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The Crystallography of Acridine. Part II. The Structure of Acridine III

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Acridine III is monoclinic with $a = 11{\cdot}375$, $b = 5{\cdot}988$, $c = 13{\cdot}647$ Å, $\beta = 98° \, 58'$, $Z = 4$ and space group $P2_1/n$. Its structure has been determined from the intensities of 2057 reflexions (1375 observed) estimated by visual and (in part) by photometric methods from equi-inclination Cu $K\alpha$ Weissenberg photographs. Trial-and-error methods and Harker–Kasper inequalities were used to determine the approximate structure. Refinement has proceeded by successive calculations of ϱ_o and $(\varrho_o - \varrho_c)$ in three dimensions. The structure is a modification of the anthracene structure with the polar molecules arranged in antiparallel pairs. The molecules, which are subject to rigid-body vibrations and librations, are distorted slightly from the planar and symmetrical form in ways which suggest that molecular interactions are responsible. Intramolecular bond lengths have been determined with a mean standard deviation of about 0·010 Å. They agree very closely with corresponding bond lengths in anthracene; maximum deviation in C–C bonds 0·009 Å. The results are discussed briefly with respect to the molecular-orbital method of bond-length prediction.

Introduction

Determinations of the structures of many aromatic hydrocarbons (e.g. Robertson, 1951), including accurate re-determinations of the structures of naphthalene (Abrahams, Robertson & White, 1949) and anthracene (Sinclair, Robertson & Mathieson, 1950; Ahmed & Cruickshank, 1952), have provided excellent experimental data against which theoretical calculations of bond lengths in these molecules can be checked. The agreement achieved between observed and calculated bond lengths has shown that bond lengths in condensed-ring aromatic hydrocarbon molecules can now be predicted with confidence to within 0·02 Å (Coulson, Daudel & Robertson, 1951). The situation is not so satisfactory, however, when the molecules contain hetero-atoms, for reasons which have been discussed by Coulson (1951).

Development of the theory of hetero-nuclear molecules appears to have been hindered by a shortage of sufficiently accurate measurements. The work now to be described was undertaken to provide such experimental data for acridine, $C_{13}H_9N$, a heterocyclic molecule which is derived from anthracene by the replacement of one *meso* CH group by nitrogen. Fig. 1 shows the structural formula with the atoms numbered in the order preferred by Albert (1951) in his exhaustive account of this compound and its important derivatives. The structure of phenazine, the related compound in which both anthracene *meso* CH groups are replaced by nitrogen, has been determined by Herbstein & Schmidt (1955).

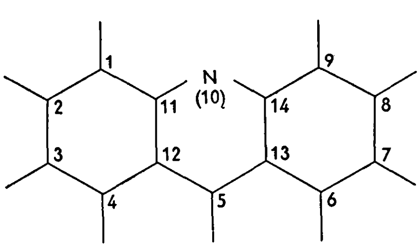

Fig. 1. Structural formula of acridine.

At an early stage in the investigation a second problem of considerable interest appeared: acridine was found to exhibit polymorphism to a remarkable degree. The existence of five distinct polymorphic forms has been reported by Kofler (1943). Crystallographic data for three forms, two of which appear to be stable under normal conditions of temperature and pressure, have been published in Part I (Lowde, Phillips & Wood, 1953). A fourth form has been described elsewhere (Phillips, 1954*a*). Herbstein & Schmidt (1955) also have published preliminary X-ray data for four forms, one of which (β-acridine in their notation) has not been described elsewhere. The simplest form, the only one in which the asymmetric unit is a single molecule, was chosen for the first complete investigation aimed at accurate measurement of the acridine molecule. The analysis of the structure of this form, designated acridine III, is described here. The relation of its structure to those of the other forms, and the polymorphism of acridine in general, will be discussed in detail elsewhere when the other structure determinations have been completed.

Crystal data

The unit-cell dimensions have been re-determined from measurements of high-angle reflexions recorded in the Straumanis setting on oscillation photographs. Fe radiation, $\lambda(K\alpha_1) = 1{\cdot}93597$ Å, was used. The crystals are monoclinic prismatic (Lowde *et al.*, 1953) with

$a=11\cdot375\pm0\cdot003$, $b=5\cdot988\pm0\cdot003$, $c=13\cdot647\pm0\cdot003$ Å; $\beta=98°\ 58'$;

$U=918\cdot2$ Å^3; $D_m=1\cdot293$ g.ml.$^{-1}$; $Z=4$; $D_x=1\cdot294$ g.ml.$^{-1}$; $F(000)=376$. Linear absorption coefficient, for X-rays with wavelength 1·542 Å: $\mu=7\cdot01$ cm.$^{-1}$.

Reflexions $h0l$ are absent when $h+l$ is odd and reflexions $0k0$ are absent when k is odd. All other reflexions are present in all orders. The space group thus is $P2_1/n$–C_{2h}^5, a convenient reorientation of No. 14 (*International Tables*, 1952) giving β near 90°.

Intensity measurements

All the reflexions observable within the limit for copper radiation were recorded on equi-inclination Weissenberg photographs of crystals mounted for rotation about the [010], [100], [101] and [001] axes. Most of the intensity measurements were made from photographs, taken in the first two orientations, of all layer lines with equi-inclination angles less than 40° (k = 0–5; h = 0–9). The crystals were mounted on glass fibres with 'Duco' cement and were reduced to rough cylinders about 0·5 mm. long and 0·3 mm. in diameter by brushing with a camel hair brush moistened with ethanol.

Each exposure was made with a set of three sheets of Kodak no-screen X-ray film. Photographs of two types were taken. Integrated photographs (Wiebenga & Smits, 1950) with about 24 hr. exposure time were used for estimation of the stronger intensities. Measurements of relative photographic density in these integrated-reflexion spots were made with a simple split-beam photometer designed in this laboratory by Barnes & Hanson (1950, unpublished). A calibration wedge was printed on each film so that the X-ray intensity versus photographic density relationship could be checked. It was linear over the measured range.

The photometric measurements were supplemented by visual estimations from non-integrated photographs with exposure times up to 100 hr. These photographs and the oscillation photographs used for lattice-parameter measurements showed a fairly heavy background scatter and some strong diffuse reflexions. The visual estimations were made by comparison with timed exposures of a single reflexion. Film-factors were estimated separately for each set of photographs. The densities of both the extended and the contracted reflexion spots were estimated from [010]-axis upper-level photographs, and the harmonic mean of the two measurements was accepted whenever the distortions were small ($(A+\Delta A)/A < 1\cdot5$). The densities of the extended spots alone were estimated from the less symmetrical [100]-axis photographs and these estimations, together with all visual estimations of severely distorted reflexion spots, were corrected by the method described elsewhere (Phillips, 1954*b*).

There are 2057 possible reflexions but only 1375 were observed. This large proportion of very weak reflexions is an indication of the high dispersion of the intensity distribution characteristic of highly pseudo-symmetric structures. A detailed analysis has shown that the distribution is closely tricentric (Rogers & Wilson, 1953). The intensities of 579 reflexions were estimated once, 656 twice (i.e. on separate photographs taken around different axes) and 140 three times. The intensities of 435 reflexions were estimated at least once photometrically. Lorentz polarization factors were derived from charts due to Lu (1943) and Cochran (1948). No absorption corrections were applied. The estimations from the various layer lines were brought to the same relative scale by the use of factors A_h, B_k calculated from the equations

$$A_h \sum_k \sum_l I_h^{\frac{1}{2}} - \sum_k B_k \sum_l I_k^{\frac{1}{2}} = 0\,,$$

$$B_k \sum_h \sum_l I_k^{\frac{1}{2}} - \sum_h A_h \sum_l I_h^{\frac{1}{2}} = 0\,,$$

where I_h and I_k are respectively the corrected intensities derived from the hth and kth layer lines with crystals rotated about [100] and [010] and the summations include all the reflexions whose intensities were estimated from both sets of photographs. Visual and photometric estimations were correlated by comparison of results for the many reflexions common to both sets. No systematic variation of the correlating factor, such as might be expected to arise from variations in the shape of non-integrated reflexion spots, was observed. The photometric measurements were given twice the weight of visual estimations in the calculation of average values for the relative intensities. The absolute scale was determined by comparison of the observed and calculated structure-amplitudes. The observed structure amplitudes are listed in Table 1, together with the final calculated values.

Comparison of the observed and calculated structure amplitudes shows also that intensities observed for the most intense low-angle reflexions are all smaller than the corresponding calculated values. The effect probably is due to secondary extinction with little primary extinction, since crystals dipped repeatedly into liquid air showed only a small increase in these intensities. The powder photograph (Lowde *et al.*, 1953), from which intensities unaffected by primary and less affected by secondary extinction might be estimated, is not well enough resolved for use in estimating all the seriously affected intensities and enough others to provide adequate correlation. Analytical correction of extinction errors has not been attempted. The reflexions most seriously affected are $10\bar{1}$, 202, 301, 110, 212, 211, 311.

Structure determination

The projection of the structure on (010) was first determined (Phillips, 1950). The $h0l$ reflexions, which are present only when $h+l = 2n$ because of the diagonal glide plane, are strong only when $h+l = 4n$.

