

The Crystal and Molecular Structure of Dicinnamyl Disulfide

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The crystal and molecular structure of dicinnamyl disulfide has been reinvestigated with a new set of data obtained from single crystals. The unit cell and space group are the same as in a previous study, but, contrary to the earlier work, no unusual structural features in the form of bifurcated C-H...S hydrogen bonds were found. The major difference in molecular structure is the torsion angle at the carbon-carbon single bond adjacent to the disulfide group. In the present work this angle is about 120°, a value which does not lead to the close intramolecular H...S contacts brought about the value of nearly 0° of the former study. A relationship between the new positional parameters and those of the previous work causes the calculated F values to be very nearly the same if l is even. Major discrepancies between observed F values when l is odd are noted. These discrepancies could be explained by the existence of two different conformers of this substance with the same unit cell and space group.

Introduction

The molecule of dicinnamyl disulfide, $C_6H_5-CH=CH-CH_2-S-S-CH_2-CH=CH-C_6H_5$, as it occurs in crystals studied by Lee & Bryant (1971) (LB), was found by them to contain a most unusual structural feature consisting of intramolecular H...S distances of 2.28 and 2.32 Å which were interpreted as indicating bifurcated C-H...S hydrogen bonds, the carbon atoms in question being those attached to the phenyl groups. This feature prompted us to reinvestigate this structure, but because the data set published by LB is incomplete (*e.g.* all $1k1$ and $1k3$ are missing from their F table) a new set of data was collected in this laboratory.

Experimental

Crystals were obtained by evaporation from acetone. Precession and Weissenberg photography confirmed the symmetry and space group, $Fdd2$, reported by LB.

Least-squares treatment of the 2θ values greater than 40° for 12 reflections observed with a Picker four-circle diffractometer gave the following lattice constants: $a = 17.390$ (2), $b = 34.220$ (6), $c = 5.4053$ (8) Å; the differences, respectively, of -0.2, +0.1, and -0.3% from those of LB are not significant. The unit cell contains eight molecules which lie on twofold axes parallel to z .

A crystal approximately 0.17 × 0.17 × 0.17 mm was used to collect a complete set of data out to $2\theta = 125^\circ$ with a Picker diffractometer equipped with Cu $K\alpha$ radiation and a graphite monochromator. The $\theta-2\theta$ scan mode was used, with a scan rate of 2.0° min⁻¹ and a 20 s count for each background. The intensities of three standard reflections, collected every 50 reflections, remained constant during the data collection. A total of 722 unique non-zero reflections was observed. Of these, a total of 705 reflections had F_{obs} greater than $2\sigma_{F_{obs}}$ (counting statistics). The number of reflections listed by LB is 660.

Table 1. Positional and thermal parameters (with estimated standard deviations) for the sulfur and carbon atoms

All values $\times 10^4$. Temperature factor = $\exp[-(h^2b_{11} + k^2b_{22} + l^2b_{33} + hkb_{12} + hlb_{13} + klb_{23})]$.

	x	y	z	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
S	177.3 (0.6)	281.0 (0.3)	0	52	12	412	6	20	29
C(1)	855 (3)	318 (1)	-2649 (11)	54	11	466	-3	5	-6
C(2)	1436 (2)	0 (1)	-2705 (10)	39	11	425	-4	10	0
C(3)	1492 (2)	-257 (1)	-4507 (8)	44	11	352	-7	13	-12
C(4)	2035 (2)	-582 (1)	-4663 (9)	41	10	348	-8	-37	-6
C(5)	2602 (2)	-649 (1)	-2897 (10)	51	14	423	3	-2	1
C(6)	3090 (3)	-965 (2)	-3065 (14)	60	17	583	17	-31	-31
C(7)	3017 (3)	-1223 (1)	-4990 (14)	70	14	674	10	-152	-14
C(8)	2474 (3)	-1161 (1)	-6788 (13)	78	12	651	-9	-175	48
C(9)	1979 (3)	-838 (1)	-6641 (10)	52	14	442	-11	-52	25

