Journal of Organometallic Chemistry
Elsevier Sequoia S.A., Lausanne
Printed in The Netherlands

THE CRYSTAL STRUCTURE OF DIMETHYLTIN DIISOTHIOCYANATE

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(Received December 19th, 1969)

SUMMARY

The crystal structure of $Me_2Sn(NCS)_2$ has been determined by single crystal X-ray diffraction. The crystals are orthorhombic, *Pmmn*, $a=9.79(2)$, $b=7.88(2)$, $c=5.57(2)$ Å, $Z=2$. The structure consists of rather distorted tetrahedral dimethyltin diisothiocyanate molecules, linked by S–Sn interactions to form infinite chains along the Z axis with a repeat unit of one molecule. The Sn–C(H$_3$) bonds lie in a plane perpendicular to that of the planar $Sn(NCS)_2$ groups; the NCS units are vitually linear. The principal bond lengths and angles are: Sn–N 2.139(13), N–C 1.174(14), C–S 1.571(13), S–Sn 3.202(7), Sn–C(H$_3$) 2.089(14) Å; ∠Sn–N–C 163.5(1.1), ∠N–C–S 177.2(1.2), ∠C–S–Sn 90.4(0.5), ∠(H$_3$)C–Sn–C(H$_3$) 148.9(0.9)°.

INTRODUCTION

In the crystal structure[1,2] of trimethyltin isothiocyanate, the molecules are arranged in infinite zig-zag =S–Sn–N=C=S–Sn– chains, linear except at sulphur. Mössbauer[3] and IR[4] spectra of solid dimethyltin diisothiocyanate are reasonably consistent with an analogous crystal structure for this compound, the coordination of the tin atoms being intermediate between tetrahedral and octahedral. In view of possible ambiguities in the interpretation of the spectroscopic data, we have determined the crystal structure of dimethyltin diisothiocyanate. When our determination had reached an advanced stage, we learnt that Prof. D. Britton and Dr. Y. M. Chow had recently completed a determination of the same structure; we are grateful to these authors for a preprint of this work[5]. The two structures are consistent within experimental error. The determination reported here appears to be the more precise; the estimated standard deviations in the bond lengths and angles are lower by a factor of about three, probably because the refinement was based on more reflections (334 instead of 147).

EXPERIMENTAL

The sample was prepared by the reaction of Me_2SnCl_2 and NaNCS (mole ratio 1/2) in ethanol; the precipitate of NaCl was filtered off, the solvent was removed under vacuum, and the crystals were obtained by two recrystallisations from benzene.

(*Continued p.* 614)

TABLE 1

OBSERVED AND CALCULATED STRUCTURE FACTORS

Since the interlayer scale factors were refined, it is necessary to multiply these values (which are on an absolute scale) by 1.155, 0.999, 0.804, 0.853 and 0.772 for *hk*0, *hk*1, ..., *hk*4 for use in the weighting scheme.

