[Chem. Pharm. Bull. 18(7)1362—1368(1970)]

UDC 547.597.02 : 548.73 : 582.53604 : 615.322.110.4

Biological-Active Triterpenes of Alismatis Rhizoma. III.[1] The X-Ray Crystallography of Alisol A (23, 24)-Acetonide 11-Monobromoacetate[2]

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(Received January 23, 1970)

The X-ray analysis on alisol A (23, 24)-acetonide 11-monobromoacetate was undertaken establishing the molecular structure to be **1**.

The usefulness of applying the least-squares method at an early stage for distinguishing real atoms from spurious peaks in the maps of minimum functions was again demonstrated.

Elucidation of the chemical structure of alisol A,[1a] one of the hypocholesterolemic triterpenes isolated from a Chinese crude drug "Zexie", the rhizoma of *Alisma Plantago-aquatica* L. var. *orientale* SAMUELS., was done in the preceding paper,[1b] however, the study led to no unequivocal proof of the structure of the compound; the stereochemistry of the compound, especially that of the ring junctures was left almost unclarified.

In order to establish the whole structure of alisol A, X-ray analysis was undertaken. In the course of our attempts to prepare suitable crystals of various derivatives containing a heavy atom, we found that alisol A (23, 24)-acetonide 11-monobromoacetate gave beautiful pillars upon recrystallization from methanol. It was therefore decided to perform the X-ray analysis on the acetonide monobromoacetate $C_{35}H_{55}O_6Br$.

Experimental

Alisol A (23, 24)-acetonide 11-monobromoacetate was prepared by bromoacetylation of alisol A (23, 24)-acetonide with bromoacetyl chloride in N,N-dimethylformamide. Treatment of the bromoacetate with aqueous sodium carbonate solution followed by aqueous acetic acid gave alisol A.[1b] Single crystals for the present work were obtained from a methanol solution. A crystal, $0.40\times0.40\times2.0$ mm was chosen. Weissenberg photographs obtained with Cu-$K\alpha$ radiation showed that the crystal is orthorhombic with the unit cell parameters, $a=18.18\pm0.04$ Å, $b=24.63\pm0.04$ Å and $c=7.75\pm0.03$ Å, where the c-axis is parallel to the needle axis of the crystal. Systematic extinctions of ($h00$) when h is odd, ($0k0$) when k is odd and ($00l$) when l is odd, led to the space group $P2_12_12_1$. Assuming four molecules in the unit cell, the density is calculated to be 1.243 gcm^{-3}, which agrees well with the observed density 1.247 gcm^{-3} (measured by floatation method). Intensity measurements were carried out with a Hilger and Watt's linear diffractometer using Mo-$K\alpha$ radiation (7 layers about the c-axis). 2237 independent reflexion intensities were thus obtained. The absolute structure factors were deduced by the ordinary procedure.

Results and Discussion

Determination of the Structure

The three-dimensional Patterson function was computed with sharpening coefficients $[F_0^2/(\hat{f})^2]\exp[2(B-B')(\sin^2\theta/\lambda^2)]$, where $B-B'$ was chosen as zero and $\hat{f}=\sum f_i/\sum Z_i$ in the usual notation. The three highest peaks (0.50, 0.07, 0.30), (0.12, 0.50, 0.20) and (0.38, 0.43, 0.50) in the three Harker sections H (1/2, v, w), H (u, 1/2, w) and H (u, v, 1/2), respectively,

1) *a*) Part I: T. Murata, Y. Imai, T. Hirata and M. Miyamoto, *Chem. Pharm. Bull.* (Tokyo), **18**, 1347 (1970); *b*) Part II: T. Murata and M. Miyamoto, *ibid.*, **18**, 1354 (1970).
2) A. preliminary report on this work appeared in *Tetrahedron Letters*, **1968**, 103; *cf.* also *ibid.*, **1968**, 849.
3) Location: *Juso-Nishino-cho, Higashiyodogawa-ku, Osaka.*

Table I. Final Atomic Co-ordinates and Temperature Factors, with the Estimated Standard Deriviations in Parrentheses

Atom	x/a	y/b	z/c	b
Br	0.0588 (01)	0.2868 (01)	0.6510 (04)	7.9 (0.1)
O (1)	0.1685 (08)	0.0502 (05)	0.8499 (24)	6.1 (0.4)
O (2)	0.2228 (07)	0.2849 (05)	0.6475 (21)	5.2 (0.3)
O (3)	0.3850 (10)	0.5061 (07)	0.6040 (27)	8.0 (0.5)
O (4)	0.4094 (08)	0.5703 (06)	0.8084 (23)	6.7 (0.4)
O (5)	0.5383 (16)	0.5801 (11)	0.0591 (42)	8.6 (0.8)
O (6)	0.2406 (08)	0.2827 (06)	0.3634 (25)	7.2 (0.4)
C (1)	0.2578 (12)	0.1633 (09)	0.6589 (37)	5.7 (0.5)
C (2)	0.2430 (11)	0.1028 (08)	0.6682 (32)	4.6 (0.5)
C (3)	0.2205 (11)	0.0771 (07)	0.8422 (33)	4.5 (0.5)
C (4)	0.2695 (12)	0.0910 (09)	0.9957 (32)	5.1 (0.5)
C (5)	0.3032 (11)	0.1486 (08)	0.9722 (31)	4.6 (0.5)
C (6)	0.3383 (12)	0.1746 (08)	0.1337 (35)	5.3 (0.5)
C (7)	0.3997 (12)	0.2133 (09)	0.0678 (30)	5.1 (0.5)
C (8)	0.3895 (11)	0.2421 (07)	0.8945 (28)	4.2 (0.5)
C (9)	0.3080 (11)	0.2422 (08)	0.8391 (34)	4.9 (0.5)
C (10)	0.2678 (10)	0.1876 (07)	0.8436 (32)	4.3 (0.4)
C (11)	0.2978 (11)	0.2778 (08)	0.6827 (29)	4.5 (0.5)
C (12)	0.3239 (11)	0.3371 (08)	0.6712 (33)	4.7 (0.5)
C (13)	0.4032 (11)	0.3377 (08)	0.7558 (29)	4.3 (0.5)
C (14)	0.4141 (12)	0.3008 (09)	0.9194 (31)	5.3 (0.5)
C (15)	0.4964 (12)	0.3082 (08)	0.9492 (31)	4.8 (0.5)
C (16)	0.5296 (13)	0.3480 (09)	0.8092 (37)	6.4 (0.6)
C (17)	0.4634 (11)	0.3639 (08)	0.7053 (29)	4.4 (0.5)
C (18)	0.3765 (12)	0.3299 (08)	0.0787 (31)	5.0 (0.5)
C (19)	0.1938 (12)	0.1970 (08)	0.9318 (31)	5.1 (0.5)
C (20)	0.4729 (13)	0.3973 (09)	0.5363 (35)	6.0 (0.6)
C (21)	0.5261 (15)	0.3726 (11)	0.3985 (41)	7.8 (0.8)
C (22)	0.4938 (13)	0.4581 (09)	0.5746 (31)	5.6 (0.6)
C (23)	0.4437 (12)	0.4839 (08)	0.7103 (29)	4.7 (0.5)
C (24)	0.4728 (13)	0.5325 (10)	0.8098 (37)	6.9 (0.7)
C (25)	0.5127 (14)	0.5295 (10)	0.9849 (38)	6.9 (0.7)
C (26)	0.5770 (16)	0.4963 (11)	0.9222 (42)	8.4 (0.8)
C (27)	0.4687 (23)	0.5048 (16)	0.1361 (70)	14.4 (1.4)
C (30)	0.2236 (12)	0.0877 (09)	0.1625 (36)	5.7 (0.5)
C (31)	0.3285 (16)	0.0460 (12)	0.0050 (44)	8.7 (0.8)
C (32)	0.4333 (12)	0.2197 (08)	0.7387 (29)	5.0 (0.5)
C (33)	0.3575 (18)	0.5536 (13)	0.6900 (51)	10.5 (1.0)
C (34)	0.2914 (21)	0.5357 (15)	0.8052 (60)	13.0 (1.2)
C (35)	0.3502 (27)	0.5921 (19)	0.5350 (73)	17.2 (1.8)
C (36)	0.2009 (12)	0.2888 (09)	0.4865 (30)	5.1 (0.5)
C (37)	0.1193 (18)	0.3050 (13)	0.4530 (47)	9.8 (1.0)

led to the co-ordinates of the bromine atom as (0.06, 0.28, 0.64). A minimum function was calculated at this stage leading to a map from which 43 peaks were chosen mainly from consideration of their height and all were tentatively assigned to carbon atoms. The least squares treatment was then applied only to the temperature factors of these 43 atoms.[4] After three cycles of the treatments, it was observed that the temperature factor values of 24 atoms are not diverged. Subsequent Fourier synthesis calculated with the phase angles based on these 24 atoms revealed 41 atoms which visuallized the molecule of alisol A (23, 24)-acetonide 11-monobromoacetate except one atom (carbon or oxygen) at the end of the side chain (C_{25}).