Table 1. *Observed and calculated structure factors*

hkℓ	$\lvert F\rvert_o$	F_c
0,0,0	-	376.0
0,0,2	*0.5	- 0.9
0,0,4	25.8	-27.3
0,0,6	*0.8	+ 1.8
0,0,8	7.2	+ 7.6
0,0,10	3.5	- 3.9
0,0,12	20.7	-21.5
0,0,14	9.8	-10.2
0,0,16	*0.9	
1,0,15	*1.2	+ 1.4
1,0,13	10.6	-11.0
1,0,11	5.4	+ 5.3
1,0,9	2.9	+ 3.2
1,0,7	18.7	-18.1
1,0,5	3.8	- 3.3
1,0,3	46.9	+52.8
1,0,1	9.2	+ 9.7
1,0,$\bar{1}$	55.6	+74.8
1,0,$\bar{3}$	0.8	+ 0.8
1,0,$\bar{5}$	10.0	-11.0
1,0,$\bar{7}$	1.1	- 0.8
1,0,$\bar{9}$	5.7	- 5.6
1,0,$\overline{11}$	5.2	+ 7.3
1,0,$\overline{13}$	25.4	-26.8
1,0,$\overline{15}$	*1.3	+ 0.5
1,0,$\overline{17}$	2.0	+ 2.5
2,0,16	*1.0	+ 1.2
2,0,14	9.7	+ 9.9
2,0,12	*1.2	+ 1.4
2,0,10	2.8	- 2.3
2,0,8	4.3	- 3.9
2,0,6	3.1	+ 3.0
2,0,4	*1.0	- 0.5
2,0,2	90.7	-109.6
2,0,0	2.3	0
2,0,$\bar{2}$	40.4	-43.8
2,0,$\bar{4}$	6.0	+ 5.8
2,0,$\bar{6}$	8.3	+ 7.3
2,0,$\bar{8}$	*1.3	+ 0.2
2,0,$\overline{10}$	3.8	- 2.2
2,0,$\overline{12}$	19.2	+22.2
2,0,$\overline{14}$	1.6	- 0.5
2,0,$\overline{16}$	*0.7	- 1.5
3,0,15	2.6	+ 2.9
3,0,13	2.6	+ 1.8
3,0,11	*1.2	- 0.4
3,0,9	11.7	+10.9
3,0,7	4.6	- 5.1
3,0,5	11.4	+10.7
3,0,3	4.6	- 5.2
3,0,1	129.0	-149.7
3,0,$\bar{1}$	6.5	+ 5.3
3,0,$\bar{3}$	31.6	+32.8
3,0,$\bar{5}$	7.4	- 6.4
3,0,$\bar{7}$	10.1	-11.3
3,0,$\bar{9}$	1.9	+ 2.2
3,0,$\overline{11}$	34.6	+37.4
3,0,$\overline{13}$	3.8	+ 6.0
3,0,$\overline{15}$	*1.2	- 0.1
3,0,$\overline{17}$	*0.8	- 0.5
4,0,16	*0.8	+ 1.0
4,0,14	*1.0	- 0.9
4,0,12	*1.2	- 0.8
4,0,10	*1.2	+ 0.7
4,0,8	28.8	+28.5
4,0,6	3.0	+ 2.9
4,0,4	9.6	-10.2
4,0,2	5.5	- 6.2
4,0,0	7.0	+ 7.5
4,0,$\bar{2}$	3.3	- 2.4
4,0,$\bar{4}$	35.4	-35.9
4,0,$\bar{6}$	6.5	- 6.2
4,0,$\bar{8}$	6.2	+ 5.9
4,0,$\overline{10}$	11.1	-13.3
4,0,$\overline{12}$	21.0	+22.3
4,0,$\overline{14}$	1.7	- 1.5
4,0,$\overline{16}$	2.2	- 2.2
5,0,15	*1.0	+ 1.8
5,0,13	*1.2	- 1.4
5,0,11	*1.2	+ 0.7
5,0,9	7.6	+ 7.9
5,0,7	3.8	+ 3.6
5,0,5	2.3	+ 3.3
5,0,3	9.5	+ 9.8
5,0,1	7.0	- 6.4
5,0,$\bar{1}$	9.4	-10.5
5,0,$\bar{3}$	3.2	+ 5.7
5,0,$\bar{5}$	31.3	-29.7
5,0,$\bar{7}$	1.2	- 1.2
5,0,$\bar{9}$	*1.1	- 0.7
5,0,$\overline{11}$	9.5	-11.6
5,0,$\overline{13}$	2.5	- 0.6
5,0,$\overline{15}$	2.0	+ 2.3
5,0,$\overline{17}$	2.4	+ 2.1
6,0,14	*1.0	+ 1.7
6,0,12	*1.2	+ 0.5
6,0,10	13.3	-13.3
6,0,8	4.6	+ 3.9
6,0,6	5.6	- 4.5
6,0,4	1.9	- 0.3
6,0,2	6.3	- 7.0
6,0,0	3.2	- 3.2
6,0,$\bar{2}$	5.5	+ 5.0
6,0,$\bar{4}$	12.7	+12.6
6,0,$\bar{6}$	8.5	+ 7.8
6,0,$\bar{8}$	1.0	+ 1.3
6,0,$\overline{10}$	14.7	-15.2
6,0,$\overline{12}$	3.5	+ 2.4
6,0,$\overline{14}$	2.5	+ 1.9
6,0,$\overline{16}$	3.1	- 2.4
7,0,13	*1.0	- 1.1
7,0,11	2.8	- 3.1
7,0,9	13.2	-13.3
7,0,7	1.7	- 1.5
7,0,5	6.2	+ 5.6
7,0,3	1.3	- 0.9
7,0,1	2.0	- 2.6
7,0,$\bar{1}$	*1.3	- 1.4
7,0,$\bar{3}$	45.1	+45.2
7,0,$\bar{5}$	2.6	+ 3.3
7,0,$\bar{7}$	*1.3	- 1.0
7,0,$\bar{9}$	3.7	+ 3.6
7,0,$\overline{11}$	3.0	- 3.9
7,0,$\overline{13}$	1.4	- 0.9
7,0,$\overline{15}$	2.0	- 2.7
8,0,12	*1.0	+ 1.2
8,0,10	4.2	- 3.6
8,0,8	2.5	- 2.1
8,0,6	*1.4	- 0.4
8,0,4	21.6	+21.0
8,0,2	1.1	+ 1.2
8,0,0	5.0	+ 4.6
8,0,$\bar{2}$	5.7	- 5.5
8,0.$\bar{4}$	18.7	+19.0
8,0,$\bar{6}$	*1.2	+ 0.1
8,0,$\bar{8}$	6.8	- 7.6
8,0,$\overline{10}$	4.1	- 4.0
8,0,$\overline{12}$	1.1	- 1.6
8,0,$\overline{14}$	*1.2	+ 1.0
8,0,$\overline{16}$	4.0	- 4.1
9,0,11	*1.0	+ 2.6
9,0,9	*1.1	- 1.2
9,0,7	2.6	+ 2.1
9,0,5	3.6	+ 3.4
9,0,3	14.0	+13.6
9,0,1	*1.2	- 0.3
9,0,$\bar{1}$	13.8	-13.0
9,0,$\bar{3}$	*1.2	- 2.3
9,0,$\bar{5}$	3.9	- 3.5
9,0,$\bar{7}$	*1.2	+ 0.6
9,0,$\bar{9}$	8.3	- 8.7
9,0,$\overline{11}$	*1.2	- 1.9
9,0,$\overline{13}$	*1.1	- 0.2
9,0,$\overline{15}$	5.0	+ 4.5
10,0,10	*1.0	+ 1.2
10,0,8	*1.2	+ 0.4
10,0,6	6.8	- 5.6
10,0,4	2.6	+ 2.2
10,0,2	*1.2	- 2.4
10,0,0	*1.2	+ 1.3
10,0,$\bar{2}$	15.1	-14.9
10,0,$\bar{4}$	*1.2	+ 0.9
10,0,$\bar{6}$	3.3	+ 0.8
10,0,$\bar{8}$	3.9	+ 3.8
10,0,$\overline{10}$	2.6	- 2.2
10,0,$\overline{12}$	*1.2	+ 0.3
10,0,$\overline{14}$	3.6	+ 3.8
11,0,9	*1.0	+ 1.0
11,0,7	*1.2	- 0.5
11,0,5	13.9	-12.3
11,0,3	*1.2	+ 0.6
11,0,1	*1.2	+ 1.9
11,0,$\bar{1}$	*1.2	+ 0.7
11,0,$\bar{3}$	3.7	- 4.8
11,0,$\bar{5}$	*1.0	+ 0.2
11,0,$\bar{7}$	10.3	+10.9
11,0,$\bar{9}$	4.1	+ 3.9
11,0,$\overline{11}$	2.5	+ 1.3
11,0,$\overline{13}$	4.0	- 3.0
12,0,8	*0.8	- 0.6
12,0,6	*1.0	- 1.4
12,0,4	4.6	- 2.5
12,0,2	*1.0	+ 0.2
12,0,0	*1.0	
12,0,$\bar{2}$	2.6	- 2.3
12,0,$\bar{4}$	*1.0	+ 0.6
12,0,$\bar{6}$	*1.0	- 1.0
12,0,$\bar{8}$	9.3	+ 9.1
12,0,$\overline{10}$	*0.7	- 0.3
12,0,$\overline{12}$	*0.8	- 1.1
13,0,5	*0.8	- 0.5
13,0,3	*0.9	+ 0.5
13,0,1	2.0	- 0.8
13,0,$\bar{1}$	6.3	- 4.6
13,0,$\bar{3}$	*0.9	- 0.7
13,0,$\bar{5}$	*0.9	- 0.4
13,0,$\bar{7}$	2.6	- 2.9
13,0,$\bar{9}$	*0.8	+ 0.5
14,0,2	*0.8	- 0.5
14,0,0	*0.8	
14,0,$\bar{2}$	*0.8	+ 0.4
14,0,$\bar{4}$	*0.8	+ 0.1
14,0,$\bar{6}$	4.6	- 5.2
14,0,$\bar{8}$	*0.7	- 0.4
0,1,1	48.3	+53.0
0,1,2	3.1	+ 2.8
0,1,3	9.1	+ 8.9
0,1,4	3.4	+ 3.1
0,1,5	7.9	+ 9.2
0,1,6	8.0	- 8.5
0,1,7	14.9	-15.0
0,1,8	2.0	- 1.5
0,1,9	3.6	+ 4.1
0,1,10	1.0	+ 1.2
0,1,11	2.3	+ 1.6
0,1,12	2.1	- 3.4
0,1,13	14.2	-14.9
0,1,14	3.1	- 3.5
0,1,15	*1.0	- 0.2
0,1,16	2.2	- 1.1
1,1,16	*0.9	+ 0.5
1,1,15	4.8	+ 5.6
1,1,14	2.6	+ 2.7
1,1,13	4.4	- 6.2
1,1,12	*1.3	+ 0.3
1,1,11	3.1	+ 2.1
1,1,10	*1.2	- 0.7
1,1,9	2.8	- 3.0
1,1,8	9.2	- 8.7
1,1,7	17.1	+18.3
1,1,6	10.0	-10.5
1,1,5	0.5	- 0.1
1,1,4	5.1	- 4.8
1,1,3	14.2	+15.3
1,1,2	23.1	-26.0
1,1,1	12.9	-14.4
1,1,0	63.6	+75.2
1,1,$\bar{1}$	23.5	-24.5
1,1,$\bar{2}$	20.1	-21.0
1,1,$\bar{3}$	5.2	+ 4.9
1,1,$\bar{4}$	12.7	-13.4
1,1,$\bar{5}$	15.4	+16.6
1,1,$\bar{6}$	35.9	+38.4
1,1,$\bar{7}$	4.8	- 5.5
1,1,$\bar{8}$	1.3	+ 1.6
1,1,$\bar{9}$	3.4	- 4.5
1,1,$\overline{10}$	5.0	- 5.6
1,1,$\overline{11}$	*1.6	- 0.9
1,1,$\overline{12}$	3.1	- 3.4
1,1,$\overline{13}$	9.5	+ 9.5
1,1,$\overline{14}$	3.7	- 5.2
1,1,$\overline{15}$	*1.5	- 0.5
1,1,$\overline{16}$	*1.4	+ 0.1
1,1,$\overline{17}$		- 1.3
2,1,16	*1.2	+ 1.0
2,1,15	5.9	+ 6.2
2,1,14	12.1	+13.2
2,1,13	1.7	+ 1.6
2,1,12	1.8	- 2.9
2,1,11	2.5	+ 3.2
2,1,10	*1.7	- 0.5
2,1,9	1.3	- 0.6
2,1,8	4.1	+ 5.1
2,1,7	23.9	-24.3
2,1,6	27.5	+29.7
2,1,5	*1.0	+ 0.8
2,1,4	2.1	+ 3.4
2,1,3	9.0	- 8.1
2,1,2	88.7	-98.3
2,1,1	79.7	-85.2
2,1,0	7.3	- 7.0
2,1,$\bar{1}$	14.9	-15.4
2,1,$\bar{2}$	*0.8	- 0.3
2,1,$\bar{3}$	21.4	+20.6
2,1,$\bar{4}$	1.8	+ 1.7
2,1,$\bar{5}$	19.7	+19.8
2,1,$\bar{6}$	22.1	-23.0
2,1,$\bar{7}$	8.2	+ 8.3
2,1,$\bar{8}$	3.4	- 3.3
2,1,$\bar{9}$	*1.4	+ 1.0
2,1,$\overline{10}$	5.7	+ 5.8
2,1,$\overline{11}$	15.8	+14.8
2,1,$\overline{12}$	3.6	+ 3.9
2,1.$\overline{13}$	1.7	- 1.9
2,1,$\overline{14}$	2.5	+ 3.5
2,1,$\overline{15}$	*1.4	+ 0.3
2,1,$\overline{16}$	*1.2	0
3,1,16		+ 0.1
3,1,15	5.8	+ 5.6
3,1,14	2.9	+ 3.2
3,1,13	3.4	+ 4.9
3,1,12	*1.9	- 1.5
3,1,11	*1.9	+ 0.8
3,1,10	2.9	- 3.6
3,1,9	2.0	- 3.6
3,1,8	8.5	+ 7.6
3,1,7	8.4	+ 7.7
3,1,6	3.8	- 3.2
3,1,5	9.4	- 8.6
3,1,4	9.2	- 9.3
3,1,3	1.7	- 0.7
3,1,2	53.7	-56.4
3,1,1	94.6	-101.9
3,1,0	11.5	+11.4
3,1,$\bar{1}$	3.2	+ 2.6
3,1,$\bar{2}$	9.6	+ 9.4
3,1,$\bar{3}$	4.9	+ 4.8
3,1,$\bar{4}$	14.9	-14.9
3,1,$\bar{5}$	3.6	+ 3.3
3,1,$\bar{6}$	17.6	+16.8
3,1,$\bar{7}$	8.9	- 8.5
3,1,$\bar{8}$	1.9	+ 2.5
3,1,$\bar{9}$	3.0	+ 3.0
3,1,$\overline{10}$	*1.4	+ 1.0
3,1,$\overline{11}$	12.9	+15.5
3,1,$\overline{12}$	15.7	+16.8
3,1,$\overline{13}$	1.7	+ 2.0
3,1,$\overline{14}$	*1.4	- 0.4
3,1,$\overline{15}$	1.0	- 0.8
3,1,$\overline{16}$	2.9	- 1.5
3,1,$\overline{17}$	0.6	+ 0.9
4,1,16		- 3.7
4,1,15	1.9	- 1.5
4,1,14	4.9	+ 4.8
4,1,13	2.5	- 3.6
4,1,12	*1.7	- 1.5
4,1,11	*1.9	+ 1.1
4,1,10	*1.2	- 0.9
4,1,9	9.5	+ 9.6
4,1,8	10.0	-10.6
4,1,7	1.4	+ 1.3
4,1,6	*1.0	+ 0.3
4,1,5	2.5	- 2.4
4,1,4	2.1	+ 2.7
4,1,3	30.8	+32.4
4,1,2	1.4	+ 0.4
4,1,1	5.0	- 5.0
4,1,0	*0.7	+ 0.2
4,1,$\bar{1}$	8.4	+ 7.4
4,1,$\bar{2}$	6.1	- 6.4
4,1,$\bar{3}$	5.4	+ 6.0
4,1,$\bar{4}$	20.4	+20.4
4,1,$\bar{5}$	30.1	-28.3
4,1,$\bar{6}$	6.6	+ 7.2
4,1,$\bar{7}$	*1.0	- 0.6
4,1,$\bar{8}$	10.3	+ 9.8
4,1,$\bar{9}$	1.6	- 2.4
4,1,$\overline{10}$	8.6	- 9.8
4,1,$\overline{11}$	2.5	+ 2.4
4,1,$\overline{12}$	3.4	+ 4.9
4,1,$\overline{13}$	3.9	- 2.2
4,1,$\overline{14}$	1.3	+ 0.4
4,1,$\overline{15}$	*1.0	- 0.7
4,1,$\overline{16}$	*0.9	- 0.2
4,1,$\overline{17}$	2.1	+ 3.3
5,1,15	*1.2	- 5.6
5,1,14	*1.4	- 0.1
5,1,13	*1.6	+ 0.4
5,1,12	*1.7	+ 0.8
5,1,11	*1.7	+ 0.1
5,1,10	1.6	- 1.6
5,1,9	2.1	- 2.6
5,1,8	7.0	+ 7.6
5,1,7	10.5	- 9.9
5,1,6	1.9	+ 2.1
5,1,5	*1.0	+ 0.5
5,1,4	13.2	+12.8
5,1,3	66.4	+66.4
5,1,2	18.1	+18.4
5,1,1	5.5	- 4.4
5,1,0	8.2	+ 8.3
5,1,$\bar{1}$	1.7	+ 1.1
5,1,$\bar{2}$	10.5	-10.6
5,1,$\bar{3}$	3.2	- 2.4
5,1,$\bar{4}$	15.8	-15.9
5,1,$\bar{5}$	29.8	+26.4
5,1,$\bar{6}$	4.3	- 4.8
5,1,$\bar{7}$	2.3	- 1.8
5,1,$\bar{8}$	*1.1	- 0.9
5,1,$\bar{9}$	14.3	-14.8
5,1,$\overline{10}$	19.9	-20.6
5,1,$\overline{11}$	10.4	-11.9
5,1,$\overline{12}$	*1.2	+ 0.5
5,1,$\overline{13}$	1.4	- 1.6
5,1,$\overline{14}$	2.4	+ 1.8
5,1,$\overline{15}$	*1.0	+ 1.0
5,1,$\overline{16}$	*0.9	
5,1,$\overline{17}$	0.8	- 1.3
6,1,14	*1.3	- 0.9
6,1,13	*1.5	0
6,1,12	*1.0	- 0.2
6,1,11	2.0	- 1.6
6,1,10	5.9	- 4.1
6,1,9	3.2	- 2.8
6,1,8	1.5	- 1.1
6,1,7	1.7	+ 0.6
6,1,6	2.7	+ 2.6
6,1,5	*1.0	- 0.2
6,1,4	2.2	+ 2.4
6,1,3	7.3	+ 7.2
6,1,2	43.9	+42.2
6,1,1	12.6	-12.7
6,1,0	1.2	- 0.5
6,1,$\bar{1}$	5.4	- 5.5
6,1,$\bar{2}$	6.5	- 7.0
6,1,$\bar{3}$	18.9	+16.7
6,1,$\bar{4}$	*0.9	+ 0.6
6,1,$\bar{5}$	6.9	- 7.5
6,1,$\bar{6}$	2.1	+ 3.7
6,1,$\bar{7}$	3.2	- 3.0
6,1,$\bar{8}$	1.5	+ 1.9
6,1,$\bar{9}$	1.6	- 1.3
6,1,$\overline{10}$	25.3	-27.0
6,1,$\overline{11}$	2.8	- 3.6
6,1,$\overline{12}$	*1.1	- 1.8
6,1,$\overline{13}$	*1.1	+ 0.3
6,1,$\overline{14}$	*1.1	+ 0.6
6,1,$\overline{15}$	*0.9	+ 0.6
7,1,13	*1.2	+ 0.3
7,1,12	*1.4	- 0.3
7,1,11	1.3	- 0.3
7,1,10	3.0	- 3.9
7,1,9	5.0	- 4.5
7,1,8	*1.1	+ 0.7
7,1,7	1.3	- 1.3
7,1,6	*1.1	- 0.3
7,1,5	2.8	- 2.9
7,1,4	6.6	- 7.5
7,1,3	4.5	+ 5.2
7,1,2	8.4	- 8.5
7,1,1	2.3	- 1.8
7,1,0	4.6	+ 4.9
7,1,$\bar{1}$	2.7	+ 1.5
7,1,$\bar{2}$	6.2	+ 5.4
7,1,$\bar{3}$	0.7	- 2.9
7,1,$\bar{4}$	10.3	+ 9.1
7,1,$\bar{5}$	1.7	+ 1.1
7,1,$\bar{6}$	1.0	- 1.0
7,1,$\bar{7}$	6.0	- 4.9
7,1,$\bar{8}$	7.0	+ 7.3
7,1,$\bar{9}$	11.5	+11.5
7,1,$\overline{10}$	*1.1	+ 0.1
7,1,$\overline{11}$	7.9	- 9.0
7,1,$\overline{12}$	4.1	+ 3.2
7,1,$\overline{13}$	*1.0	- 0.6
7,1,$\overline{14}$	*0.9	+ 0.4
7,1,$\overline{15}$	1.0	+ 1.3
7,1,$\overline{16}$	4.3	- 4.0
8,1,12		+ 1.3
8,1,11	*1.8	+ 1.2
8,1,10	2.8	- 2.9
8,1,9	1.5	+ 1.4
8,1,8	2.7	+ 1.8
8,1,7	1.1	- 0.8
8,1,6	*1.4	- 0.5
8,1,5	1.5	- 1.8
8,1,4	17.8	-17.0
8,1,3	3.9	+ 4.7
8,1,2	4.6	+ 3.2
8,1,1	1.1	+ 1.7
8,1,0	2.4	+ 2.6
8,1,$\bar{1}$	3.5	- 3.3
8,1,$\bar{2}$	1.5	- 2.5
8,1,$\bar{3}$	5.8	+ 4.8
8,1,$\bar{4}$	2.4	- 3.4
8,1,$\bar{5}$	2.5	- 1.0
8,1,$\bar{6}$	2.3	+ 1.6
8.1.$\bar{7}$	1.2	- 0.2
8,1,$\bar{8}$	12.0	+12.0
8,1,$\bar{9}$	8.4	+ 6.4
8,1,$\overline{10}$	4.4	+ 5.7
8,1,$\overline{11}$	*1.4	- 0.2
8,1,$\overline{12}$	1.3	+ 0.8
8,1,$\overline{13}$	4.1	- 2.3
8,1,$\overline{14}$	2.0	- 1.0
9,1,11	8.9	+ 6.9
9,1,10	*1.4	- 0.1
9,1,9	*1.6	- 1.4
9,1,8	*1.6	+ 0.1
9,1,7	*1.7	+ 0.2
9,1,6	*1.9	+ 0.2
9,1,5	*1.9	- 0.9
9,1,4	5.0	+ 6.0
9,1,3	7.2	- 6.6
9,1,2	*1.9	+ 0.9
9,1,1	*1.8	- 0.1
9,1,0	*1.8	- 1.4
9,1,$\bar{1}$	7.0	- 5.7
9,1,$\bar{2}$	10.0	- 8.6
9,1,$\bar{3}$	3.6	- 3.9
9,1,$\bar{4}$	*1.7	+ 0.6
9,1,$\bar{5}$	*1.7	- 2.7
9,1,$\bar{6}$	*1.9	+ 0.2
9,1,$\bar{7}$	3.3	- 2.6
9,1,$\bar{8}$	*1.9	- 1.0
9,1,$\bar{9}$	16.5	+15.7
9,1,$\overline{10}$	4.8	- 4.0
9,1,$\overline{11}$	*1.6	- 0.3
9,1,$\overline{12}$	*1.6	- 0.3
9,1,$\overline{13}$	3.3	- 0.8
10,1,10	6.0	+ 5.6
10,1,9	*1.4	- 0.4
10,1,8	*1.5	+ 0.8
10,1,7	*1.6	- 0.3
10,1,6	2.3	- 2.5
10,1,5	2.9	- 2.8
10,1,4	1.7	- 1.9
10,1,3	1.8	+ 1.8
10,1,2	*1.9	+ 0.8
10,1,1	*1.9	- 1.5
10,1,0	*1.9	+ 1.1
10,1,$\bar{1}$	3.1	- 2.6
10,1,$\bar{2}$	18.6	-17.3
10,1,$\bar{3}$	2.5	+ 2.8
10,1,$\bar{4}$	1.8	- 2.2
10,1,$\bar{5}$	1.8	+ 1.2
10,1,$\bar{6}$	1.8	+ 2.5
10,1,$\bar{7}$	1.8	- 1.5
10,1,$\bar{8}$	3.5	- 3.2
10,1,$\bar{9}$	*1.7	- 1.1
10,1,$\overline{10}$	2.7	+ 3.8
10,1,$\overline{11}$	1.6	+ 0.6
10,1,$\overline{12}$	1.4	+ 1.0
10,1,$\overline{13}$		- 0.1
11,1,9	*1.2	- 0.5
11,1,8	*1.5	+ 0.1
11,1,7	*1.6	- 0.5
11,1,6	3.2	- 2.6
11,1,5	*1.6	- 1.0
11,1,4	2.3	- 2.9
11,1,3	*1.7	- 0.1
11,1,2	*1.7	- 1.2
11,1,1	*1.7	- 0.6
11,1,0	5.0	+ 4.5
11,1,$\bar{1}$	*1.8	+ 1.6
11,1,$\bar{2}$	3.5	+ 3.4
11,1,$\bar{3}$	6.6	- 5.1
11,1,$\bar{4}$	1.7	+ 1.2
11,1,$\bar{5}$	*1.7	- 0.1
11,1,$\bar{6}$	*1.7	+ 0.6
11,1,$\bar{7}$	*1.7	- 0.7
11,1,$\bar{8}$	4.2	+ 3.9
11,1,$\bar{9}$	*1.6	+ 0.2
11,1,$\overline{10}$	*1.6	+ 0.3
11,1,$\overline{11}$	*1.5	+ 0.2
12,1,7	*1.2	+ 0.8
12,1,6	*1.5	- 0.4
12,1,5	4.5	- 3.4
12,1,4	*1.5	- 0.6
12,1,3	*1.6	+ 1.3
12,1,2	*1.6	- 0.2
12,1,1	*1.6	+ 2.2
12,1,0	12.4	+ 9.2
12,1,$\bar{1}$	*1.6	- 1.9
12,1,$\bar{2}$	*1.6	+ 1.1
12,1,$\bar{3}$	*1.6	+ 0.8
12,1,$\bar{4}$	*1.6	+ 2.2
12,1,$\bar{5}$	*1.6	- 0.4
12,1,$\bar{6}$	*1.6	+ 0.3
12,1,$\bar{7}$	*1.6	+ 1.6
12,1,$\bar{8}$	2.8	- 2.4
12,1,$\bar{9}$	*1.6	+ 1.6
13,1,5	*1.3	- 0.1
13,1,4	*1.5	+ 0.6
13,1,3	*1.5	- 0.2
13,1,2	*1.6	- 1.1
13,1,1	*1.6	- 0.6
13,1,0	3.3	- 2.4
13,1,$\bar{1}$	12.8	+ 12.6
13,1,$\bar{2}$	4.4	- 3.5
13,1,$\bar{3}$	*1.5	+ 0.4
13,1,$\bar{4}$	*1.5	- 0.8
13,1,$\bar{5}$	2.6	- 2.9
13,1,$\bar{6}$	2.8	- 2.4
13,1,$\bar{7}$	*1.5	- 0.8
14,1,2	*1.1	- 0.5
14,1,1	*1.1	+ 0.3
14,1,0	*1.2	+ 0.3
14,1,$\bar{1}$	4.9	- 3.3
14,1,$\bar{2}$	3.6	+ 2.5
14,1,$\bar{3}$	*1.2	+ 0.1
0,2,0	47.7	-50.2
0,2,1	10.2	-11.2
0,2,2	4.8	+ 3.8
0,2,3	0.7	+ 0.6
0,2,4	4.5	+ 4.5
0,2,5	21.2	-23.8
0,2,6	13.2	+15.2
0,2,7	12.8	-12.6
0,2,8	13.6	-15.2
0,2,9	4.3	- 4.5
0,2,10	2.5	- 1.9
0,2,11	*1.2	+ 0.8
0,2,12	6.3	+ 7.9
0,2,13	5.4	- 6.3
0,2,14	*1.1	+ 0.3
0,2,15	*1.0	+ 1.1
0,2,16	*0.8	
1,2,16	*1.1	+ 0.1
1,2,15	3.1	+ 2.9
1,2,14	2.0	+ 1.7
1,2,13	2.6	+ 3.4
1,2,12	5.3	- 6.9
1,2,11	2.0	+ 2.7
1,2,10	1.7	- 2.4
1,2,9	1.6	- 1.2
1,2,8	11.7	-11.4
1,2,7	23.9	-26.3
1,2,6	5.6	+ 6.9
1,2,5	7.5	+ 7.9
1,2,4	1.9	- 0.7
1,2,3	2.3	+ 1.1
1,2,2	3.1	+ 3.4
1,2,1	3.8	- 4.1
1,2,0	21.9	+24.2
1,2,$\bar{1}$	34.5	-38.4
1,2,$\bar{2}$	17.6	+17.5
1,2,$\bar{3}$	1.2	+ 1.4
1,2,$\bar{4}$	16.9	+16.7
1,2,$\bar{5}$	35.3	+38.1
1,2,$\bar{6}$	4.1	+ 5.2
1,2,$\bar{7}$	2.9	+ 3.1
1,2,$\bar{8}$	5.9	+ 5.8
1,2,$\bar{9}$	1.6	+ 0.9
1,2,$\overline{10}$	5.0	- 6.1
1,2,$\overline{11}$	2.5	- 4.8
1,2,$\overline{12}$	*1.9	- 1.5
1,2,$\overline{13}$	3.7	+ 4.7
1,2,$\overline{14}$	*1.7	- 1.2
1,2,$\overline{15}$	*1.5	+ 0.2
1,2,$\overline{16}$	*1,2	- 0.7
2,2,16	*1.0	+ 0.9
2,2,15	5.8	+ 5.0
2,2,14	2.7	+ 2.0
2,2,13	*1.7	- 1.0
2,2,12	2.6	+ 3.1
2,2,11	*1.5	- 0.6
2,2,10	*1.5	- 2.0
2,2,9	5.0	+ 4.8
2,2,8	3.5	- 4.1
2,2,7	1.5	+ 2.1
2,2,6	4.1	- 4.2
2,2,5	5.8	+ 6.4
2,2,4	1.5	- 0.2
2,2,3	3.5	+ 4.1
2,2,2	8.8	- 8.2
2,2,1	22.7	-22.4
2,2,0	10.9	-11.2
2,2,$\bar{1}$	18.9	+18.2
2,2,$\bar{2}$	8.5	+ 7.4
2,2,$\bar{3}$	5.2	- 7.6
2,2,$\bar{4}$	12.1	-12.1
2,2,$\bar{5}$	5.1	+ 5.2
2,2,$\bar{6}$	34.7	+36.2
2,2,$\bar{7}$	16.5	-17.0
2,2,$\bar{8}$	*1.4	- 1.2
2,2,$\bar{9}$	2.2	- 1.9
2,2,$\overline{10}$	1.4	- 2.0
2,2,$\overline{11}$	1.4	- 1.8
2,2,$\overline{12}$	1.4	- 2.2
2,2,$\overline{13}$	*1.5	- 0.2
2,2,$\overline{14}$	1.3	+ 1.7
2,2,$\overline{15}$	1.9	- 1.0
2,2,$\overline{16}$	*1.0	+ 0.9
3,2,15	*1.2	+ 1.4
3,2,14	7.6	+ 7.9
3,2,13	4.8	- 4.5
3,2,12	1.8	- 0.8
3,2,11	*1.4	- 0.1
3,2,10	2.2	+ 2.1
3,2,9	6.0	+ 6.5
3,2,8	6.2	+ 5.8
3,2,7	2.6	- 2.1
3,2,6	4.7	+ 3.8
3,2,5	7.8	- 7.8
3,2,4	3.6	- 3.1
3,2,3	0.8	+ 1.2
3,2,2	23.6	-24.7
3,2,1	25.9	+27.3
3,2,0	11.8	-12.8
3,2,$\bar{1}$	3.2	- 2.0
3,2,$\bar{2}$	3.5	- 3.6
3,2,$\bar{3}$	10.9	-10.2
3,2,$\bar{4}$	23.6	-23.4
3,2,$\bar{5}$	10.4	- 9.7
3,2,$\bar{6}$	7.6	- 8.8
3,2,$\bar{7}$	6.4	+ 8.4
3,2,$\bar{8}$	1.5	+ 1.3
3,2,$\bar{9}$	2.6	+ 3.8
3,2,$\overline{10}$	*1.3	+ 1.3
3,2,$\overline{11}$	3.8	- 6.2
3,2,$\overline{12}$	3.3	+ 5.4
3,2,$\overline{13}$	1.7	- 1.9
3,2,$\overline{14}$	*1.3	+ 0.4
3,2,$\overline{15}$	0.9	- 0.6
3,2,$\overline{16}$	3.4	+ 2.7
4,2,15	*1.1	- 1.6
4,2,14	*1.5	- 1.0
4,2,13	*1.6	+ 1.2
4,2,12	*1.7	0
4,2,11	*1.2	+ 1.1