h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$
2	0	0	43.7	33.4	7	3	1	21.9	21.9	3	4	2	13.9	14.7	1	4	3	27.0	30.4
4	0	0	98.2	87.5	8	3	1	6.7	5.1	4	4	2	26.9	27.6	2	4	3	9.9	9.3
6	0	0	49.1	47.3	9	3	1	15.9	16.1	5	4	2	30.4	30.6	3	4	3	24.9	25.3
8	0	0	25.1	25.6	0	4	1	36.7	37.9	6	4	2	20.1	20.1	4	4	3	24.4	24.6
10	0	0	17.0	18.9	1	4	1	10.8	10.3	8	4	2	11.9	10.6	5	4	3	16.2	16.0
1	1	0	77.9	78.4	2	4	1	58.9	63.9	9	4	2	12.2	11.6	6	4	3	10.3	8.9
3	1	0	66.8	68.2	3	4	1	37.1	34.7	0	5	2	10.8	10.6	7	4	3	17.8	16.1
5	1	0	38.1	35.5	4	4	1	34.9	32.7	1	5	2	26.4	30.0	8	4	3	7.5	7.0
7	1	0	53.4	48.5	6	4	1	27.9	28.7	2	5	2	22.5	23.9	9	4	3	6.1	6.7
9	1	0	8.7	9.0	7	4	1	15.0	14.1	3	5	2	24.3	25.5	0	5	3	19.4	21.8
11	1	0	12.1	12.4	8	4	1	19.1	19.1	4	5	2	11.5	10.3	1	5	3	16.4	17.9
0	2	0	109.5	111.6	10	4	1	7.2	6.9	5	5	2	27.4	25.2	2	5	3	19.9	23.1
4	2	0	53.9	61.4	0	5	1	14.5	15.3	6	5	2	11.3	11.5	3	5	3	24.7	24.0
6	2	0	32.4	32.4	1	5	1	37.2	38.6	7	5	2	8.5	7.8	4	5	3	13.9	13.2
8	2	0	17.5	17.7	3	5	1	31.8	31.8	8	5	2	5.8	5.8	5	5	3	9.6	7.6
10	2	0	12.9	14.1	4	5	1	17.9	17.2	9	5	2	11.1	10.6	6	5	3	14.5	13.9
1	3	0	62.1	69.2	5	5	1	28.4	25.8	0	6	2	17.3	18.6	7	5	3	14.8	13.4
3	3	0	54.4	55.7	7	5	1	15.5	15.3	1	6	2	12.9	13.6	0	6	3	21.2	23.3
5	3	0	29.5	31.0	9	5	1	10.5	10.6	2	6	2	24.2	25.8	1	6	3	16.1	16.7
7	3	0	45.8	38.6	0	6	1	19.4	19.7	3	6	2	5.8	5.2	2	6	3	10.4	11.2
9	3	0	7.8	8.5	2	6	1	28.8	32.3	4	6	2	18.1	18.4	3	6	3	14.8	14.2
11	3	0	10.3	10.1	3	6	1	12.5	13.5	5	6	2	13.6	13.8	4	6	3	18.4	17.2
0	4	0	93.5	98.0	4	6	1	17.1	17.2	6	6	2	12.8	13.0	5	6	3	10.3	8.9
2	4	0	28.0	29.1	6	6	1	14.9	15.2	8	6	2	8.8	7.3	6	6	3	8.9	8.2
4	4	0	52.4	53.6	7	6	1	5.9	5.5	0	7	2	9.8	8.4	7	6	3	10.4	9.4
6	4	0	33.5	31.1	8	6	1	11.4	10.6	1	7	2	15.4	16.4	0	7	3	10.7	12.4
8	4	0	17.9	17.3	0	7	1	9.5	7.7	2	7	2	12.4	12.6	1	7	3	9.5	9.9
10	4	0	12.9	12.6	1	7	1	21.1	21.5	3	7	2	13.9	14.3	2	7	3	13.6	13.3
1	5	0	41.1	43.0	3	7	1	17.9	18.7	4	7	2	7.4	7.2	3	7	3	11.4	12.3
3	5	0	34.6	34.3	4	7	1	9.0	8.9	5	7	2	14.1	13.6	4	7	3	8.4	8.0
5	5	0	20.5	21.4	5	7	1	15.7	14.3	6	7	2	7.5	6.8	5	7	3	4.6	5.0
7	5	0	23.1	23.9	7	7	1	9.9	9.6	7	7	2	5.1	5.0	6	7	3	8.2	8.0
9	5	0	6.5	6.6	0	8	1	12.2	12.8	0	8	2	8.5	9.8	0	8	3	7.7	9.2
0	6	0	49.1	46.7	2	8	1	18.8	17.7	1	8	2	9.8	9.4	1	8	3	8.5	9.5
2	6	0	14.4	13.4	3	8	1	8.6	8.3	2	8	2	10.6	12.0	2	8	3	3.7	5.1
4	6	0	22.6	25.3	4	8	1	12.5	10.8	3	8	2	6.4	5.5	3	8	3	8.4	8.3
6	6	0	16.1	15.6	6	8	1	9.0	9.0	4	8	2	9.3	9.5					
8	6	0	9.1	8.6	7	8	1	4.1	3.5	5	8	2	8.2	8.4	1	0	4	32.1	36.2