4) M. Nishikawa, K. Kamiya, M. Tomita, Y. Okamoto, T. Kikuchi, K. Osaki, Y. Tomiie, I. Nitta and K. Goto, *J. Chem. Soc.* (*B*), **1968**, 652.

Stereo-model indicated a boat conformation of the B ring of the molecule. But another possibility which led to a chair conformation of the B ring could not be excluded completely. This was discarded, however, after confirmation and refinement of the above atoms by least-squares method with diagonal approximation. The second Fourier synthesis was then carried out based on the refined atomic parameters, and the all atoms including the one at the end of the side chain, described above, were thus confirmed. Further refinement was done by five cycles of least-squares treatments and the final atomic co-ordinates thus obtained are listed in Table I.[5] R-value was 0.142 at this stage.

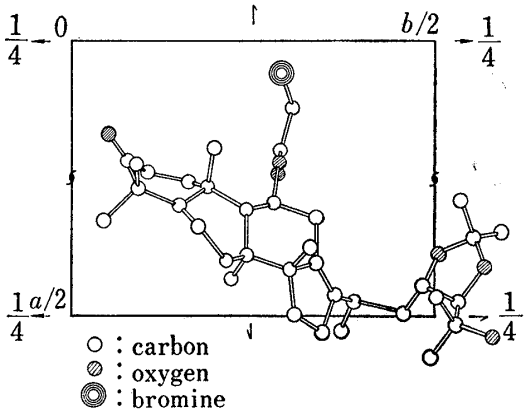

Fig. 1. Stereo-model of Alisol A (23, 24)-Acetonide 11-Monobromoacetate

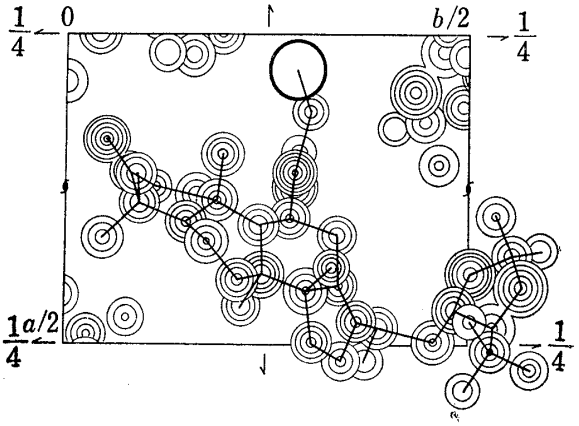

Fig. 2. The Final Three-dimensional Electron-density Distribution shown by Means of Superimposed Contour Sections Projected on (001)

Table II. Bond Lengths (Angstroms)

Br—C (37)	1.94	C (8)–C (32)	1.55
O (1)–C (21)	1.16	C (9)–C (10)	1.53
O (2)–C (11)	1.40	C (9)–C (11)	1.51
O (2)–C (36)	1.31	C (10)–C (19)	1.53
O (3)–C (23)	1.45	C (11)–C (12)	1.54
O (3)–C (33)	1.44	C (12)–C (13)	1.58
O (4)–C (24)	1.48	C (13)–C (14)	1.57
O (4)–C (33)	1.38	C (13)–C (17)	1.33
O (5)–C (25)	1.45	C (14)–C (15)	1.52
O (6)–C (36)	1.21	C (14)–C (18)	1.58
C (1)–C (2)	1.51	C (15)–C (16)	1.58
C (1)–C (10)	1.56	C (16)–C (17)	1.50
C (2)–C (3)	1.54	C (17)–C (20)	1.56
C (3)–C (4)	1.53	C (20)–C (21)	1.56
C (4)–C (5)	1.56	C (20)–C (22)	1.57
C (4)–C (30)	1.54	C (22)–C (23)	1.53
C (4)–C (31)	1.54	C (23)–C (24)	1.52
C (5)–C (6)	1.54	C (24)–C (25)	1.54
C (5)–C (10)	1.53	C (25)–C (26)	1.54
C (6)–C (7)	1.55	C (25)–C (27)	1.51
C (7)–C (8)	1.53	C (33)–C (34)	1.56
C (8)–C (9)	1.54	C (33)–C (35)	1.54
C (8)–C (14)	1.53	C (36)–C (37)	1.56

5) As to the numbering of the carbon atoms of the molecule (1), the conventional numbering system for tetracyclic triterpenes is followed in this work. *cf.* G. Ourisson, P. Crabbé and O.R. Rodig, "Tetracyclic Triterpenes," Hermann, Paris, 1964, p. 19.

Table III. Bond Angles (Degrees)

C(11)–O(2)–C(36)	119	C(12)–C(13)–C(17)	129
C(23)–O(3)–C(33)	107	C(14)–C(13)–C(17)	114
C(24)–O(4)–C(33)	110	C(8)–C(14)–C(13)	114
C(2)–C(1)–C(10)	111	C(8)–C(14)–C(15)	115
C(1)–C(2)–C(3)	119	C(8)–C(14)–C(18)	114
O(1)–C(3)–C(2)	120	C(13)–C(14)–C(15)	100
O(1)–C(3)–C(4)	124	C(13)–C(14)–C(18)	108
C(2)–C(3)–C(4)	116	C(15)–C(14)–C(18)	105
C(3)–C(4)–C(5)	110	C(14)–C(15)–C(16)	110
C(3)–C(4)–C(30)	109	C(15)–C(16)–C(17)	103
C(3)–C(4)–C(31)	106	C(13)–C(17)–C(16)	112
C(5)–C(4)–C(30)	111	C(13)–C(17)–C(20)	127
C(5)–C(4)–C(31)	113	C(16)–C(17)–C(20)	120
C(30)–C(4)–C(31)	107	C(17)–C(20)–C(21)	116
C(4)–C(5)–C(6)	116	C(17)–C(20)–C(22)	112
C(4)–C(5)–C(10)	119	C(21)–C(20)–C(22)	110
C(6)–C(5)–C(10)	116	C(20)–C(22)–C(23)	112
C(5)–C(6)–C(7)	107	O(3)–C(23)–C(22)	102
C(6)–C(7)–C(8)	119	O(3)–C(23)–C(24)	104
C(7)–C(8)–C(9)	111	C(22)–C(23)–C(24)	118
C(7)–C(8)–C(14)	107	O(4)–C(24)–C(23)	103
C(7)–C(8)–C(32)	117	O(4)–C(24)–C(25)	114
C(9)–C(8)–C(14)	108	C(23)–C(24)–C(25)	125
C(9)–C(8)–C(32)	106	O(5)–C(25)–C(24)	117
C(14)–C(8)–C(32)	107	O(5)–C(25)–C(26)	102
C(8)–C(9)–C(10)	117	O(5)–C(25)–C(27)	110
C(8)–C(9)–C(11)	110	C(24)–C(25)–C(26)	116
C(10)–C(9)–C(11)	118	C(24)–C(25)–C(27)	96
C(1)–C(10)–C(5)	114	C(26)–C(25)–C(27)	116
C(1)–C(10)–C(9)	112	O(3)–C(33)–O(4)	108
C(1)–C(10)–C(19)	111	O(3)–C(33)–C(34)	108
C(5)–C(10)–C(9)	111	O(3)–C(33)–C(35)	100
C(5)–C(10)–C(19)	100	O(4)–C(33)–C(34)	103
C(9)–C(10)–C(19)	107	O(4)–C(33)–C(35)	113
O(2)–C(11)–C(9)	110	C(34)–C(33)–C(35)	124
O(2)–C(11)–C(12)	100	O(2)–C(36)–O(6)	124
C(9)–C(11)–C(12)	124	O(2)C–(36)–C(37)	118
C(11)–C(12)–C(13)	105	O(6)–C(36)–C(37)	118
C(12)–C(13)–C(14)	116	Br–C(37)–C(36)	110

