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A Refinement of the Crystal Structure of Azobenzene

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The crystal structure of *trans*-azobenzene has been refined further, from three-dimensional X-ray diffraction data by electronic computing methods. The structure previously published by de Lange, Robertson & Woodward has been confirmed as essentially correct and this refinement introduces only minor modifications to the atomic coordinates. The two molecules comprising the asymmetric unit are still apparently non-equivalent, but from the anisotropic thermal parameters which have been calculated, one of these two molecules is able to adopt more than one configuration, and the averaging of this disorder accounts for the variations in bond lengths and inter-bond angles.

Introduction

Previous X-ray work on azobenzene included the determination of the unit-cell dimensions and space group by Prasad (1930) and an approximate analysis of the structure by de Lange, Robertson & Woodward (1939). The latter work, which involved Fourier refinement of the **b** projection only, showed the two non-equivalent molecules to have slightly different geometrical configurations, but the magnitude of the differences was within the limits of experimental error, and no attempt was made to refine the y atomic coordinates. In order to assist our understanding of the behaviour of more complex azo compounds, it was decided to reinvestigate azobenzene to ascertain the significance of these differences between the molecules.

Experimental

A commercial sample of azobenzene was recrystallized from ethanol. The diamond-shaped plates were tabular on (001) with **a** bisecting the acute angle. Large plates were allowed to grow, and suitable sections for X-ray photography were cut with a sharp edge. The unit-cell dimensions were redetermined from the measurements of high order reflexions on Weissenberg photographs to be:

$$a=12\cdot144,\ b=5\cdot756,\ c=15\cdot396\ \text{Å},\ \beta=114°8'.$$

The previous orientation of the unit cell, which shows the relationship to the dibenzyl series, is here retained, despite c being the longest axis. For four molecules of $C_{12}H_{10}N_2$ per unit cell, the calculated specific gravity is 1·230, while that determined by flotation in sodium iodide solution was 1·224. The space group is unambiguously $P2_1/a$. Weissenberg photographs for intensity measurement were obtained about **a** (zero and ten layers), **b** (zero and four layers), and about [101] (zero layer only), thus covering almost all the sphere of reflexion obtainable with Cu $K\alpha$ radiation. Multiple films, in two sets of three, were used, and the intensities of the reflexions estimated by comparison with a time-exposure calibrated film strip. The zero layers about **a** and **b** were also measured on an integrating micro-densitometer. Correction factors for polarization and the geometry of the system were applied in the usual way, and experimental structure amplitudes of 2225 planes obtained. Of these, 579 were too weak on the longest exposed photograph to be assigned accurate values, so that 1646 terms were actually used in the refinement.

Refinement of the structure

The approximate structure was already known, so that was used as the starting point. Several sets of **b** electron density projections and $h0l$ structure amplitudes were calculated manually to improve the x and z coordinates. The best electron density projection is reproduced in Fig. 1, from which it may be seen that there was some uncertainty in fixing the atomic centres owing to several pairs of atoms overlapping. The best x and z values were used together with the y coordinates of de Lange, Robertson & Woodward (1939) to calculate some $F(hkl)$. Only about the first 175 terms gave sufficiently good agreement with experiment to be certain of their phases. Successive sets of syntheses along lines parallel to **b** were computed manually, using Beevers–Lipson strips, followed by recalculation of structure factors until the phases of almost all the terms were known. All the terms were then used for computing sections perpendicular to **b** through each of the atoms in turn, with the result shown in Fig. 2, which is a composite electron density map.

The atomic coordinates from these last lines and sections were then used to compute three cycles of structure factors and differential syntheses on the Man-

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chester University Mark 1 electronic computer; these were kindly carried out by Dr P. J. Wheatley. For the second and third of these cycles, hydrogen atoms and individual isotropic temperature factors for the carbon and nitrogen atoms were incorporated in the syntheses. The hydrogen positional coordinates were not refined by the computer, nor were the temperature factors; these were used to improve the structure amplitude agreement, which was reduced to 13·6% at this stage. The positions of the hydrogen atoms are shown clearly on the **b** difference projection, using values of F(obs) − F(cal), for carbon and nitrogen only, as coefficients; the projection is reproduced in Fig. 3.