1	7	0	22.6	24.6
3	7	0	20.3	19.1
5	7	0	14.4	14.1
7	7	0	13.7	13.0
0	8	0	24.2	25.2
2	8	0	12.4	11.5
4	8	0	16.9	15.0
6	8	0	11.9	10.3
1	9	0	11.2	11.2
1	0	1	19.8	15.5
2	0	1	126.1	100.2
3	0	1	71.2	66.0
4	0	1	56.9	49.1
5	0	1	5.3	4.3
6	0	1	46.8	44.2
7	0	1	26.0	23.9
8	0	1	28.4	28.6
10	0	1	7.4	9.6
11	0	1	5.6	7.5
12	0	1	5.5	9.1
0	1	1	35.0	40.5
1	1	1	69.1	74.5
3	1	1	63.0	59.9
4	1	1	41.8	41.6
5	1	1	54.3	50.9
7	1	1	27.4	25.8
8	1	1	5.8	5.8
9	1	1	17.4	19.7
11	1	1	6.0	7.1
0	2	1	30.0	32.4
1	2	1	14.1	9.4
2	2	1	76.4	80.0
3	2	1	40.2	40.0
4	2	1	36.5	34.6
5	2	1	13.6	12.9
6	2	1	37.8	33.8
7	2	1	16.4	15.2
8	2	1	23.7	22.6
9	2	1	5.6	3.4
12	2	1	5.8	7.4
0	3	1	25.8	29.7
1	3	1	60.2	61.6
3	3	1	45.1	49.0
4	3	1	30.6	31.2
5	3	1	40.5	40.6
1	9	1	9.7	9.8
3	9	1	8.6	9.0
4	9	1	4.1	3.4
5	9	1	6.5	6.4
1	0	2	60.0	58.1
2	0	2	82.3	68.8
3	0	2	22.0	20.4
4	0	2	49.4	41.0
5	0	2	55.6	51.0
6	0	2	33.5	30.4
8	0	2	14.7	15.3
9	0	2	18.8	18.3
10	0	2	10.9	11.3
0	1	2	10.6	12.5
1	1	2	50.4	57.2
2	1	2	55.4	54.0
3	1	2	56.9	47.0
4	1	2	19.3	16.4
5	1	2	56.4	48.9
6	1	2	25.6	22.3
7	1	2	12.1	11.4
8	1	2	11.6	10.9
9	1	2	18.2	19.6
0	2	2	32.8	36.3
1	2	2	29.8	31.7
2	2	2	62.6	64.9
3	2	2	5.2	4.9
4	2	2	42.1	40.1
5	2	2	40.4	35.2
6	2	2	30.3	29.2
8	2	2	14.6	15.1
9	2	2	13.7	13.1
10	2	2	10.3	11.0
0	3	2	14.7	14.1
1	3	2	42.0	45.5
2	3	2	39.4	41.7
3	3	2	39.6	37.8
4	3	2	14.9	15.0
5	3	2	42.1	38.6
6	3	2	18.9	18.3
7	3	2	10.0	10.0
8	3	2	9.7	9.1
9	3	2	15.7	15.8
0	4	2	22.8	25.6
1	4	2	29.9	33.7
2	4	2	35.7	42.6
6	8	2	6.0	6.5
0	9	2	6.8	4.4
1	9	2	7.9	8.0
2	9	2	5.3	5.0
3	9	2	6.1	7.2
1	0	3	61.5	47.5
2	0	3	10.5	8.5
3	0	3	44.1	38.5
4	0	3	50.7	39.3
5	0	3	26.4	23.7
6	0	3	13.6	12.0
7	0	3	25.0	23.7
8	0	3	10.8	9.8
9	0	3	10.3	9.7
10	0	3	7.6	8.6
0	1	3	41.3	41.5
1	1	3	27.2	31.1
2	1	3	43.9	43.9
3	1	3	50.2	47.7
4	1	3	26.5	23.1
5	1	3	12.4	9.3
6	1	3	27.6	25.6
7	1	3	28.8	26.3
8	1	3	14.3	14.0
0	2	3	58.4	61.2
1	2	3	35.0	37.5
2	2	3	16.4	19.3
3	2	3	31.1	30.6
4	2	3	44.5	42.4
5	2	3	20.7	18.7
6	2	3	17.7	16.2
7	2	3	20.9	19.7
8	2	3	13.1	12.1
9	2	3	9.8	7.8
10	2	3	8.9	9.4
0	3	3	28.3	33.6
1	3	3	21.6	24.9
2	3	3	29.0	35.4
3	3	3	34.1	36.9
4	3	3	21.4	19.4
5	3	3	6.4	8.4
6	3	3	22.6	20.9
7	3	3	22.0	20.6
8	3	3	12.5	11.8
0	4	3	32.2	34.6
2	0	4	25.4	22.2
3	0	4	35.5	35.7
5	0	4	17.0	18.0
6	0	4	9.4	10.2
7	0	4	17.3	22.3
0	1	4	49.5	60.4
1	1	4	22.0	23.4
2	1	4	16.6	18.3
3	1	4	8.1	8.9
4	1	4	36.4	38.2
5	1	4	16.2	17.0
6	1	4	15.8	18.1
0	2	4	15.6	16.1
1	2	4	35.5	36.3
2	2	4	26.3	25.9
3	2	4	37.2	34.9
5	2	4	18.5	19.0
6	2	4	11.4	12.6
7	2	4	18.9	21.4
0	3	4	55.3	47.9
1	3	4	20.6	18.4
2	3	4	17.1	16.1
4	3	4	30.7	30.6
5	3	4	12.1	13.3
6	3	4	14.2	15.2
0	4	4	9.3	7.9
1	4	4	28.2	24.7
2	4	4	15.8	14.2
3	4	4	25.6	23.3
5	4	4	12.8	13.6
7	4	4	12.4	14.5
0	5	4	34.3	31.2
1	5	4	15.3	12.6
2	5	4	13.7	12.6
3	5	4	3.7	6.2
4	5	4	22.9	20.4
5	5	4	9.4	8.9
6	5	4	10.4	11.0
0	6	4	11.2	8.2
1	6	4	21.5	17.1
2	6	4	14.2	10.9
3	6	4	19.0	15.3
5	6	4	10.0	10.4
0	7	4	21.9	16.7
1	7	4	8.2	6.7
2	7	4	10.1	8.0
1	8	4	9.1	8.6