Stereo-model of the molecule is given in Fig. 1, and the bond lengths and angles are shown in Tables II and III, respectively. The average estimated standard deviations of carbon-oxygen, carbon-bromine and carbon-carbon bond lengths are 0.03, 0.02 and 0.03 Å, respectively. The mean estimated standard deviation of the bond angles is 1.7°. The final electron density synthesis was calculated with phase angles on the basis of above results, and is shown in Fig. 2 by means of superimposed contour sections projected on (001). The observed and calculated structure factors are shown in Table IV.

The determination of the absolute configuration of the alisol A acetonide monomoacetate was carried out using the anomalous dispersion of the bromine atom. Upon calculating the structure factors of the crystal, taking the anomalous dispersion into account, intensities of certain pairs of reflexions (hkl) and $(\bar{h}\bar{k}\bar{l})$ differed to some extent. Of these, 15 pairs are listed in Table V. All data were consistent with the calculated differences based on the configuration as shown in Fig. 1, thus establishing the absolute configuration as drawn in the figure.

It was hardly possible to distinguish crystallographically beyond all doubt the correct assignment of the three atoms, C_{26}, C_{27} and O_5 at the end of the side chain. However, the chemical evidence that the periodate oxidation of alisol A gave acetone and the tetranor-

TABLE IVa. Observed and Calculated Structure Factors

L	H	K	FO	FC
	0	2	113.3	141.3
	0	4	160.1	166.1
	0	6	24.1	17.0
	0	8	112.9	104.2
	0	10	87.2	90.1
	0	12	52.9	57.9
	0	14	67.5	79.5
	0	16	66.8	68.0
	0	18	20.1	23.7
	0	20	12.0	6.6
	0	22	10.0	5.2
	0	24	19.9	18.5
	0	26	14.3	10.1
	1	2	5.3	7.3
	1	3	8.5	11.5
	1	4	9.5	11.8
	1	5	146.8	150.3
	1	6	194.3	200.2
	1	7	42.1	43.7
	1	8	140.0	142.8
	1	9	78.6	77.0
	1	10	62.0	65.5
	1	11	56.7	49.8
	1	12	43.3	39.0
	1	13	21.0	13.1
	1	14	11.6	13.0
	1	15	22.4	18.9
	1	16	28.2	24.4
	1	17	5.2	11.5
	1	18	16.0	17.1
	1	19	29.2	26.4
	1	20	16.8	20.8
	1	21	34.4	35.7
	1	22	26.1	24.2
	1	26	12.3	9.1
	2	0	39.6	42.0
	2	1	10.3	10.4
	2	2	65.2	58.8
	2	3	54.3	51.6
	2	4	64.1	67.4
	2	5	117.7	111.4
	2	6	80.8	70.0
	2	7	6.0	7.8
	2	8	61.5	53.3
	2	9	65.4	65.3
	2	10	16.4	23.9
	2	11	79.2	66.3
	2	12	60.9	54.5
	2	13	77.6	75.2
	2	14	22.4	30.3
	2	15	24.7	25.8
	2	18	7.4	5.6
	2	23	4.8	0.9
	2	24	11.0	2.0
	2	25	8.7	1.0
	3	1	171.9	178.1
	3	2	71.2	71.9
	3	3	82.1	83.3
	3	4	5.0	1.4
	3	5	17.1	19.0
	3	6	48.1	49.1
	3	7	38.6	39.2
	3	8	37.4	31.1
	3	9	17.1	14.5
	3	10	11.2	8.6
	3	11	81.2	65.1
	3	12	14.9	8.0
	3	14	17.3	12.7
	3	16	2.7	3.3
	3	17	21.7	20.3
	3	20	14.1	16.6
	3	21	2.7	1.3
	3	22	22.9	19.2
	3	23	9.3	4.1
	4	0	121.0	125.5
	4	1	69.3	63.1
	4	2	4.0	2.3
	4	3	87.0	89.2
	4	4	40.7	40.1
	4	5	31.8	31.6
	4	6	41.3	33.8
	4	7	8.6	7.1
	4	8	8.0	12.0
	4	9	26.1	12.5
	4	10	42.2	38.5
	4	11	9.0	7.5
	4	12	45.7	40.5
	4	13	19.0	21.0
	4	16	46.5	39.2
	4	17	15.3	18.9
	4	18	21.0	18.6
	4	19	17.2	11.8
	4	21	2.4	0.3
	4	22	12.6	7.7
	4	23	8.0	5.4
	4	24	9.2	6.0
	5	1	3.9	4.8
	5	2	61.0	48.4
	5	3	7.1	10.8
	5	4	4.9	15.3
	5	5	147.9	145.7
	5	6	25.0	19.8
	5	7	92.4	90.9
	5	8	73.1	69.8
	5	9	66.6	72.9
	5	10	6.2	0.4
	5	11	91.0	90.8
	5	12	20.6	19.6
	5	13	19.8	18.4
	5	14	12.7	19.8
	5	15	23.8	20.4
	5	16	12.1	9.7
0	5	17	14.8	18.1
	5	18	8.6	3.8
	5	19	17.6	20.4
	5	21	12.4	15.3
	5	22	4.7	1.4
	5	25	9.7	8.9
	6	0	81.0	77.5
	6	1	88.6	87.6
	6	2	107.8	106.9
	6	3	82.7	93.4
	6	4	52.1	49.4
	6	5	28.8	24.7
	6	6	45.4	36.7
	6	7	24.9	15.4
	6	9	33.9	38.5
	6	10	98.8	92.4
	6	11	49.8	41.4
	6	12	5.7	2.9
	6	13	45.5	45.7
	6	14	63.9	64.4
	6	15	32.5	29.1
	6	16	19.8	25.5
	6	17	29.9	26.3
	6	20	9.1	7.8
	6	21	3.9	0.8
	6	23	22.8	20.8
	6	24	12.1	4.7
	7	1	11.7	7.3
	7	2	52.5	55.7
	7	3	14.2	15.6
	7	4	97.7	95.4
	7	5	8.8	1.7
	7	6	79.7	77.3
	7	7	27.5	19.5
	7	8	74.0	65.0
	7	9	69.8	64.0
	7	10	44.9	44.9
	7	11	8.8	2.4
	7	12	43.6	38.1
	7	14	14.6	13.5
	7	17	18.0	16.5
	7	18	29.4	27.7
	7	19	5.8	5.8
	7	20	21.0	15.4
	7	21	9.2	5.2
	7	22	16.1	11.6
	7	23	11.1	4.8
	7	24	7.7	8.3
	8	0	58.6	49.7
	8	1	11.8	13.6
	8	2	80.3	68.0
	8	3	46.2	44.4
	8	4	65.7	56.1
	8	5	11.7	18.4
	8	6	11.8	14.7
	8	7	12.3	10.4
	8	8	2.5	4.0
	8	9	26.0	25.7
	8	10	12.9	10.8
	8	12	50.6	44.4
	8	14	57.1	53.3
	8	15	8.5	9.6
	8	16	24.0	20.8
	8	17	11.3	11.1
	8	19	10.6	6.9
	8	22	10.4	6.8
	8	23	6.7	0.4
	9	1	5.7	0.3
	9	2	20.8	24.1
	9	3	34.4	32.4
	9	4	38.0	32.6
	9	6	24.6	25.5
	9	7	20.5	15.7
	9	8	50.1	48.7
	9	9	15.2	13.7
	9	10	6.6	3.2
	9	12	14.6	8.5
	9	13	11.3	6.0
	9	16	9.9	1.0
	9	18	19.1	13.4
	9	20	4.0	5.8
	9	21	10.2	12.2
	9	22	22.8	14.9
	9	23	5.7	0.1
	10	0	8.2	11.4
	10	2	11.8	8.2
	10	3	9.3	1.8
	10	5	13.0	8.0
	10	6	4.1	8.1
	10	7	2.1	2.7
	10	9	14.1	13.6
	10	10	25.3	23.7
	10	12	18.5	17.9
	10	14	14.5	12.2
	10	15	9.9	9.9
	10	16	22.0	15.8
	10	18	17.6	14.4
	10	19	8.6	3.6
	10	20	7.8	6.4
	10	22	9.6	4.5
	11	1	19.8	21.1
	11	2	22.3	22.2
	11	3	26.2	26.0
	11	4	56.7	54.3
	11	5	14.1	9.2
	11	6	58.2	56.8
	11	7	28.6	27.8
	11	8	28.1	22.0
	11	9	22.8	21.9
	11	10	12.1	8.2
	11	13	8.3	11.8
	11	14	15.3	12.4
0	11	15	16.1	14.3
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	11	17	18.2	16.7
	11	18	22.1	17.1
	11	20	11.7	7.0
	12	0	9.0	7.7
	12	1	50.7	58.9
	12	2	13.4	17.2
	12	3	32.1	32.3
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	12	5	26.2	27.4
	12	6	10.4	6.7
	12	7	16.6	17.0
	12	8	23.0	24.0
	12	9	9.3	7.5
	12	10	9.4	8.4
	12	11	7.3	7.8
	12	12	4.5	1.6
	12	13	21.5	20.0
	12	14	25.2	19.1
	12	15	17.6	18.8
	12	16	7.3	4.9
	12	17	26.6	19.2
	12	20	12.2	4.4
	13	1	18.8	13.6
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	13	4	14.1	23.5
	13	5	40.3	40.2
	13	7	35.1	25.2
	13	9	31.6	26.8
	13	10	3.6	0.0
	13	11	22.3	21.5
	13	12	7.4	2.4
	13	16	4.2	2.3
	13	17	11.1	3.5
	13	18	6.6	0.3
	13	19	13.5	10.8
	14	1	17.9	21.0
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	14	3	22.3	24.6
	14	4	21.3	19.9
	14	7	16.0	19.0
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	14	9	22.6	24.5
	14	10	18.2	19.0
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	14	12	11.4	14.7
	14	13	5.6	7.4
	14	14	14.8	11.7
	14	17	14.2	5.3
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	15	2	10.6	10.2
	15	3	4.1	2.6
	15	5	13.6	13.1
	15	7	8.0	3.4
	15	9	5.2	7.8
	15	13	11.5	7.4
	15	14	8.9	1.2
	15	15	4.9	3.9
	16	0	13.8	10.0
	16	2	18.5	21.4
	16	6	9.6	6.3
	16	10	3.8	7.9
	16	12	15.3	8.8
	17	1	7.5	1.8
	17	2	9.9	8.4
	17	4	21.3	22.5
	17	6	10.9	9.0
	17	7	16.4	13.0
	17	8	21.6	18.9
	17	9	5.8	4.7
	17	10	10.3	11.7
L	17	11	9.5	4.3
	17	12	9.2	5.2
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	0	5	46.9	41.3
	0	6	9.1	13.5
	0	7	33.6	25.4
	0	8	42.0	49.3
	0	9	6.5	7.5
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	0	12	15.3	11.4
