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Studies on Pillaromycin A. V.[1] The X-Ray Analysis of Pillaronone Monobromoacetate

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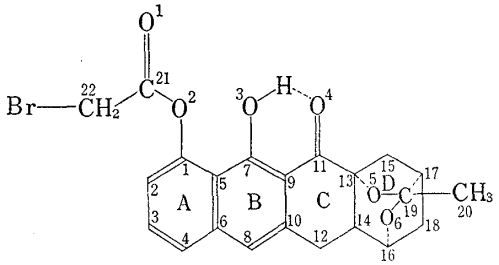
In order to confirm the chemical and stereochemical structure of pillaronone, a derivative of the aglycone obtained from pillaromycin A, an X-ray analysis of its monobromoacetate has been carried out. Pillaronone monobromoacetate crystallizes in the orthorhombic system, space group $P2_12_12_1$-D_2^4, with four molecules of $C_{22}H_{19}O_6Br$ in a unit cell. Application of a three-dimensional Patterson superposition method facilitated the elucidation of the structure which is given in formula (I). The final R-value was 0.133. The absolute configuration was determined using the anomalous dispersion of the bromine atom. The molecular structure in the crystal was also discussed.

Pillaromycin A,[3] $C_{28}H_{28}O_{11}$, is an anti-tumor antibiotic compound produced by *Streptomyces flavovirens* No. 65786. The present X-ray work[4] has been carried out in order to establish the chemical and stereochemical structure of pillaronone, $C_{20}H_{18}O_5$, which is prepared by catalytic reduction of pillaromycinone. A preliminary study indicated that the monobromoacetate of pillaronone was the most promising for this purpose.

Experimental

Preparation of the Crystal——To a solution of 100 mg of pillaronone in 2 ml of benzene was added dropwise 0.2 ml of monobromoacetic acid anhydride. After being stirred for 6 hr at 70° to complete the reaction, the mixture was poured into ice water and extracted with benzene. The benzene solution was washed with water and dried over anhydrous sodium sulfate. The solution was concentrated under a reduced pressure, petroleum ether was added and crystals separated were recrystallized several times from a mixture of ethyl acetate, ethyl ether and petroleum ether to afford yellow prisms, mp 194°. *Anal.* Calcd. for $C_{22}H_{19}O_6Br$: C, 57.53; H, 4.17; Br, 17.40. Found: C, 57.56; H, 4.14; Br, 17.37. IR ν_{max}^{KBr} cm^{-1}: 1772 (s), 1630 (vs). UV λ_{max}^{EtOH} mμ ($\varepsilon \times 10^{-3}$): 270 (37), 290 (6.8), 300 (6.8), 395 (6.5).



I

Measurements of X-ray Diffraction——Oscillation and Weissenberg photographs were taken about the two principal crystal axes, using CuKα radiation. The specimens used had the following cross-sections at right angles to the axis of rotation; 0.2×0.3 mm for the c axis rotation, 0.2×0.2 mm for the a axis. Intensities of reflections were visually estimated from multiple film integrating Weissenberg photographs (8 layers about the c axis and 12 layers about the a axis, respectively) and obtained 2000 independent data (excluding zero intensity reflections). The linear absorption coefficient for CuKα radiation was estimated to be $\mu = 21.84$ cm^{-1}. Thus the absorption and extinction errors were neglected. The absolute structure factors were deduced from these intensity data by the ordinary procedure, using a NEAC 2206 electronic computer.

1) Part IV: M. Asai, E. Mizuta, K. Mizuno, A. Miyake and S. Tatsuoka, *Chem. Pharm. Bull.* (Tokyo), **18**, 1720 (1970).
2) Location: *a*) *Juso, Higashiyodogawa-ku, Osaka*; *b*) *Uegahara, Nishinomiya.*
3) M. Asai, *Chem. Pharm. Bull.* (Tokyo), **18**, 1699 (1970).
4) Preliminary report, H. Matsuda, M. Nishikawa, K. Kamiya, M. Asai, Y. Tomiie, I. Nitta, S. Yamamura, Y. Hirata; presented at 9th Symposium on the Chemistry of Natural Products, Osaka, Oct., 1965.

Result and Discussion

Unit Cell and Space Group

It has been found from X-ray examination that the crystal belongs to the orthorhombic system with the unit cell parameters, $a=12.46$, $b=19.54$, $c=7.85$ Å.

The space group was uniquely determined to be $P2_12_12_1$-D_2^4. Judging from the cell size and the space group, it is clear that the unit cell contains four molecules as the calculated density becomes a reasonable value, 1.596 g cm^{-3} (D_0=1.591 g cm^{-3}).

Determination of the Structure

As the first step of the structure analysis, a three-dimensional Patterson function $P(uvw)$ was calculated. The coordinates of the bromine atom were determined without ambiguity from the three Harker sections, one of which at $v=1/2$ is shown in Fig. 1. With phase angles calculated from these coordinates, a three-dimensional Fourier synthesis was calculated. In co-operation with this analysis, a minimum function diagram was obtained with four superposition of bromine vectors. In spite of quite different principles, the results obtained by these two methods were fully consistent with each other and the positions of all light atoms except hydrogen were determined undoubtedly.

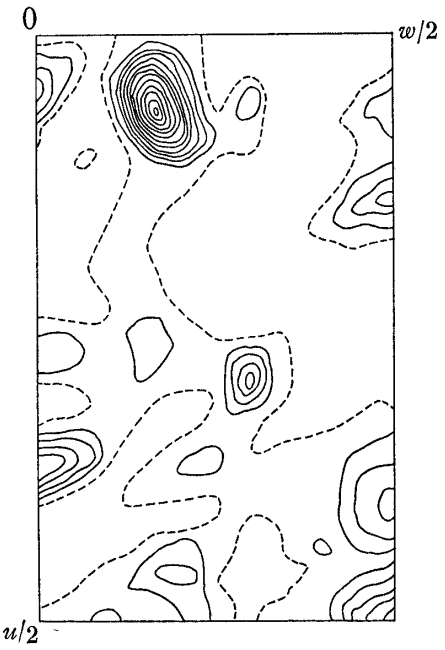


Fig. 1. Harker Section at $v=1/2$

The refinement of the atomic positions was carried out using the least squares method, and in accordance, R-value fell to 0.133. Maps of the final electron density distribution are shown in Fig. 2a and 2b. The final x, y and z atomic parameters and temperature factors are listed in Table I. The observed and the calculated structure factors are listed in Table II.

Absolute Configuration

Of the 20 pairs of reflections for which the intensity differences between $I(hkl)$ and $I(\bar{h}\bar{k}\bar{l})$ were expected to be discernible, 14 pairs showed significant differences in the lth layer Weissenberg photographs. The results are shown in Table III. The comparison between observed and calculated intensities indicated that the assumed configuration was actually correct. The absolute configuration was then established as (I). In this paper, all figures are drawn in such a way that they represent the correct configuration.

Molecular and Crystal Structure

The bond lengths and angles, calculated from the atomic parameters of Table I, are shown in Fig. 3 and Table IV. The mean values of bond lengths are 1.40 Å for aromatic carbon bond, 1.53 Å for C–C single bond, 1.42 Å for C–O single bond and 1.24 Å for C–O double bond. These values seem to be quite reasonable.

The rings A and B form a naphthalene skeleton and C(11), C(12), C(13), O(2), O(3) and O(4) atoms are nearly on this plane. The OH group attached to the ring B seems to form a strong six-membered ring hydrogen bond with the carbonyl group at C(11), the bond between O(3) and C(7) being somewhat contracted.