Table 1 (*cont.*)

hkℓ	$\lvert F\rvert_o$	F_c
4,2,10	1.4	+ 1.9
4,2,9	5.7	+ 4.0
4,2,8	5.1	- 4.8
4,2,7	1.7	- 2.1
4,2,6	1.6	- 1.0
4,2,5	2.3	+ 0.6
4,2,4	13.6	+12.6
4,2,3	27.0	+26.8
4,2,2	12.5	+13.5
4,2,1	29.9	-31.5
4,2,0	25.1	+26.7
4,2,$\bar{1}$	4.6	- 4.5
4,2,$\bar{2}$	2.6	- 3.0
4,2,$\bar{3}$	4.0	- 3.1
4,2,$\bar{4}$	29.1	-26.8
4,2,$\bar{5}$	2.0	- 2.2
4,2,$\bar{6}$	6.1	+ 5.9
4,2,$\bar{7}$	2.6	- 3.5
4,2,$\bar{8}$	*1.1	+ 2.0
4,2,$\bar{9}$	*1.2	+ 0.9
4,2,$\bar{10}$	4.2	- 2.8
4,2,$\bar{11}$	*1.2	+ 2.4
4,2,$\bar{12}$	8.9	-10.1
4,2,$\bar{13}$	3.9	+ 4.2
4,2,$\bar{14}$	*1.1	- 0.2
4,2,$\bar{15}$	*1.1	+ 0.6
4,2,$\bar{16}$	1.5	+ 2.0
5,2,14	*1.2	+ 0.6
5,2,13	*1.3	+ 0.1
5,2,12	*1.6	- 1.3
5,2,11	*1.7	+ 0.9
5,2,10	2.3	- 2.4
5,2,9	2.8	- 2.1
5,2,8	8.2	+ 8.6
5,2,7	6.2	- 8.1
5,2,6	3.7	+ 3.8
5,2,5	1.9	+ 0.6
5,2,4	14.8	+12.6
5,2,3	35.0	+35.8
5,2,2	27.0	+25.0
5,2,1	4.5	+ 4.4
5,2,0	3.6	- 2.0
5,2,$\bar{1}$	11.0	-11.6
5,2,$\bar{2}$	*1.0	- 0.3
5,2,$\bar{3}$	4.9	+ 3.8
5,2,$\bar{4}$	5.4	- 5.1
5,2,$\bar{5}$	*1.0	+ 1.1
5,2,$\bar{6}$	*1.0	+ 1.0
5,2,$\bar{7}$	*1.1	- 0.3
5,2,$\bar{8}$	2.8	- 2.2
5,2,$\bar{9}$	8.8	- 8.6
5,2,$\bar{10}$	14.4	-14.4
5,2,$\bar{11}$	2.3	- 2.4
5,2,$\bar{12}$	1.4	+ 2.2
5,2,$\bar{13}$	1.6	- 2.2
5,2,$\bar{14}$	*1.0	+ 0.5
5,2,$\bar{15}$	*1.0	- 1.2
6,2,14	*1.4	+ 1.2
6,2,13	*1.5	+ 0.1
6,2,12	*0.9	+ 0.1
6,2,11	*1.1	+ 0.6
6,2,10	1.3	+ 1.0
6,2,9	2.7	- 2.1
6,2,8	4.7	- 5.6
6,2,7	4.0	+ 4.5
6,2,6	5.8	+ 6.1
6,2,5	4.7	- 4.9
6,2,4	1.6	- 1.9
6,2,3	25.7	+24.4
6,2,2	6.2	- 6.9
6,2,1	3.9	- 3.6
6,2,0	*0.9	- 0.4
6,2,$\bar{1}$	1.7	- 1.0
6,2,$\bar{2}$	5.4	+ 5.0
6,2,$\bar{3}$	8.4	+ 7.7
6,2,$\bar{4}$	1.1	- 0.3
6,2,$\bar{5}$	6.6	- 5.5
6,2,$\bar{6}$	*1.0	- 1.7
6,2,$\bar{7}$	6.4	- 4.9
6,2,$\bar{8}$	2.2	+ 1.5
6,2,$\bar{9}$	2.0	- 2.1
6,2,$\bar{10}$	3.7	- 2.4
6,2,$\bar{11}$	7.6	- 9.7
6,2,$\bar{12}$	2.5	+ 2.1
6,2,$\bar{13}$	*1.0	0
6,2,$\bar{14}$	2.3	+ 1.4
6,2,$\bar{15}$	0.7	- 0.9
6,2,$\bar{16}$	3.0	- 3.0
7,2,13	*1.2	- 1.1
7,2,12	*1.5	- 0.1
7,2,11	*0.9	- 0.3
7,2,10	7.1	- 6.4
7,2,9	10.3	+12.1
7,2,8	1.2	-·1.1
7,2,7	1.5	- 0.6
7,2,6	4.3	- 4.0
7,2,5	18.2	-17.6
7,2,4	22.4	-21.5
7,2,3	3.4	- 4.1
7,2,2	9.9	+ 8 8
7,2,1	5.3	- 4.8
7,2,0	1.6	+ 1.4
7,2,$\bar{1}$	2.4	+ 3.0
7,2,$\bar{2}$	1.6	- 2.5
7,2,$\bar{3}$	5.9	- 7.1
7,2,$\bar{4}$	8.3	+ 9.1
7,2,$\bar{5}$	3.8	- 4.1
7,2,$\bar{6}$	1.5	+ 2.3
7,2,$\bar{7}$	*1.9	- 0.2
7,2,$\bar{8}$	13.1	+11.7
7,2,$\bar{9}$	11.0	+10.5
7,2,$\bar{10}$	3.0	- 3.5
7,2,$\bar{11}$	7.3	+ 7.2
7,2,$\bar{12}$	*1.0	- 0.8
7,2,$\bar{13}$	*1.0	- 0.9
7,2,$\bar{14}$	*0.9	0
7,2,$\bar{15}$	2.7	- 1.5
8,2,12		+ 0.8
8,2,11	2.4	+ 1.6
8,2,10	5.4	+ 4.5
8,2,9	8.3	- 9.7
8,2,8	3.6	+ 4.6
8,2,7	1.4	- 1.9
8,2,6	1.3	- 0.2
8,2,5	10.2	- 9.5

hkℓ	$\lvert F\rvert_o$	F_c
8,2,4	20.3	-19.7
8,2,3	7.1	- 6.8
8,2,2	1.3	+ 1.9
8,2,1	1.9	- 3.2
8,2,0	3.5	+ 5.4
8,2,$\bar{1}$	1.7	+ 2.2
8,2,$\bar{2}$	1.7	- 1.5
8,2,$\bar{3}$	11.6	+12.4
8,2,$\bar{4}$	19.8	-20.1
8,2,$\bar{5}$	9.5	+ 7.9
8,2,$\bar{6}$	1.3	+ 0.6
8,2,$\bar{7}$	3.5	+ 2.7
8,2,$\bar{8}$	17.5	+17.2
8,2,$\bar{9}$	17.9	+17.3
8,2,$\bar{10}$	2.0	+ 1.9
8,2,$\bar{11}$	2.1	- 1.8
8,2,$\bar{12}$	*1.2	- 0.4
8,2,$\bar{13}$	2.6	- 1.7
8,2,$\bar{14}$	1.0	+ 0.7
9,2,11	*1.0	- 1.3
9,2,10	2.1	+ 1.2
9,2,9	4.1	+ 3.7
9,2,8	1.6	- 1.3
9,2,7	*1.7	- 1.6
9,2,6	4.7	+ 4.6
9,2,5	*1.9	- 0.1
9,2,4	8.2	- 8.2
9,2,3	*1.8	+ 1.4
9,2,2	2.6	+ 2.7
9,2,1	*1.9	- 0.4
9,2,0	*1.9	+ 2.8
9,2,$\bar{1}$	2.5	- 2.6
9,2,$\bar{2}$	10.1	- 9.3
9,2,$\bar{3}$	*1.8	- 0.7
9,2,$\bar{4}$	10.1	+ 8.8
9,2,$\bar{5}$	*1.9	+ 1.1
9,2,$\bar{6}$	*1.9	- 0.8
9,2,$\bar{7}$	6.3	- 5.8
9,2,$\bar{8}$	*1.9	+ 1.3
9,2,$\bar{9}$	6.3	+ 5.7
9,2,$\bar{10}$	*1.7	+ 3.6
9,2,$\bar{11}$	*1.6	- 1.1
10,2,10	*1.0	- 5.3
10,2,9	*1.4	+ 0.6
10,2,8	*1.6	- 0.1
10,2,7	3.0	+ 2.3
10,2,6	8.2	+ 7.2
10,2,5	6.9	+ 6.1
10,2,4	*1.8	- 0.6
10,2,3	*1.9	- 0.6
10,2,2	*1.9	- 2.0
10,2,1	2.6	- 1.9
10,2,0	*1.9	- 1.0
10,2,$\bar{1}$	4.6	- 3.0
10,2,$\bar{2}$	13.9	+13.6
10,2,$\bar{3}$	10.3	-10.5
10,2,$\bar{4}$	*1.9	+ 0.6
10,2,$\bar{5}$	*1.9	- 1.9
10,2,$\bar{6}$	2.5	- 2.4
10,2,$\bar{7}$	10.6	-10.6
10,2,$\bar{8}$	7.7	- 6.5
10,2,$\bar{9}$	3.4	+ 2.7
10,2,$\bar{10}$	2.4	- 1.5
10,2,$\bar{11}$	*0.7	0
11,2,8		+ 0.2
11,2,7		0
11,2,6	2.7	+ 2.0
11,2,5	3.4	+ 2.6
11,2,4	*1.6	+ 0.5
11,2,3	*1.7	- 1.0
11,2,2	*1.7	+ 1.1
11,2,1	*1.7	+ 0.7
11,2,0	4.3	+ 3.0
11,2,$\bar{1}$	1.9	+ 2.3
11,2,$\bar{2}$	11.5	-11.2
11,2,$\bar{3}$	12.7	+12.7
11,2,$\bar{4}$	2.5	- 2.9
11,2,$\bar{5}$	*1.7	+ 0.7
11,2,$\bar{6}$	*1.7	- 0.3
11,2,$\bar{7}$	11.4	-10.2
11,2,$\bar{8}$	8.4	- 7.0
11,2,$\bar{9}$	*1.7	+ 0.7
12,2,6	*1.2	+ 0.1
12,2,5	3.4	+ 2.2
12,2,4	*1.5	- 1.1
12,2,3	*1.5	+ 0.3
12,2,2	*1.5	+ 0.4
12,2,1	*1.6	- 0.1
12,2,0	2.4	+ 2.5
12,2,$\bar{1}$	10.1	+ 8.1
12,2,$\bar{2}$	*1.7	+ 1.3
12,2,$\bar{3}$	5.2	- 3.6
12,2,$\bar{4}$	*1.6	- 1.2
12,2,$\bar{5}$	1.6	+ 1.6
12,2,$\bar{6}$	4.3	+ 3.0
12,2,$\bar{7}$	*1.5	- 0.8
12,2,$\bar{8}$	2.1	- 1.9
12,2,$\bar{9}$	*1.4	- 0.6
13,2,4		+ 0.7
13,2,3		+ 0.9
13,2,2	*1.4	- 0.4
13,2,1	1.8	- 0.4
13,2,0	6.3	+ 3.6
13,2,$\bar{1}$	7.7	- 4.9
13,2,$\bar{2}$	4.7	+ 3.3
13,2,$\bar{3}$	*1.5	+ 0.1
13,2,$\bar{4}$	*1.5	+ 0.3
13,2,$\bar{5}$	*1.5	+ 1.0
13,2,$\bar{6}$	3.3	+ 2.7
0,3,1	10.2	-10.3
0,3,2	6.6	+ 6.8
0,3,3	3.3	+ 1.7
0,3,4	5.6	- 4.6
0,3,5	2.5	- 3.7
0,3,6	1.8	- 0.3
0,3,7	2.4	- 1.7
0,3,8	7.5	- 8.4
0,3,9	1.1	- 0.2
0,3,10	*1.1	+ 0.1
0,3,11	1.1	- 1.4
0,3,12	2.6	+ 0.8
0,3,13	4.3	+ 5.4
0,3,14	1.9	+ 1.3

hkℓ	$\lvert F\rvert_o$	F_c
0,3,15	1.5	- 1.1
0.3,16	*1.2	
1,3,15	2.2	- 2.5
1,3,14	1.2	- 1.5
1,3,13	1.7	- 2.2
1,3,12	3.8	+ 4.6
1,3,11	1.8	- 2.6
1,3,10	*1.3	+ 1.5
1,3,9	*1.3	- 0.5
1,3,8	8.2	- 9.2
1,3,7	15.3	-13.4
1,3,6	3.0	+ 3.1
1,3,5	5.8	+ 6.7
1,3,4	3.5	+ 2.4
1,3,3	10.7	+ 9.9
1,3,2	22.5	+23.1
1,3,1	6.3	+ 7.8
1,3,0	23.8	-25.4
1,3,$\bar{1}$	29.3	+32.2
1,3,$\bar{2}$	5.6	- 6.4
1,3,$\bar{3}$	*1.0	+ 0.8
1,3,$\bar{4}$	1.4	+ 1.5
1,3,$\bar{5}$	*1.9	+ 0.5
1,3,$\bar{6}$	4.2	+ 4.9
1,3,$\bar{7}$	1.2	+ 2.0
1,3,$\bar{8}$	*1.2	- 2.5
1,3,$\bar{9}$	*1.3	+ 2.0
1,3,$\bar{10}$	2.1	- 3.6
1,3,$\bar{11}$	5.2	- 6.3
1,3,$\bar{12}$	*2.7	+ 0.3
1,3,$\bar{13}$	4.3	- 4.9
1,3,$\bar{14}$	3.1	+ 3.9
1,3,$\bar{15}$	*1.9	- 1.0
1,3,$\bar{16}$	*1.5	- 0.1
2,3,15	1.5	- 0.9
2,3,14	*2.0	- 1.8
2,3,13	*2.3	- 0.4
2,3,12	1.6	- 1.6
2,3,11	*1.4	- 1.7
2,3,10	4.0	+ 5.5
2,3,9	5.2	+ 5.8
2,3,8	*1.5	+ 0.8
2,3,7	7.9	+ 8.0
2,3,6	12.4	-14.4
2,3,5	5.6	+ 5.5
2,3,4	2.2	- 2.8
2,3,3	11.1	+11.3
2,3,2	26.2	+29.3
2,3,1	16.5	+15.5
2,3,0	8.0	+ 9.1
2,3,$\bar{1}$	16.6	-17.1
2,3,$\bar{2}$	1.9	- 1.7
2,3,$\bar{3}$	7.3	- 5.8
2,3,$\bar{4}$	8.5	- 8.8
2,3,$\bar{5}$	3.7	+ 3.3
2,3,$\bar{6}$	3.5	+ 6.1
2,3,$\bar{7}$	5.0	- 5.5
2,3,$\bar{8}$	1.6	+ 0.8
2,3,$\bar{9}$	1.4	- 2.4
2,3,$\bar{10}$	4.5	- 6.6
2,3,$\bar{11}$	8.0	- 9.3
2,3,$\bar{12}$	2.0	- 2.5
2,3,$\bar{13}$	3.1	+ 3.7
2,3,$\bar{14}$	4.2	- 4.3
2,3,$\bar{15}$	1.9	+ 1.7
2,3,$\bar{16}$	*0.7	- 0.2
3,3,15	*1.3	- 0.2
3,3,14	*1.9	- 1.1
3,3,13	*2.3	+ 0.4
3,3,12	*1.3	+ 0.5
3,3,11	2.6	+ 3.2
3,3,10	6.1	+ 5.2
3,3,9	10.5	+11.1
3,3,8	2.2	+ 2.0
3,3,7	11.5	-13.1
3,3,6	16.6	+18.9
3,3,5	3.2	- 4.3
3,3,4	5.2	- 4.7
3,3,3	1.9	- 2.1
3,3,2	5.8	+ 5.2
3,3,1	1.6	- 0.8
3,3,0	3.4	+ 4.1
3,3,$\bar{1}$	*1.1	- 0.9
3,3,$\bar{2}$	*1.1	+ 1.7
3,3,$\bar{3}$	15.8	-16.0
3,3,$\bar{4}$	18.3	-18.9
3,3,$\bar{5}$	6.6	- 7.1
3,3,$\bar{6}$	9.1	-10.0
3,3,$\bar{7}$	7.9	+ 7.4
3,3,$\bar{8}$	3.3	- 2.1
3,3,$\bar{9}$	5.4	+ 6.2
3,3,$\bar{10}$	*1.6	+ 1.5
3,3,$\bar{11}$	4.3	- 5.2
3,3,$\bar{12}$	5.1	- 6.6
3,3,$\bar{13}$	1.3	+ 1.8
3,3,$\bar{14}$	0.9	+ 1.9
3,3,$\bar{15}$	2.5	+ 1.5
3,3,$\bar{16}$	1.1	+ 1.2
4,3,14	*1.5	+ 1.3
4,3,13	*2.2	- 0.6
4,3,12	*1.0	- 0.3
4,3,11	*1.2	- 0.7
4,3,10	*1.2	- 0.8
4,3,9	4.4	+ 6.5
4,3,8	15.6	+14.6
4,3,7	6.3	- 6.2
4,3,6	8.5	- 8.2
4,3,5	2.7	- 4.2
4,4,4	3.1	- 2.4
4,3,3	3.6	- 3.8
4,3,2	*1.0	+ 0.6
4,3,1	12.3	+12.1
4,3,0	10.8	-10.1
4,3,$\bar{1}$	3.7	- 3.7
4,3,$\bar{2}$	*1.0	+ 0.2
4,3,$\bar{3}$	10.9	-12.1
4,3,$\bar{4}$	16.4	-16.7
4,3,$\bar{5}$	3.7	+ 5.0
4,3,$\bar{6}$	3.2	+ 3.3
4,3,$\bar{7}$	5.0	- 3.4
4,3,$\bar{8}$	3.2	+ 1.3
4,3,$\bar{9}$	5.7	+ 7.1
4,3,$\bar{10}$	3.7	+ 4.9
4,3,$\bar{11}$	*1.2	- 0.7
4.3,$\bar{12}$	3.5	+ 4.2