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	0	16	31.4	33.7
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	0	18	29.7	32.3
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	0	20	28.2	26.2
	0	21	11.8	6.0
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	0	24	14.0	13.9
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	1	4	74.7	68.9
	1	5	124.7	127.6
	1	6	128.9	132.1
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	1	8	72.5	71.2
	1	9	16.7	16.1
	1	10	73.4	67.8
	1	11	14.7	12.9
	1	12	58.8	59.2
	1	13	8.1	13.2
	1	14	22.9	25.9
	1	15	24.9	26.9
	1	16	22.8	21.7
1	1	17	25.1	28.0
	1	18	23.3	24.2
	1	19	10.7	7.2
	1	20	5.5	3.9
	2	0	104.9	101.3
	2	1	126.8	124.6
	2	2	194.3	205.7
	2	3	131.1	135.0
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	2	5	78.1	71.0
	2	6	67.5	63.0
	2	7	36.6	36.9
	2	8	37.1	43.4
	2	9	85.1	82.3
	2	10	20.1	21.9
	2	11	14.0	23.3
	2	12	42.8	39.9
	2	14	9.4	13.2
	2	16	20.0	22.2
	2	17	17.2	14.6
	2	18	14.6	15.8
	2	19	19.5	15.1
	2	20	4.6	8.5
	2	21	7.0	8.4
	2	22	5.9	10.2
	2	23	12.0	4.6
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	3	6	32.2	32.6
	3	7	18.8	14.8
	3	8	25.9	21.3
	3	9	10.7	13.5
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	3	11	66.1	59.2
	3	12	16.5	14.4
	3	13	40.3	46.5
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	3	17	13.8	17.4
	3	18	14.4	11.2
	3	19	6.4	3.3
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	4	5	108.7	112.6
	4	6	30.0	29.7
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	4	8	51.2	45.4
	4	9	61.2	58.5
	4	10	32.9	30.3
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	4	13	9.7	8.7
	4	14	24.8	24.2
	4	15	3.1	6.0
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	4	19	35.4	32.5
	4	21	15.2	14.0
	4	22	6.0	8.2
	4	23	14.9	14.6
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	5	2	54.7	61.1
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	5	7	27.4	25.1
	5	8	82.0	71.4
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	5	11	65.6	59.0
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	5	13	29.2	28.8
	5	14	24.8	18.5
	5	15	34.8	30.3
	5	17	14.5	11.4
	5	18	17.2	14.0
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	5	20	17.2	11.5
	5	21	7.7	4.3
	5	22	9.0	10.3
	6	0	27.8	29.5
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	6	16	16.1	17.9
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	7	0	7.4	6.4
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	7	4	37.7	32.0
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	7	6	28.8	26.2
	7	7	62.0	57.1
	7	8	44.6	34.7
	7	9	44.7	40.4
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	7	12	47.6	39.0
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	7	14	20.6	22.4
	7	15	28.0	23.7
	7	16	18.4	17.4
	7	18	17.6	14.8
	7	19	17.3	18.4
	7	20	18.2	12.2
	7	21	9.2	12.0
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	8	0	53.8	55.2
	8	1	31.5	34.5
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	8	7	20.9	17.7
	8	8	34.0	34.0
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	8	16	15.5	15.4
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	9	3	42.5	43.9
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	9	10	16.6	13.0
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	10	19	4.9	2.9
	11	0	27.6	31.1
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	11	2	27.9	28.9
	11	3	13.7	14.1
	11	4	9.6	12.0
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	11	6	18.3	20.4
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	11	8	9.8	10.6
	11	9	16.8	11.0
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	11	12	5.1	2.0
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	11	14	17.9	12.6
	11	15	15.2	16.6
	11	16	16.0	9.1
	11	17	14.8	13.4
	11	18	9.2	9.4
	11	19	10.6	7.8
	12	0	22.5	20.7
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	13	4	17.5	19.6
	13	5	19.2	26.2
	13	6	15.2	12.2
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	14	12	9.4	5.4
	15	0	9.4	11.3
	15	5	10.7	14.8
	15	6	10.9	15.1
	15	7	11.7	11.0
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	15	11	5.8	7.5
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	16	4	9.4	11.7
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	16	6	8.8	14.8
L	16	7	8.0	12.3
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	8	7	64.4	65.0
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	8	9	45.0	41.3
	8	10	20.8	14.4
	8	11	36.4	30.1
	8	12	7.7	7.6
	8	13	6.7	5.8
	8	14	1.7	1.9
	8	16	20.4	15.6
	8	17	30.6	24.0
	8	19	13.9	16.7
	9	0	19.8	31.6
	9	1	30.2	31.1
	9	2	14.2	14.2
	9	3	34.8	38.1
	9	4	33.1	36.0
	9	5	24.4	21.0
	9	6	10.9	9.0
	9	8	37.4	36.9
	9	9	16.2	22.9
	9	10	20.1	18.3
	9	11	28.6	23.8
	9	12	12.9	13.5
	9	13	39.4	34.3
	9	14	15.7	15.4
	9	15	31.2	27.1
	9	16	14.9	10.9
	9	17	15.8	14.4
	9	18	13.2	10.6
	9	19	11.9	11.7
	10	0	41.0	45.4
	10	1	9.9	14.9
2	10	2	34.7	42.7
	10	3	11.6	16.8
	10	4	30.2	29.0
	10	5	24.7	23.0
	10	6	30.6	30.5
	10	7	35.5	34.7
	10	8	11.7	14.8
	10	9	7.5	14.3
	10	10	19.1	18.5
	10	11	14.0	7.8
	10	12	26.7	25.9
	10	14	11.7	14.3
	10	15	22.4	22.1
	10	16	6.7	5.7
	10	17	19.3	15.2
	11	0	22.6	27.2
	11	1	26.0	30.0
	11	3	8.8	4.2
	11	4	20.7	26.0
	11	5	18.4	18.6
	11	6	10.2	12.7
	11	7	11.8	9.7
	11	8	10.0	13.0
	11	9	18.7	18.8
	11	10	13.1	12.9
	11	11	20.8	17.3
	11	13	18.8	18.0
	11	14	4.3	2.8
	11	15	6.8	10.4
	11	16	12.1	12.2
	11	17	12.4	14.0
	12	0	6.7	0.9
	12	1	13.5	20.4
	12	2	20.8	25.0
	12	3	24.3	26.2
	12	4	22.9	25.2
	12	5	21.3	23.6
	12	6	22.1	22.0
	12	7	13.0	17.5
	12	8	20.2	18.2
	12	10	19.6	17.2
	12	11	11.0	7.8
	12	12	30.7	26.2
	12	13	14.8	12.5
	12	15	8.7	4.8
	13	0	28.5	29.8
	13	2	25.5	26.3
	13	4	33.6	38.8
	13	5	7.5	9.8
	13	6	13.4	16.4
	13	8	7.7	10.9
	13	9	18.2	21.6
	13	10	11.5	14.0
	13	11	19.4	19.5
	13	14	20.2	16.2
	14	3	10.7	13.3
	14	6	12.8	16.0
	14	7	16.7	16.2
	14	8	18.2	17.2
	14	10	9.9	11.8
	15	0	9.2	10.7
	15	1	11.3	11.7
	15	2	15.0	11.2
	15	8	9.4	8.1
	15	9	2.7	3.5
	15	10	11.9	8.5
	16	3	13.1	10.5
L	16	5	15.1	18.9
	16	6	10.1	11.1
3	0	2	13.9	9.5
	0	3	55.3	60.6
	0	4	23.3	21.2
	0	5	8.7	9.0
	0	6	64.3	59.0
	0	7	16.6	2.1
	0	8	65.3	53.9
	0	9	20.3	17.7
	0	10	10.2	7.8
	0	11	60.2	59.8
	0	12	7.4	2.5
	0	13	49.1	54.3
	0	14	13.0	15.7
	0	15	53.3	58.8
	0	16	6.3	3.8
	0	17	31.8	34.1
	0	19	13.2	16.0
	0	22	6.2	8.9
	1	0	50.0	50.5
	1	1	25.6	33.1
	1	2	49.9	50.5
	1	3	30.0	27.2
	1	4	36.9	35.4
	1	5	37.7	32.3
	1	6	72.0	77.3
	1	7	69.7	75.2
	1	8	43.3	41.5
	1	9	33.7	32.5
	1	10	21.7	18.3
	1	11	25.2	31.3
	1	12	19.2	26.3
	1	13	12.8	16.1
	1	14	25.1	26.6
	1	15	11.1	10.2
	1	16	17.5	20.0
	1	17	18.1	22.4
	1	18	20.6	14.7
	1	19	24.8	29.7
	1	20	20.5	16.8
	2	0	18.6	21.6
	2	1	20.7	25.9
	2	2	62.3	68.2
	2	3	36.3	39.2