The juncture between the ring C, which is puckered at C(14), and the ring D, which is of a chair form, is in a *trans*-fusion. The atoms, O(5), O(6) and C(19) are axially bonded to

TABLE I. The Final Atomic Coordinates and Temperature Factors

Atom	a/x	b/y	c/z	b
Br	0.0266	0.8425	0.1649	4.65
O(1)	0.4733	0.5466	0.1030	4.82
O(2)	0.3614	0.0445	0.2810	3.31
O(3)	0.3253	0.9376	0.4792	3.31
O(4)	0.2314	0.1672	0.1442	4.29
O(5)	0.2292	0.3042	0.1965	3.21
O(6)	0.1911	0.4144	0.1084	3.57
C(1)	0.3982	0.9960	0.1536	3.01
C(2)	0.4367	0.0200	0.0084	4.00
C(3)	0.0372	0.0239	0.3782	4.32
C(4)	0.0541	0.0945	0.3987	3.93
C(5)	0.3808	0.9248	0.1877	2.78
C(6)	0.4038	0.8792	0.0531	3.03
C(7)	0.3439	0.8963	0.3464	3.10
C(8)	0.3837	0.8078	0.0728	3.79
C(9)	0.3234	0.8246	0.3578	2.69
C(10)	0.3463	0.7806	0.2125	3.33
C(11)	0.2179	0.2038	0.0177	3.73
C(12)	0.3176	0.7044	0.2200	3.63
C(13)	0.2447	0.2795	0.0254	2.85
C(14)	0.3275	0.6785	0.4036	3.09
C(15)	0.1352	0.7066	0.4950	3.37
C(16)	0.2950	0.6002	0.4262	3.19
C(17)	0.3636	0.3691	0.0524	3.28
C(18)	0.1780	0.5888	0.4020	3.53
C(19)	0.2720	0.3742	0.1797	3.38
C(20)	0.2969	0.3998	0.3591	4.52
C(21)	0.4323	0.0654	0.3921	3.32
C(22)	0.3769	0.1141	0.5059	4.91

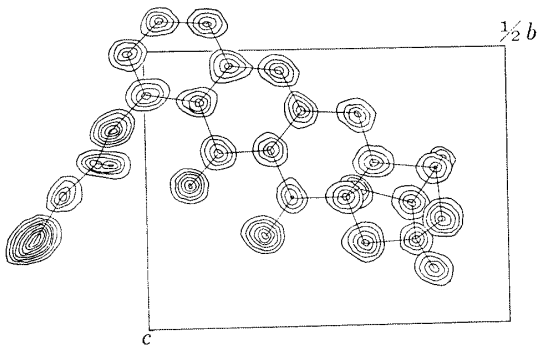


Fig. 2a. The Final Three-dimensional Electron Density Distribution shown by Means of Superimposed Contour Section Projected on (100)

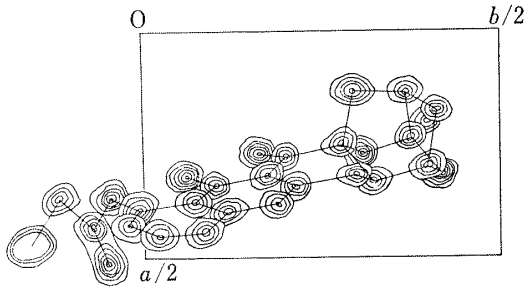


Fig. 2b. Projected on (001)

the ring D and form a ketal, leading to a cage structure of this part of the molecule. Thus considerable steric strains seem to exist at C(15) and C(18) so that the bond angles C(13)–C(15)–C(17) and C(16)–C(18)–C(17) are smaller than 100°.

