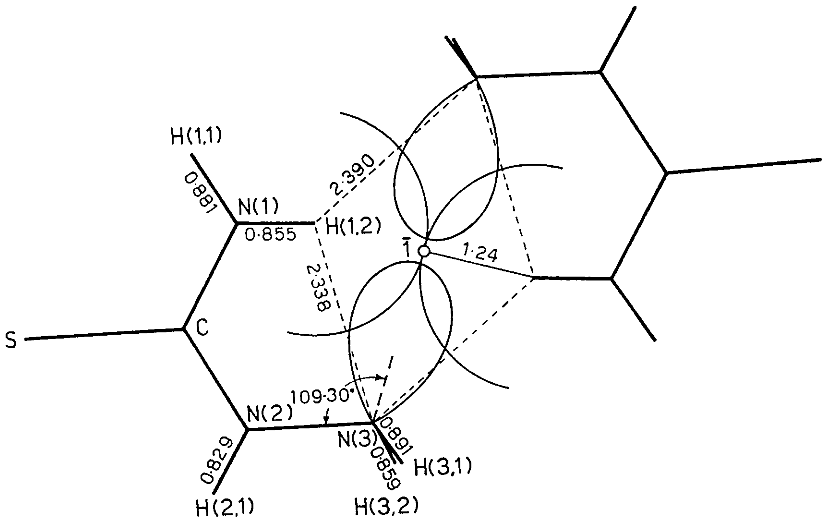

Fig. 2. Arrangement of two centrosymmetrical molecules with respect to $\bar{1}(\frac{1}{2}\frac{1}{2}\frac{1}{2})$.

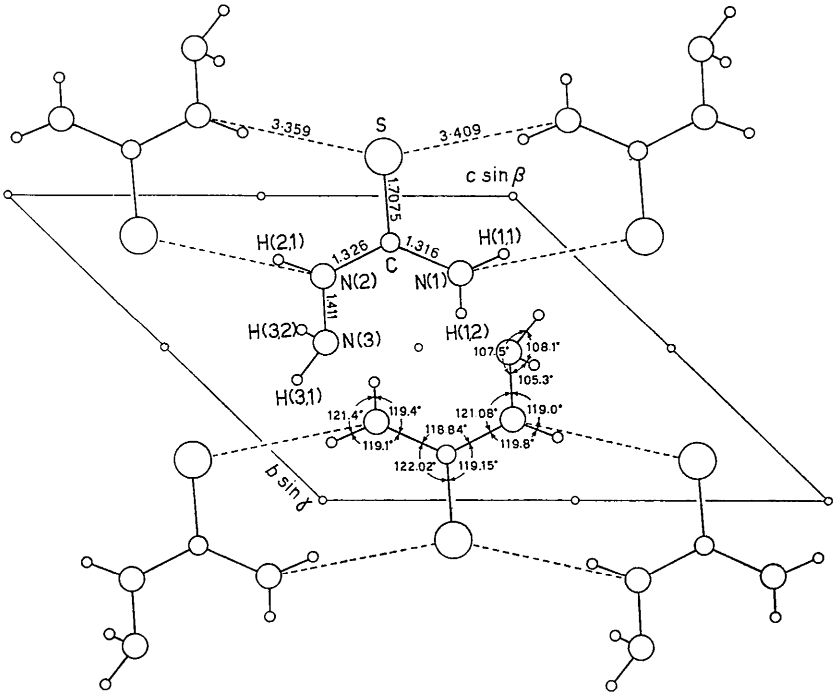

Fig. 3. Diagrammatic projection of the structure along [100].

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