Table IVb. Observed and Calculated Structure Factors

L	H	K	FO	FC
3	2	4	33.3	36.6
	2	5	44.0	39.1
	2	6	67.6	69.4
	2	7	26.5	26.0
	2	8	47.8	44.5
	2	9	24.7	33.9
	2	10	33.8	32.9
	2	11	27.2	22.1
	2	12	9.5	11.6
	2	13	38.5	35.2
	2	14	10.3	11.5
	2	15	19.9	14.6
	2	16	7.2	6.1
	2	18	16.0	13.2
	2	20	7.9	6.8
	3	0	13.5	2.2
	3	1	27.4	19.3
	3	2	45.3	42.6
	3	3	52.7	52.7
	3	4	12.2	17.6
	3	5	58.4	65.2
	3	6	11.4	16.3
	3	7	33.8	29.5
	3	8	47.7	45.3
	3	9	31.1	31.4
	3	10	15.9	18.4
	3	12	11.7	9.3
	3	13	14.8	17.4
	3	14	24.1	23.0
	3	17	9.1	4.1
	3	19	11.4	14.1
	3	20	8.4	14.0
	4	0	112.2	113.4
	4	1	46.3	44.1
	4	2	18.7	17.1
	4	3	38.1	31.3
	4	5	25.3	25.0
	4	6	40.2	36.7
	4	7	7.0	6.4
	4	8	47.2	43.1
	4	9	16.1	9.9
	4	10	20.7	18.9
	4	11	13.2	19.9
	4	12	34.0	34.0
	4	13	17.0	9.4
	4	14	34.6	38.8
	4	15	9.9	4.8
	4	16	36.6	34.8
	4	17	19.5	14.2
	4	18	15.8	15.7
	5	1	33.6	33.9
	5	2	36.3	34.2
	5	3	33.5	37.8
	5	4	53.4	50.8
	5	5	36.1	36.2
	5	6	85.3	90.8
	5	7	23.4	27.7
	5	8	51.0	51.8
	5	9	15.5	20.7
	5	10	46.7	50.2
	5	11	7.2	11.1
	5	12	22.3	20.8
	5	13	7.3	3.4
	5	14	13.7	9.6
	5	15	7.6	6.3
	5	16	41.3	40.6
	5	18	11.1	12.9
	6	0	39.1	40.4
	6	1	62.0	61.5
	6	2	40.0	36.2
	6	3	24.7	25.7
	6	4	64.1	63.2
	6	5	46.9	53.0
	6	6	5.7	11.5
	6	8	32.0	30.8
	6	9	27.7	28.1
	6	10	42.0	36.9
	6	11	24.1	21.2
	6	12	43.1	42.1
	6	13	17.7	20.5
	6	15	20.2	21.2
	6	16	14.2	15.0
	6	17	19.0	22.5
	6	18	8.0	11.0
	7	1	47.3	41.1
	7	2	33.7	35.9
	7	3	34.6	37.5
	7	4	33.6	30.1
	7	5	35.3	30.0
	7	6	23.8	21.4
	7	7	49.6	53.6
	7	9	18.5	18.1
	7	10	15.2	18.9
	7	11	15.2	16.5
	7	12	15.6	15.7
	7	13	17.1	16.3
	7	14	16.2	15.3
	7	15	10.5	9.4
	7	16	13.6	10.2
	7	17	24.5	22.3
	7	18	7.6	8.8
	8	0	4.4	4.5
	8	1	35.3	40.9
	8	2	10.4	8.3
	8	3	28.7	26.8
	8	4	17.7	20.7
	8	5	25.8	24.3
	8	6	19.1	13.2
	8	7	20.9	21.2
	8	8	21.3	17.4
	8	9	14.3	16.6
	8	10	27.3	25.5