TABLE II. Observed and Calculated Structure Factors

K L	FO	FC	H K L	FO	FC	H K L	FQ	FC	H K L	FO	FC	H K L	FQ	FC	H K L	FO	FC	H K L	FO	FC	H K L	FO	FC	H K L	FO	FC
H 2 0	26.9	13.4	0 3 6	13.1	11.0	1 20 2	7.4	5.5	1 12 8	3.1	1.9	2 14 4	23.9	20.6	3 9 1	40.0	34.5	3 12 6	11.1	10.1	4 1 3	40.3	35.4	5 4 0	56.9	58.4
4 0	132.6	145.8	4 6	24.8	20.1	21 2	9.7	11.6	14 8	6.9	7.2	15 4	11.4	12.6	10 1	39.1	34.8	13 6	15.1	13.6	2 3	45.2	42.5	5 0	25.1	18.4
0 6 0	52.3	57.0	5 6	19.5	14.4	22 2	6.6	5.8	15 8	3.7	4.8	16 4	8.4	9.7	11 1	23.6	19.8	14 6	20.6	18.6	3 3	19.9	19.2	6 0	12.6	6.3
8 0	88.8	82.1	6 6	48.6	47.3	23 2	15.5	12.8	1 9	6.1	5.2	17 4	14.7	10.3	12 1	11.1	18.5	18 6	10.0	9.2	4 3	17.9	16.8	7 0	76.7	74.9
10 0	34.2	9.0	8 6	6.5	5.7	24 2	10.9	10.5	3 9	10.9	14.7	19 4	16.9	13.6	13 1	26.0	24.4	0 7	27.1	31.7	5 3	51.6	54.8	8 0	14.7	20.0
12 0	19.1	23.7	9 6	10.5	7.1	0 3	19.9	14.2	7 9	9.5	13.7	20 4	7.9	9.0	14 1	7.7	4.8	1 7	23.6	25.2	6 3	26.6	26.7	9 0	46.9	38.1
14 0	7.5	8.5	10 6	24.9	19.4	1 3	36.3	40.5	8 9	2.8	5.7	21 4	5.9	5.6	15 1	23.9	22.1	2 7	12.2	12.4	7 3	19.2	14.2	10 0	12.9	18.1
16 0	34.0	35.6	11 6	4.5	6.9	2 3	9.5	9.1	9 9	4.5	7.0	22 4	9.4	9.3	16 1	24.6	26.1	3 7	6.5	5.3	8 3	26.6	18.0	11 0	31.0	23.4
18 0	20.2	20.2	12 6	17.9	16.2	3 3	85.7	83.8	1 0	4.6	7.7	0 5	1.7	3.4	17 1	5.3	6.1	4 7	25.2	25.2	9 3	29.5	25.2	12 0	13.0	10.3
20 0	11.5	12.1	14 6	14.3	13.6	4 3	46.4	33.3	2 0	3.1	5.0	1 5	21.9	19.4	18 1	19.0	18.0	5 7	8.4	8.6	10 3	40.3	39.0	13 0	40.5	43.9
22 0	12.4	13.0	16 6	11.5	11.6	5 3	25.5	19.7	3 0	3.6	7.0	2 5	33.2	29.9	19 1	4.7	7.8	6 7	4.1	3.3	11 3	25.2	23.8	14 0	19.6	17.2
24 0	7.5	7.1	17 6	3.4	1.7	6 3	46.7	35.9	0 0	55.6	38.4	3 5	29.5	28.5	20 1	13.3	11.2	7 7	11.7	16.0	12 3	15.3	12.7	15 0	16.6	15.9
1 1	54.7	41.7	18 6	12.0	12.6	7 3	73.8	70.5	1 0	57.2	59.0	4 5	41.2	41.5	21 1	6.1	7.1	8 7	8.7	4.6	13 3	20.0	21.8	16 0	14.1	10.3
2 1	100.7	136.4	20 6	2.8	3.4	8 3	15.7	13.7	2 0	87.7	86.6	5 5	40.2	36.1	22 1	6.6	8.5	9 7	8.4	10.0	14 3	31.2	29.6	17 0	17.9	22.6
3 1	39.2	42.3	2 7	39.8	53.4	9 3	70.5	69.2	3 0	18.9	16.0	6 5	6.1	8.4	23 1	6.6	6.6	10 7	18.2	18.6	15 3	18.3	15.1	18 0	14.1	14.6
4 1	94.8	114.5	3 7	7.7	9.6	10 3	21.6	14.7	4 0	6.5	7.3	7 5	5.9	9.1	24 1	5.1	7.0	11 7	6.1	7.6	16 3	9.8	10.8	20 0	2.5	1.8
5 1	39.7	32.8	4 7	6.3	6.6	11 3	15.7	14.0	5 0	34.5	35.9	8 5	48.7	45.8	0 2	23.4	21.5	12 7	8.7	12.0	17 3	26.3	29.4	21 0	8.3	8.4
6 1	78.7	78.7	5 7	19.7	24.8	12 3	24.0	19.2	6 0	102.8	101.5	9 5	27.4	19.7	1 2	77.6	76.0	13 7	8.0	9.2	18 3	7.4	6.0	22 0	2.0	2.1
7 1	18.1	15.4	6 7	3.3	2.1	13 3	34.1	29.9	8 0	43.7	36.8	10 5	24.2	22.6	2 2	22.1	18.7	14 7	11.5	10.0	19 3	3.2	1.6	23 0	3.5	6.9
8 1	7.3	12.3	7 7	7.8	8.2	14 3	11.5	7.7	9 0	88.0	75.7	11 5	16.9	16.4	3 2	36.1	33.1	15 7	5.0	4.7	21 3	11.1	10.3	0 1	66.1	75.2
9 1	60.8	59.4	8 7	17.1	16.7	15 3	42.2	40.2	10 0	108.8	107.9	12 5	30.5	31.6	4 2	40.6	38.4	16 7	6.4	5.7	22 3	11.0	13.8	1 1	45.8	40.0
10 1	18.3	22.2	9 7	10.0	11.6	16 3	31.1	27.7	11 0	10.8	15.8	13 5	17.3	13.7	5 2	52.7	47.9	17 7	3.1	5.4	0 4	52.8	55.1	2 1	15.7	15.2
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16 1	13.2	10.0	13 7	10.3	10.7	21 3	4.1	4.4	16 0	26.7	29.0	18 5	6.8	8.6	9 2	28.7	25.2	3 8	15.3	16.2	4 4	28.1	26.1	6 1	43.6	43.7
17 1	16.0	15.6	14 7	6.5	4.9	23 3	7.5	9.3	17 0	14.9	13.0	20 5	13.3	13.3	10 2	20.0	21.3	4 8	6.1	6.3	5 4	46.9	43.8	7 1	41.8	35.0
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23 1	5.0	6.4	3 8	13.6	8.0	2 4	32.7	33.2	20 0	7.3	10.0	1 6	25.8	27.6	13 2	15.8	17.0	7 8	13.0	11.0	8 4	32.7	27.8	10 1	31.6	28.5
24 1	6.2	10.1	4 8	13.5	14.4	3 4	36.0	30.8	22 0	16.4	16.6	2 6	22.7	24.2	14 2	47.6	42.9	8 8	6.9	6.1	9 4	35.3	35.8	11 1	43.2	42.3
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1 2	21.0	7.0	6 8	6.3	6.6	5 4	74.6	74.9	24 0	2.0	1.8	4 6	24.8	24.3	16 2	13.9	14.0	10 8	12.1	4.3	11 4	34.0	30.7	13 1	10.3	12.1
2 2	32.3	32.9	7 8	15.1	14.3	6 4	18.2	9.8	0 1	13.1	8.5	5 6	22.1	21.0	17 2	16.0	17.1	11 8	8.4	11.0	12 4	35.5	29.7	14 1	27.2	24.6
3 2	106.6	129.5	8 8	6.0	5.7	7 4	37.3	35.2	1 1	133.0	184.0	6 6	31.6	31.8	18 2	8.7	8.8	12 8	4.4	7.8	13 4	20.5	14.4	15 1	7.1	9.2
4 2	7.7	9.2	9 8	12.5	14.1	8 4	30.9	28.1	2 1	34.1	40.0	7 6	5.6	9.1	19 2	13.2	12.6	13 8	3.8	5.0	14 4	13.4	12.5	16 1	14.3	13.7
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6 2	67.3	65.2	11 8	6.3	1.1	10 4	32.1	24.4	4 1	105.9	110.6	10 6	25.3	20.5	21 2	7.9	11.2	1 9	8.8	9.2	16 4	12.5	12.2	18 1	4.0	4.8
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9 2	41.5	38.7	4 9	3.6	6.4	13 4	5.8	10.6	7 1	70.2	69.7	13 6	5.0	8.1	1 3	61.9	63.4	4 9	4.7	6.1	19 4	15.7	14.9	21 1	6.2	8.2