hkℓ	$\lvert F\rvert_o$	F_c
4,3,$\bar{13}$	1.0	- 2.5
4,3,$\bar{14}$	*1.0	+ 0.4
4,3,$\bar{15}$	*0.9	0
4,3,$\bar{16}$	*0.6	+ 1.0
5,3,14	*1.2	+ 0.3
5,3,13	*1.9	- 0.4
5,3,12	*2.2	+ 1.3
5,3,11	2.8	- 3.1
5,3,10	3.6	- 3.2
5,3,9	3.7	+ 3.6
5,3,8	15.5	-16.4
5,3,7	12.7	+14.1
5,3,6	*1.2	- 1.1
5,3,5	3.3	+ 4.0
5,3,4	2.0	- 0.3
5,3,3	2.2	- 3.4
5,3,2	2.9	- 2.7
5,3,1	2.6	- 2.5
5,3,0	*1.1	- 0.3
5,3,$\bar{1}$	5.3	+ 6.3
5,3,$\bar{2}$	13.1	+11.9
5,3,$\bar{3}$	12.2	+12.8
5,3,$\bar{4}$	2.0	+ 2.5
5,3,$\bar{5}$	21.8	-25.3
5,3,$\bar{6}$	15.3	+16.3
5,3,$\bar{7}$	3.8	- 4.5
5,3,$\bar{8}$	2.3	+ 2.8
5,3,$\bar{9}$	3.5	+ 4.8
5,3,$\bar{10}$	4.4	+ 4.1
5,3,$\bar{11}$	*1.2	+ 0.3
5,3,$\bar{12}$	2.9	- 3.3
5,3,$\bar{13}$	2.4	+ 1.8
5,3,$\bar{14}$	*1.0	- 1.0
5,3,$\bar{15}$	2.5	- 1.8
6,3,13	*1.3	- 0.3
6,3,12	1.7	- 1.3
6,3,11	3.6	- 3.1
6,3,10	5.1	- 4.4
6,3,9	1.2	+ 0.9
6,3,8	11.2	+11.4
6,3,7	10.6	-10.4
6,3,6	1.2	- 1.8
6,3,5	3.5	- 2.8
6,3,4	3.4	- 3.8
6,3,3	3.2	+ 3.6
6,3,2	*1.1	- 0.1
6,3,1	*2.4	- 1.3
6,3,0	3.8	+ 4.3
6,3,$\bar{1}$	1.9	0
6,3,$\bar{2}$	11.0	+ 9.9
6,3,$\bar{3}$	11.0	+11.8
6,3,$\bar{4}$	*1.1	- 1.4
6,3,$\bar{5}$	16.2	+19.1
6,3,$\bar{6}$	13.8	-14.7
6,3,$\bar{7}$	2.6	- 1.5
6,3,$\bar{8}$	3.4	- 3.8
6,3,$\bar{9}$	*1.3	- 1.5
6,3,$\bar{10}$	4.2	+ 4.3
6,3,$\bar{11}$	2.9	+ 3.0
6,3,$\bar{12}$	0.9	- 1.1
6,3,$\bar{13}$	*1.0	- 0.1
6,3,$\bar{14}$	2.0	- 1.9
6,3,$\bar{15}$	4.1	- 3.9
7,3,12	*1.5	+ 0.1
7,3,11	*0.7	+ 0.5
7,3,10	2.1	+ 1.6
7,3,9	7.2	- 6.4
7,3,8	1.1	+ 2.0
7,3,7	2.5	+ 1.8
7,3,6	2.9	+ 0.9
7,3,5	10.2	-10.2
7,3,4	9.3	- 9.3
7,3,3	2.2	- 1.6
7,3,2	2.0	- 3.2
7,3,1	*1.0	+ 0.3
7,3,0	2.4	+ 3.0
7,3,$\bar{1}$	1.9	- 2.2
7,3,$\bar{2}$	6.9	+ 8.1
7,3,$\bar{3}$	15.5	+15.4
7,3,$\bar{4}$	13.4	-14.9
7,3,$\bar{5}$	3.7	- 3.6
7,3,$\bar{6}$	6.4	+ 3.9
7,3,$\bar{7}$	2.7	+ 3.2
7,3,$\bar{8}$	2.9	+ 1.9
7,3,$\bar{9}$	3.1	+ 2.9
7,3,$\bar{10}$	1.6	+ 1.4
7,3,$\bar{11}$	*1.0	- 1.1
7,3,$\bar{12}$	1.0	- 0.2
7,3,$\bar{13}$	*0.8	0
7,3,$\bar{14}$	*0.8	0
8,3,11	*1.5	+ 0.9
8,3,10	2.0	- 2.7
8,3,9	6.9	+ 7.5
8,3,8	5.9	- 5.7
8,3,7	1.1	- 0.5
8,3,6	1.8	- 1.3
8,3,5	8.1	- 7.8
8,3,4	8.9	- 8.2
8,3,3	*1.4	+ 1.5
8,3,2	1.1	+ 1.7
8,3,1	2.1	+ 2.2
8,3,0	1.1	- 1.5
8,3,$\bar{1}$	6.5	- 6.1
8,3,$\bar{2}$	4.3	- 4.9
8,3,$\bar{3}$	7.2	- 8.3
8,3,$\bar{4}$	19.4	+21.5
8,3,$\bar{5}$	6.4	- 7.2
8,3,$\bar{6}$	3.9	+ 4.2
8,3,$\bar{7}$	*1.4	- 0.5
8,3,$\bar{8}$	3.4	+ 2.2
8,3,$\bar{9}$	4.3	+ 4.2
8,3,$\bar{10}$	*1.3	+ 0.8
8,3,$\bar{11}$	1.0	+ 0.8
8,3,$\bar{12}$	1.0	- 0.1
8,3,$\bar{13}$	0.8	+ 0.8
8,3,$\bar{14}$	2.2	+ 2.3
9,3,10	*1.4	- 0.9
9,3,9	2.8	- 2.6
9,3,8	3.5	+ 1.3
9,3,7	3.0	+ 2.6
9,3,6	8.0	+ 7.1
9,3,5	*2.7	+ 3.0
9,3,4	2.6	- 2.1
9,3,3	*2.7	- 2.0

hkℓ	$\lvert F\rvert_o$	F_c
9,3,2	*2.7	+ 1.8
9,3,1	*2.7	- 2.5
9,3,0	*2.7	- 0.1
9,3,$\bar{1}$	3.5	- 1.7
9,3,$\bar{2}$	2.7	+ 1.0
9,3,$\bar{3}$	3.6	+ 3.9
9,3,$\bar{4}$	10.3	-11.2
9,3,$\bar{5}$	5.9	+ 6.5
9.3.$\bar{6}$	*2.8	- 0.3
9,3,$\bar{7}$	4.7	- 4.0
9,3,$\bar{8}$	*2.7	+ 1.5
9,3,$\bar{9}$	*2.6	+ 0.2
9,3,$\bar{10}$	3.3	- 2.2
9,3,$\bar{11}$	*2.2	+ 0.4
9,3,$\bar{12}$	*1.6	- 0.7
9,3,$\bar{13}$	2.9	+ 0.5
10,3,9	*1.2	+ 0.3
10,3,8	*1.7	+ 1.1
10,3,7	4.4	+ 2.6
10,3,6	10.8	+ 9.1
10,3,5	6.9	+ 5.8
10,3,4	*2.6	- 1.9
10,3,3	2.6	+ 3.8
10,3,2	*2.7	- 1.4
10,3,1	*2.8	- 1.7
10,3,0	*2.8	+ 1.0
10,3,$\bar{1}$	*2.8	- 1.5
10,3,$\bar{2}$	7.6	- 5.7
10,3,$\bar{3}$	6.6	+ 7.0
10,3,$\bar{4}$	*2.8	+ 0.2
10,3,$\bar{5}$	2.9	- 1.4
10,3,$\bar{6}$	7.2	- 6.4
10,3,$\bar{7}$	11.6	-10.3
10,3,$\bar{8}$	5.7	- 5.3
10,3,$\bar{9}$	*2.3	- 1.0
10,3,$\bar{10}$	*2.2	+ 0.9
10,3,$\bar{11}$	*1.9	+ 0.5
10,3,$\bar{12}$	*1.5	+ 0.6
11,3,7	*1.4	- 0.5
11,3,6	4.7	+ 4.5
11,3,5	7.2	+ 5.9
11,3,4	*2.2	- 2.2
11,3,3	*2.3	- 1.5
11,3,2	*2.4	- 0.1
11,3,1	*2.4	- 0.3
11,3,0	*2.6	+ 1.3
11,3,$\bar{1}$	2.5	+ 1.2
11,3,$\bar{2}$	3.6	+ 3.4
11,3,$\bar{3}$	6.3	- 6.1
11,3,$\bar{4}$	2.5	- 0.9
11,3,$\bar{5}$	*2.4	0
11,3,$\bar{6}$	3.1	- 1.4
11,3,$\bar{7}$	6.3	- 5.7
11,3,$\bar{8}$	3.3	- 3.1
11,3,$\bar{9}$	*2.0	0
11,3,$\bar{10}$	*1.7	- 0.2
11,3,$\bar{11}$	*1.4	+ 0.5
12,3,5	1.9	- 1.2
12,3,4	*1.7	+ 2.6
12,3,3	*1.8	- 0.4
12,3,2	*2.0	+ 1.1
12,3,1	*2.2	- 0.3
12,3,0	*2.2	+ 0.5
12,3,$\bar{1}$	*2.2	- 0.9
12,3,$\bar{2}$	*2.2	- 0.7
12,3,$\bar{3}$	*2.2	+ 1.6
12,3,$\bar{4}$	*2.2	+ 0.1
12,3,$\bar{5}$	5.0	+ 4.0
12,3,$\bar{6}$	5.9	+ 5.1
12,3,$\bar{7}$	*1.8	- 0.7
12,3,$\bar{8}$	3.3	- 3.2
12,3,$\bar{9}$	2.6	+ 2.4
13,3,0	*1.7	+ 0.8
13,3,$\bar{1}$	*1.7	- 0.5
13,3,$\bar{2}$	*1.7	- 0.1
13,3,$\bar{3}$	*1.7	+ 0.3
13,3,$\bar{4}$	*1.7	+ 0.6
13,3,$\bar{5}$	*1.7	+ 3.9
13,3,$\bar{6}$	4.2	+ 6.3
0,4,0	*1.1	+ 3.5
0,4,1	4.7	- 4.8
0,4,2	1.6	+ 0.5
0,4,3	3.1	+ 2.6
0,4,4	13.6	-18.2
0,4,5	20.5	+24.5
0,4,6	7.5	- 9.1
0,4,7	1.4	+ 1.7
0,4,8	8.2	+10.7
0,4,9	2.8	+ 1.9
0,4,10	2.6	+ 3.8
0,4,11	1.2	+ 1.4
0,4,12	*1.1	+ 2.2
0,4,13	1.2	+ 1.1
1,4,14	*0.8	- 1.0
1,4,13	*1.0	+ 0.6
1,4,12	1.1	- 1.7
1,4,11	0.8	+ 0.2
1,4,10	*1.3	- 0.4
1,4,9	*1.3	+ 1.1
1,4,8	3.9	+ 5.7
1,4,7	5.6	+ 6.0
1,4,6	4.2	- 4.6
1,4,5	13.7	-15.8
1,4,4	10.2	+11.5
1,4,3	7.4	+ 8.8
1,4,2	14.1	+14.0
1,4,1	4.4	+ 5.9
1,4,0	*1.1	+ 0.4
1,4,$\bar{1}$	6.1	- 5.4
1,4,$\bar{2}$	1.6	- 2.8
1,4,$\bar{3}$	0.9	- 0.3
1,4,$\bar{4}$	1.9	- 1.5
1,4,$\bar{5}$	13.1	-12.2
1,4,$\bar{6}$	1.7	- 0.1
1,4,$\bar{7}$	3.6	+ 4.9
1,4,$\bar{8}$	9.3	-10.9
1,4,$\bar{9}$	6.9	+ 6.0
1,4,$\bar{10}$	1.9	- 1.9
1,4,$\bar{11}$	1.5	- 2.3
1,4,$\bar{12}$	*1.1	+ 0.8
1,4,$\bar{13}$	*1.1	+ 0.8
1,4,$\bar{14}$	*0.9	- 1.5

hkℓ	$\lvert F\rvert_o$	F_c
2,4,14	*1.0	- 1.6
2,4,13	*1.3	+ 0.2
2,4,12	*1.5	+ 0.8
2,4,11	1.3	+ 0.8
2,4,10	1.7	- 1.2
2,4,9	*1.5	0
2,4,8	*1.5	- 1.4
2,4,7	9.2	-10.5
2,4,6	16.6	+18.9
2,4,5	1.7	- 0.4
2,4,4	1.9	+ 0.6
2,4,3	1.6	- 0.8
2,4,2	11.7	+11.5
2,4,1	2.3	+ 1.9
2,4,0	2.2	- 2.8
2,4,$\bar{1}$	5.3	+ 5.7
2,4,$\bar{2}$	0.9	+ 1.4
2,4,$\bar{3}$	7.4	+ 7.7
2,4,$\bar{4}$	8.6	+10.1
2,4,$\bar{5}$	6.3	- 7.1
2,4,$\bar{6}$	14.7	-14.3
2,4,$\bar{7}$	11.5	+12.3
2,4,$\bar{8}$	*1.5	- 2.4
2,4,$\bar{9}$	1.4	- 0.1
2,4,$\bar{10}$	2.1	- 3.7
2,4,$\bar{11}$	2.6	- 3.8
2,4,$\bar{12}$	1.6	- 1.0
2,4,$\bar{13}$	*1.1	- 1.0
2,4,$\bar{14}$	2.5	+ 2.3
3,4,14	*0.6	- 0.2
3,4,13	*1.1	- 0.4
3,4,12	*1.1	- 0.1
3,4,11	1.4	- 1.8
3,4,10	2.2	+ 0.8
3,4,9	3.0	+ 2.0
3,4,8	2.8	+ 2.3
3,4,7	8.4	+ 9.5
3,4,6	17.8	-20.3
3,4,5	6.4	+ 9.0
3,4,4	8.0	- 9.6
3,4,3	3.6	- 3.5
3,4,2	6.2	+ 7.2
3,4,1	1.4	- 1.3
3,4,0	1.7	- 2.7
3,4,$\bar{1}$	1.4	+ 1.8
3,4,$\bar{2}$	3.9	- 4.8
3,4,$\bar{3}$	3.9	+ 5.0
3,4,$\bar{4}$	5.4	+ 5.8
3,4,$\bar{5}$	*1.3	+ 0.4
3,4,$\bar{6}$	13.4	+13.8
3,4,$\bar{7}$	20.9	-22.6
3,4,$\bar{8}$	6.9	+ 5.8
3,4,$\bar{9}$	*1.4	- 0.1
3,4,$\bar{10}$	2.3	+ 2.5
3,4,$\bar{11}$	1.7	+ 0.7
3,4,$\bar{12}$	*1.3	+ 0.3
3,4,$\bar{13}$	*1.2	0
3,4,$\bar{14}$	*0.9	- 0.5
4,4,12	2.5	- 0.5
4,4,11	1.6	- 1.5
4,4,10	*1.1	+ 0.2
4,4,9	1.9	+ 1.2
4,4,8	2.3	- 2.5
4,4,7	1.2	- 1.0
4,4,6	7.0	+ 7.3
4,4,5	7.0	- 4.8
4,4,4	12.2	-13.6
4,4,3	11.9	-11.3
4,4,2	3.1	- 4.2
4,4,1	1.8	- 0.9
4,4,0	*1.2	+ 1.6
4,4,$\bar{1}$	1.8	+ 1.6
4,4,$\bar{2}$	3.0	+ 2.7
4,4,$\bar{3}$	3.0	+ 2.6
4,4,$\bar{4}$	2.6	- 0.2
4,4,$\bar{5}$	8.1	- 8.2
4,4,$\bar{6}$	4.1	- 4.1
4,4,$\bar{7}$	14.6	+14.4
4,4,$\bar{8}$	2.5	- 1.1
4,4,$\bar{9}$	6.5	+ 6.4
4,4,$\bar{10}$	4.5	+ 5.4
4,4,$\bar{11}$	1.7	- 1.6
4,4,$\bar{12}$	*1.0	+ 0.2
4,4,$\bar{13}$	*1.0	+ 2.1
4,4,$\bar{14}$	*0.8	- 0.2
5,4,12	*1.1	- 1.0
5,4,11	3.3	- 3.1
5,4,10	3.1	- 3.3
5,4,9	*1.1	+ 0.1
5,4,8	6.0	+ 7.0
5,4,7	7.8	- 8.8
5,4,6	*1.2	+ 1.8
5,4,5	1.6	- 1.0
5,4,4	3.5	- 3.0
5,4,3	8.3	- 8.1
5,4,2	*1.2	- 0.1
5,4,1	2.3	+ 2.9
5,4,0	*1.2	- 2.2
5,4,$\bar{1}$	3.8	+ 2.0
5,4,$\bar{2}$	5.3	+ 5.1
5,4,$\bar{3}$	1.6	+ 0.8
5,4,$\bar{4}$	*1.2	+ 1.1
5,4,$\bar{5}$	19.4	+16.8
5,4,$\bar{6}$	9.6	- 9.5
5,4,$\bar{7}$	1.7	- 0.3
5,4,$\bar{8}$	1.4	- 2.5
5,4,$\bar{9}$	5.1	+ 6.2
5,4,$\bar{10}$	4.8	+ 5.6
5,4,$\bar{11}$	*1.1	+ 0.8
5,4,$\bar{12}$	*1.0	+ 0.3
5,4,$\bar{13}$	1.6	- 1.3
5,4,$\bar{14}$	1.1	- 0.6
6,4,11	3.8	- 3.0
6,4,10	4.4	- 4.0
6,4,9	1.0	0
6,4,8	1.6	- 2.6
6,4,7	5.3	+ 5.4
6,4,6	0.9	+ 1.6
6,4,5	5.4	+ 5.6
6,4,4	1.7	+ 2.4
6,4,3	2.5	- 3.8
6,4,2	2.5	- 4.1
6,4,1	3.4	+ 4.0
6,4,0	1.0	+ 0.4
6.4.$\bar{1}$	4.2	+ 5.3