L	H	K	FO	FC
3	8	11	24.8	22.4
	8	12	8.3	11.8
	8	13	30.2	24.6
	8	14	16.9	10.1
	8	15	27.8	25.4
	8	16	14.6	13.8
	8	17	11.8	8.9
	8	18	8.0	4.0
	9	0	39.5	41.3
	9	1	10.4	7.2
	9	2	8.6	6.8
	9	3	15.3	11.8
	9	4	28.6	29.6
	9	6	6.3	1.6
	9	8	14.3	19.1
	9	9	18.3	26.3
	9	11	21.3	23.0
	9	13	16.3	18.7
	9	15	6.8	8.5
	9	16	10.8	12.1
	10	0	4.4	6.5
	10	2	9.5	16.9
	10	3	27.1	27.5
	10	4	10.0	16.6
	10	5	11.8	17.4
	10	6	5.3	11.3
	10	7	11.8	14.1
	10	9	10.6	9.3
	10	10	6.9	10.1
	10	11	17.0	23.1
	10	12	7.8	12.1
	10	13	8.6	7.4
	10	14	21.8	19.7
	10	15	13.6	16.2
	10	16	8.4	3.7
	11	0	11.1	17.4
	11	1	2.9	5.7
	11	2	12.5	8.8
	11	3	24.4	24.1
	11	4	24.5	29.7
	11	6	25.2	23.2
	11	7	24.2	25.4
	11	8	19.2	16.8
	11	9	20.1	24.8
	11	10	8.2	7.4
	11	11	2.0	0.9
	11	13	11.3	12.7
	12	0	23.7	22.8
	12	1	6.8	9.4
	12	2	40.1	46.7
	12	3	7.2	9.3
	12	4	14.1	17.6
	12	5	11.2	12.2
	12	6	14.4	15.8
	12	8	8.0	10.4
	12	9	18.9	16.7
	12	10	17.3	14.0
	12	11	12.0	9.7
	12	12	27.3	25.7
	12	13	12.6	12.5
	13	2	11.2	8.3
	13	3	7.8	9.4
	13	4	15.0	13.6
	13	6	16.7	14.9
	13	7	16.6	18.5
	13	8	27.5	29.3
	14	0	6.6	2.5
	14	2	11.3	12.9
	14	3	18.5	18.9
	14	4	8.7	11.2
	14	6	8.2	13.4
L	15	0	9.1	4.5
	15	1	7.7	7.8
4	0	2	46.7	62.6
	0	4	58.8	54.6
	0	5	83.9	80.6
	0	6	13.5	7.9
	0	7	45.4	41.5
	0	8	9.6	11.1
	0	9	35.2	44.1
	0	10	16.8	26.0
	0	11	17.3	27.9
	0	12	11.3	14.1
	0	13	15.7	5.4
	0	14	19.1	18.7
	0	15	30.6	27.1
	0	16	8.2	1.5
	0	17	7.3	3.8
	0	18	11.4	13.2
	0	19	6.5	4.4
	0	22	4.0	0.9
	1	0	19.1	32.9
	1	1	15.6	23.4
	1	2	34.1	46.5
	1	3	31.8	40.5
	1	4	44.6	48.3
	1	5	21.3	25.8
	1	6	22.8	23.1
	1	7	15.8	12.6
	1	8	26.3	29.9
	1	9	42.2	34.6
	1	10	24.4	22.5
	1	11	28.8	25.2
	1	12	13.5	11.8
	1	13	17.6	16.6
	1	14	18.9	13.4
	1	16	17.8	20.3
	1	17	25.1	28.9
	1	21	9.9	5.7
	2	0	13.1	15.9
	2	1	25.0	26.6
	2	2	4.6	5.6

L	H	K	FO	FC
4	2	3	28.0	29.3
	2	4	12.9	15.7
	2	5	29.4	31.8
	2	6	8.7	10.1
	2	7	7.2	10.1
	2	8	11.3	10.8
	2	9	28.8	29.8
	2	12	20.7	18.9
	2	14	19.5	17.0
	2	15	28.0	27.2
	2	16	7.3	8.3
	2	17	22.0	16.9
	2	18	11.8	-6.1
	2	19	11.3	6.3
	2	21	3.0	1.4
	3	0	11.8	5.2
	3	1	8.7	9.9
	3	2	39.0	43.6
	3	3	14.1	14.5
	3	4	18.6	25.5
	3	5	30.9	36.6
	3	6	15.2	15.2
	3	7	22.4	32.7
	3	8	21.4	16.3
	3	9	18.3	21.8
	3	10	8.2	3.5
	3	11	9.2	9.2
	3	12	18.3	22.1
	3	13	6.9	6.6
	3	14	12.2	10.5
	3	15	14.2	14.5
	3	16	12.8	9.7
	3	17	20.9	21.5
	3	18	14.7	17.9
	3	20	7.4	5.0
	4	0	10.1	8.6
	4	1	45.5	53.6
	4	2	23.3	23.6
	4	3	47.7	48.8
	4	4	37.1	35.2
	4	5	15.7	13.1
	4	6	28.8	22.2
	4	8	43.4	42.5
	4	10	8.2	14.0
	4	11	17.5	26.9
	4	12	14.9	10.4
	4	13	10.0	12.8
	4	14	8.4	12.1
	4	16	7.1	10.5
	4	17	13.6	13.1
	4	18	5.2	8.7
	4	19	21.5	18.2
	5	0	39.3	45.4
	5	1	8.1	11.3
	5	3	27.5	27.5
	5	4	21.3	16.8
	5	5	46.0	50.7
	5	6	15.7	8.9
	5	7	54.9	60.3
	5	8	7.2	9.0
	5	9	33.6	32.3
	5	10	15.9	15.3
	5	11	25.8	26.2
	5	12	12.5	12.8
	5	13	13.6	10.2
	5	14	8.7	20.6
	5	15	8.3	8.7
	5	16	12.6	15.6
	5	17	11.2	14.0
	5	18	8.2	9.5
	6	0	27.8	34.6
	6	1	45.3	52.5
	6	2	23.2	25.7
	6	3	12.9	9.9
	6	4	24.6	22.7
	6	5	29.0	36.5
	6	6	9.9	10.3
	6	7	14.2	14.3
	6	8	29.0	33.9
	6	9	21.6	24.2
	6	10	18.9	24.5
	6	11	18.9	17.9
	6	12	16.5	21.4
	6	14	17.0	15.0
	6	15	24.8	21.4
	6	16	12.9	12.1
	6	17	19.0	13.1
	7	1	12.4	19.8
	7	2	18.4	23.1
	7	3	27.4	35.4
	7	4	19.8	22.2
	7	5	6.3	10.2
	7	6	34.3	37.5
	7	7	8.7	9.4
	7	8	9.7	11.9
	7	9	21.8	18.3
	7	10	28.3	23.3
	7	11	19.2	18.8
	7	13	14.7	10.5
	7	14	28.4	28.9
	7	17	6.5	5.0
	7	18	6.5	5.9
	8	1	16.6	21.9
	8	2	17.8	17.3
	8	3	10.4	7.1
	8	4	13.7	11.9
	8	5	18.5	21.7
	8	6	18.6	20.0
	8	7	10.6	7.2
	8	8	23.8	27.5
	8	9	19.3	20.8
	8	10	7.6	10.0