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11 2	32.7	26.2	6 9	3.4	4.5	15 4	15.1	10.7	9 1	28.1	21.1	15 6	4.2	4.1	3 3	58.8	65.2	7 9	7.3	6.0	21 4	2.2	3.0	23 1	4.4	4.4
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13 2	53.4	57.1	11 9	7.5	10.2	17 4	16.2	16.9	11 1	29.3	30.8	17 6	3.2	4.8	5 3	17.2	15.3	9 9	5.0	6.1	0 5	22.6	24.7	1 2	49.3	41.0
15 2	33.0	27.4	H 1 0	43.0	49.6	19 4	6.2	4.8	12 1	46.2	44.9	0 7	21.7	19.2	6 3	44.2	31.3	4 0 0	12.3	37.0	1 5	21.2	19.4	2 2	48.6	42.1
16 2	21.6	19.6	2 0	58.6	65.0	20 4	7.6	8.1	13 1	10.4	6.3	1 7	20.7	18.8	7 3	23.2	20.7	1 0	87.6	24.0	2 5	37.3	38.1	3 2	20.2	18.4
19 2	22.9	23.8	1 3 0	54.2	44.1	21 4	11.4	11.6	14 1	46.8	45.2	2 7	23.6	24.4	8 3	50.8	50.0	2 0	51.3	62.5	3 5	40.3	39.4	4 2	47.8	44.2
20 2	10.3	9.7	4 0	50.0	43.8	22 4	3.6	4.2	15 1	17.3	17.2	3 7	10.1	10.4	9 3	38.0	38.3	3 0	28.3	17.2	4 5	31.7	32.3	5 2	60.5	50.9
21 2	5.5	5.5	5 0	48.1	39.2	0 5	55.0	62.5	16 1	7.9	9.2	4 7	17.2	20.7	10 3	29.1	23.3	4 0	36.8	37.3	5 5	34.2	28.6	6 2	69.8	66.7
23 2	4.6	6.5	6 0	11.9	2.6	1 5	16.0	17.9	17 1	18.2	20.1	5 7	15.6	14.0	11 3	12.6	10.1	5 0	49.4	48.2	6 5	25.1	27.6	7 2	42.5	38.0
24 2	5.1	5.0	7 0	48.6	38.1	2 5	23.9	21.6	18 1	28.3	26.3	6 7	9.0	7.1	12 3	23.4	21.5	6 0	87.1	81.8	7 5	19.5	19.1	8 2	27.7	21.5
1 3	110.9	84.6	8 0	129.9	131.3	3 5	39.9	41.3	19 1	4.4	5.7	7 7	17.9	18.0	13 3	42.6	42.0	7 0	74.8	67.4	8 5	20.0	19.0	9 2	38.7	36.5
2 3	6.8	7.4	9 0	77.7	68.1	4 5	42.8	42.3	20 1	16.6	17.1	8 7	20.2	20.6	14 3	51.3	48.3	8 0	6.6	12.6	9 5	14.8	10.9	10 2	39.5	39.3
3 3	32.8	20.8	10 0	49.6	37.0	6 5	47.0	41.4	21 1	5.6	6.1	9 7	5.5	8.3	15 3	13.3	5.8	9 0	46.1	28.6	11 5	21.5	18.5	11 2	22.6	25.1
4 3	17.8	11.5	11 0	14.1	15.2	7 5	22.4	18.1	23 1	7.3	6.8	10 7	8.1	10.6	16 3	18.7	12.6	10 0	49.8	49.2	12 5	11.2	13.6	12 2	24.1	22.6
5 3	33.0	18.3	12 0	49.4	48.8	8 5	13.1	9.7	24 1	8.3	10.7	11 7	15.9	14.5	17 3	16.6	12.9	11 0	49.7	48.9	13 5	3.5	8.4	13 2	24.2	24.1
6 3	24.5	19.8	13 0	19.9	15.9	9 5	15.6	13.7	0 2	25.6	28.9	12 7	10.1	8.0	18 3	8.3	7.0	12 0	34.5	36.2	15 5	16.6	13.1	14 2	17.1	13.9
7 3	60.8	65.3	14 0	35.3	36.5	10 5	40.9	40.2	1 2	21.0	15.7	13 7	7.8	5.3	19 3	14.4	17.0	13 0	15.2	9.1	16 5	14.9	13.1	15 2	12.0	10.6
8 3	2.8	2.1	15 0	9.8	9.0	12 5	13.4	12.4	2 2	42.1	38.9	14 7	13.3	13.4	20 3	12.9	10.8	14 0	3.5	2.2	17 5	6.1	7.6	16 2	23.8	19.0
9 3	37.3	36.4	16 0	15.1	17.3	13 5	7.5	1.9	3 2	103.0	107.3	15 7	7.3	7.3	21 3	5.3	6.6	15 0	16.9	20.5	18 5	5.6	4.9	17 2	3.5	6.3
10 3	14.0	13.4	17 0	27.5	22.8	14 5	8.9	8.8	4 2	36.2	28.4	17 7	8.1	8.5	23 3	7.5	8.8	16 0	39.2	40.0	19 5	4.3	3.9	18 2	18.2	16.0
11 3	48.0	46.3	18 0	29.8	30.1	15 5	11.1	10.6	5 2	40.2	28.7	0 8	5.1	5.1	0 4	7.0	2.5	17 0	11.8	12.4	0 6	31.2	30.9	19 2	13.1	15.5
13 3	21.7	16.6	19 0	15.3	12.5	16 5	19.0	17.6	6 2	18.3	19.7	1 8	18.5	18.7	1 4	57.7	63.0	19 0	13.4	9.7	1 6	25.2	22.8	20 2	8.3	11.5
14 3	22.2	14.5	20 0	12.9	15.1	17 5	12.0	7.1	7 2	63.4	62.8	2 8	13.5	13.4	2 4	37.6	36.4	20 0	7.6	9.9	2 6	30.4	27.8	21 2	8.5	8.3
15 3	49.9	51.3	21 0	12.1	10.2	19 5	9.1	8.4	9 2	30.7	31.3	3 8	12.4	14.0	3 4	19.3	16.7	21 0	15.8	15.0	3 6	26.4	25.0	22 2	5.7	5.5
17 3	22.5	23.6	22 0	4.9	6.2	21 5	3.1	2.6	10 2	37.4	33.0	4 8	7.1	8.1	4 4	23.8	19.9	22 0	11.3	11.3	4 6	23.8	21.9	23 2	3.0	6.0
21 3	18.5	20.5	23 0	7.6	7.1	0 6	23.3	26.6	11 2	35.0	29.6	6 8	8.4	8.3	5 4	30.9	30.1	0 1	95.6	97.4	5 6	20.5	15.9	0 3	15.8	13.7
23 3	4.6	6.0	24 0	9.6	11.0	1 6	22.5	27.3	12 2	32.9	28.1	7 8	10.6	10.6	6 4	20.5	19.1	1 1	53.0	46.9	6 6	32.8	33.4	1 3	30.0	28.3
0 4	98.1	69.2	0 1	6.6	5.6	2 6	33.3	40.0	13 2	32.0	35.7	8 8	9.2	8.2	7 4	40.0	41.0	2 1	62.4	64.0	7 6	23.8	19.4	2 3	62.4	61.4
1 4	47.6	32.4	1 1	38.0	33.7	3 6	13.4	11.0	14 2	37.2	34.1	9 8	11.7	11.5	8 4	36.9	33.3	3 1	50.4	43.2	8 6	12.6	9.9	3 3	52.6	54.2
2 4	23.9	21.8	2 1	98.4	105.8	4 6	32.6	35.5	15 2	18.8	15.7	10 8	8.4	7.4	9 4	26.2	20.2	4 1	50.4	49.5	9 6	12.7	12.7	4 3	55.0	51.8
3 4	11.1	18.4	3 1	82.0	82.8	5 6	5.7	8.9	16 2	21.8	20.4	11 8	3.9	7.2	10 4	21.0	19.2	5 1	14.6	18.3	10 6	23.5	20.7	5 3	22.0	17.4
4 4	68.1	67.7	4 1	73.7	77.2	6 6	27.0	24.6	17 2	12.8	12.7	12 8	7.3	8.3	11 4	38.3	35.1	6 1	35.4	26.8	11 6	19.8	13.5	6 3	29.4	27.7
6 4	36.3	32.0	5 1	10.4	11.9	7 6	9.7	6.0	18 2	21.0	23.1	13 8	9.3	11.2	12 4	35.6	31.4	7 1	30.3	29.0	12 6	5.6	6.0	7 3	52.6	50.5
7 4	35.0	37.0	6 1	89.8	88.2	8 6	33.5	32.5	19 2	8.9	12.4	1 9	10.1	10.7	13 4	12.9	14.7	8 1	52.5	52.8	13 6	5.5	5.8	8 3	37.1	32.7
8 4	12.4	5.5	7 1	8.5	9.7	9 6	11.3	10.1	20 2	5.1	4.9	2 9	4.1	7.9	14 4	24.5	20.6	9 1	44.5	44.0	14 6	6.1	6.6	9 3	24.9	24.6