Table 1 (*cont.*)

hkℓ	$\|F\|_o$	F_o
6,4,$\bar{2}$	9.4	+ 8.5
6,4,$\bar{3}$	7.7	+ 6.9
6,4,$\bar{4}$	*1.1	+ 0.9
6,4,$\bar{5}$	13.8	-13.8
6,4,$\bar{6}$	12.6	+12.7
6,4,$\bar{7}$	5.6	- 5.6
6,4,$\bar{8}$	3.6	- 4.1
6,4,$\bar{9}$	*1.1	- 0.1
6,4,$\overline{10}$	*1.1	+ 0.3
6,4,$\overline{11}$	0.9	- 0.8
6,4,$\overline{12}$	0.9	- 0.4
6,4,$\overline{13}$	*0.8	- 0.7
6,4,$\overline{14}$	1.1	- 1.8
7,4,10	*0.8	- 1.2
7,4,9	*0.8	+ 0.3
7,4,8	1.0	+ 0.8
7,4,7	1.3	- 0.1
7,4,6	1.6	+ 0.1
7,4,5	3.2	+ 4.1
7,4,4	2.5	+ 2.2
7,4,3	*1.1	0
7,4,2	3.8	+ 0.7
7,4,1	2.9	- 4.6
7,4,0	1.8	- 1.3
7,4,$\bar{1}$	4.9	- 5.0
7,4,$\bar{2}$	1.3	+ 0.5
7,4,$\bar{3}$	1.3	+ 3.2
7,4,$\bar{4}$	4.9	+ 3.7
7,4,$\bar{5}$	1.7	+ 1.4
7,4,$\bar{6}$	4.5	- 4.9
7,4,$\bar{7}$	3.3	- 4.0
7,4,$\bar{8}$	6.4	- 6.2
7,4,$\bar{9}$	3.5	- 3.7
7,4,$\overline{10}$	1.5	+ 1.2
7,4,$\overline{11}$	1.2	+ 1.6
7,4,$\overline{12}$	1.0	- 1.1
7,4,$\overline{13}$	2.1	+ 1.3
8,4,10	0.5	- 0.8
8,4,9	0.8	+ 0.2
8,4,8	*1.0	- 0.6
8,4,7	*1.1	- 1.0
8,4,6	*1.2	+ 0.4
8,4,5	1.8	+ 1.6
8,4,4	3.0	+ 2.6
8,4,3	1.0	- 1.1
8,4,2	3.7	- 4.2
8,4,1	2.2	+ 3.1
8,4,0	7.1	- 5.0
8,4,$\bar{1}$	8.7	- 8.4
8,4,$\bar{2}$	2.6	- 2.5
8,4,$\bar{3}$	2.2	+ 1.2
8,4,$\bar{4}$	5.9	- 5.3
8,4,$\bar{5}$	1.9	+ 2.6
8,4,$\bar{6}$	2.7	+ 2.7
8,4,$\bar{7}$	*1.3	+ 1.3
8,4,$\bar{8}$	2.6	- 2.8
8,4,$\bar{9}$	2.8	- 2.5
8,4,$\overline{10}$	*1.1	0
8,4,$\overline{11}$	1.5	- 1.2
8,4,$\overline{12}$	2.6	+ 1.8
9,4,8	*1.0	+ 1.4
9,4,7	*1.3	- 0.1
9,4,6	1.4	+ 1.7
9,4,5	*1.5	+ 0.1
9,4,4	1.6	- 1.5
9,4,3	4.4	+ 5.4
9,4,2	2.3	- 2.8
9,4,1	1.7	- 0.7
9,4,0	2.3	- 3.2
9,4,$\bar{1}$	6.9	- 6.2
9,4,$\bar{2}$	4.4	- 3.5
9,4,$\bar{3}$	*1.7	+ 1.9
9,4,$\bar{4}$	*1.7	+ 2.8
9,4,$\bar{5}$	*1.7	+ 0.1
9,4,$\bar{6}$	*1.6	+ 1.6
9,4,$\bar{7}$	*1.6	+ 1.7
9,4,$\bar{8}$	2.1	- 1.9
9,4,$\bar{9}$	2.2	- 2.4
9,4,$\overline{10}$	3.4	+ 2.4
9,4,$\overline{11}$	*1.1	0
10,4,6	3.6	+ 3.1
10,4,5	2.7	+ 1.6
10,4,4	*1.3	+ 1.1
10,4,3	4.9	- 4.8
10,4,2	2.6	+ 3.1
10,4,1	1.6	+ 1.1
10,4,0	2.6	+ 3.3
10,4,$\bar{1}$	*1.6	- 1.3
10,4,$\bar{2}$	2.2	- 2.8
10,4,$\bar{3}$	*1.6	+ 1.1
10,4,$\bar{4}$	*1.6	- 0.8
10,4,$\bar{5}$	2.3	- 1.4
10,4,$\bar{6}$	*1.5	- 0.1
10,4,$\bar{7}$	*1.5	+ 0.1
10,4,$\bar{8}$	*1.3	+ 0.2
10,4,$\bar{9}$	*1.3	+ 0.9
10,4,$\overline{10}$	3.6	- 3.2
11,4,5	*0.7	- 0.5
11,4,4	*1.0	- 0.1
11,4,3	2.1	+ 1.9
11,4,2	2.1	- 1.5
11,4,1	2.6	+ 2.0
11,4,0	4.4	+ 3.0
11,4,$\bar{1}$	*1.5	+ 0.1
11,4,$\bar{2}$	*1.5	+ 1.3
11,4,$\bar{3}$	4.8	- 3.1
11,4,$\bar{4}$	*1.5	+ 0.4
11,4,$\bar{5}$	*1.3	- 0.9
11,4,$\bar{6}$	*1.3	+ 0.6
11,4,$\bar{7}$	*1.1	- 0.5
11,4,$\bar{8}$	*1.0	- 1.0
12,4,2	*1.0	+ 0.4
12,4,1	*1.0	+ 0.8
12,4,0	2.2	+ 1.5
12,4,$\bar{1}$	*1.1	- 0.5
12,4,$\bar{2}$	*1.1	- 1.5
12,4,$\bar{3}$	4.4	+ 2.8
12,4,$\bar{4}$	*1.1	- 1.0
12,4,$\bar{5}$	3.6	+ 2.6
12,4,$\bar{6}$	*1.0	+ 2.0
0,5,1	1.7	- 2.3
0,5,2	2.2	+ 5.9
0,5,3	1.7	- 3.4
0,5,4	10.9	+11.3
0,5,5	13.5	-15.8
0,5,6	*1.1	- 0.7
0,5,7	4.4	+ 4.7
0,5,8	11.8	+12.6
0,5,9	6.7	+ 8.3
0,5,10	1.2	+ 1.5
0,5,11	0.9	+ 0.9
0,5,12	*0.8	- 0.9
1,5,13	*0.5	+ 0.1
1,5,12	1.8	+ 2.0
1,5,11	3.0	- 1.9
1,5,10	4.1	- 4.1
1,5,9	2.9	- 3.4
1,5,8	3.9	+ 4.7
1,5,7	1.7	+ 1.5
1,5,6	1.2	- 3.1
1,5,5	8.8	+12.1
1,5,4	9.0	-10.2
1,5,3	3.2	+ 2.7
1,5,2	*1.0	+ 1.1
1,5,1	1.6	- 3.7
1,5,0	0.8	+ 1.1
1,5,$\bar{1}$	*0.6	+ 0.2
1,5,$\bar{2}$	1.5	- 1.2
1,5,$\bar{3}$	9.8	+ 9.8
1,5,$\bar{4}$	1.0	- 2.7
1,5,$\bar{5}$	10.5	-11.0
1,5,$\bar{6}$	3.8	- 2.6
1,5,$\bar{7}$	*1.1	+ 0.6
1,5,$\bar{8}$	9.1	+11.8
1,5,$\bar{9}$	6.8	- 7.8
1,5,$\overline{10}$	1.0	+ 0.6
1,5,$\overline{11}$	1.9	- 2.2
1,5,$\overline{12}$	1.1	- 0.2
1,5,$\overline{13}$	*0.6	+ 1.3
2,5,12	3.8	- 3.7
2,5,11	*0.8	+ 0.1
2,5,10	7.3	- 6.9
2,5,9	5.9	- 7.0
2,5,8	*1.1	+ 1.3
2,5,7	1.8	+ 1.9
2,5,6	4.1	- 4.5
2,5,5	*1.1	+ 2.4
2,5,4	2.9	+ 1.1
2,5,3	*1.2	+ 3.2
2,5,2	*1.2	- 0.7
2,5,1	*1.1	- 1.0
2,5,0	*1.1	+ 0.4
2,5,$\bar{1}$	*1.1	- 2.8
2,5,$\bar{2}$	8.8	+ 8.0
2,5,$\bar{3}$	17.5	+18.1
2,5,$\bar{4}$	10.8	+13.3
2,5,$\bar{5}$	0.9	+ 1.2
2,5,$\bar{6}$	4.3	+ 3.3
2,5,$\bar{7}$	5.4	- 6.5
2,5,$\bar{8}$	4.8	- 5.6
2,5,$\bar{9}$	2.7	+ 2.5
2,5,$\overline{10}$	*1.1	+ 0.6
2,5,$\overline{11}$	*1.1	- 0.6
2,5,$\overline{12}$	*0.8	+ 1.1
2,5,$\overline{13}$	1.4	- 0.6
3,5,12	*0.7	+ 0.2
3,5,11	1.6	- 1.7
3,5,10	4.2	- 4.0
3,5,9	4.3	- 4.8
3,5,8	*1.1	+ 0.3
3,5,7	*1.1	- 0.1
3,5,6	3.0	+ 4.5
3,5,5	3.2	- 2.4
3,5,4	2.5	- 1.7
3,5,3	2.8	- 4.0
3,5,2	*1.2	- 0.7
3,5,1	1.9	- 3.1
3,5,0	3.0	+ 0.1
3,5,$\bar{1}$	2.5	+ 4.8
3,5,$\bar{2}$	0.8	+ 1.2
3,5,$\bar{3}$	15.5	+13.1
3,5,$\bar{4}$	13.0	+12.6
3,5,$\bar{5}$	1.9	- 3.3
3,5,$\bar{6}$	6.6	- 6.3
3,5,$\bar{7}$	8.5	+ 9.8
3,5,$\bar{8}$	4.3	- 6.0
3,5,$\bar{9}$	1.0	+ 1.8
3,5,$\overline{10}$	*1.1	- 0.7
3,5,$\overline{11}$	1.0	- 0.7
3,5,$\overline{12}$	*0.8	+ 0.1
3,5,$\overline{13}$	0.8	- 1.3
4,5,11	2.2	+ 1.5
4,5,10	1.8	+ 1.1
4,5,9	1.4	+ 1.4
4,5,8	1.7	- 0.8
4,5,7	*1.1	+ 0.4
4,5,6	1.5	- 1.9
4,5,5	1.4	- 1.9
4,5,4	4.7	- 3.8
4,5,3	3.6	- 4.2
4,5,2	1.1	+ 2.9
4,5,1	3.1	+ 3.5
4,5,0	4.8	- 6.8
4,5,$\bar{1}$	1.9	- 0.5
4,5,$\bar{2}$	7.5	- 8.9
4,5,$\bar{3}$	2.0	+ 2.8
4,5,$\bar{4}$	7.2	+ 7.3
4,5,$\bar{5}$	2.6	+ 0.6
4,5,$\bar{6}$	1.2	+ 1.5
4,5,$\bar{7}$	4.4	- 5.2
4,5,$\bar{8}$	2.0	+ 2.4
4,5,$\bar{9}$	1.5	+ 1.9
4,5,$\overline{10}$	*1.0	+ 0.9
4,5,$\overline{11}$	*0.8	+ 0.8
5,5,11	*0.5	+ 1.6
5,5,10	*0.8	+ 0.3
5,5,9	*0.8	- 0.5
5,5,8	2.3	+ 1.4
5,5,7	2.2	- 2.1
5,5,6	1.0	- 0.1
5,5,5	1.8	+ 3.3
5,5,4	1.9	- 1.8
5,5,3	2.2	- 2.0
5,5,2	*1.1	- 0.3
5,5,1	2.7	- 4.1
5,5,0	5.3	+ 7.3
5,5,$\bar{1}$	7.1	- 7.7
5,5,$\bar{2}$	8.0	-10.4
5,5,$\bar{3}$	3.8	- 4.4
5,5,$\bar{4}$	2.7	+ 1.4
5,5,$\bar{5}$	1.1	0
5,5,$\bar{6}$	1.9	+ 2.9
5,5,$\bar{7}$	1.0	+ 2.7
5,5,$\bar{8}$	*1.1	- 0.2
5,5,$\bar{9}$	*1.1	+ 0.4
5,5,$\overline{10}$	*1.0	+ 0.5
5,5,$\overline{11}$	*0.9	- 0.8
5,5,$\overline{12}$	0.8	- 0.9
6,5,10	*0.5	- 0.2
6,5,9	*0.8	- 0.5
6,5,8	1.9	- 1.4
6,5,7	5.1	+ 3.9
6,5,6	2.0	+ 0.7
6,5,5	5.4	+ 5.7
6,5,4	1.3	+ 0.9
6,5,3	1.6	- 0.5
6,5,2	3.9	+ 4.8
6,5,1	1.8	- 2.4
6,5,0	2.7	- 3.4
6,5,$\bar{1}$	1.9	+ 1.3
6,5,$\bar{2}$	4.9	- 4.0
6,5,$\bar{3}$	3.1	- 2.4
6,5,$\bar{4}$	1.6	+ 2.7
6,5,$\bar{5}$	*1.1	- 1.0
6,5,$\bar{6}$	*1.1	+ 0.3
6,5,$\bar{7}$	2.9	- 1.9
6,5,$\bar{8}$	3.2	- 2.5
6,5,$\bar{9}$	1.4	- 1.4
6,5,$\overline{10}$	*0.8	+ 0.1
6,5,$\overline{11}$	*0.8	+ 1.2
6,5,$\overline{12}$	1.2	+ 0.5
7,5,9	*0.5	+ 1.4
7,5,8	1.0	+ 0.9
7,5,7	2.6	- 2.3
7,5,6	4.1	+ 3.5
7,5,5	5.2	+ 4.8
7,5,4	3.3	+ 3.4
7,5,3	*1.0	- 0.2
7,5,2	5.0	- 4.7
7,5,1	4.1	+ 5.3
7,5,0	1.0	- 1.4
7,5,$\bar{1}$	*1.0	+ 1.6
7,5,$\bar{2}$	2.2	- 2.6
7,5,$\bar{3}$	2.6	- 1.4
7,5,$\bar{4}$	*1.0	+ 0.4
7,5,$\bar{5}$	*1.0	- 0.3
7,5,$\bar{6}$	3.5	- 2.5
7,5,$\bar{7}$	*0.9	- 0.8
7,5,$\bar{8}$	4.9	- 4.1
7,5,$\bar{9}$	*0.8	- 0.7
7,5,$\overline{10}$	*0.8	+ 0.3
7,5,$\overline{11}$	2.6	- 2.7
8,5,8	*0.5	+ 1.9
8,5,7	1.5	- 1.6
8,5,6	1.9	- 1.2
8,5,5	1.4	+ 1.0
8,5,4	1.2	+ 1.4
8,5,3	*1.1	- 0.3
8,5,2	1.4	+ 2.1
8,5,1	5.3	- 4.7
8,5,0	*1.1	0
8,5,$\bar{1}$	1.5	- 1.6
8,5,$\bar{2}$	*1.1	- 0.3
8,5,$\bar{3}$	*1.1	+ 0.7
8,5,$\bar{4}$	4.2	- 4.0
8,5,$\bar{5}$	2.7	+ 1.8
8,5,$\bar{6}$	2.9	+ 1.7
8,5,$\bar{7}$	*0.8	+ 0.8
8,5,$\bar{8}$	2.8	- 2.4
8,5,$\bar{9}$	1.4	- 1.2
8,5,$\overline{10}$	1.0	+ 0.7
9,5,6	*0.5	- 2.9
9,5,5	*0.7	- 0.1
9,5,4	1.5	+ 1.1
9,5,3	2.0	- 1.7
9,5,2	*0.8	+ 0.2
9,5,1	1.9	+ 1.6
9,5,0	1.7	- 1.2
9,5,$\bar{1}$	2.0	- 2.8
9,5,$\bar{2}$	1.4	- 1.2
9,5,$\bar{3}$	*0.8	- 0.5
9,5,$\bar{4}$	5.6	+ 4.7
9,5,$\bar{5}$	3.3	- 2.8
9,5,$\bar{6}$	5.4	+ 4.6
9,5,$\bar{7}$	3.1	+ 2.4
9,5,$\bar{8}$	0.9	+ 0.8
9,5,$\bar{9}$	1.0	+ 0.8
10,5,4	*0.5	- 0.1
10,5,3	*0.7	+ 0.2
10,5,2	*0.7	+ 0.7
10,5,1	2.9	+ 2.4
10,5,0	2.4	+ 1.8
10,5,$\bar{1}$	*0.8	+ 0.5
10,5,$\bar{2}$	1.5	+ 0.6
10,5,$\bar{3}$	1.5	- 0.6
10,5,$\bar{4}$	2.0	- 1.4
10,5,$\bar{5}$	3.3	+ 1.9
10,5,$\bar{6}$	1.6	+ 1.7
10,5,$\bar{7}$	3.4	+ 3.0
11,5,1	*0.5	+ 2.6
11,5,0	*0.5	+ 2.7
11,5,$\bar{1}$	*0.5	0
11,5,$\bar{2}$	1.9	- 1.6
11,5,$\bar{3}$	3.3	+ 2.8
11,5,$\bar{4}$	*0.5	- 2.4
0,6,0	1.7	- 1.4
0,6,1	1.1	- 1.0
0,6,2	1.1	+ 1.0
0,6,3	3.0	+ 5.1
0,6,4	*1.2	- 0.1
0,6,5	*1.2	+ 1.1
0,6,6	1.3	- 0.6
0,6,7	*1.0	- 0.2
0,6,8	3.1	+ 3.3
0,6,9	3.0	+ 3.5
0,6,10	2.6	- 3.2
1,6,10	2.3	- 3.5
1,6,9	2.0	- 1.9
1,6,8	1.6	+ 1.3
1,6,7	*1.1	- 0.8
1,6,6	*1.2	- 0.1
1,6,5	*1.2	+ 1.7
1,6,4	*1.2	0
1,6,3	1.7	+ 3.8
1,6,2	1.7	+ 2.6
1,6,1	*1.3	- 0.4
1,6,0	0.8	- 0.8
1,6,$\bar{1}$	3.1	- 5.0
1,6,$\bar{2}$	3.2	+ 2.0
1,6,$\bar{3}$	4.5	+ 5.1
1,6,$\bar{4}$	*1.2	+ 1.7
1,6,$\bar{5}$	*1.2	- 0.3
1,6,$\bar{6}$	*1.1	+ 1.1
1,6,$\bar{7}$	*1.1	- 0.9
1,6,$\bar{8}$	2.9	- 3.0
1,6,$\bar{9}$	0.6	+ 1.3
1,6,$\overline{10}$	2.7	- 3.7
2,6,9	4.8	- 5.0
2,6,8	1.2	+ 1.8
2,6,7	2.1	+ 1.3
2,6,6	2.8	- 2.1
2,6,5	*1.4	+ 1.0
2,6,4	1.3	+ 1.5
2,6,3	*1.5	- 1.2
2,6,2	*1.5	+ 1.0
2,6,1	*1.5	- 2.4
2,6,0	*1.5	+ 0.7
2,6,$\bar{1}$	7.6	+ 8.8
2,6,$\bar{2}$	*1.5	- 1.4
2,6,$\bar{3}$	9.8	+10.4
2,6,$\bar{4}$	3.1	+ 3.4
2,6,$\bar{5}$	*1.4	- 0.1
2,6,$\bar{6}$	*1.4	+ 0.9
2,6,$\bar{7}$	*1.3	- 0.5
2,6,$\bar{8}$	*1.2	+ 0.4
2,6,$\bar{9}$	*1.0	+ 0.2
2,6,$\overline{10}$	1.6	- 1.8
2,6,$\overline{11}$	*0.4	- 1.0
3,6,9	3.5	- 3.2
3,6,8	*1.0	+ 0.4
3,6,7	*1.1	- 0.3
3,6,6	4.0	+ 3.6
3,6,5	3.1	- 2.8
3,6,4	1.1	- 1.7
3,6,3	1.1	- 0.5
3,6,2	1.2	+ 2.3
3,6,1	2.1	+ 1.8
3,6,0	1.1	+ 1.4
3,6,$\bar{1}$	3.9	- 3.9
3,6,$\bar{2}$	4.3	+ 5.3
3,6,$\bar{3}$	4.5	+ 5.7
3,6,$\bar{4}$	4.1	+ 4.5
3,6,$\bar{5}$	*1.3	- 1.3
3,6,$\bar{6}$	*1.3	- 2.0
3,6,$\bar{7}$	*1.2	+ 1.1
3,6,$\bar{8}$	*1.1	- 0.3
3,6,$\bar{9}$	1.0	+ 2.0
3,6,$\overline{10}$	*0.8	+ 0.3
3,6,$\overline{11}$	*0.5	- 0.6
4,6,9	*0.6	- 1.2
4,6,8	*0.7	+ 1.0
4,6,7	*0.9	+ 0.1
4,6,6	4.7	- 4.6
4,6,5	4.8	+ 4.0
4,6,4	2.0	- 1.8
4,6,3	*1.1	+ 1.4
4,6,2	*1.1	+ 0.1
4,6,1	2.9	- 2.5
4,6.0	2.2	+ 3.6
4,6,$\bar{1}$	7.0	- 7.0
4,6,$\bar{2}$	7.9	- 9.8
4,6,$\bar{3}$	1.2	- 0.8
4,6,$\bar{4}$	2.8	+ 2.5
4,6,$\bar{5}$	*1.1	- 0.7
4,6,$\bar{6}$	*1.0	+ 1.6
4,6,$\bar{7}$	2.7	- 2.2
4,6,$\bar{8}$	2.8	+ 2.4
4,6,$\bar{9}$	*0.8	+ 0.8
4,6,$\overline{10}$	*0.7	+ 0.8
5,6,7	2.6	+ 1.6
5,6,6	0.8	+ 0.1
5,6,5	1.8	- 0.8
5,6,4	*0.9	- 0.2
5,6,3	*1.0	+ 0.8
5,6,2	*1.0	0
5,6,1	1.0	- 0.6
5,6,0	5.8	- 5.6
5,6,$\bar{1}$	4.4	- 4.3
5,6,$\bar{2}$	10.8	-11.8
5,6,$\bar{3}$	3.1	- 2.4
5,6,$\bar{4}$	1.7	+ 1.1
5,6,$\bar{5}$	2.4	- 1.9
5,6,$\bar{6}$	2.7	+ 2.7
5,6,$\bar{7}$	2.3	+ 1.8
5,6,$\bar{8}$	2.4	- 1.9
5,6,$\bar{9}$	1.0	+ 0.6
5,6,$\overline{10}$	*0.6	- 0.2
6,6,7	*0.6	- 2.9
6,6,6	3.0	+ 2.5
6,6,5	1.7	+ 0.9
6,6,4	1.1	+ 0.9
6,6,3	*0.8	- 0.7
6,6,2	*0.8	- 0.5
6,6,1	*1.0	- 0.3
6,6,0	3.9	+ 3.3
6,6,$\bar{1}$	2.1	- 1.8
6,6,$\bar{2}$	5.7	- 6.2
6,6,$\bar{3}$	3.3	- 2.6
6,6,$\bar{4}$	1.4	+ 1.9
6,6,$\bar{5}$	3.7	+ 2.9
6,6,$\bar{6}$	4.4	- 3.9
6,6,$\bar{7}$	1.7	+ 0.8
6,6,$\bar{8}$	*0.7	- 1.4
6,6,$\bar{9}$	*0.7	+ 0.8
7,6,5	1.6	+ 1.3
7,6,4	*0.8	- 0.3
7,6,3	*0.8	- 0.6
7,6,2	*0.8	+ 0.5
7,6,1	2.3	+ 1.8
7,6,0	5.1	+ 5.2
7,6,$\bar{1}$	4.7	+ 5.7
7,6,$\bar{2}$	1.2	- 1.0
7,6,$\bar{3}$	1.9	- 1.7
7,6,$\bar{4}$	1.9	- 0.6
7,6,$\bar{5}$	2.3	- 2.8
7,6,$\bar{6}$	3.7	+ 3.2
7,6,$\bar{7}$	2.4	- 2.2
8,6,3	0.6	+ 0.5
8,6,2	1.0	+ 1.6
8,6,1	1.3	- 1.7
8,6,0	5.3	+ 4.4
8,6,$\bar{1}$	3.9	+ 4.3
8,6,$\bar{2}$	*1.0	0
8,6,$\bar{3}$	1.6	- 0.8
8,6,$\bar{4}$	2.6	+ 1.6
8,6,$\bar{5}$	1.8	- 2.0
8,6,$\bar{6}$	*0.7	- 0.5
0,7,1	0.7	- 0.4
0,7,2	2.4	+ 3.1
0,7,3	3.1	+ 3.7
0,7,4	*0.9	- 0.2
0,7,5	2.3	+ 2.6
0,7,6	*0.7	+ 0.8
0,7,7	*0.6	- 1.0
1,7,7	*0.4	0
1,7,6	*0.7	+ 0.1
1,7,5	2.2	- 2.4
1,7,4	4.1	+ 4.8
1,7,3	0.9	+ 1.4
1,7,2	2.3	+ 2.8
1,7,1	*1.0	- 1.9
1,7,0	*1.0	- 0.2
1,7,$\bar{1}$	0.9	+ 0.8
1,7,$\bar{2}$	2.2	- 2.4
1,7,$\bar{3}$	3.0	- 4.1
1,7,$\bar{4}$	1.0	+ 1.2
1,7,$\bar{5}$	0.9	+ 1.2
1,7,$\bar{6}$	*0.8	+ 0.1
1,7,$\bar{7}$	*0.5	- 0.1
2,7,5	1.1	- 0.9
2,7,4	4.9	- 5.0
2,7,3	2.7	+ 2.2
2,7,2	*1.0	+ 1.1
2,7,1	*1.1	0
2,7,0	*1.1	+ 0.4
2,7,$\bar{1}$	*1.1	- 1.1
2,7,$\bar{2}$	*1.1	+ 0.9
2,7,$\bar{3}$	2.1	- 1.8
2,7,$\bar{4}$	*1.0	- 0.8
2,7,$\bar{5}$	*0.9	- 0.3
2,7,$\bar{6}$	*0.8	- 0.2
2,7,$\bar{7}$	*0.7	+ 1.1
3,7,6	*0.5	- 2.5
3,7,5	*0.8	- 0.2
3,7,4	2.0	- 3.0
3,7,3	1.1	- 0.1
3,7,2	*1.0	+ 0.4
3,7,1	*1.0	+ 0.8
3,7,0	*1.0	+ 0.3
3,7,$\bar{1}$	*1.0	+ 0.9
3,7,$\bar{2}$	*1.0	- 0.8
3,7,$\bar{3}$	*1.0	- 0.2
3,7,$\bar{4}$	*1.0	- 0.7
3,7,$\bar{5}$	0.7	+ 0.6
3,7,$\bar{6}$	1.1	+ 1.0
3,7,$\bar{7}$	*0.5	- 1.5
4,7,4	*0.7	- 0.8
4,7,3	*0.7	- 1.0
4,7,2	1.1	+ 0.7
4,7,1	*0.8	- 0.5
4,7,0	*0.8	+ 0.6
4,7,$\bar{1}$	2.1	- 1.7
4,7,$\bar{2}$	2.1	- 2.0
4,7,$\bar{3}$	*0.8	- 0.7
4,7,$\bar{4}$	*0.8	- 0.5
4,7,$\bar{5}$	*0.7	- 0.3
5,7,3	*0.6	- 0.6
5,7,2	*0.7	+ 0.4
5,7,1	*0.7	- 1.0
5,7,0	2.7	- 3.6
5,7,$\bar{1}$	*0.7	- 0.2
5,7,$\bar{2}$	2.6	- 3.5
5,7,$\bar{3}$	*0.7	+ 0.4
5,7,$\bar{4}$	*0.7	+ 0.6