(Found: C, 18.0; H, 2.5; N, 9.7. $C_4H_6N_2S_2Sn$ calcd.: C, 18.1; H, 2.3; N, 10.6%.) The mass spectrum exhibited a parent ion and was entirely consistent with the proposed formula.

Intensities were determined visually from equi-inclination Weissenberg photographs taken with a crystal mounted about the Z (needle) axis (in a sealed Lindemann glass capillary tube) and Ni-filtered Cu-$K\alpha$ radiation (layers $hk0$–$hk4$ inclusive). Lorentz, polarisation and absorption corrections were applied, the latter being calculated exactly for a cylinder with $\mu \cdot r = 1.3$, where μ is the linear absorption coefficient and r the radius of the cylinder. Unit-cell dimensions were obtained from powder photographs, taken with a Guinier focussing camera and silicon internal calibrant ($a = 5.4306$ Å), and indexed using the approximate unit-cell constants and intensity data from the Weissenberg photographs. The estimated standard deviations in the unit-cell dimensions were multiplied by a factor of two to allow for possible systematic errors.

Crystal data

$C_4H_6N_2S_2Sn$, $M = 264.9$, orthorhombic, $a = 9.79(2)$*, $b = 7.88(2)$, $c = 5.57(2)$, $U = 429.7$, $d_c = 2.05$ for $Z = 2$. This value of Z was consistent with the interpretation of the Patterson function. Systematic absences: $hk0$, $h+k \neq 2n$. These determine the space group as $Pmmn$, $P2_1mn$ or $Pm2_1n$ (with the axes in the same orientation).

Structure solution and refinement

In the centrosymmetric space group $Pmmn$, the tin atoms lie in special positions $\pm(0.25, 0.25, z)$ on the line of intersection of the mirror planes; the thiocyanate and methyl groups must lie in special positions either in the mirror plane perpendicular to Y or in that perpendicular to X. From the Patterson and Fourier syntheses it was clear that the NCS groups lay in the former mirror plane and the carbon atoms of the methyl groups in the latter.

The structure was refined in $Pmmn$ by full-matrix least-squares, initially with isotropic temperature factors for all the atoms; the weighting scheme adopted on the basis of an analysis of variance was: $w = 1/(100 + F_o^2)$. In the final stage of the refinement a total of 25 parameters were varied simultaneously, including four inter-layer scale factors, anisotropic temperature factors for the tin and sulphur atoms only, and the Zachariasen secondary extinction coefficient[6]. Complex neutral-atom scattering factors were employed for all atoms[7,8]. The final value of $R = \Sigma w^{\frac{1}{2}} \cdot \Delta / \Sigma w^{\frac{1}{2}} \cdot F_o$ ($\Delta = |F_o| - |F_c|$) was 0.083 for a total of 334 unique observed reflections; the corresponding unweighted "R index" was 0.080. A final difference Fourier revealed no pronounced features.