Structure refinement

A three-dimensional electron-density function, phased with the position of the sulfur atom given by LB, revealed clearly the positions of the nine carbon atoms in the asymmetric unit, and refinement by least-squares calculations which allowed for isotropic thermal motion of these ten atoms converged smoothly in 11 block-diagonal cycles to an *R* of 10.5%. The nine hydrogen atoms were then placed in their expected positions, assigned isotropic *B* values of 6 Å², and the refinement of the heavy atoms, with anisotropic thermal parameters, was continued to an *R* of 4.6% for *F*_{obs} greater than 2σ_{*F*obs} after five additional cycles, in which the parameters changed only slightly. Hydrogen-atom coordinates were recalculated after each cycle using the new heavy-atom coordinates. A dif-

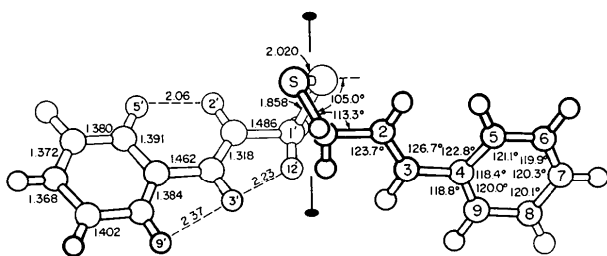


Fig. 1. Interatomic distances and bond angles in the molecule of dicinnamyl disulfide.

ference Fourier map calculated using all 722 reflections and all atom positions showed maximum fluctuations in the difference function of less than 3% of the carbon atom peak values found in the three-dimensional electron-density function. The final parameters are given in Tables 1 and 2. Observed and calculated structure factors are shown in Table 3; Table 4 gives an agreement analysis between *F*_{obs} and *F*_{calc} similar to that given by LB.

Table 2. Calculated hydrogen positions

	All values × 10 ⁴		
	<i>x</i>	<i>y</i>	<i>z</i>
H(11)	1174	0584	-2368
H(12)	0513	0282	-4288
H(2)	1832	-0022	-1187
H(3)	1096	-0225	-6022
H(5)	2659	-0449	-1366
H(6)	3528	-1009	-1687
H(7)	3386	-1475	-5089
H(8)	2426	-1362	-8316
H(9)	1558	-0790	-8061

Discussion

A view of the molecule perpendicular to both the two-fold axis and the C(7)-C(7') vector, including the bond distances and angles, is shown in Fig. 1. The S-S' bond length of 2.020 (2) Å, although shorter than the average value of 2.060 (3) Å observed in the S₈ mole-

Table 3. Observed and calculated structure factors

Reflections marked with an asterisk were omitted from the least-squares refinement.