* Indicates minimum observable amplitude for unobserved reflexion.

They show that the four molecules in the unit-cell are arranged at nearly equal intervals along [101] in such a way as to make the reflexions 301, 202, $70\bar{3}$ and $3,0,\overline{11}$ very strong. A trial structure based on these considerations was quickly refined by successive Fourier syntheses of the electron density distribution. The projection of one molecule is shown in Fig. 2(*a*). Difference syntheses gave good indications of hydrogen atom positions but did not differentiate conclusively between the nitrogen and the poorly resolved CH group in the *meso* positions. The molecules are not well resolved in the other projections of the structure so that it was decided to try the use of a direct method to determine the molecular arrangement in three dimensions.

The structure factors were put on an absolute scale

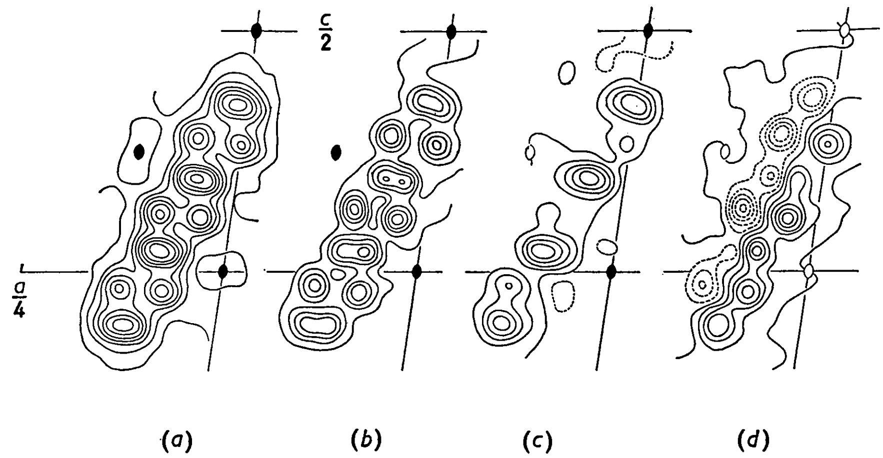

Fig. 2. (*a*) Electron density in one molecule projected on (010). Contour lines at intervals of 1 e.Å^{-2}. (*b*, *c*, *d*) Corresponding $h1l$ generalized projections $|\sigma|$, C and S. Negative contours broken; symmetry elements after Cochran (1952).

by comparing the observed and calculated $F(h0l)$'s. Unitary structure factors, $U(hkl) = F(hkl)/F_{\max.}(hkl)$, were then calculated for the $h0l$, $h1l$, and $h2l$ reflexions and Harker–Kasper (1948) inequalities in the form recommended by Grison (1951) were used to determine the signs of the $h1l$ structure factors. Fifteen reflexions in the limited set considered have $U > 0{\cdot}5$ so that the inequalities proved very powerful. The signs of $31 F(h1l)$'s were quickly determined.

These structure factors were used in the calculation of a generalized projection (Cochran & Dyer, 1952) of the electron density on (010). The functions calculated were

$$\sigma_1(X, Z) = b\int_0^1 \varrho(X, Y, Z)\exp(2\pi iY)dY$$
$$= C_1(X, Z)+iS_1(X, Z)\ ,$$

where $C_1(X, Z)$ and $S_1(X, Z)$ are given by

$$C_1(X, Z) = \frac{1}{B}\sum_{h,l=0}^{\infty}\sum^{h+k+l=2n}[\{F(h1l)+F(h1\bar{l})\}\cos 2\pi hX\cos 2\pi lZ$$
$$-\{F(h1l)-F(h1\bar{l})\}\sin 2\pi hX\sin 2\pi lZ]$$

and

$$S_1(X, Z) = \frac{1}{B}\sum_{h,l=0}^{\infty}\sum^{h+k+l=2n+1}[\{F(h1l)+F(h1\bar{l})\}\sin 2\pi hX\cos 2\pi lZ$$
$$+\{F(h1l)-F(h1\bar{l})\cos 2\pi hX\sin 2\pi lZ]\ .$$

The atomic peaks in $C_1(X, Z)$ and $S_1(X, Z)$ have heights proportional to $\cos 2\pi Y_i$ and $\sin 2\pi Y_i$ respectively, where Y_i is the fractional y coordinate of the corresponding atom. These syntheses showed clearly the orientation of the molecules in the unit cell and yielded y coordinates which were used with x, z coordinates from the zero-level projection in a calculation of all the $F(h1l)$'s. Satisfactory agreement was obtained between the observed and calculated structure factors; reliability index

$$R = \Sigma||F_o|-|F_c||\div\Sigma|F_o| = 0{\cdot}28\ .$$

Table 2. *Refinement of atomic coordinates*

(1) Fractional atomic coordinates derived from [010] projections.
(2) Atomic peak positions in $\varrho_o(x, y, z)$ calculated using signs from (1).
(3) Atomic coordinates corrected from difference synthesis.
(4) Atomic peak positions in $\varrho_o(x, y, z)$ calculated using signs from (3).
(5) Atomic peak positions in $\varrho_c(x, y, z)$ calculated from (3).
(6) Atomic coordinates corrected by back-shift method from (4) and (5).

		(1)	(2)	(3)	(4)	(5)	(6)
C_1	X	0·143	0·1395	0·1388	0·1387	0·1390	0·1385
	Y	0·289	0·2890	0·2911	0·2900	0·2894	0·2917
	Z	0·958	0·9573	0·9571	0·9567	0·9567	0·9571
C_2	X	0·199	0·2020	0·2021	0·2028	0·2022	0·2027
	Y	0·220	0·2251	0·2248	0·2252	0·2245	0·2255
	Z	0·888	0·8860	0·8851	0·8858	0·8856	0·8853
C_3	X	0·260	0·2612	0·2623	0·2619	0·2611	0·2631
	Y	0·014	0·0155	0·0172	0·0177	0·0166	0·0183
	Z	0·891	0·8914	0·8906	0·8916	0·8915	0·8907
C_4	X	0·252	0·2530	0·2543	0·2545	0·2535	0·2553
	Y	0·867	0·8762	0·8758	0·8761	0·8762	0·8757
	Z	0·967	0·9690	0·9683	0·9685	0·9682	0·9686
C_5	X	0·181	0·1804	0·1804	0·1797	0·1800	0·1801
	Y	0·800	0·8001	0·7976	0·8013	0·8002	0·7987
	Z	0·125	0·1265	0·1265	0·1259	0·1263	0·1261
C_6	X	0·111	0·1086	0·1080	0·1069	0·1076	0·1073
	Y	0·746	0·7466	0·7446	0·7458	0·7454	0·7450
	Z	0·283	0·2844	0·2845	0·2849	0·2846	0·2848
C_7	X	0·051	0·0476	0·0473	0·0475	0·0475	0·0473
	Y	0·836	0·8267	0·8272	0·8281	0·8284	0·8269
	Z	0·355	0·3543	0·3554	0·3551	0·3544	0·3561
C_8	X	0·992	0·9924	0·9936	0·9928	0·9932	0·9932
	Y	0·043	0·0410	0·0402	0·0408	0·0400	0·0410
	Z	0·345	0·3445	0·3452	0·3447	0·3444	0·3455
C_9	X	0·998	0·9994	0·9997	0·0004	0·0004	0·9997
	Y	0·169	0·1687	0·1708	0·1682	0·1684	0·1706
	Z	0·264	0·2637	0·2633	0·2634	0·2634	0·2633

Table 2 (*cont.*)

		(1)	(2)	(3)	(4)	(5)	(6)
$N_{(10)}$	X	0·073	0·0703	0·0703	0·0703	0·0706	0·0700
	Y	0·230	0·2252	0·2264	0·2238	0·2253	0·2249
	Z	0·110	0·1096	0·1095	0·1096	0·1096	0·1095
C_{11}	X	0·135	0·1331	0·1325	0·1313	0·1317	0·1321
	Y	0·150	0·1529	0·1498	0·1506	0·1506	0·1498
	Z	0·039	0·0389	0·0394	0·0396	0·0388	0·0402
C_{12}	X	0·190	0·1895	0·1897	0·1896	0·1897	0·1896
	Y	0·941	0·9368	0·9353	0·9377	0·9378	0·9352
	Z	0·045	0·0451	0·0446	0·0449	0·0448	0·0447
C_{13}	X	0·123	0·1188	0·1181	0·1177	0·1180	0·1178
	Y	0·885	0·8757	0·8765	0·8758	0·8766	0·8757
	Z	0·198	0·1982	0·1985	0·1987	0·1987	0·1985
C_{14}	X	0·066	0·0642	0·0639	0·0633	0·0635	0·0637
	Y	0·092	0·0927	0·0909	0·0923	0·0924	0·0908
	Z	0·190	0·1878	0·1879	0·1876	0·1878	0·1877

The signs were used in a recalculation of the generalized projection which gave the maps shown in Fig. 2. It may be noticed, incidentally, that Figs. 2(*a*) and 2(*b*) show the increased resolution in $|\sigma| = (C^2+S^2)^{\frac{1}{2}}$ which has been discussed elsewhere (Phillips, 1954*c*).

The *y* coordinates were further refined by calculation of the structure factors and generalized projections corresponding to the *h2l*, *h3l*, and *h5l* reflexions. The values obtained by this process are given in Table 2, column (1). They were used in the first calculation of all the structure factors. Geometrical structure factors were calculated on I.B.M. machines, using the method of Grems & Kasper (1949). The nitrogen atom was not identified and no allowance was made for hydrogen atoms. The James–Brindley carbon *f* curve (*Internationale Tabellen*, 1935) was used with a temperature factor having the constant $B = 5{\cdot}5$ Å^2, derived from a graph of $\log (\Sigma F_o/\Sigma F_c)$ against $\sin^2 \theta$. The reliability index, calculated with contributions $||F_c|-|F_o^*||$ from the unobserved reflexions with $|F_c|$ larger than the minimum observable $|F_o^*|$, was 0·26.

Refinement of the structure

Three stages of refinement have been completed, using three-dimensional Fourier methods. The syntheses of electron density and difference electron density were calculated by Dr J. Kates and Dr J. F. Hart on FERUT, the electronic digital computor at the University of Toronto, using a programme designed by Kates which is readily adaptable to all space groups (Gottlieb & Kates, 1955). The densities were calculated at intervals *a*/60, *b*/30 and *c*/60.

The signs determined in the first full structure-factor calculations were used with the observed structure amplitudes in a Fourier synthesis of the electron-density distribution. The atomic peak positions found by graphical interpolation are listed in Table 2, column (2). The nitrogen atom was characterized by a peak significantly larger than that in the alternative *meso* position and it was included separately in subsequent structure-factor calculations. The atomic peaks also showed a variation in height very similar to that observed in anthracene (Sinclair *et al.*, 1950), but no allowance for variation in individual atomic temperature factors was included in the second structure-factor calculation. McWeeny (1951) scattering factors for carbon (valence states) and nitrogen were used at this stage. Their use involved a revision of the apparent best mean isotropic temperature factor constant from 5·5 Å^2 to 4·1 Å^2 and a change in the scale of F_o by a factor 1·25. The reliability index was reduced to 0·17.