After an interval of several years, it became possible to carry out refinement of anisotropic temperature parameters on our own Ferranti Pegasus computer, using programs prepared by Cruickshank & Pilling (1961). A further seven cycles of structure factor and least-squares refinement calculations were carried out, with the eventual reduction of the residual R to 6·9%. At this stage the shifts indicated both for the coordinates and for the anisotropic temperature factors were less than the corresponding standard deviations, and the refinement process was judged to be complete for the experimental data available. Table 1 gives the final atomic coordinates and temperature factors. Table 2 gives the experimental and calculated structure am-

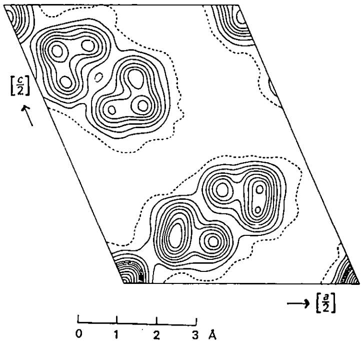

Fig. 1. Two-dimensional projection along **b**. Contours are at intervals of 1 e.Å^{-2}, the broken line being the zero contour.

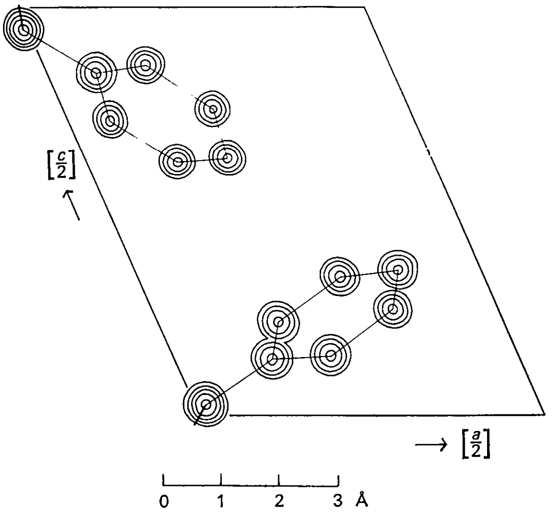

Fig. 2. Composite three-dimensional electron density map, showing sections through each atom. Contours are at intervals of 1 e.Å^{-3}, the lowest being 3 e.Å^{-3}.

Table 1. *Atomic coordinates and thermal parameters*

The values of B are defined by the coefficients in the expression

$$\exp\left[-\tfrac{1}{4}(h^2a^{*2}B_{11}+2hka^*b^*B_{12}+\ldots)\right]$$

used in the calculation of the structure amplitudes.

	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{12}	B_{23}	B_{13}
C(1)	0·1409	0·1338	0·0678	3·25	3·55	3·50	−0·24	0·13	1·28
C(2)	0·1732	0·3451	0·1142	4·36	3·84	4·82	−0·31	−0·58	1·71
C(3)	0·2940	0·3898	0·1709	4·83	4·14	4·85	−0·89	−0·87	1·39
C(4)	0·3808	0·2304	0·1780	3·81	5·27	4·29	−0·71	0·05	0·90
C(5)	0·3482	0·0214	0·1290	3·55	4·83	4·47	0·18	0·06	1·28
C(6)	0·2285	−0·0275	0·0741	3·88	3·72	3·78	−0·06	−0·14	1·28
N(1)	0·0145	0·1027	0·0114	3·43	3·41	4·22	−0·09	−0·22	1·33
C(7)	0·0705	−0·0469	0·4211	4·01	6·32	4·09	0·73	0·74	1·43
C(8)	0·0621	−0·2299	0·3615	4·83	5·60	6·01	−0·22	−0·62	1·87
C(9)	0·1310	−0·2320	0·3096	5·33	6·66	5·04	0·26	−0·93	1·75
C(10)	0·2072	−0·0521	0·3163	5·00	7·91	4·22	0·55	0·30	1·93
C(11)	0·2151	0·1338	0·3756	5·48	6·50	4·90	−0·55	0·38	1·57
C(12)	0·1471	0·1369	0·4284	4·50	5·67	4·12	0·24	−0·52	1·26
N(2)	−0·0082	−0·0744	0·4720	5·60	5·40	5·76	−0·03	−0·84	1·58
H(2)	0·1052	0·4741	0·1064	Mean isotropic B=3·60					
H(3)	0·3192	0·5513	0·2099						
H(4)	0·4743	0·2669	0·2218						
H(5)	0·4167	−0·1034	0·1340						
H(6)	0·2032	−0·1904	0·0363						
H(8)	0·0014	−0·3721	0·3557	Mean isotropic B=4·80					
H(9)	0·1247	−0·3767	0·2635						
H(10)	0·2611	−0·0547	0·2756						
H(11)	0·2747	0·2769	0·3807						
H(12)	0·1534	0·2813	0·4753						