<i>hkl</i>	<i>F</i> _o	<i>F</i> _c	<i>hkl</i>	<i>F</i> _o	<i>F</i> _c	<i>hkl</i>	<i>F</i> _o	<i>F</i> _c	<i>hkl</i>	<i>F</i> _o	<i>F</i> _c	<i>hkl</i>	<i>F</i> _o	<i>F</i> _c	<i>hkl</i>	<i>F</i> _o	<i>F</i> _c	<i>hkl</i>	<i>F</i> _o	<i>F</i> _c
0 0 0	1264	1271	2 2 2	197	201	4 4 4	81	81	6 6 6	41	41	8 8 8	24	24	10 10 10	14	14	12 12 12	8	8
0 2 0	258	258	2 4 0	154	150	4 6 0	107	107	6 8 0	67	67	8 10 0	47	47	10 12 0	30	30	12 14 0	19	19
0 4 0	79	78	2 6 0	80	78	4 8 0	43	43	6 10 0	25	25	8 12 0	13	13	10 14 0	7	7	12 16 0	4	4
0 6 0	25	25	2 8 0	37	36	4 10 0	19	19	6 12 0	11	11	8 14 0	6	6	10 16 0	3	3	12 18 0	2	2
0 8 0	15	15	2 10 0	22	21	4 12 0	11	11	6 14 0	7	7	8 16 0	4	4	10 18 0	2	2	12 20 0	1	1
0 10 0	9	9	2 12 0	13	13	4 14 0	7	7	6 16 0	4	4	8 18 0	3	3	10 20 0	1	1	12 22 0	1	1
0 12 0	5	5	2 14 0	8	8	4 16 0	4	4	6 18 0	3	3	8 20 0	2	2	10 22 0	1	1	12 24 0	1	1
0 14 0	3	3	2 16 0	5	5	4 18 0	3	3	6 20 0	2	2	8 22 0	1	1	10 24 0	1	1	12 26 0	1	1
0 16 0	2	2	2 18 0	3	3	4 20 0	2	2	6 22 0	1	1	8 24 0	1	1	10 26 0	1	1	12 28 0	1	1
0 18 0	1	1	2 20 0	2	2	4 22 0	1	1	6 24 0	1	1	8 26 0	1	1	10 28 0	1	1	12 30 0	1	1
0 20 0	1	1	2 22 0	1	1	4 24 0	1	1	6 26 0	1	1	8 28 0	1	1	10 30 0	1	1	12 32 0	1	1
0 22 0	1	1	2 24 0	1	1	4 26 0	1	1	6 28 0	1	1	8 30 0	1	1	10 32 0	1	1	12 34 0	1	1
0 24 0	1	1	2 26 0	1	1	4 28 0	1	1	6 30 0	1	1	8 32 0	1	1	10 34 0	1	1	12 36 0	1	1
0 26 0	1	1	2 28 0	1	1	4 30 0	1	1	6 32 0	1	1	8 34 0	1	1	10 36 0	1	1	12 38 0	1	1
0 28 0	1	1	2 30 0	1	1	4 32 0	1	1	6 34 0	1	1	8 36 0	1	1	10 38 0	1	1	12 40 0	1	1
0 30 0	1	1	2 32 0	1	1	4 34 0	1	1	6 36 0	1	1	8 38 0	1	1	10 40 0	1	1	12 42 0	1	1
0 32 0	1	1	2 34 0	1	1	4 36 0	1	1	6 38 0	1	1	8 40 0	1	1	10 42 0	1	1	12 44 0	1	1
0 34 0	1	1	2 36 0	1	1	4 38 0	1	1	6 40 0	1	1	8 42 0	1	1	10 44 0	1	1	12 46 0	1	1
0 36 0	1	1	2 38 0	1	1	4 40 0	1	1	6 42 0	1	1	8 44 0	1	1	10 46 0	1	1	12 48 0	1	1
0 38 0	1	1	2 40 0	1	1	4 42 0	1	1	6 44 0	1	1	8 46 0	1	1	10 48 0	1	1	12 50 0	1	1
0 40 0	1	1	2 42 0	1	1	4 44 0	1	1	6 46 0	1	1	8 48 0	1	1	10 50 0	1	1	12 52 0	1	1
0 42 0	1	1	2 44 0	1	1	4 46 0	1	1	6 48 0	1	1	8 50 0	1	1	10 52 0	1	1	12 54 0	1	1
0 44 0	1	1	2 46 0	1	1	4 48 0	1	1	6 50 0	1	1	8 52 0	1	1	10 54 0	1	1	12 56 0	1	1
0 46 0	1	1	2 48 0	1	1	4 50 0	1	1	6 52 0	1	1	8 54 0	1	1	10 56 0	1	1	12 58 0	1	1
0 48 0	1	1	2 50 0	1	1	4 52 0	1	1	6 54 0	1	1	8 56 0	1	1	10 58 0	1	1	12 60 0	1	1
0 50 0	1	1	2 52 0	1	1	4 54 0	1	1	6 56 0	1	1	8 58 0	1	1	10 60 0	1	1	12 62 0	1	1
0 52 0	1	1	2 54 0	1	1	4 56 0	1	1	6 58 0	1	1	8 60 0	1	1	10 62 0	1	1	12 64 0	1	1
0 54 0	1	1	2 56 0	1	1	4 58 0	1	1	6 60 0	1	1	8 62 0	1	1	10 64 0	1	1	12 66 0	1	1
0 56 0	1	1	2 58 0	1	1	4 60 0	1	1	6 62 0	1	1	8 64 0	1	1	10 66 0	1	1	12 68 0	1	1