L	H	K	FO	FC
4	8	12	6.1	13.0
	8	13	7.1	15.4
	8	14	12.2	9.4
	8	15	12.9	17.1
	8	16	10.0	4.9
	9	2	15.8	19.2
	9	3	20.3	24.5
	9	6	11.2	11.6
	9	8	15.3	18.9
	9	9	13.3	7.8
	9	10	10.3	11.1
	9	11	4.0	3.6
	10	0	20.5	26.9
	10	1	8.7	10.7
	10	2	18.6	23.1
	10	5	12.2	9.0
	10	6	7.9	11.1
	10	8	12.9	15.4
	10	10	9.9	14.8
	10	12	13.2	12.1
	10	13	7.9	8.9
	10	14	12.8	9.2
	10	15	10.1	8.5
	11	1	13.9	17.8
	11	3	14.7	19.9
	11	4	14.9	16.3
	11	5	10.5	10.3
	11	6	18.6	22.4
	11	7	22.4	28.0
	11	9	21.7	25.8
	11	10	19.2	18.0
	11	11	17.0	13.5
	11	12	9.0	4.1
	12	1	21.0	23.8
	12	2	7.6	9.5
	12	3	19.8	20.9
	12	4	17.4	18.1
	12	5	21.7	24.1
	12	6	21.0	21.8
	12	7	15.4	14.2
	12	8	7.4	10.6
	12	9	10.6	14.1
	12	10	3.7	7.6
	12	12	6.8	10.7
	13	0	15.9	19.4
	13	3	13.4	20.3
	13	4	18.7	25.3
	13	5	6.6	10.9
	13	6	6.4	9.8
	13	7	7.5	10.5
	13	8	8.4	3.8
	14	0	4.9	1.0
	14	2	4.1	9.0
	14	3	4.5	10.7
	14	5	4.5	7.8
	14	6	3.9	5.9
L	14	7	4.4	10.3
	15	1	7.6	5.1
5	0	2	5.5	1.3
	0	3	14.9	17.8
	0	4	37.9	55.5
	0	5	8.4	10.3
	0	6	26.4	25.9
	0	7	16.4	13.0
	0	8	27.3	32.0
	0	9	2.7	1.5
	0	10	36.8	25.7
	0	11	12.7	15.1
	0	12	14.8	13.4
	0	14	4.8	4.5
	0	16	15.1	13.1
	0	17	7.2	3.6
	0	18	16.6	13.1
	0	19	10.7	8.9
	1	1	23.2	34.2
	1	2	24.7	35.8
	1	3	15.6	15.5
	1	5	15.8	18.1
	1	6	4.1	7.5
	1	7	7.0	6.9
	1	8	16.1	11.8
	1	9	14.3	8.4
	1	10	24.9	23.6
	1	11	9.1	10.5
	1	13	6.1	7.3
	1	14	13.9	15.7
	1	15	3.9	4.6
	1	16	17.6	14.1
	1	17	13.6	7.0
	2	0	10.7	8.0
	2	2	23.1	24.8
	2	3	31.8	29.8
	2	4	11.7	14.2
	2	5	30.9	31.6
	2	6	22.2	21.4
	2	7	28.5	29.9
	2	8	15.7	17.8
	2	9	32.1	30.4
	2	10	19.8	14.5
	2	12	5.8	10.1
	2	13	3.7	5.4
	2	14	6.3	5.8
	2	15	8.2	9.3
	2	17	15.8	15.5
	2	19	10.2	6.7
	3	0	30.2	29.0
	3	1	13.7	17.5
	3	2	22.6	27.5
	3	3	18.2	26.7
	3	4	18.5	19.5
	3	5	12.1	14.0
	3	6	2.8	3.5

L	H	K	FO	FC
5	3	8	13.8	10.6
	3	9	18.6	16.7
	3	10	12.9	15.5
	3	11	23.0	21.8
	3	12	14.1	10.2
	3	13	20.3	21.4
	3	14	5.6	5.4
	3	15	17.3	16.0
	3	16	7.5	6.7
	3	17	7.5	4.7
	3	18	3.9	3.8
	3	19	7.5	2.7
	3	20	2.9	4.3
	4	0	11.3	17.1
	4	1	26.0	29.6
	4	2	8.8	17.7
	4	3	28.9	27.6
	4	4	9.6	5.0
	4	5	19.8	21.5
	4	6	9.3	6.5
	4	7	15.4	20.4
	4	8	14.2	9.7
	4	9	12.2	9.6
	4	10	13.2	13.7
	4	11	14.7	15.8
	4	12	17.7	17.8
	4	16	9.2	6.1
	4	17	10.1	9.2
	4	18	8.5	10.0
	5	1	22.8	28.6
	5	2	22.8	25.3
	5	3	23.8	25.5
	5	4	7.5	9.7
	5	9	15.6	21.7
	5	10	5.7	4.5
	5	11	14.3	14.8
	5	12	5.9	4.9
	5	13	14.7	13.5
	5	14	3.0	1.0
	5	15	11.9	11.4
	6	1	11.0	12.0
	6	2	8.4	12.5
	6	3	19.6	18.8
	6	4	5.7	9.2
	6	5	18.3	25.8
	6	6	15.7	22.6
	6	7	22.6	23.9
	6	8	23.7	23.2
	6	9	13.3	18.9
	6	10	15.4	19.2
	6	12	9.4	7.4
	6	14	14.6	14.4
	6	15	4.4	4.5
	6	17	9.5	5.0
	6	18	5.0	5.5
	6	19	12.3	4.3
	7	0	21.5	19.7
	7	1	8.6	10.6
	7	2	19.6	25.0
	7	3	11.6	10.5
	7	4	13.3	14.9
	7	5	21.9	20.2
	7	8	7.2	5.0
	7	9	6.9	7.6
	7	10	16.9	20.1
	7	11	8.0	3.2
	7	12	11.8	15.2
	7	13	9.9	11.6
	7	14	25.0	24.3
	7	15	4.2	7.1
	7	16	16.7	14.7
	7	17	10.0	5.2
	8	0	12.2	15.8
	8	2	24.1	28.1
	8	3	8.0	11.7
	8	4	31.5	40.2
	8	5	17.6	16.3
	8	6	26.1	28.8
	8	8	23.9	23.8
	8	9	12.7	12.7
	8	10	12.3	13.2
	8	11	11.7	8.6
	8	12	11.8	6.8
	8	13	7.2	9.5
	8	14	8.1	4.0
	8	16	7.2	5.1
	9	0	19.5	23.2
	9	2	12.1	19.3
	9	3	10.1	14.9
	9	4	13.4	17.0
	9	5	14.2	15.5
	9	7	10.4	9.1
	9	8	7.9	7.1
	9	10	9.5	9.8
	9	11	4.5	6.6
	9	12	10.7	12.6
	9	13	8.7	4.7
	9	14	11.5	9.0
	9	15	9.2	2.4
	10	2	5.5	5.5
	10	3	6.6	8.7
	10	4	11.7	15.2
	10	5	9.4	11.8
	10	6	12.7	17.0
	10	8	13.7	11.8
	10	13	11.8	4.9
	10	15	6.7	2.9
	11	1	8.6	11.7
	11	2	12.4	16.2
	11	6	8.2	12.9
	11	8	12.5	5.8
	11	10	13.3	7.8