9 4	45.8	48.8	8 1	56.7	46.6	10 6	25.8	19.9	23 2	4.3	6.1	3 9	4.0	1.4	15 4	14.0	12.7	10 1	40.6	38.2	15 6	4.9	6.4	10 3	17.1	10.5
10 4	21.0	16.6	9 1	70.0	66.1	11 6	19.1	18.3	0 3	42.1	40.2	4 9	5.6	5.7	16 4	25.4	24.5	11 1	33.6	32.4	16 6	11.1	8.8	11 3	5.9	5.7
11 4	22.1	17.1	10 1	49.2	49.1	12 6	19.3	14.4	1 3	83.6	87.3	5 9	15.8	16.8	17 4	19.1	16.8	12 1	21.6	19.1	17 6	6.7	7.6	12 3	17.9	10.1
12 4	63.1	64.5	11 1	37.5	36.8	13 6	10.0	10.4	2 3	34.6	31.0	6 9	3.7	2.8	18 4	13.7	14.3	13 1	23.1	23.1	1 7	15.2	18.6	13 3	35.3	32.2
13 4	67.9	68.9	12 1	52.9	52.8	14 6	25.5	21.6	3 3	49.5	39.9	H 7 9	6.1	6.0	21 4	8.0	8.9	14 1	21.4	21.9	2 7	14.2	11.9	14 3	19.7	17.7
14 4	25.8	17.2	13 1	21.3	21.3	15 6	7.8	7.8	4 3	68.5	63.5	9 9	6.7	9.1	0 5	33.4	33.7	15 1	29.5	27.4	3 7	14.9	16.6	15 3	10.7	13.3
15 4	13.2	14.4	14 1	10.4	9.4	18 6	9.1	9.3	5 3	92.2	94.3	3 1 0	134.9	176.4	1 5	29.0	27.8	16 1	5.1	7.7	4 7	19.4	21.2	18 3	12.4	12.6
16 4	17.4	17.6	15 1	14.6	12.2	20 6	4.4	6.4	6 3	53.5	47.0	2 0	131.8	157.2	2 5	12.4	17.2	17 1	14.4	17.1	5 7	8.6	11.0	19 3	10.5	11.7
17 4	9.4	6.2	16 1	42.0	42.4	0 7	15.7	16.0	7 3	66.9	66.0	3 0	49.9	43.3	3 5	25.7	27.5	18 1	20.6	18.8	6 7	7.2	4.2	20 3	8.1	8.7
18 4	9.1	4.7	17 1	5.8	6.1	1 7	8.8	9.7	8 3	8.1	6.0	4 0	64.0	63.6	4 5	30.0	27.3	19 1	8.4	10.3	7 7	8.4	10.4	21 3	3.7	4.6
19 4	14.5	14.2	19 1	22.1	22.1	2 7	24.7	29.9	9 3	39.5	34.8	5 0	18.5	20.1	5 5	26.0	24.8	22 1	6.5	6.4	8 7	15.0	14.3	22 3	3.3	6.5
21 4	8.7	9.6	20 1	15.8	12.1	3 7	35.3	39.0	10 3	19.9	15.2	6 0	12.7	17.9	6 5	34.4	37.7	24 1	7.1	7.5	9 7	14.3	18.0	0 4	48.2	49.9
22 4	3.9	4.4	21 1	5.3	4.9	4 7	13.7	9.5	11 3	52.6	52.9	7 0	7.4	2.5	7 5	31.8	28.6	0 2	53.9	43.7	10 7	8.2	9.2	1 4	38.5	34.7
1 5	16.8	13.2	22 1	13.8	15.8	5 7	4.4	7.2	12 3	16.5	11.1	8 0	58.1	61.6	8 5	8.0	7.2	1 2	35.9	34.5	11 7	3.9	3.3	2 4	25.1	23.2
2 5	52.3	50.8	23 1	4.3	3.8	6 7	23.5	25.6	13 3	37.7	34.2	9 0	32.8	27.7	9 5	8.4	10.6	2 2	44.9	41.1	12 7	10.5	5.7	3 4	30.6	26.9
3 5	13.0	14.6	24 1	3.4	2.3	7 7	6.5	3.4	14 3	24.8	16.7	10 0	24.4	28.8	10 5	17.2	15.8	3 2	87.2	79.5	13 7	5.6	6.2	4 4	41.5	44.0
4 5	26.4	23.6	0 2	37.3	43.5	8 7	5.6	10.9	15 3	26.3	22.6	11 0	23.2	17.3	11 5	24.7	20.1	4 2	91.4	83.4	14 7	8.8	7.6	5 4	17.1	18.3
5 5	35.4	35.2	1 2	62.5	64.1	9 7	10.9	9.4	16 3	17.7	14.7	12 0	57.1	52.6	12 5	27.9	28.5	5 2	52.5	50.2	1 8	10.6	8.2	6 4	36.8	37.7
6 5	17.2	15.3	2 2	73.0	77.1	10 7	23.1	23.2	17 3	11.6	13.2	13 0	10.2	14.6	13 5	27.1	25.5	6 2	52.1	47.6	2 8	4.7	6.8	7 4	9.4	11.1
7 5	11.9	8.0	3 2	14.8	7.6	11 7	12.6	12.5	21 3	7.5	9.5	14 0	25.8	28.1	14 5	4.6	4.7	7 2	56.8	53.6	3 8	18.3	21.1	8 4	13.4	16.8
8 5	30.0	26.1	4 2	57.0	54.8	12 7	9.4	10.6	23 3	7.5	8.7	15 0	3.0	5.8	15 5	10.0	12.5	8 2	70.4	71.0	4 8	13.0	9.6	9 4	42.5	31.9
9 5	12.3	2.9	5 2	120.6	122.8	13 7	7.8	6.7	0 4	47.8	47.9	16 0	3.2	3.0	16 5	19.0	18.1	9 2	53.3	49.4	5 8	4.5	3.8	10 4	15.7	19.9
10 5	18.6	21.7	6 2	53.3	44.5	14 7	5.1	1.8	1 4	54.3	54.7	17 0	3.2	6.2	17 5	14.2	9.7	10 2	10.6	10.9	6 8	4.4	6.6	11 4	28.4	28.8
11 5	30.8	30.8	7 2	33.1	29.6	15 7	4.2	5.6	2 4	38.8	30.1	18 0	7.2	11.5	18 5	11.3	12.0	12 2	13.5	16.7	8 8	7.1	6.1	12 4	14.9	14.1
13 5	15.0	20.5	8 2	33.2	34.4	16 7	9.1	10.1	3 4	71.6	67.1	20 0	25.7	23.8	21 5	3.2	5.6	13 2	32.1	30.0	9 8	10.5	12.3	13 4	21.7	21.2
14 5	26.8	27.8	9 2	45.5	43.9	17 7	4.1	1.4	4 4	61.7	56.2	24 0	4.2	6.1	1 6	16.9	15.7	14 2	20.6	18.4	10 8	5.3	3.5	14 4	9.5	9.2
15 5	16.5	11.0	10 2	14.4	16.7	1 8	21.3	21.9	5 4	17.8	16.2	0 1	144.7	182.6	2 6	50.8	52.3	15 2	34.0	31.2	12 8	4.4	6.1	15 4	26.8	20.9
16 5	4.7	3.9	11 2	43.9	40.5	3 8	6.1	9.7	6 4	38.2	33.0	1 1	87.8	93.4	3 6	15.5	19.8	16 2	16.1	12.6	0 9	5.2	7.5	17 4	5.0	7.5
17 5	17.1	15.6	12 2	29.9	22.2	4 8	7.4	10.6	7 4	34.6	29.0	2 1	96.3	91.8	4 6	24.8	26.9	17 2	14.5	12.3	1 9	8.1	7.6	20 4	9.1	8.8
18 5	20.9	20.8	13 2	46.0	41.0	5 8	8.5	9.1	8 4	20.8	18.7	3 1	35.0	33.9	5 6	7.1	8.4	18 2	12.7	13.6	4 9	3.4	4.4	0 5	24.8	25.7
19 5	2.7	0.9	14 2	31.8	27.7	6 8	7.2	7.3	9 4	30.0	31.3	4 1	69.8	62.0	6 6	9.8	8.3	19 2	19.3	20.1	H 5 9	7.4	7.6	1 5	29.1	26.2
20 5	5.4	6.3	15 2	41.6	37.9	7 8	12.2	14.8	10 4	27.1	27.3	5 1	70.4	67.3	7 6	25.2	29.3	20 2	6.7	6.8	6 9	6.3	8.3	2 5	13.7	15.6
21 5	4.3	6.4	17 2	23.9	25.6	8 8	7.9	8.5	11 4	27.1	22.7	6 1	77.7	71.2	8 6	21.9	21.1	22 2	10.0	10.2	5 1 0	27.4	25.9	3 5	18.9	17.0
0 6	63.3	55.5	18 2	19.3	17.1	9 8	6.6	6.4	12 4	19.7	16.5	7 1	39.8	33.6	10 6	17.6	18.0	23 2	5.9	7.8	2 0	64.2	66.3	4 5	22.0	24.0
2 6	21.4	23.6	19 2	4.2	3.5	11 8	12.2	13.4	13 4	21.8	21.0	8 1	15.1	17.1	11 6	10.7	9.4	0 3	33.2	34.7	3 0	68.0	67.5	5 5	17.4	15.5