Refinement was continued by calculation of a complete three-dimensional difference synthesis. Gradients at atomic positions in this synthesis were derived by graphical methods and used in the calculation of shifts in the atomic coordinates from the formula

$$\Delta x_n = (\partial(\varrho_o-\varrho_c)/\partial x)_n/(\partial^2\varrho_o/\partial x^2)_n\,.$$

The atomic peak curvatures were calculated from the three-dimensional electron-density distribution corrected for the change in scale. The new atomic coordinates are given in Table 2, column (3). The mean shift in an atomic position was 0·012 Å and the maximum shift was 0·021 Å.

The difference synthesis showed clearly that the atoms are not all subject to the same thermal motion. Atoms 10, 11, 12, 5, 13 and 14 were associated with roughly equal peaks of difference density while atoms 1, 4, 6 and 9 were associated with smaller peaks and atoms 2, 3, 7 and 8 with holes. Separate isotropic temperature factors were calculated for each atomic group. The temperature-factor constants were not estimated from the difference-density curvatures (Cochran, 1951) but in an approximate way from the variation in atomic peak height with temperature factor. A three-dimensional Fourier transform of the difference between atomic scattering factors with slightly differing temperature coefficients showed that, for a mean B near 4·0 Å^2, a change in the temperature factor constant $\Delta B = 0{\cdot}5$ Å^2 gives rise to a change of approximately $\Delta\varrho = 0{\cdot}4$ e.Å^{-3} in the peak height of a carbon atom. This relationship was used in calculations of temperature-factor constants for the separate atomic groups from the mean variations in difference density at the atomic positions. These temperature-factor constants are listed under $B_i(1)$ in Table 3.

Recalculation of the structure factors using the new

Table 3. *Temperature-factor constants for separate atomic groups*

Group	Atoms	$B_i(1)$	$B_i(2)$
A_1	5, 10	$3{\cdot}7_5$ Å^2	$4{\cdot}0_3$ Å^2
A_2	11, 12, 13, 14	$3{\cdot}7_5$	$3{\cdot}6_8$
B	1, 4, 6, 9	4·4	$4{\cdot}5_8$
C	2, 3, 7, 8	4·8	$5{\cdot}1_6$

atomic coordinates and temperature factors gave $R = 0{\cdot}15_5$.

Good hydrogen atom peaks also appeared in the difference synthesis but their shapes clearly were affected by the variations in difference density associated with the heavier atoms. Hydrogen atom positions (listed in Table 4) consistent with these peaks, but calculated by assuming a C–H bond length 1·08 Å (Pauling, 1940) and trigonal bond configuration at the carbon atoms, were used in a calculation of the hydrogen atom contributions to the structure factors. The hydrogen scattering curve adopted was that given by McWeeny (1951) with $B = 5{\cdot}0$ Å^2. The reliability index was reduced to $0{\cdot}13_3$. These structure factors, together with the observed amplitudes, are shown in Table 1.

Table 4. *Hydrogen atom positions*

	Assumed			Measured		
	X	*Y*	*Z*	*X*	*Y*	*Z*
H_1	0·096	0·448	0·953	0·100	0·409	0·960
H_2	0·207	0·336	0·825	—	—	—
H_3	0·311	0·963	0·834	0·299	0·992	0·823
H_4	0·297	0·717	0·974	0·290	0·755	0·984
H_5	0·223	0·636	0·133	0·210	0·631	0·125
H_6	0·151	0·584	0·292	0·145	0·597	0·288
H_7	0·041	0·724	0·419	0·036	0·760	0·431
H_8	0·946	0·105	0·401	0·951	0·137	0·397
H_9	0·957	0·332	0·256	0·960	0·336	0·251

A second three-dimensional difference synthesis was then calculated, using the latest calculated structure factors without hydrogen contributions and leaving out the planes seriously affected by extinction which have been listed above. About one hundred high-order reflexions of small amplitude were included for the first time in this calculation. The unobserved reflexions were discounted although detailed consideration (Hamilton, 1955) suggests that this may not be the best course even though most of the unobserved reflexions have calculated structure amplitudes less than the minimum observable. A section in a related* difference synthesis in the best plane of the molecule, obtained by interpolation from values of $(\varrho_o-\varrho_c)$ calculated at intervals $a/60$, $b/30$, $c/60$, is shown in Fig. 3. The electron-density distribution in the crystal also was recalculated in patches near the heavy-atom positions so that allowance could be made for the change in scale and for the correction of some 30 signs since the original calculation. Corrections to the atomic coordinates were then calculated by Booth's (1946) back-shift method, which is equivalent to the direct use of the difference synthesis (e.g. Cruickshank, 1952), but which involves simpler calculations from the density distributions. The electron-density distribution corresponding to the calculated structure factors was obtained by combining the ϱ_o and $(\varrho_o-\varrho_c)$ syntheses. Peak positions in the ϱ_o and ϱ_c syntheses were then determined by the method due to Shoemaker, Donohue, Schomaker & Corey (1950). The exponential function

$$\varrho(x, y, z) = \exp\,(p-\tfrac{1}{2}rx^2-\tfrac{1}{2}sy^2-\tfrac{1}{2}tz^2 +ux+vy+wz+lyz+mzx+nxy)$$

was adjusted, by least-squares calculations of the best constants, to fit each atomic peak in turn. The peak positions and shape parameters were then determined analytically. Peak positions in the 'observed' and 'calculated' densities are listed in Table 2, columns (4) and (5) respectively; the corrected atomic coordinates are listed in column (6). The average correction to an atomic position was 0·008 Å and the largest was 0·012 Å. The importance of finite-series errors can be seen from comparison of the final atomic coordinates, column (6), with the atomic positions, column (4), in the observed electron-density distribution. The average finite-series correction per coordinate is $0{\cdot}005_7$ Å, very nearly identical with that in anthracene (Ahmed & Cruickshank, 1952). The structure factors have not been recalculated.

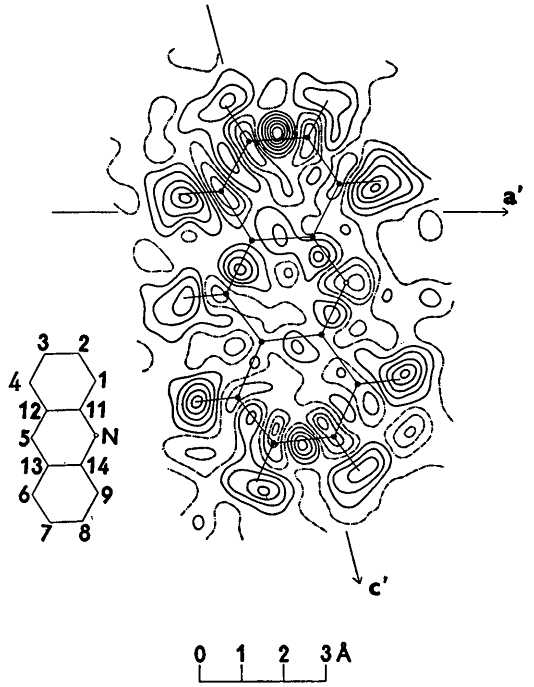

Fig. 3.* Section through the final three-dimensional difference synthesis in the best plane of the molecule. **a**′ and **c**′ are the lines in which this plane cuts (001) and (100). Contours at intervals of 0·1 e.Å^{-3}. Broken lines: negative contours; chain lines: zero contours.

The positions of hydrogen atom peaks in the difference synthesis are listed in Table 4. All but H_2 are clearly resolved. The peak heights and curvatures are given in Table 5.

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* Fig. 3 was derived from a synthesis in which reflexions *hk*0 and 0*kl* were given half weight. The corrected synthesis, used in the refinement, shows the same features with an average increase of about 10% in peak heights.

Table 5. *Shape parameters of hydrogen atom peaks in the difference synthesis*

	Heights (e.Å^{-3})	Curvatures (e.Å^{-5})			
	ϱ_m	$-A_{hh}$	$-A_{kk}$	$-A_{ll}$	A_{lh}
H_1	0·54	4·0	2·9	4·0	1·2
H_2	—	—	—	—	—
H_3	0·39	1·9	2·8	3·4	1·1
H_4	0·62	4·2	4·9	4·7	0·3
H_5	0·53	3·9	3·0	3·1	1·1
H_6	0·58	3·5	4·3	3·7	0·8
H_7	0·51	3·9	4·1	4·2	0·8
H_8	0·42	2·7	1·4	3·2	2·0
H_9	0·54	3·4	2·1	3·7	1·4

The heights and curvatures of atomic peaks in the observed and calculated electron-density distributions are given in Table 6. There is good general agreement, but apparently the mean isotropic temperature factors used were not the best possible. Corrected values of the constants are given in Table 3 under $B_i(2)$. Group A now has been divided into two parts because atoms 5 and 10, associated with holes in the difference density (Fig. 3), appear to have a B_i larger than that appropriate to other atoms at the same distance from the molecular centre. Further refinement of the structure depends upon more detailed consideration of the thermal motion.

Molecular thermal motion

Higgs (1955) has shown that the mean square amplitude of vibration of an atom in a molecular crystal is given by equation

$$\overline{u_i^2} = (\overline{u_i^2})_{\text{int.}} + (\overline{u_i^2})_{\text{r.b.}} ,$$

where the two components are respectively the contributions due to internal modes of vibration within the molecule and to rigid-body vibrations of the molecule as a whole. The former is much the smaller since the intramolecular forces are much stronger than the intermolecular forces. Thus, for example, in naphthalene $(\overline{u_i^2})_{\text{int.}}$ contributes only about 4% of $\overline{u_i^2}$. The displacements due to rigid-body vibrations can be resolved into components $P+QR_i^2$ which arise respectively from translation and restricted rotation (libration) of the molecule, so that

$$\overline{u_i^2} \simeq (\overline{u_i^2})_{\text{r.b.}} = P+QR_i^2 , \qquad (1)$$

where R_i is the distance of the atom from the molecular centre. If the small contributions of internal modes of vibration are ignored, therefore, the relation of $\overline{u_i^2}$ and R_i^2 should be very nearly linear.

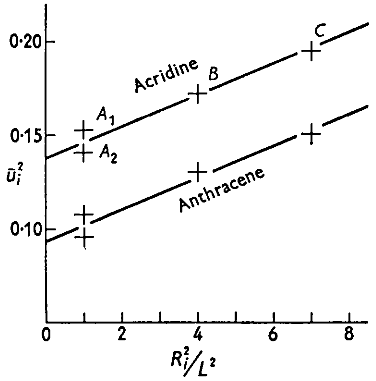


Fig. 4. Graphs of $\overline{u_i^2}$ versus R_i^2/L^2 for atomic groups in acridine and anthracene.

Values of $\overline{u_i^2} = 3B_i/8\pi^2$ for the different atomic groups are plotted in Fig. 4 against R_i^2/L^2. It is assumed that the heavy atoms lie at the vertices of regular hexagons with side L. Corresponding results for anthracene (Higgs, 1955) are plotted for comparison. Both sets are consistent with Higgs's picture of the molecular vibrations. It is clear also that despite differences in molecular arrangement discussed below, the thermal motions of molecules in the two structures are very similar. The variation of $\overline{u_i^2}$ with R_i is the same, indicating the presence of very similar librations, but the constant term P (equation (1)) is larger for acridine. This result indicates that translational vibra-

Table 6. *Shape parameters of atomic peaks in ϱ_o and ϱ_c*

	Heights (e.Å^{-3})		Curvatures (e.Å^{-5})							
	ϱ_m		$-A_{hh}$		$-A_{kk}$		$-A_{ll}$		A_{lh}	
	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.
C_1	6·97	7·11	57·1	57·4	54·2	57·3	54·3	55·1	4·8	6·7
C_2	6·20	6·52	47·0	48·6	40·1	47·8	49·0	50·0	6·6	7·2
C_3	6·00	6·34	45·3	47·5	36·9	46·7	45·3	48·9	8·2	8·3
C_4	6·66	6·97	56·4	55·9	50·2	56·6	49·8	54·4	9·3	7·8
C_5	7·75	7·89	74·1	71·2	66·0	69·1	58·5	63·9	9·0	10·2
C_6	6·83	6·96	55·7	54·4	52·8	55·8	48·5	52·6	4·8	7·4
C_7	6·24	6·50	47·5	49·2	38·8	47·3	48·7	49·0	7·3	7·1
C_8	6·32	6·56	51·6	50·0	41·0	48·4	49·5	51·1	11·5	9·4
C_9	7·03	7·04	58·9	55·5	55·3	56·9	53·5	54·5	10·4	8·8
$N_{(10)}$	9·40	9·70	81·9	80·4	80·0	82·2	74·6	79·6	10·7	11·1
C_{11}	8·11	8·06	76·5	71·8	67·4	67·2	66·6	69·3	7·5	9·5
C_{12}	7·91	7·85	73·6	67·7	64·2	66·9	62·4	65·9	6·9	7·7
C_{13}	7·90	7·84	73·4	67·7	61·1	63·4	63·8	65·5	8·0	8·7
C_{14}	7·99	7·93	74·1	68·4	65·7	66·8	63·3	67·3	10·0	10·1

tions may be more severe in acridine, in accord with its lower melting point (110° C. compared with 210° C. for anthracene), but it may be due in part at least to the effects of unequally compensated systematic errors in the intensity measurements from which the two structures have been determined and to a greater degree of static disorder in acridine.

The molecules thus appear to be subject to rigid-body vibrations and librations with $(\overline{u_i^2})^{\frac{1}{2}}$ due to vibrations alone of about 0·37 Å. The difference density distribution, which is very similar indeed to that in anthracene at this stage of refinement (Cruickshank, private communication), is entirely consistent with this picture. Thus, for example, the large peaks in the bonds C_2–C_3 and C_7–C_8 and the relative diffuseness of the hydrogen atoms 2, 3, 7 and 8 are due predominantly to librations about an axis perpendicular to the molecular plane, the effects of which increase with distance from the molecular centre. No detailed allowance has been made for this thermal motion in the present refinement.

Accuracy of the structure determination

Standard errors for the intensity measurements have been calculated from the residuals among the two or three measurements on some 800 reflexions. They show that the standard error in estimating a structure factor can be represented over the greater part of the intensity range by the expression $\sigma(F) = 0{\cdot}2F^{\frac{1}{2}}$ or, less exactly, by $\sigma(F) = 0{\cdot}07F$. It follows, by the application of a formula quoted by Lipson & Cochran (1954, Table 288), that the standard deviation in electron density due to random errors in the intensity measurements is about $\sigma(\varrho) = 0{\cdot}08$ e.Å^{-3}. Corresponding deviations in atomic coordinates are given in Table 7 for the three groups of carbon atoms and the well resolved hydrogen atoms. These are not, however, the only errors to be considered. There are systematic errors in the measurements and, although a refinement technique has been used which can eliminate finite-series errors, some errors remain owing to the use of an imperfect model of the structure.

Table 7. *Standard errors in the atomic coordinates*

Values in Ångström units

	From $\sigma(F)=0{\cdot}07F$			From $\Delta F=F_o-F_c$		
Atoms	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$
Group A_2	0·0032	0·0038	0·0039	0·0035	0·0044	0·0041
Group B	0·0037	0·0040	0·0042	0·0046	0·0054	0·0051
Group C	0·0040	0·0052	0·0040	0·0055	0·0074	0·0055
H	0·029	0·033	0·029	0·069	0·084	0·069

A contribution from absorption and reflexion-spot-shape errors is included in the estimation of $\sigma(F)$, since residuals between measurements from different crystals in different orientations were used. Since the crystals were small and regularly shaped, the remaining systematic errors due to these effects are likely to be fairly smooth functions of sin θ and to be allowed for to some extent in the apparent temperature factor. Their existence will not much affect the determination of atomic positions but it must limit detailed analysis of the atomic peak shapes. Similarly, the larger extinction errors will affect seriously only the investigation of fine detail in the electron distribution.

The systematic features in the difference density that indicate the positions of hydrogen atoms and show the nature of the molecular thermal motion also affect the atomic coordinates, which have been determined by reducing to zero the difference density gradient at each atomic position. Thus, for example, the hydrogen atom peaks overlap neighbouring carbon atom positions so that the difference gradient at these positions in fact should not be zero. The errors due to this cause are about 0·003 Å, given by the gradient in a hydrogen peak at the carbon atom position divided by the carbon-atom-peak curvature. Other features in the difference density introduce errors with this same order of magnitude but often, fortunately, in more or less opposed directions. In fact these various systematic errors to some extent cancel one another. There is, therefore, some justification for the common practice of treating them as additional random errors. The standard deviations in atomic coordinates may then be calculated from the residuals $\Delta F = F_o - F_c$ by the use of Cruickshank's (1949) formulae. This method has been adopted here. The differences between the observed and calculated structure factors from which the final difference synthesis was calculated were used in calculating the principal standard deviations in difference-density gradient, $\sigma(A_h) = 0{\cdot}262$, $\sigma(A_k) = 0{\cdot}287$ and $\sigma(A_l) = 0{\cdot}264$ e.Å^{-4}. Combined with the atomic peak curvatures in formulae quoted by Parry (1954), they give standard deviations in atomic coordinates shown in Table 7. Judged in comparison with those derived from the experimental errors alone, these values appear to include a rather small allowance for the additional errors which have been reviewed above. It is probable, however, that they can be used safely, with proper significance criteria (Cruickshank & Robertson, 1953) in discussion of the structural details.

A further systematic error remains to be considered. Cox, Cruickshank & Smith (1955) have shown that molecular librations can have a large effect on the apparent atomic positions. Atoms are observed displaced towards an axis of libration by distances which depend in a complicated way on their perpendicular distance from the axis and on the amplitude of the librations. If no particular axis of libration is favoured, the error in an atomic position is directed along the line joining the atom to the molecular centre. Further refinement of anthracene (Cruickshank, private communication) has shown that the maximum error in an atomic position there is 0·006 Å and that librations about the length of the molecule predominate slightly.

A very similar result is to be expected here, since the thermal motion has been shown to be very much the same as that in anthracene. It is probably close enough for the present purpose to suppose that the atoms in groups *A*, *B* and *C* have been observed too near the molecular centre by 0·002, 0·004 and 0·006 Å respectively. The bond angles are little affected, but the measured intramolecular bond lengths must be too short by amounts up to about 0·003 Å. Intermolecular distances may be in error by 0·012 Å.

These errors in atomic positions are restricted to the plane of the molecule and so may be ignored in discussion of the small deviations from molecular planarity. They are also symmetrical in the molecule and do not affect the comparison of equivalent molecular parameters, i.e. those related by the apparent molecular symmetry. They do not affect comparisons of corresponding bond lengths in acridine and anthracene at this stage of refinement (Ahmed & Cruickshank, 1952). But they do, of course, affect comparison of the results with those predicted by theory or obtained by any other method. Since accurate correction is not at the moment practicable, the likely errors due to this effect have been added to standard deviations derived from those in Table 7 to give the final estimations of accuracy in the structural parameters.

Description of the structure

Arrangement of the molecules

The crystal structure is shown in Fig. 5 in a convenient orientation for comparison with the structure of anthracene shown in Fig. 2 of Sinclair *et al.* (1950). The two structures are clearly related, but in acridine III the non-centrosymmetrical molecules are arranged in antiparallel pairs about the centres of symmetry instead of being arranged individually at centres of symmetry as in anthracene. This pairing of the molecules is very similar to that in pyrene (Robertson & White, 1947) and benzperylene (White, 1948) shown clearly by Wyckoff (1953, Figs. XIV*A*, 22*b* and 23*b*). The parallel molecules do not lie directly over each other but are staggered, as shown in Fig. 6(*a*), in a way similar to that found in ovalene (Donaldson & Robertson, 1953) with no two atoms overlapping exactly. Parallel molecules in pyrene and benzperylene, and also in α-phenazine in which the molecular packing is of a different type (Herbstein & Schmidt, 1955), appear to be superimposed more nearly like adjacent layers in graphite. The intermolecular spacing in acridine is 3·47 Å, rather larger than that in ovalene (3·45 Å), benzperylene (3·38 Å) or graphite (3·41 Å) but less than that in pyrene (3·53 Å). Corresponding interatomic distances are shown in Fig. 6. The shortest are N–C'_{12} (3·484 Å), C_1–C'_{13} (3·476 Å) and H_1–C'_{13} (3·47 Å).