0 58 0	1	1	2 60 0	1	1	4 62 0	1	1	6 64 0	1	1	8 66 0	1	1	10 68 0	1	1	12 70 0	1	1
0 60 0	1	1	2 62 0	1	1	4 64 0	1	1	6 66 0	1	1	8 68 0	1	1	10 70 0	1	1	12 72 0	1	1
0 62 0	1	1	2 64 0	1	1	4 66 0	1	1	6 68 0	1	1	8 70 0	1	1	10 72 0	1	1	12 74 0	1	1
0 64 0	1	1	2 66 0	1	1	4 68 0	1	1	6 70 0	1	1	8 72 0	1	1	10 74 0	1	1	12 76 0	1	1
0 66 0	1	1	2 68 0	1	1	4 70 0	1	1	6 72 0	1	1	8 74 0	1	1	10 76 0	1	1	12 78 0	1	1
0 68 0	1	1	2 70 0	1	1	4 72 0	1	1	6 74 0	1	1	8 76 0	1	1	10 78 0	1	1	12 80 0	1	1
0 70 0	1	1	2 72 0	1	1	4 74 0	1	1	6 76 0	1	1	8 78 0	1	1	10 80 0	1	1	12 82 0	1	1
0 72 0	1	1	2 74 0	1	1	4 76 0	1	1	6 78 0	1	1	8 80 0	1	1	10 82 0	1	1	12 84 0	1	1
0 74 0	1	1	2 76 0	1	1	4 78 0	1	1	6 80 0	1	1	8 82 0	1	1	10 84 0	1	1	12 86 0	1	1
0 76 0	1	1	2 78 0	1	1	4 80 0	1	1	6 82 0	1	1	8 84 0	1	1	10 86 0	1	1	12 88 0	1	1
0 78 0	1	1	2 80 0	1	1	4 82 0	1	1	6 84 0	1	1	8 86 0	1	1	10 88 0	1	1	12 90 0	1	1
0 80 0	1	1	2 82 0	1	1	4 84 0	1	1	6 86 0	1	1	8 88 0	1	1	10 90 0	1	1	12 92 0	1	1
0 82 0	1	1	2 84 0	1	1	4 86 0	1	1	6 88 0	1	1	8 90 0	1	1	10 92 0	1	1	12 94 0	1	1
0 84 0	1	1	2 86 0	1	1	4 88 0	1	1	6 90 0	1	1	8 92 0	1	1	10 94 0	1	1	12 96 0	1	1
0 86 0	1	1	2 88 0	1	1	4 90 0	1	1	6 92 0	1	1	8 94 0	1	1	10 96 0	1	1	12 98 0	1	1
0 88 0	1	1	2 90 0	1	1	4 92 0	1	1	6 94 0	1	1	8 96 0	1	1	10 98 0	1	1	12 100 0	1	1
0 90 0	1	1	2 92 0	1	1	4 94 0	1	1	6 96 0	1	1	8 98 0	1	1	10 100 0	1	1	12 102 0	1	1
0 92 0	1	1	2 94 0	1	1	4 96 0	1	1	6 98 0	1	1	8 100 0	1	1	10 102 0	1	1	12 104 0	1	1
0 94 0	1	1	2 96 0	1	1	4 98 0	1	1	6 100 0	1	1	8 102 0	1	1	10 104 0	1	1	12 106 0	1	1
0 96 0	1	1	2 98 0	1	1	4 100 0	1	1	6 102 0	1	1	8 104 0	1	1	10 106 0	1	1	12 108 0	1	1
0 98 0	1	1	2 100 0	1	1	4 102 0	1	1	6 104 0	1	1	8 106 0	1	1	10 108 0	1	1	12 110 0	1	1
0 100 0	1	1	2 102 0	1	1	4 104 0	1	1	6 106 0	1	1	8 108 0	1	1	10 110 0	1	1	12 112 0	1	1
0 102 0	1	1	2 104 0	1	1	4 106 0	1	1	6 108 0	1	1	8 110 0	1	1	10 112 0	1	1	12 114 0	1	1
0 104 0	1	1	2 106 0	1	1	4 108 0	1	1	6 110 0	1	1	8 112 0	1	1	10 114 0	1	1	12 116 0	1	1
0 106 0	1	1	2 108 0	1	1	4 110 0	1	1	6 112 0	1	1	8 114 0	1	1	10 116 0	1	1	12 118 0	1	1
0 108 0	1	1	2 110 0	1	1	4 112 0	1	1	6 114 0	1	1	8 116 0	1	1	10 118 0	1	1	12 120 0	1	1
0 110 0	1	1	2 112 0	1	1	4 114 0	1	1	6 116 0	1	1	8 118 0	1	1	10 120 0	1	1	12 122 0	1	1
0 112 0	1	1	2 114 0	1	1	4 116 0	1	1	6 118 0	1	1	8 120 0	1	1	10 122 0	1	1	12 124 0	1	1
0 114 0	1	1	2 116 0	1	1	4 118 0	1	1	6 120 0	1	1	8 122 0	1	1	10 124 0	1	1	12 126 0	1	1
0 116 0	1	1	2 118 0	1	1	4 120 0	1	1	6 122 0	1	1	8 124 0	1	1	10 126 0	1	1	12 128 0	1	1
0 118 0	1	1	2 120 0	1	1	4 122 0	1	1	6 124 0	1	1	8 126 0	1	1	10 128 0	1	1	12 130 0	1	1
0 120 0	1	1	2 122 0	1	1	4 124 0	1	1	6 126 0	1	1	8 128 0	1	1	10 130 0	1	1	12 132 0	1	1