Table II. (continued)

HKL FO FC	HKL FO FC	HKL FO FC	HKL FO FC	H K L FO FC	HKL FO FC	HKL FO FC	KL FO FC	K L FO FC
865 24.5 22.8	652 30.427.8	678 4.5 6.2	71 527.627.4	819 2 2.0 2.3	93114.312.5	1090 20.020.1	101 12.2 7.4	2 313.715.8
75 16.6 14.1	62 22.216.4	8812.6 12.2	2510.718.4	20 210.011.1	4136.934.9	100 14.913.7	111 17.421.0	3 3 9.610.6
85 12.1 12.4	72 22.917.7	108 5.8 5.8	4517.518.3	0 339.843.5	5127.430.4	110 18.722.0	121 10.3 8.7	4 316.419.0
95 8.4 8.7	82 38.932.7	118 5.6 8.9	5544.345.9	1 322.816.7	6115.516.3	120 10.510.1	131 6.1 3.6	5 3 5.410.8
105 20.4 20.6	92 10.913.8	128 5.2 6.4	6512.814.3	2 321.118.5	7116.515.1	170 11.315.0	141 9.0 8.8	6 3 9.111.2
115 19.0 17.6	102 23.415.1	0914.3 14.9	7518.319.2	3 3 5.8 9.9	8122.225.4	180 7.2 3.6	151 7.2 7.7	7 3 7.2 7.2
125 21.6 23.6	112 16.014.4	29 5.0 9.0	8521.625.0	4 329.729.8	91 4.1 7.4	11 20.427.5	171 1.6 3.3	8 3 6.9 4.8
135 17.9 15.9	122 12.010.4	39 3.4 3.3	9516.917.5	5 3 8.1 7.6	10113.7 7.6	21 14.715.6	181 2.2 2.6	9 3 3.3 4.7
145 12.5 11.5	132 18.417.1	49 4.6 6.4	10515.111.9	6 334.838.0	11132.731.1	31 26.932.8	02 35.941.7	10 313.012.7
155 14.4 16.0	142 25.123.3	H59 4.2 6.3	11518.419.7	7 3 3.9 7.2	12116.311.7	41 23.220.8	12 13.012.5	12 3 3.6 7.0
165 12.5 12.1	152 17.415.9	69 6.6 9.0	12513.510.0	8 3 9.9 9.1	131 8.2 6.7	51 10.9 9.8	22 4.3 2.3	13 3 5.6 7.0
175 8.7 8.4	162 22.119.2	720 6.8 15.9	13514.010.1	9 3 6.2 2.1	15118.719.0	61 14.317.4	32 16.016.5	2 4 9.613.8
185 4.8 5.6	172 13.812.8	3054.2 53.4	14512.2 7.5	10 316.819.9	161 3.8 5.1	71 30.331.8	42 19.421.6	3 4 6.7 9.2
195 3.3 1.1	182 11.215.0	40 5.1 12.1	165 2.6 7.0	11 320.318.0	17114.013.6	81 21.618.6	52 4.9 5.5	4 414.914.8
205 5.2 7.1	212 8.0 8.8	5037.3 29.0	175 6.8 8.8	12 317.017.9	181 3.8 6.5	91 23.121.6	62 16.818.2	5 4 9.2 9.7
26 12.3 11.8	03 49.052.0	6029.5 23.0	185 5.4 5.2	13 3 4.7 3.4	201 6.0 6.9	111 15.610.7	72 18.621.2	6 4 6.4 5.4
36 27.7 26.9	13 35.938.5	7037.9 39.6	06 3.8 3.2	14 3 5.4 7.2	0250.953.3	121 7.1 8.8	82 11.212.8	7 4 6.2 7.8
46 14.5 16.7	23 17.618.6	8052.2 47.7	1620.223.4	15 310.2 8.7	1237.333.9	131 18.921.1	92 18.017.8	8 4 8.412.1
56 24.3 23.8	33 19.311.7	9035.2 35.7	2612.513.1	16 311.913.9	2223.823.7	151 12.416.0	102 10.314.2	9 4 8.0 7.5
66 11.7 7.7	43 36.939.0	11026.5 25.5	3633.234.9	17 3 3.9 7.0	3217.921.9	161 14.917.2	112 5.4 5.4	3 512.614.1
76 25.5 24.4	53 36.233.2	12013.1 7.6	4614.510.5	18 3 6.3 7.9	4236.439.9	191 5.9 9.6	122 14.515.9	5 5 5.3 5.7
86 20.5 19.6	63 43.844.8	13027.5 29.9	5614.210.7	19 3 3.9 2.9	5218.816.8	12 24.623.0	132 10.810.5	6 5 3.6 4.5
96 20.1 19.9	73 24.425.5	140 5.9 7.3	6616.915.5	20 3 5.0 7.6	6230.031.7	22 10.912.1	142 2.8 4.5	7 5 6.0 7.5
116 11.9 9.0	83 6.6 4.4	15016.5 15.8	76 5.5 5.5	0 425.319.8	7219.915.2	32 5.5 6.2	152 7.1 9.0	10 5 5.3 8.3
126 23.6 17.6	93 21.416.1	16013.4 18.2	86 5.1 5.8	1 4 8.4 7.6	8219.312.4	42 19.222.1	03 11.6 6.8	11 5 3.5 7.0
136 7.8 10.0	103 27.526.0	18014.0 9.4	9618.118.3	2 420.321.2	9218.915.3	52 12.014.1	13 8.5 9.3	H1 6 9.811.6
146 13.3 13.7	113 27.223.4	19013.1 13.9	126 9.5 7.8	3 414.212.1	10226.427.6	62 5.3 3.0	23 16.121.6	2 012.413.8
156 9.4 7.1	123 50.946.9	21010.5 9.5	136 3.5 6.7	4 424.826.2	11223.419.5	72 11.912.0	33 9.0 6.7	133 013.920.3
166 4.4 4.9	133 28.617.6	0125.7 16.6	146 4.0 6.8	5 422.121.4	12216.618.4	82 26.730.5	43 17.421.4	6 013.614.2
176 8.7 8.6	143 6.0 6.7	1176.7 84.9	156 7.9 8.0	6 427.830.1	13218.915.1	92 20.418.5	53 6.1 6.3	7 021.022.2
186 5.3 6.2	153 9.4 7.7	21 5.4 8.4	166 4.5 4.0	7 414.3 9.5	142 3.2 3.8	122 11.613.7	63 10.3 8.9	13 0 7.7 7.6
07 20.4 16.8	163 16.517.1	3113.8 12.6	07 3.3 4.3	8 429.932.2	152 6.5 5.9	142 10.111.0	73 9.211.0	0 1 6.7 8.3
17 10.3 12.0	173 2.8 6.6	4131.3 29.8	1711.813.0	9 4 4.5 6.9	162 8.311.7	152 6.8 7.6	83 10.416.2	1 120.621.9
27 18.3 18.2	183 9.9 9.3	5157.0 59.9	27 6.3 6.6	10 415.516.5	172 5.8 4.6	172 8.4 9.9	93 9.010.5	3 111.4 9.5
37 6.8 8.0	193 5.6 5.2	6130.8 28.4	4712.811.9	11 423.922.9	182 8.9 8.6	182 6.0 7.7	103 9.011.5	4 113.815.0
47 7.7 4.9	203 3.1 2.5	7146.7 45.4	57 8.410.8	12 413.613.0	192 9.4 8.5	03 20.126.7	113 2.8 4.4	5 1 9.112.3
57 17.0 20.7	213 7.1 8.8	8140.7 31.1	67 4.4 6.6	13 413.111.4	0315.1 9.0	13 28.524.6	123 7.4 9.6	7 112.313.2
67 19.0 19.5	04 17.219.5	9124.7 20.0	8712.512.1	14 419.919.5	1310.1 7.3	23 23.421.5	133 8.110.7	8 1 4.2 9.3
77 9.3 12.1	14 34.931.1	10138.5 27.8	97 7.3 6.5	15 410.612.3	2327.220.8	33 8.9 3.4	143 12.215.7	9 1 5.7 5.3
87 8.0 9.0	24 27.931.3	11125.8 26.6	107 5.8 4.3	16 4 1.5 4.1	33 8.9 6.3	43 14.315.8	153 5.4 5.0	10 1 3.8 8.6
97 10.4 12.6	34 29.726.1	12110.9 13.2	117 6.2 7.7	17 4 6.5 4.2	4323.925.5	53 11.2 8.1	04 13.915.1	11 1 6.2 9.0
107 7.1 6.1	44 25.629.4	131 5.1 3.4	127 3.4 5.4	19 4 3.2 2.7	5321.115.4	63 26.427.5	14 3.5 1.9	14 1 6.2 8.6
117 11.1 12.6	54 22.719.3	14139.1 35.3	137 3.6 5.3	0 516.518.5	6311.814.4	73 15.810.4	34 8.7 6.9	0 212.816.2
127 5.5 8.6	64 13.814.2	15112.2 11.5	18 7.610.2	1 515.016.5	7315.212.6	83 14.211.8	44 19.617.7	2 2 5.2 4.6
137 9.4 11.3	74 20.716.9	17123.9 22.8	28 2.7 5.5	2 526.822.9	8333.134.9	93 9.4 8.2	54 13.413.9	3 2 6.3 9.5
147 5.8 5.2	84 19.319.5	18113.8 13.9	38 6.4 7.6	3 519.118.5	93 9.510.8	103 21.121.2	64 10.311.8	4 2 6.2 9.1
157 6.3 6.5	94 11.1 9.3	191 5.5 5.5	48 8.510.1	4 520.719.1	10321.119.5	123 9.110.9	74 13.615.3	5 2 6.111.4
167 5.5 6.7	104 18.417.6	201 5.6 3.0	68 8.5 9.6	5 517.417.0	11323.214.0	143 8.4 7.3	84 6.2 5.2	7 213.414.5
08 25.1 22.4	114 20.414.3	211 6.8 7.7	78 3.1 5.7	7 5 9.510.9	12311.610.7	163 11.312.4	94 6.6 9.5	9 2 4.3 6.5
18 9.5 10.6	124 13.415.5	221 5.4 6.3	88 4.6 4.5	8 513.410.1	133 5.3 5.5	14 11.3 8.6	104 7.6 8.9	10 2 4.1 6.5
28 16.3 18.8	134 11.213.9	0222.9 10.9	108 4.4 6.7	9 521.222.1	14313.215.2	24 14.417.0	124 10.711.7	1 3 8.4 8.4
48 8.5 7.9	144 20.420.4	1232.6 34.5	H1 9 2.5 3.3	10 519.118.5	163 8.2 6.8	34 17.816.0	134 4.2 7.0	2 3 6.810.8
58 8.3 8.0	154 5.4 8.4	2230.4 22.3	39 2.5 6.2	11 513.210.2	173 2.6 3.6	44 23.620.7	144 2.7 3.0	3 313.514.3
68 4.7 4.8	164 9.312.3	3252.6 44.8	00 6.5 7.6	12 513.913.0	183 7.2 9.9	54 23.723.1	05 7.5 6.8	4 3 6.6 6.4