After this refinement in the centrosymmetric space group $Pmmn$ had been completed, a number of other models were tested using Hamilton's "R factor ratio" test[9]. A generalised index $R_G = \Sigma w \cdot \Delta^2 / \Sigma w \cdot F_o^2$ was calculated for each of the refinements described below, and compared with the value of 0.1053 found for the final refinement described above.

(A). When the Zachariasen extinction coefficient was fixed at zero, R_G con-

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* Estimated standard deviations (in parentheses) are given in units of the least significant digit of the quantity to which they refer.

verged to 0.1088. This one parameter hypothesis was rejected at the 0.5% significance level. Clearly it is important to refine or correct for extinction in fairly rigid structures of this type.

(*B*). A structure in the space group $P2_1mn$ with an "all *trans*" arrangement and approximate local centre of symmetry at the tin atom (suggested by Chow[5] as a possible, though inferior, interpretation of the Patterson function) refined to R_G = 0.2301, and so could be immediately rejected.

(*C*). A refinement in the space group $P2_1mn$ was performed with all the atomic positions displaced a little from their centrosymmetric values; R_G converged to 0.1008 (0.1016 for the enantiomeric structure). For eight extra parameters these are just significant at the 1% and 2.5% levels respectively. Although this structure would thus be preferred (although not decisively) on the basis of the statistical test, it was rejected because of the very poor agreement between chemically equivalent bond lengths (*e.g.* Sn–N = 2.30 and 1.97 Å).

(*D*). An analogous refinement in the space group $Pm2_1n$ gave R_G = 0.1028 (0.1030 for the enantiomeric structure); this is only significant at the 10% level, and so was rejected.

These refinements confirm that the centrosymmetric structure is correct. Observed and calculated structure factors are shown in Table 1, and the results from the final full-matrix least-squares cycle are given in Tables 2 and 3. These results together with the full covariance matrix and the estimated standard deviations in the unit-cell dimensions were used to calculate the bond lengths and angles given in Table 4; no corrections have been applied for libration. Fig. 1 shows the structure in projection down the *Y* and *X* axes.

TABLE 2

ATOM COORDINATES AND ISOTROPIC VIBRATIONAL AMPLITUDES ($Å^2 \times 1000$) WITH ESTIMATED STANDARD DEVIATIONS

Atom	*x/a*	*y/b*	*z/c*	$\bar{U}$
N	0.1002(13)	0.2500	0.2648(22)	68(3)
C	0.0461(13)	0.2500	0.0768(20)	56(3)
C(1)	0.2500	−0.0054(16)	0.6448(26)	68(3)

The Zachariasen secondary extinction coefficient refined to the value 3.7(1.0) × 10^{-6}.

TABLE 3

ATOM COORDINATES AND ANISOTROPIC VIBRATIONAL AMPLITUDES ($Å^2 \times 1000$) WITH ESTIMATED STANDARD DEVIATIONS

Atom	*x/a*	*y/b*	*z/c*	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Sn	0.2500	0.2500	0.5442(2)	65(1)	57(1)	35(4)	0	0	0
S	−0.0332(4)	0.2500	−0.1683(7)	67(2)	104(2)	57(5)	0	−11(1)	0

The anisotropic temperature factor takes the form:

$$\exp\left[-2\pi^2(U_{11}\cdot h^2\cdot a^{*2}+U_{22}\cdot k^2\cdot b^{*2}+U_{33}\cdot l^2\cdot c^{*2}+2U_{23}\cdot k\cdot l\cdot b^*\cdot c^*+2U_{31}\cdot l\cdot h\cdot c^*\cdot a^*+2U_{12}\cdot h\cdot k\cdot a^*\cdot b^*)\right]$$

TABLE 4

INTERATOMIC DISTANCES (Å) AND BOND ANGLES (°)

Sn–N	2.139(13)	N–C	1.174(14)
C–S	1.571(13)	Sn–C(1)	2.089(14)
S′–Sn	3.202(7)		
Sn–N–C	163.5(1.1)	N–C–S	177.2(1.2)
C(1)–Sn–N	101.3(0.3)	C(1)–Sn–S	82.3(0.2)
C(1)–Sn–C(1)′	148.9(0.9)	C′–S′–Sn	90.4(0.5)
S′–Sn–N	163.3(0.3) (*trans*)	S′–Sn–N	76.7(0.4) (*cis*)
N–Sn–N′	86.6(0.5)	S′–Sn–S″	120.0(0.3)