Table 4. Agreement analysis for dicinnamyl disulfide

sin θ	Number of reflections	R (%)	Layer	Number of reflections	R (%)
0.0-0.1	2	1.8	hk0	145	5.7
0.1-0.2	10	4.1	hk1	151	4.7
0.2-0.3	22	5.8	hk2	134	4.1
0.3-0.4	40	4.2	hk3	119	4.0
0.4-0.5	65	4.8	hk4	90	3.4
0.5-0.6	93	3.8	hk5	57	5.0
0.6-0.7	129	4.0	hk6	9	6.3
0.7-0.8	165	4.5			
0.8-0.9	179	6.3			

cule in orthorhombic sulfur (Caron & Donohue, 1965), is not different from those observed in dibenzyl disulfide (van Dijk & Visser, 1971) and diphenyl disulfide (Lee & Bryant, 1969), 2.015 (3) and 2.027 (5) Å, respectively. The C-S bond distance of 1.858 (6) Å, although longer than the normally observed value of 1.815 Å for C-S single bonds (Cox & Jeffrey, 1951), is not different from the value 1.844 (12) Å in dibenzyl disulfide, but longer than those of 1.79 and 1.81 Å in diphenyl disulfide and 1.75 and 1.77 Å in 2,2'-diaminodiphenyl disulfide (Lee & Bryant, 1970).

The C-S-S-C torsion angle is 74.3°, and thus smaller than the values 92.1, 83.8 and 89.5° in the disulfides cited in the preceding paragraph, as well as the value 85.3° found for the S-S-S-S torsion angle in the infinite chains in fibrous sulfur (Lind & Geller, 1969). No simple *ad hoc* explanation for these variations has occurred to us.

The six carbon atoms of the benzene ring are closely coplanar, the average distance from the best plane being 0.008 Å. The four carbon atoms associated with the double bond are also closely coplanar, the average distance from the best plane in this case being 0.007 Å. These two planes are almost, but not quite, coincident. The C(3)-C(4) bond is bent out of the plane of the benzene ring so that C(3) is 0.059 Å from that plane, and a twist about the C(3)-C(4) bond brings C(2) 0.154 Å from the plane. These distortions, as well as the observation that the angle C(5)-C(4)-C(3) is 4.0° larger than the angle C(9)-C(4)-C(3), may result from the repulsion between H(2) and H(5). In an undistorted molecule this distance would be only 1.84 Å, well below the observed value of 2.06 Å. (This distance is still smaller than the value 2.40 Å expected on the basis of the commonly cited van der Waals radius of 1.20 Å for hydrogen.) Other bond distances and angles have the expected values and require no special comments.

The torsion angle about the C(1)-C(2) bond is 117.4°, or close to the value 120° predicted by the bent-bonds concept of the double bond. With this conformational angle the ethylenic group is twisted away from the disulfide group (see Fig. 1) with the result that H(3) is over 3.9 Å away from both of the sulfur atoms, or well in excess of the sum of the two van der Waals radii. There are thus no unusually short intramolecular S...H interactions of the kind termed C-H...S hydrogen bonds by LB.

The torsion angle about the S-C(1) bond is 48.0°. We are not aware of any theory which predicts the geometry of S-S-C-C systems. The observed value in dibenzyl disulfide is 73.4°, but that molecule is not strictly comparable with the dicinnamyl compound.

The dicinnamyl disulfide molecule may be thought of as made up of three parts: the disulfide group, -C(1')-S'-S-C(1)-, and two identical cinnamyl groups, C₆H₅-CH=CH-C(1)H₂-, the C(1) atoms of which are in common with the former. In both the present study and in that of LB the geometries of these two groups are essentially the same: we find an -S-S- torsion angle of 74.3°, not far from the value of 66.4° found by LB, and in both studies the cinnamyl groups were found to be almost coplanar and *trans*, as expected. The remaining two degrees of freedom are: the torsion angle at S-C(1), which is 48.0° and not too different from the value of 55.1° reported LB; and the torsion angle at C(1)-C(2), which is 117.4° and markedly different from the 2.2° obtained by LB.