L	H	K	FO	FC
5	11	11	10.0	5.6
	11	12	7.6	7.5
	12	2	10.0	13.4
	12	4	9.4	12.7
	12	5	13.2	12.5
	12	6	10.1	13.5
	12	7	8.2	5.5
	12	8	10.0	6.1
	12	9	11.6	6.7
	13	1	16.8	14.4
	13	2	5.9	5.0
	13	3	7.9	11.9
	14	1	2.8	3.9
	14	2	12.1	8.9
	14	3	7.0	4.3
	14	6	9.0	6.7
	14	7	8.6	9.4
L	15	0	13.2	8.0
	15	2	6.9	7.6
6	0	1	2.4	4.3
	0	2	5.5	12.9
	0	3	3.6	0.0
	0	5	24.3	25.5
	0	6	21.4	20.9
	0	7	24.9	26.0
	0	8	11.9	11.5
	0	9	9.8	8.7
	0	10	9.1	4.7
	0	11	8.9	6.0
	0	12	7.9	7.4
	0	13	8.0	1.0
	0	14	4.5	9.8
	0	15	3.6	5.0
	0	16	13.3	16.7
	0	17	6.9	2.7
	1	0	3.5	5.9
	1	1	20.6	26.3
	1	5	22.9	27.9
	1	6	17.3	25.6
	1	10	5.7	9.4
	1	11	18.2	24.6
	1	14	12.6	6.8
	1	15	19.8	11.8
	1	16	7.1	4.3
	1	17	5.4	3.5
	2	0	27.6	32.9
	2	1	20.5	25.6
	2	2	10.9	16.1
	2	3	10.8	17.2
	2	8	3.5	4.3
	2	10	5.7	2.3
	2	11	5.9	6.3
	2	12	12.0	11.8
	2	13	14.4	12.9
	2	14	7.4	4.8
	2	15	10.5	8.8
	3	0	6.3	6.3
	3	1	15.4	16.8
	3	2	5.4	6.4
	3	3	16.2	15.3
	3	4	14.4	10.3
	3	7	20.2	22.5
	3	8	8.4	11.3
	3	9	14.8	17.4
	3	11	6.3	2.2
	3	16	6.6	0.2
	4	0	29.1	36.1
	4	1	27.7	38.8
	4	2	17.9	15.8
	4	3	20.6	26.3
	4	4	2.9	2.0
	4	5	4.1	6.9
	4	6	4.2	6.3
	4	7	10.1	6.8
	4	8	11.0	7.0
	4	9	7.6	8.7
	4	12	10.2	9.5
	4	13	7.3	8.5
	4	15	8.1	8.5
	4	16	15.8	11.8
	5	1	7.4	6.8
	5	2	7.8	7.6
	5	3	3.9	3.3
	5	5	18.1	23.8
	5	9	11.6	16.0
	5	10	12.0	14.0
	5	11	15.4	19.4
	5	13	5.8	1.5
	5	14	4.8	6.5
	6	0	6.8	3.8
	6	1	2.8	5.4
	6	2	15.9	16.0
	6	3	13.8	12.1
	6	4	4.6	8.0
	6	7	5.2	8.0
	6	8	10.9	8.5
	6	9	8.0	11.0
	6	10	20.5	19.3
	6	11	14.9	13.0
	6	12	12.4	9.3
	6	14	8.7	6.3
	6	15	14.6	12.2
	7	1	15.1	20.2
	7	2	10.0	16.4
	7	3	19.8	23.5
	7	4	18.6	22.0
	7	6	7.8	12.3
	7	8	10.4	11.3
	7	10	8.8	6.9
	7	11	9.2	14.7
	7	12	4.5	3.7
	7	13	6.9	7.0

L	H	K	FO	FC
6	7	15	7.6	4.5
	8	0	11.6	17.6
	8	1	6.6	8.9
	8	2	11.4	16.5
	8	7	12.8	12.9
	8	8	3.2	3.5
	8	9	12.2	6.3
	8	11	4.1	7.3
	8	12	11.5	5.7
	8	13	4.2	0.8
	9	0	9.0	9.1
	9	3	8.9	7.6
	9	4	3.6	6.5
	9	7	6.5	6.5
	9	10	9.8	8.0
	9	12	5.5	3.7
	10	0	16.5	18.2
	10	1	3.5	5.6
	10	6	4.2	7.6
	10	9	5.9	3.7
	10	10	9.2	6.7
	10	11	7.9	10.0
	11	2	6.4	8.4
	11	4	9.2	10.6
	11	5	8.0	7.1
	11	6	4.4	6.6
	12	1	10.0	9.8
	12	3	9.0	5.4
	12	5	11.7	7.2
	12	6	10.6	7.7
	13	1	6.1	7.0
L	13	2	9.3	7.1
	0	2	2.2	0.8
7	0	3	26.0	31.9
	0	4	7.3	10.4
	0	8	13.1	11.8
	0	9	9.4	12.0
	0	10	9.1	10.8
	0	11	14.8	16.1
	0	13	6.7	12.4
	1	6	15.5	14.3
	1	7	7.8	8.7
	1	8	13.9	9.7
	1	9	15.0	13.5
	1	11	12.4	13.2
	1	12	8.4	3.8
	2	0	8.1	12.1
	2	2	11.8	15.9
	2	3	5.7	4.5
	2	4	11.4	11.3
	2	6	3.0	1.8
	2	9	6.8	9.1
	2	10	3.9	7.0
	2	12	5.1	5.5
	2	13	7.2	1.1
	3	0	4.0	2.0
	3	1	10.9	14.3
	3	9	5.5	1.1
	3	11	8.8	7.7
	3	12	4.4	1.8
	4	1	1.1	0.3
	4	2	4.5	4.9
	4	4	4.7	4.7
	4	6	5.1	1.0
	4	8	7.2	4.2
	4	10	5.3	2.0
	4	12	4.2	3.4
	5	0	19.1	22.7
	5	1	8.0	6.3
	5	2	11.6	12.9
	5	4	9.9	12.7
	5	6	11.3	14.7
	5	8	21.8	25.7
	5	9	10.0	4.9
	5	10	11.4	5.3
	5	11	5.0	6.2
	6	0	26.8	27.2
	6	2	14.4	20.4
	6	3	8.9	9.5
	6	4	6.5	8.8
	6	6	8.6	7.0
	6	7	2.5	2.7
	6	8	4.4	3.5
	6	10	11.7	12.0
	7	7	10.6	8.7
	7	8	8.5	5.9
	7	9	11.4	8.0
	8	0	3.4	3.9
	8	2	7.2	3.2
	8	6	5.9	3.3
	8	7	3.2	4.8
	9	0	5.6	2.1
	9	1	3.9	2.2
	9	2	8.2	3.6
	9	4	4.3	2.1
	9	5	5.3	5.0

Table V. Comparison of Observed and Calculated Intensity Differences used to Establish the Absolute Configuration

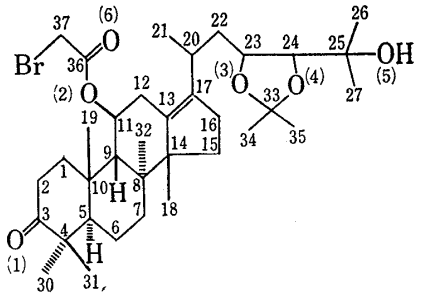
h	k	l	$Io(hkl)/Io(\bar{h}\bar{k}\bar{l})$	$\lvert Fc(hkl)\rvert/\lvert Fc(\bar{h}\bar{k}\bar{l})\rvert$
16	2	1	>1	1.15
3	8	1	<1	0.83
5	17	1	<1	0.88
9	5	1	>1	1.12
11	3	1	<1	0.90
6	2	1	>1	1.18
2	17	1	<1	0.92
3	12	1	<1	0.88
1	11	1	<1	0.70
2	10	1	<1	0.89
6	2	2	>1	1.35
10	3	2	>1	1.16
2	12	2	>1	1.11
2	11	2	>1	1.23
3	16	2	<1	0.86

aldehyde clearly indicated that one of the three atoms is oxygen and the others are carbon.[1b] Thus we tentatively assigned the atom having the shortest bond length to C_{25} as oxygen.

Discussion

On the basis of above results the molecular structure of alisol A (23, 24)-acetonide 11-monobromoacetate is written as **1**.[5] The absolute configuration of the side chain is 20*R*, 23*S*, 24*R*; the relative configuration at C_{23} and C_{24} is *threo*. The B ring of the molecule is clearly shown to exist in a boat conformation; the B/C ring juncture is a type of 8α-methyl-9β-proton, a ring junction of fusidane and protostane triterpene,[6] thus leading the 11β-hydroxyl group to an equatorial configuration. In addition to this, the A ring of the molecule also exists in a boat conformation, which is presumably due to diaxial interaction between the two methyl groups at C_4 (β) and C_{10}. Thus alisol A has been shown to be the first triterpene of protostane series.[6b]

Previously, some of the authors proposed that the least-squares method applied to temperature factors only would be a short cut in the structural elucidation of complex organic molecules.[4] In the present work, thus we picked out 43 maxima from the superposition map mainly by considering their height, and then tested whether the peaks are true or not by initially assigning the same temperature factors to them. By the calculation of the first electron density distribution with phase angles based on the survived 24 atoms, we succeeded in elucidating the structure without any difficulty. It took only ten days to clarify the structure since we undertook the X-ray analysis. Thus the usefulness of this technique was demonstrated again in the work.



1

Acknowledgement The authors thank Dr. S. Tatsuoka and Dr. Y. Abe, of the Division for their encouragement throughout this work.

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