Table 2. *Observed and calculated structure amplitudes*

H	K	L	F(OBS)	F(CAL)
0	0	1	7.9	+7.5
0	0	2	38.0	+38.0
0	0	3	8.3	+7.2
0	0	4	55.2	−55.2
0	0	5	6.1	−4.2
0	0	6	15.6	+15.3
0	0	7	9.5	+9.9
0	0	8	14.7	−13.9
0	0	9	2.0	+2.4
0	0	10	6.8	+6.2
0	0	11	4.8	+4.6
0	0	12	6.2	+6.2
0	0	13	4.7	+4.5
0	0	14	3.1	+3.2
0	0	16	9.5	+9.8
0	0	17	5.3	+4.8
0	0	18	1.9	−2.1
2	0	−18	3.9	−4.5
2	0	−17	8.5	+8.6
2	0	−16	7.3	−6.8
2	0	−15	4.0	+3.9
2	0	−14	5.3	+5.9
2	0	−13	9.5	−9.0
2	0	−12	2.7	−2.1
2	0	−10	12.1	+10.7
2	0	−9	3.8	−3.8
2	0	−8	12.8	+12.5
2	0	−7	2.7	+2.8
2	0	−6	17.4	+15.2
2	0	−5	68.1	+69.0
2	0	−4	118.2	+116.6
2	0	−3	70.0	+68.9
2	0	−2	29.4	−29.3
2	0	−1	5.8	+5.0
2	0	0	28.8	−28.6
2	0	1	72.3	−71.2
2	0	2	110.8	+109.9
2	0	3	46.9	−46.9
2	0	4	1.7	+1.2
2	0	5	4.7	−4.3
2	0	6	16.4	+15.8
2	0	7	16.0	+15.4
2	0	8	20.0	+19.3
2	0	9	22.8	+23.2
2	0	10	6.8	−7.5
2	0	11	8.1	+7.5
2	0	12	3.4	+3.2
2	0	16	3.4	−2.9
4	0	−18	1.4	+1.7
4	0	−17	2.7	−3.4
4	0	−16	8.2	−7.9
4	0	−15	9.1	+9.0
4	0	−14	3.1	−2.9
4	0	−13	12.1	+11.8
4	0	−12	9.8	+10.7
4	0	−11	8.0	+7.6
4	0	−10	4.0	+4.1
4	0	−9	11.0	+9.2
4	0	−8	39.4	+38.8
4	0	−7	17.0	−17.6
4	0	−6	35.6	+35.1
4	0	−4	2.9	−2.2
4	0	−3	18.9	+19.5
4	0	−2	27.3	+26.7
4	0	−1	13.5	−13.4
4	0	0	3.8	−2.5
4	0	1	3.2	−3.6
4	0	2	27.1	+27.6
4	0	3	25.8	+25.6
4	0	4	43.8	+43.9
4	0	5	17.8	+16.6
4	0	6	5.8	+5.3
4	0	7	10.4	−9.3
4	0	8	3.4	+3.5
4	0	9	5.8	−5.3
4	0	11	8.5	+8.4
4	0	12	3.7	−4.0
4	0	13	5.3	+5.3
4	0	15	1.6	−1.6
6	0	−19	1.0	+0.7
6	0	−18	1.2	+2.2
6	0	−17	6.8	+5.9
6	0	−16	4.3	+4.9
6	0	−15	4.0	+2.9
6	0	−14	1.9	−1.0
6	0	−13	2.2	+1.7
6	0	−11	5.8	+4.3
6	0	−10	7.1	+8.3
6	0	−9	25.1	+24.5
6	0	−8	11.4	+10.7
6	0	−7	8.1	+7.2
6	0	−6	26.9	+36.8
6	0	−5	35.0	−34.6
6	0	−4	21.9	+21.8
6	0	−2	14.9	+14.7
6	0	−1	27.7	+27.2
6	0	0	25.0	+24.8
6	0	1	4.0	+3.2
6	0	2	18.7	+18.7
6	0	3	15.7	−16.2
6	0	4	4.5	+4.5
6	0	5	3.4	+3.1
6	0	7	11.6	+11.2
6	0	8	6.5	+6.1
6	0	9	3.2	+3.5
6	0	10	1.1	+0.9
6	0	11	4.0	−4.2
6	0	12	4.6	−4.7
8	0	−18	1.0	+1.8
8	0	−16	1.8	−0.8
8	0	−15	7.6	+7.2
8	0	−14	1.9	+2.9
8	0	−13	14.0	+12.9
8	0	−12	12.3	+11.9
8	0	−11	2.0	+0.9
8	0	−10	1.8	+2.1
8	0	−9	7.6	−7.6
8	0	−7	7.1	+6.3
8	0	−6	7.6	+8.6
8	0	−4	12.4	+12.8
8	0	−3	3.4	−3.4
8	0	−2	19.3	+17.3
8	0	−1	8.9	+8.3
8	0	0	10.3	+10.3