The two structures thus differ mainly only in the C(1)-C(2) torsion angle, aside from minor, and possibly insignificant, differences in the various other molecular parameters. In both structures the C(1)-C(2) bond is nearly perpendicular to the *z* axis, and rotation of the cinnamyl group of ~120° about this bond does not markedly change the *x* and *y* parameters for all atoms, gives similar *z* parameters for S, C(1) and C(2), and, for atoms C(3) through C(9), *z* parameters for one are close to $\frac{1}{2} - z$ for the other structure. This last approximation occurs because the C(1)-C(2) bond is approximately $\frac{1}{4}$ along *c* from the sulfur atoms. Details of these near-equalities are presented in Table 5.

Table 5. Parameter differences in dicinnamyl disulfide, this work vs. Lee & Bryant (1971)

	Δx (Å)	Δy (Å)	Δz^* (Å)
S	0.005	0.003	(0)
C(1)	0.101	0.021	0.096
C(2)	0.026	0.010	0.124
C(3)	0.059	0.092	0.004
C(4)	0.071	0.017	0.028
C(5)	0.031	0.031	0.046
C(6)	0.056	0.000	0.023
C(7)	0.033	0.010	0.034
C(8)	0.009	0.010	0.068
C(9)	0.002	0.014	0.044
Average	0.039	0.020	0.052
Maximum	0.101	0.092	0.124

* $\Delta z = |z_{DC} - z_{LB}|$ for S, C(1), and C(2), $\Delta z = |\frac{1}{2} - z_{DC} - z_{LB}|$ for other atoms.

Table 6. Average percent differences between $|F_{obs}|$ for this work and Lee & Bryant (1971)

Layer	$R = 100 \sum F_{DC} - F_{LB} / \sum F_{DC}$
hk0	15.4%
hk1	40.7
hk2	15.4
hk3	53.0
hk4	30.3

Table 7. *Some large differences in observed structure factors*

<i>hkl</i>	This work		Lee & Bryant		<i>hkl</i>	This work		Lee & Bryant	
	$ F_{\text{obs}} $	$ F_{\text{calc}} $	$ F_{\text{obs}} $	$ F_{\text{calc}} $		$ F_{\text{obs}} $	$ F_{\text{calc}} $	$ F_{\text{obs}} $	$ F_{\text{calc}} $
711	40	38	97	85	11,1,1	41	40	7	5
931	26	26	75	66	791	80	81	26	21
13,3,1	3	2	19	16	991	73	69	37	31
591	17	19	115	120	333	71	70	21	15
7,13,1	32	31	71	69	13,13,3	25	25	9	6
13,1,3	3	3	16	12	9,15,3	52	51	11	10
533	20	19	68	62	13,15,3	15	15	4	3
13,3,3	3	3	18	14	5,19,3	58	56	18	14
7,21,3	13	11	27	26	5,21,3	38	38	5	4
5,23,3	6	5	27	23	9,21,3	27	28	7	6
5,25,3	8	8	22	19	3,23,3	46	44	13	10

From the relationships between the two sets of parameters it follows that for *hkl* reflections with $l=2n$ the $|F|$ values should be closely similar, the differences arising only from the relatively minor differences shown in Table 5; such similarities are not predicted when l is odd. These predictions are confirmed by observation, as seen in Table 6, which gives the average percent discrepancies, by layer, between the two sets of data.

A number of very large intensity differences exist in the two data sets, some of which are presented in Table 7. Included in this table are the respective calculated values, and it may be seen that, as expected, the structure of LB agrees with *their* observed $|F|$ values, and the structure found in this work agrees with *our* observed $|F|$ values.

A possible explanation of these discrepancies is that LB actually examined a different conformer of this substance having, by coincidence, the same space group, indistinguishable unit-cell dimensions, and closely similar intensities for the *hk0*, *hk2*, and *hk4* reflections. Some support for this explanation is afforded by the information (Lee, 1974, private communication) that the intensities and indexing of their data were checked by measurements made with a Hilger linear

diffractometer at another University. The Weissenberg data, rather than the diffractometer data, were used for their refinement, because the crystal was so small that only about half the reflections could be observed by the diffractometer.

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