78 7.1 9.5	174 11.2 6.6	4219.0 18.6	1048.256.9	13 516.618.7	193 5.5 6.6	64 7.4 6.5	15 6.6 7.3	5 311.112.4
88 5.3 1.8	184 15.113.5	5230.0 30.2	2025.330.6	16 5 5.8 6.0	0416.218.0	74 7.3 4.8	25 9.710.3	6 3 8.8 9.2
118 6.5 7.9	204 8.5 8.9	6221.8 22.5	30 7.4 5.6	17 5 4.6 7.3	1415.016.3	84 10.411.2	35 1.4 1.1	8 3 6.8 9.3
138 4.3 3.1	05 18.216.7	7233.4 32.0	40 4.3 2.3	0 618.618.6	2415.417.5	94 7.3 5.7	45 9.4 5.8	9 3 7.4 7.6
19 9.1 9.1	15 39.041.5	8233.7 25.8	5056.863.0	1 621.620.6	3426.225.9	104 5.0 7.6	55 8.111.8	12 3 4.2 6.8
29 8.6 10.3	25 36.234.7	92 7.1 9.9	6029.531.0	2 6 3.3 6.7	4421.719.8	124 8.1 4.8	65 13.013.0	0 4 8.510.4
39 8.8 8.3	35 24.924.0	10244.4 48.0	7030.130.6	3 6 9.3 8.8	54 2.2 3.5	134 10.910.9	75 9.713.6	3 4 8.3 8.0
49 6.5 6.5	45 22.823.0	11218.4 17.2	8021.311.4	4 613.210.1	6421.423.7	174 2.5 3.0	85 10.110.7	5 4 7.910.5
79 6.2 6.7	55 11.4 7.7	12213.9 9.7	90 8.310.0	5 618.317.0	74 4.3 6.6	05 25.722.4	95 6.7 7.6	6 4 9.312.6
89 4.8 8.5	65 16.113.3	132 6.6 6.6	10023.316.7	6 614.610.2	94 10.9 6.8	15 16.413.8	105 7.5 9.3	7 4 8.813.2
00 65.8 65.3	75 28.833.3	14214.2 15.2	11021.526.7	7 621.523.1	10412.914.5	25 7.1 8.3	115 10.615.7	0 021.017.0
20 8.2 3.5	85 14.715.1	15216.8 13.9	12030.921.1	8 6 3.0 5.8	11410.2 8.3	35 25.123.6	125 5.6 7.9	5 010.4 8.9
30 4.5 1.0	95 20.317.6	16222.1 23.5	130 3.6 4.0	9 6 4.7 5.8	124 7.518.3	55 12.311.8	135 2.7 2.6	6 0 8.212.0
40 42.2 36.3	105 5.411.0	182 3.9 8.5	18014.0 8.0	10 6 2.9 6.0	13414.813.9	65 17.717.9	06 4.1 6.4	0 1 5.9 9.4
50 65.8 69.3	125 4.8 8.6	202 6.5 10.4	15016.519.6	11 613.415.4	144 4.1 6.6	75 11.613.8	16 9.7 9.8	1 1 7.2 7.7
60 5.7 4.5	135 21.521.6	212 6.0 6.1	170 8.5 9.3	12 6 1.5 6.0	154 7.5 7.7	85 9.7 9.1	26 1.2 4.2	2 110.112.1
70 5.0 1.9	145 15.315.4	13 2.9 6.8	190 9.1 7.7	14 6 8.6 5.9	164 8.2 9.5	95 16.414.7	36 9.3 9.9	4 1 4.0 1.7
80 31.4 31.2	155 13.910.7	2345.0 50.8	200 2.7 1.0	15 6 7.711.1	174 5.4 8.8	105 6.1 3.2	46 3.9 3.9	5 1 5.5 8.6
90 24.1 30.4	165 12.412.3	3317.2 13.6	210 7.411.3	0 711.511.9	184 5.1 5.9	135 4.4 7.8	56 8.811.6	6 1 6.6 9.4
100 37.2 30.7	185 6.8 7.0	4333.0 27.9	01 6.510.9	2 7 7.7 5.2	1523.926.2	155 6.311.3	76 5.5 4.9	7 1 3.6 7.5
110 54.3 51.5	195 5.5 5.9	53 2.3 4.1	1123.126.3	3 711.913.4	2531.932.4	06 5.1 5.9	96 5.5 7.7	8 1 6.912.0
120 16.4 20.0	06 3.9 4.2	6316.0 13.5	2119.220.3	4 7 6.1 6.6	3518.115.9	16 14.314.7	106 2.9 4.5	9 113.316.1
130 17.6 12.9	16 16.418.3	7317.3 9.0	3140.141.4	5 7 3.2 5.8	4516.511.8	26 4.0 6.6	07 2.9 5.1	10 1 5.1 7.4
140 4.0 4.9	36 25.824.4	8335.8 38.8	4127.228.2	6 7 2.3 5.7	5516.116.5	36 11.910.1	17 6.9 8.5	11 1 7.6 7.5
150 22.9 24.2	46 21.417.3	9311.6 18.5	5128.527.0	7 711.211.5	65 8.912.1	46 10.6 9.5	27 4.0 5.4	1 2 8.4 9.8
160 25.9 23.0	56 16.619.9	103 9.3 9.1	6128.627.6	9 7 5.2 6.6	75 7.4 5.3	56 13.913.3	37 3.9 4.8	2 2 6.3 7.4
190 15.6 11.1	66 11.6 9.5	11324.2 19.8	7130.432.4	10 7 4.6 4.7	9510.710.6	96 7.8 6.2	H4 7 1.6 3.6	3 2 5.4 8.8
200 9.6 7.7	76 2.9 4.1	12325.5 25.6	8114.210.4	12 7 5.7 7.5	11512.611.6	116 5.310.3	57 5.4 9.1	0 3 3.9 8.4
210 7.2 9.1	96 16.716.2	133 7.3 3.9	9133.631.6	13 7 5.8 8.4	125 7.7 7.5	07 3.1 2.7	120 013.315.1	4 3 9.712.2
220 1.9 0.7	106 14.413.5	14325.1 25.4	10123.421.9	0 810.711.3	135 1.9 1.8	37 13.518.1	3 018.714.2	5 3 6.0 9.8
01 70.9 76.3	116 16.516.9	153 8.0 10.9	11110.1 9.4	1 8 6.1 7.8	145 6.7 9.6	47 5.6 6.5	5 022.628.6	6 3 2.3 7.6
11 69.9 79.6	126 11.0 9.1	163 4.3 3.3	12123.817.1	2 812.915.2	155 2.7 5.9	57 5.8 6.0	11 011.213.8	7 3 4.3 4.3
21 56.5 60.3	136 12.411.2	173 14.2 9.7	13128.226.6	3 8 4.6 6.5	06 2.3 0.2	67 7.7 7.6	15 012.112.5	0 4 8.210.8
31 60.5 62.8	146 10.1 9.1	183 9.2 11.6	131 1.8 5.2	4 8 9.712.1	16 2.3 6.3	77 9.313.2	01 5.0 9.2	6 4 7.310.5
41 38.3 40.7	156 11.611.7	19310.2 10.5	16119.112.4	H7 8 4.3 5.4	26 2.3 2.6	H 87 1.9 4.4	1 113.214.2	15 012.2 4.6
51 19.8 19.6	166 5.1 5.8	203 8.6 11.7	171 7.4 8.3	8 8 6.6 9.6	3614.316.4	97 3.0 5.0	21 8.6 9.1	2 1 4.7 2.6
61 40.8 35.5	176 6.8 9.0	213 9.2 9.6	181 10.5 9.0	91 025.035.8	46 2.2 6.8	11 30 24.785.5	3 114.917.8	5 1 5.2 6.3
71 44.1 41.5	07 10.810.5	0435.4 39.9	191 5.3 6.6	20 1.4 0.2	5614.2 8.2	40 4.5 8.2	4 114.013.7	6 1 2.8 5.8
81 35.3 31.2	17 9.1 9.2	1413.6 10.6	201 3.5 3.1	3 030.643.5	66 2.2 5.4	50 20.823.3	5 111.013.1	7 1 2.5 3.0
91 49.3 41.9	27 10.8 8.7	2427.5 25.3	0219.417.4	4 015.211.4	7618.619.1	60 8.3 3.3	6 1 8.4 9.0	8 1 6.5 9.7
101 21.1 21.2	37 15.316.3	3427.3 23.8	1219.919.0	5 012.1 9.6	86 3.0 5.9	70 26.027.0	7 1 6.8 8.1	5 2 6.3 4.5
111 7.7 9.2	47 8.6 5.1	4427.6 31.3	2266.871.5	6 0 8.0 2.7	9616.616.4	80 13.813.6	9 112.113.2	3 3 8.517.0
121 30.1 24.6	57 9.311.8	5422.0 17.7	3216.819.8	7 019.622.2	106 3.5 6.9	90 10.916.3	10 113.312.1	E
131 17.4 19.7	67 12.611.0	6421.8 22.2	4210.714.7	8 013.711.5	136 8.611.1	100 8.4 4.8	11 113.411.4	
141 34.4 35.3	77 8.6 6.5	7426.4 28.9	52 8.4 9.5	9 029.628.2	146 5.2 7.7	110 2.8 2.0	12 1 5.7 8.9	
151 29.1 26.0	87 13.415.0	84 6.3 10.0	6215.816.4	10 010.1 1.0	07 3.8 2.9	130 11.418.3	13 1 8.4 6.6	
161 5.5 9.8	97 9.110.8	9412.5 11.5	7233.733.6	11 019.914.8	17 3.2 5.8	150 2.5 1.2	14 1 3.4 3.5	
171 18.4 14.7	107 9.311.9	10422.4 17.7	8237.340.3	13 023.023.8	28 2.8 5.8	160 13.2 8.6	15 1 4.2 5.7	
181 10.1 7.9	117 5.8 5.9	124 3.5 6.1	9217.719.5	14 0 8.3 7.8	H48 3.7 6.7	01 12.313.6	16 1 4.1 7.5	
191 19.1 18.5	127 6.7 5.6	13419.3 17.2	10219.017.8	15 034.430.6	68 3.9 8.3	11 21.021.2	1 2 9.814.7	
201 12.7 11.7	137 12.413.9	14410.8 10.2	11232.932.6	16 0 7.8 9.1	10 0 010.613.7	21 24.026.0	2 220.226.2	
211 3.4 4.6	147 1.9 6.2	15410.0 9.6	12216.917.8	17 016.116.4	1031.637.0	31 20.418.4	3 2 9.712.5	
221 3.5 5.3	18 10.811.9	16416.6 14.3	13220.717.6	18 013.710.1	20 3.8 7.8	41 5.8 5.0	5 2 8.710.4	
02 31.8 24.4	28 5.1 5.2	174 7.7 9.0	14219.122.1	19 0 9.912.7	30 8.412.8	51 10.318.3	8 213.818.9	
12 42.7 43.7	38 3.5 3.7	184 7.8 6.4	152 3.6 2.8	20 0 3.5 1.3	40 7.9 9.6	61 25.628.8	10 212.414.5	
22 85.9 83.1	48 7.0 7.6	194 8.0 7.5	162 8.0 8.7	01 7.4 9.4	5043.746.2	71 18.021.5	11 2 8.6 4.9	
32 41.8 41.4	58 3.4 3.9	204 1.8 4.5	172 4.8 4.6	1 121.126.3	6013.8 6.4	81 14.618.1	12 214.315.8	
42 9.8 10.9	68 6.110.5	0519.5 14.8	182 7.4 9.4	2 131.234.8	80 9.4 4.3	91 10.510.1	1 311.314.4	