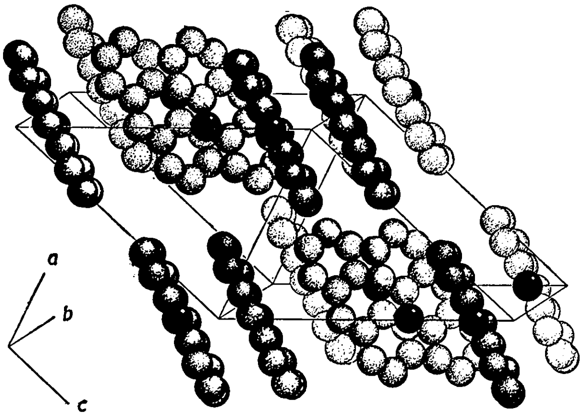
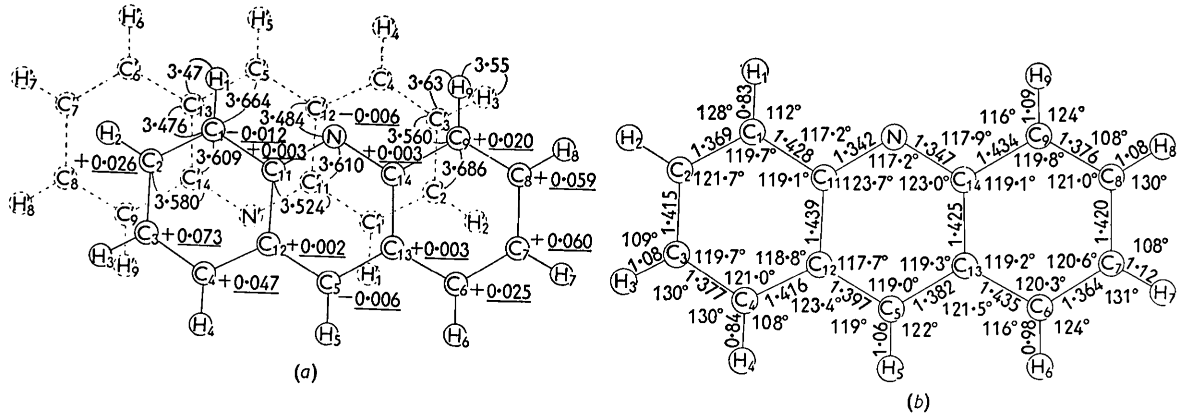


Fig. 5. Clinographic projection of the structure.

The packing of these twofold molecular groups appears to depend principally on intermolecular contacts of the kind usually found between benzene rings: all the intermolecular C–C distances concerned are longer than 3·6 Å. The separation of molecules related by the *b*-axis unit-cell translation, however, is rather less than that in anthracene. The distance N–C''_5 is



(*a*) (*b*)

Fig. 6. (*a*) Normal projection of two parallel molecules showing deviations from planarity (underlined) and interatomic distances (Å). (*b*) Measured bond lengths (Å) and angles.

3·65 Å while in anthracene the corresponding distance is 3·75 Å.

Molecular dimensions

Bond lengths and angles calculated from the final atomic coordinates in Tables 2 and 4 are shown in Fig. 6(*b*). The standard deviation for random errors in C–C type bond lengths varies between 0·006 Å for bonds in the central ring to 0·011 Å for the C_2–C_3, C_7–C_8 bonds. Standard deviations in corresponding bond angles vary between 0·3° and 0·5°. Very little significance can be attached to the observed values of bond lengths and angles which involve the positions of hydrogen atoms. Appropriate standard deviations are 0·10 Å and 6°.

The molecule is expected from its chemical properties to have an axis of symmetry passing through the atoms C_5 and N. There are, however, some quite large differences between chemically equivalent molecular parameters. The largest difference between equivalent bond lengths, 0·019 Å between C_4–C_{12} and C_{13}–C_6, is just 'possibly significant' (Cruickshank & Robertson, 1953) with $\Delta/\sigma = 1\cdot 96$. The difference between the bond angles C_4–C_{12}–C_5 and C_5–C_{13}–C_6 is 'highly significant' ($\Delta/\sigma = 4\cdot 0$), while that between the related angles C_{11}–C_{12}–C_5 and C_{14}–C_{13}–C_5 is 'significant' ($\Delta/\sigma = 3\cdot 0$). Parameter-by-parameter comparison of the two halves of the molecule suggests, therefore, that the bond configurations at atoms C_{12} and C_{13}, the positions of which are involved in all the significant differences noted above, may not be the same. A more definite conclusion about the molecular symmetry may be reached, when refinement is completed and more reliable standard deviations in atomic positions are available, by comparison of the half molecules in a multivariate significance test, similar to that described by Cruickshank & Robertson (1953), which would take into account the correlation of errors between the different parameters.

Although the molecular asymmetry may be real it is useful to work out the average values of chemically equivalent bond lengths for comparison with those observed in anthracene and phenazine and those predicted by theory. They are given in Table 8, together with standard deviations calculated on the assumption that the averaged bond lengths should in fact be equal and augmented by the likely values (0·003 Å) of systematic errors due to the molecular librations.

Table 8. *Average lengths of chemically equivalent bonds compared with those predicted by M.O. theory and those observed in anthracene and phenazine*

Values in Ångström units

Bond	Acridine	$\sigma(l)$	M.O.	Anthracene	Phenazine
C_{11}–C_1	1·431	0·008	1·400	1·424	1·408
C_1–C_2	1·372	0·009	1·380	1·371	1·375
C_2–C_3	1·417	0·010	1·400	1·408	1·412
C_3–C_4	1·370	0·009	1·375	1·371	1·375
C_4–C_{12}	1·425	0·008	1·425	1·424	1·408
C_{12}–C_5	1·389	0·007	1·415	1·396	—
C_{11}–C_{12}	1·432	0·007	1·435	1·436	1·433
C_{11}–N	1·344	0·007	1·394	—	1·345

Molecular planarity

The molecule is not strictly planar. The best plane through the heavier atoms is given by

$$0\cdot 765x'+0\cdot 415y+0\cdot 492z' = 1\cdot 736\,, \qquad (2)$$

where $x' = x+z\cos\beta$, $z' = z\sin\beta$ are atomic coordinates measured in Ångström units in the orthogonal coordinate system **a**, **b**, **c***. This was the plane used in plotting the difference-synthesis section shown in Fig. 3. The perpendicular distances of the atoms from this plane (2) are given in Table 9, where positive deviations indicate increased distance from the origin. Application of the χ^2 test (e.g. Weatherburn, 1949), with standard deviations in atomic positions perpendicular to the plane given by $\sigma_\perp^2 = l^2\sigma^2(x)+m^2\sigma^2(y)+n^2\sigma^2(z)$, shows that the deviations from planarity are 'highly significant' ($\chi^2=418\cdot 3$; $\nu=11$; $P \ll 0\cdot 001$).

Table 9. *Perpendicular distances of atoms from possible molecular planes*

Values in Ångström units

Atom	Plane (2)	Plane (3)	Plane (4)	Plane (5)
C_1	−0·021	−0·020	—	−0·012
C_2	+0·013	−0·009	—	+0·026
C_3	+0·051	+0·022	—	+0·073
C_4	+0·019	+0·006	—	+0·047
C_5	−0·038	−0·010	+0·006	−0·006
C_6	−0·009	—	−0·007	+0·025
C_7	+0·030	—	+0·008	+0·060
C_8	+0·038	—	+0·008	+0·059
C_9	+0·005	—	−0·010	+0·020
$N_{(10)}$	−0·018	+0·021	+0·007	−0·006
C_{11}	−0·013	+0·005	—	+0·003
C_{12}	−0·023	−0·013	—	+0·002
C_{13}	−0·025	—	−0·006	+0·003
C_{14}	−0·015	—	−0·005	+0·003

A likely form of molecular distortion is by bending across the line joining atoms C_5 and N. The best planes through the half-molecules having these atoms in common are

$$0\cdot 755x'+0\cdot 416y+0\cdot 506z' = 1\cdot 713 \qquad (3)$$

through atoms 10, 11, 1, 2, 3, 4, 12 and 5 and

$$0\cdot 776x'+0\cdot 412y+0\cdot 478z' = 1\cdot 691 \qquad (4)$$

through atoms 5, 13, 6, 7, 8, 9, 14 and 10. The deviations of the atoms from these planes also are shown in Table 9. χ^2 tests show that they are significant since for plane (3) $\chi^2 = 92\cdot 7$, $\nu = 5$, $P < 0\cdot 001$ and for plane (4) $\chi^2 = 20\cdot 8$, $\nu = 5$, $P \simeq 0\cdot 001$. Even when uncertainties in the estimations of $\sigma_\perp$, by as much as a factor of 2, are taken into account it seems certain that the molecule is not strictly planar and that the distortion involves more than just a bending across the C_5–N line. The deviations of the atoms from plane (4) are not so certainly significant.

It is perhaps most illuminating to consider the deviations of atoms from the best plane through the central pyridine ring alone, given by

$$0\cdot767x'+0\cdot409y+0\cdot494z' = 1\cdot719\ . \qquad (5)$$

These deviations are listed in Table 9 and underlined in Fig. 6(*a*). It is apparent that the more nearly planar half of the molecule (4) is the less involved in bonding between the two molecules. The deviations from planarity suggest that the irregularities in molecular shape can be described as the result of two distortions. The molecule appears to be bent across the line C_5–N through about 2° (calculated from planes (3) and (4)) so that the atoms C_5 and N approach the neighbouring molecule more closely. The deviation of H_5 from the best plane, which is partly responsible for the small peak height in Fig. 3. although too small itself to be significant, is consistent with this description. The less planar half of the molecule then seems to have been twisted in order to reduce the separation between C_1 and the neighbouring molecule.

Discussion

The differences between chemically equivalent molecular parameters are probably real and due to perturbation of the molecular state by molecular interactions. The striking difference in fluorescence properties between anthracene and acridine (Sangster, 1952) may be due to the same cause. The peculiarities in molecular shape certainly appear to be closely connected with the arrangement of molecules in antiparallel pairs, since similarly constituted molecules (anthracene and phenazine) in more symmetrical environments have been found to be planar. Detailed comparison of this structure with those of anthracene and the other polymorphic forms of acridine may lead to a better understanding of the intermolecular forces involved and may show also to what extent they influence the molecular dimensions.

The irregularities in molecular shape complicate further the theoretical calculation of bond lengths. The lack of planar symmetry to some extent invalidates clear-cut division of valence electrons into those of σ- or of π-character. It indicates also that the state of hybridization of some atoms, particularly C_5 and N, is not of the ideal trigonal (sp^2) type. This result is not surprising since the ready formation of addition compounds by reactions at these atoms (e.g. Albert, 1951) has shown that their state of hybridization can be changed fairly easily. The angle C_{11}–N–C_{14} (117·2°), which is in agreement with corresponding angles in phenazine (116·6° mean), is significantly less than 120°, though not by as much as the C–N–C angles in *s*-triazine (Wheatley, 1955), which are only 113·2°. It is probable, however, that these effects involve only small changes in the bond lengths (e.g. Coulson *et al.*, 1951).

Mobile bond orders and π-electron densities in acridine were calculated by Longuet-Higgins & Coulson (1947). They used Coulomb integrals $\alpha+2\beta$ for nitrogen, $\alpha+\frac{1}{4}\beta$ for carbon directly bonded to nitrogen, and α for all other carbon atoms, where α is the Coulomb integral for a carbon atom in benzene and β is the resonance integral for a C–C bond in benzene. Bond-lengths for the C–C bonds, derived from these bond orders by use of the bond-order/length curve quoted by Coulson *et al.* (1951), are given in Table 8. The C–N bond length was derived by use of the much less well established bond-order/length relationship proposed by Cox & Jeffrey (1951). There is fairly good general agreement between the observed and predicted bond lengths (maximum deviation 0·031 Å, r.m.s. deviation 0·017 Å). It is much less striking, however, than that obtained between the observed lengths of corresponding bonds in anthracene, acridine and phenazine, also shown in Table 8. The largest deviation between corresponding C–C bonds in acridine and anthracene is 0·009 Å, with r.m.s. deviation 0·005 Å. The agreement between C–C bond lengths in acridine and phenazine is less good (maximum deviation 0·023 Å, r.m.s. deviation 0·012 Å) but the agreement between C–N bond lengths in the two molecules is almost exact.

It has been recognized for some time (Coulson, 1952) that in the molecular orbital calculations quoted above too great an allowance was made for the difference in electronegativity between carbon and nitrogen. A better value for the Coulomb integral of nitrogen in this type of molecule is now known to be $\alpha+0\cdot6\beta$ (e.g. Löwdin, 1951). This conclusion is supported by the present result, which suggests that in the molecules considered carbon and nitrogen are very nearly equivalent. It is interesting to notice further that if the C–N bond is assigned the same order (1·606) as the corresponding C–C bond in anthracene the length predicted from the Cox–Jeffrey curve agrees very closely with that observed. This result must be interpreted with caution since the bond-order/length curve is not well established. The 'double-bond' length is uncertain and the intermediate points for pyridine, melanine and pyrrole are based on poor estimations of relative electronegativity in carbon and nitrogen. Revision of the curve in the light of recent measurements seems to depend on the development and extension of bond-order calculations. The present indications are that it is not much in error for bond-orders near 0·6.

The standard deviation in electron density calculated by Cruickshank's formula is only $\Delta\varrho = 0\cdot10$ e.Å^{-3} but the presence in the final difference synthesis of features due to systematic errors in the intensity estimations and the incomplete refinement of the thermal-motion parameters reduces the significance which can be attached to detail in the apparent electron-density distribution. It is interesting to notice that there is not even an indication of the accumulation of electrons at the nitrogen atom predicted by theory (Longuet-Higgins & Coulson, 1947): in fact the nitrogen atom is associated with a hole in the difference density. But this effect appears also in anthracene at this stage of refinement (Cruickshank, private communication) where it has now been shown to be due entirely to the predominance of molecular librations about the length of the molecule, the axis of lowest moment. Almost certainly it originates here in the same way.

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References

ABRAHAMS, S. C., ROBERTSON, J. M. & WHITE J. G. (1949). *Acta Cryst.* **2**, 233.

AHMED, F. R. & CRUICKSHANK, D. W. J. (1952). *Acta Cryst.* **5**, 852.

ALBERT, A. (1951). *The Acridines.* London: Arnold.

BARNES, W. H. & HANSON, A. W. (1950). Unpublished.

BOOTH, A. D. (1946). *Proc. Roy. Soc.* A, **188**, 77.

COCHRAN, W. (1948). *J. Sci. Instrum.* **25**, 253.

COCHRAN, W. (1951). *Acta Cryst.* **4**, 408.

COCHRAN, W. (1952). *Acta Cryst.* **5**, 630.

COCHRAN, W. & DYER, H. B. (1952). *Acta Cryst.* **5**, 634.

COULSON, C. A. (1951). *Proc. Roy. Soc.* A, **207**, 91.

COULSON, C. A. (1952). *Valence.* Oxford: Clarendon Press.

COULSON, C. A., DAUDEL, R. & ROBERTSON, J. M. (1951). *Proc. Roy. Soc.* A, **207**, 306.

COX, E. G. & JEFFREY, G. A. (1951). *Proc. Roy. Soc.* A, **207**, 110.

COX, E. G., CRUICKSHANK, D. W. J. & SMITH, J. A. S. (1955). *Nature, Lond.* **175**, 766.

CRUICKSHANK, D. W. J. (1949). *Acta Cryst.* **2**, 65.

CRUICKSHANK, D. W. J. (1952). *Acta Cryst.* **5**, 511.

CRUICKSHANK, D. W. J. & ROBERTSON, A. P. (1953). *Acta Cryst.* **6**, 698.

DONALDSON, D. M. & ROBERTSON, J. M. (1953). *Proc. Roy. Soc.* A, **220**, 157.

GOTTLIEB, C. C. & KATES, J. (1955). *Computers and Automation*, **4**, No. 4, 35.

GREMS, M. D. & KASPER, J. S. (1949). *Acta Cryst.* **2**, 347.

GRISON, E. (1951). *Acta Cryst.* **4**, 489.

HAMILTON, W. C. (1955). *Acta Cryst.* **8**, 185.

HARKER, D. & KASPER, J. S. (1948). *Acta Cryst.* **1**, 70.

HERBSTEIN, F. H. & SCHMIDT, G. M. J. (1955). *Acta Cryst.* **8**, 399.

HIGGS, P. W. (1955). *Acta Cryst.* **8**, 99.

International Tables for X-ray Crystallography (1952). Birmingham: Kynoch Press.

Internationale Tabellen zur Bestimmung von Kristallstrukturen (1935). Berlin: Borntraeger.

KOFLER, A. (1943). *Ber. dtsch. chem. Ges.* **76**, 871.

LIPSON, H. & COCHRAN, W. (1954). *The Determination of Crystal Structures.* London: Bell.

LONGUET-HIGGINS, H. C. & COULSON, C. A (1947). *Trans. Faraday Soc.* **43**, 87.

LOWDE, R. D., PHILLIPS, D. C. & WOOD, R. G. (1953). *Acta Cryst.* **6**, 553.

LÖWDIN, P. (1951). *J. Chem. Phys.* **19**, 1323.

LU, CHIA-SI (1943). *Rev. Sci. Instrum.* **14**, 331.

MCWEENY, R. (1951). *Acta Cryst.* **4**, 513.

PARRY, G. S. (1954). *Acta Cryst.* **7**, 313.

PAULING, L. (1940). *The Nature of the Chemical Bond.* Ithaca: Cornell University Press.

PHILLIPS, D. C. (1950). *Research, Lond.* **3**, 578.

PHILLIPS, D. C. (1954*a*). *Acta Cryst.* **7**, 649.

PHILLIPS, D. C. (1954*b*). *Acta Cryst.* **7**, 746.

PHILLIPS, D. C. (1954*c*). *Acta Cryst.* **7**, 221.

ROBERTSON, J. M. (1951). *Proc. Roy. Soc.* A, **207**, 101.

ROBERTSON, J. M. & WHITE, J. G. (1947). *J. Chem. Soc.* p. 358.

ROGERS, D. & WILSON, A. J. C. (1953). *Acta Cryst.* **6**, 439.

SANGSTER, R. C. (1952). Technical Report No. 55. Laboratory for Nuclear Science and Engineering, Massachusetts Institute of Technology.

SHOEMAKER, D. P., DONOHUE, J., SCHOMAKER, V. & COREY, R. B. (1950). *J. Amer. Chem. Soc.* **72**, 2328.

SINCLAIR, V. C., ROBERTSON, J. M. & MATHIESON, A. McL. (1950). *Acta Cryst.* **3**, 251.

WEATHERBURN, C. E. (1949). *A First Course in Mathematical Statistics.* Cambridge: University Press.

WHEATLEY, P. J. (1953). *Acta Cryst.* **6**, 369.

WHEATLEY, P. J. (1955). *Acta Cryst.* **8**, 224.

WHITE, J. G. (1948). *J. Chem. Soc.* p. 1398.

WIEBENGA, E. H. & SMITS, D. W. (1950). *Acta Cryst.* **3**, 265.

WYCKOFF, R. W. G. (1953). *Crystal Structures.* New York: Interscience Publishers.