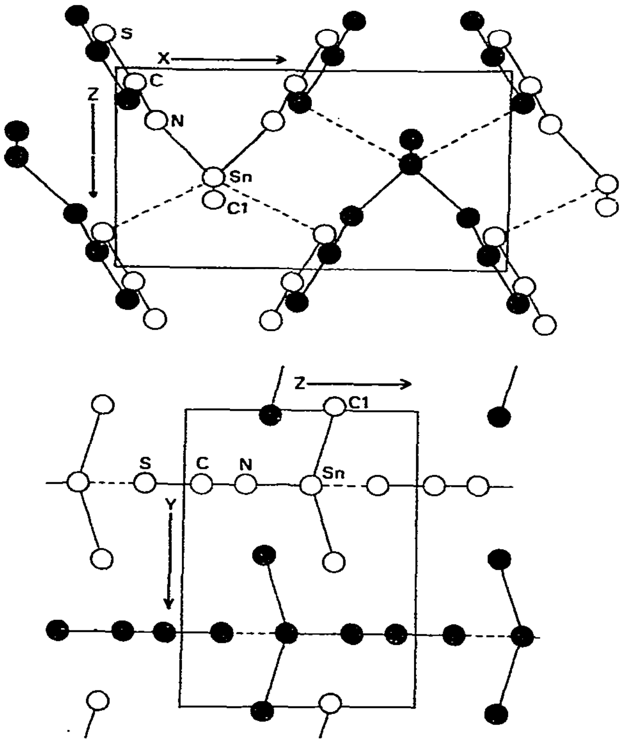

Fig. 1. The crystal structure seen in projection down the *Y* and *X* axes.

DISCUSSION

A detailed comparison of the bond lengths and angles in $Me_2Sn(NCS)_2$ with those in other thiocyanates has been given by Chow[5]. The bond lengths and angles are reasonably close to those in Me_3SnNCS[1,2], but there is a general trend to more "normal" values of the N–C and C–S bond lengths and Sn–N–C angle; the S–Sn distance is slightly greater in the dimethyl derivative. These differences could be attributed to the competition of two sulphur atoms for each tin atom, and perhaps also in part to the greater precision of the results reported here. The problems of bonding are the same, albeit less acute, than those discussed for Me_3SnNCS[1]; the Sn–N–C angles of 173(5)° in Me_3SnNCS and 163.5(1.1)° in $Me_2Sn(NCS)_2$ can be accounted for without invoking "p_π–d_π" bonding from nitrogen to tin.

ACKNOWLEDGEMENTS

We are grateful to the S.R.C. for a maintenance grant to R.A.F., and to the University Mathematical Laboratory for the provision of computing facilities. The calculations were performed using the Titan computer and programs written by G.M.S.

TABLE 5

SHORTEST NON-BONDED DISTANCES (Å), WITH TRANSFORMATIONS RELATING THE COORDINATES OF THE SECOND ATOM TO THOSE OF THE CORRESPONDING ATOM IN THE SAME ASYMMETRIC UNIT AS THE FIRST ATOM

N–S	2.744	x	y	z
N–N	2.934	$0.5-x$	y	z
Sn–S	3.202	x	y	$1+z$
N–C(1)	3.268	x	y	z
Sn–C	3.281	x	y	z
N–S	3.417	x	y	$1+z$
Sn–C	3.576	x	y	$1+z$
S–C(1)	3.581	x	y	$-1+z$
N–C	3.618	$0.5-x$	y	z
C–C(1)	3.719	x	y	$-1+z$
C–C(1)	3.811	$-x$	$-y$	$1-z$
C(1)–C(1)	3.854	x	$-0.5-y$	z
S–C(1)	3.906	$-x$	$-y$	$-z$
N–C(1)	3.965	$-x$	$-y$	$1-z$
C–S	3.975	$-x$	$1-y$	$-z$
C–C	3.992	$0.5-x$	y	z
C(1)–C(1)	4.026	x	$0.5-y$	z
N–S	4.030	$-x$	$1-y$	$-z$
S–C(1)	4.089	$-x$	$-y$	$1-z$
C–C	4.132	$-x$	$1-y$	$-z$
S–S	4.244	$-0.5-x$	y	z

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