Table III. Comparison of the Observed and Calculated Intensity Differences Used for the Establishment of Absolute Configuration

h	k	l	$F_c^2(h\bar{k}l)/F_c^2(hkl)$	$I_o(h\bar{k}l)/I_o(hkl)$	h	k	l	$F_c^2(h\bar{k}l)/F_c^2(hkl)$	$I_o(h\bar{k}l)/I_o(hkl)$
11	3	4	0.627	<1	8	9	3	2.215	>1
13	8	1	1.150	>1	13	2	3	0.824	<1
11	9	1	0.805	<1	15	5	1	0.781	<1
11	2	2	2.057	>1	11	9	1	0.805	<1
11	1	3	0.858	<1	11	10	1	0.776	<1
13	2	3	0.824	<1	10	5	1	1.078	>1
13	1	3	0.891	<1	3	20	2	0.950	<1

Table IV. Bond Angles

C(1)–O(2)–C(21)	116°	C(10)–C(12)–C(14)	109°
C(13)–O(5)–C(19)	100	O(5)–C(13)–C(11)	109
C(16)–O(6)–C(19)	102	O(5)–C(13)–C(14)	108
O(2)–C(1)–C(2)	118	O(5)–C(13)–C(15)	102
O(2)–C(1)–C(5)	124	C(11)–C(13)–C(14)	111
C(2)–C(1)–C(5)	124	C(11)–C(13)–C(15)	113
C(1)–C(2)–C(3)	120	C(14)–C(13)–C(15)	112
C(2)–C(3)–C(4)	120	C(12)–C(14)–C(13)	112
C(3)–C(4)–C(6)	121	C(12)–C(14)–C(16)	113
C(1)–C(5)–C(6)	116	C(13)–C(14)–C(16)	108
C(6)–C(5)–C(7)	117	C(13)–C(15)–C(17)	97
C(1)–C(5)–C(7)	126	O(6)–C(16)–C(14)	105
C(4)–C(6)–C(5)	118	O(6)–C(16)–C(18)	102
C(4)–C(6)–C(8)	121	C(14)–C(16)–C(18)	112
C(5)–C(6)–C(8)	120	C(15)–C(17)–C(18)	107
O(3)–C(7)–C(5)	119	C(15)–C(17)–C(19)	105
O(3)–C(7)–C(9)	121	C(18)–C(17)–C(19)	102
C(5)–C(7)–C(9)	120	C(16)–C(18)–C(17)	99
C(6)–C(8)–C(10)	124	O(5)–C(19)–(O(6)	107
C(7)–C(9)–C(10)	119	O(5)–C(19)–C(17)	106
C(7)–C(9)–C(11)	120	O(5)–C(19)–C(20)	107
C(10)–C(9)–C(11)	121	O(6)–C(19)–C(17)	108
C(8)–C(10)–C(9)	119	O(6)–C(19)–C(20)	110
C(8)–C(10)–C(12)	121	C(17)–C(19)–C(20)	118
C(9)–C(10)–C(12)	120	O(1)–C(21)–O(2)	126
O(4)–C(11)–C(9)	120	O(1)–C(21)–C(22)	128
O(4)–C(11)–C(13)	121	O(2)–C(21)–C(22)	107
C(9)–C(11)–C(13)	119	Br–C(22)–C(21)	113

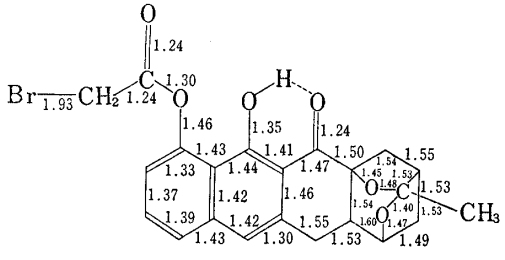

Fig. 3. Bond Distances

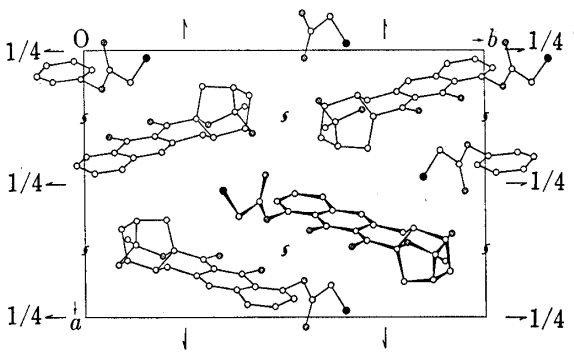

Fig. 4. Packing of the Structure Seen Down the c Axis

In Fig. 4 is shown a view of the structure along the *c* axis, together with the packing of the molecules in a unit cell. Since the molecule has no polar groups available for intermolecular hydrogen bonds, the association of the molecules seems to be mainly due to the van der Waals interactions.

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