8	0	1	11.3	+11.0
8	0	2	2.0	+1.5
8	0	3	14.3	+14.3
8	0	5	5.5	−5.2
8	0	6	1.5	+1.2
8	0	7	4.3	−4.5
8	0	8	6.5	−6.7
8	0	11	3.4	+3.3
10	0	−19	3.1	+2.6
10	0	−18	1.4	+2.3
10	0	−17	3.8	+3.1
10	0	−16	4.8	+5.8
10	0	−15	1.3	−1.0
10	0	−14	4.0	−3.0
10	0	−13	2.8	+2.1
10	0	−12	7.0	−6.1
10	0	−11	5.2	+4.3
10	0	−10	3.4	+4.1
10	0	−9	3.3	−3.9
10	0	−7	4.7	+4.2
10	0	−6	20.1	+19.7
10	0	−5	11.9	+11.9
10	0	−4	27.3	+27.1
10	0	−2	3.3	−3.0
10	0	−1	2.4	−2.5
10	0	0	1.4	−1.1
10	0	2	1.7	−1.4
10	0	4	5.0	−5.2
10	0	6	5.1	+5.4
12	0	−14	1.9	+2.8
12	0	−13	1.5	−2.3
12	0	−11	9.8	+8.4
12	0	−10	9.8	+9.2
12	0	−9	16.3	+15.5
12	0	−8	12.6	+12.2
12	0	−7	2.2	+1.7
12	0	−6	11.5	−10.5
12	0	−5	6.5	−6.4
12	0	−4	2.2	+1.5
12	0	−3	7.6	−9.1
12	0	−2	11.0	+11.9
12	0	−1	4.3	−4.3
12	0	0	2.9	−2.1
12	0	1	4.4	+4.1
12	0	2	2.2	+2.1
12	0	3	3.6	+3.3
12	0	4	1.1	+1.0
12	0	5	2.5	+2.3
14	0	−15	2.5	+2.4
14	0	−14	4.8	+5.2
14	0	−13	6.5	+5.4
14	0	−12	4.7	+5.3
14	0	−11	2.5	−2.7
14	0	−10	3.6	−3.5
14	0	−9	3.1	−3.0
14	0	−8	1.4	−1.5
14	0	−6	2.2	+2.1
14	0	−5	2.0	+2.1
14	0	−4	3.4	−3.5
14	0	−3	3.3	+3.5
14	0	−2	7.7	+6.4
14	0	0	7.9	+7.3
0	1	1	18.5	−19.0
0	1	2	68.1	−67.2
0	1	3	34.3	−34.0
0	1	4	38.5	+38.4
0	1	5	1.2	−0.7
0	1	6	14.3	−14.3
0	1	7	5.2	+5.5
0	1	8	10.8	−11.3
0	1	9	3.0	−3.1
0	1	10	4.6	+4.6
0	1	11	2.4	+2.9
0	1	12	8.8	−8.2
0	1	13	1.7	−1.8
0	1	15	2.1	+2.3
1	1	−18	1.2	−1.1
1	1	−17	8.2	+8.1
1	1	−16	3.3	+3.8
1	1	−15	1.3	+1.1
1	1	−14	3.6	+3.8
1	1	−13	2.2	−2.3
1	1	−11	2.8	−3.0
1	1	−10	6.7	+6.0
1	1	−9	8.8	−8.4
1	1	−8	12.3	−12.9
1	1	−7	3.2	+3.6
1	1	−6	2.1	+1.5
1	1	−5	33.9	+33.9
1	1	−4	64.1	+64.0
1	1	−3	63.7	+62.8
1	1	−2	28.7	+28.5
1	1	−1	10.0	+9.7
1	1	0	66.9	+66.1
1	1	1	68.7	−67.8
1	1	2	70.0	+69.2
1	1	3	25.4	−25.7
1	1	4	2.0	−1.9
1	1	5	5.4	+6.2
1	1	6	2.8	+2.3
1	1	7	5.2	+6.0
1	1	8	9.7	−9.5
1	1	9	9.5	+9.8
1	1	11	8.3	+7.9
1	1	12	13.8	+13.1
1	1	13	8.4	+8.1
1	1	14	3.5	+3.3
1	1	16	3.1	−2.6
1	1	17	4.2	−4.4
2	1	−18	0.8	−0.7
2	1	−17	1.1	+0.8
2	1	−16	3.4	+3.8
2	1	−15	1.5	−1.9
2	1	−14	4.6	−4.2
2	1	−13	5.2	+4.6
2	1	−11	3.1	+2.2
2	1	−10	15.1	+13.6
2	1	−9	16.7	−15.1
2	1	−8	4.9	−3.9
2	1	−7	12.4	+11.0
2	1	−6	34.6	+34.5
2	1	−5	30.8	+27.9
2	1	−4	21.0	+20.2
2	1	−3	25.1	−25.8
2	1	−2	31.0	−32.5
2	1	−1	39.8	−40.9
2	1	0	15.8	+15.8
2	1	1	14.9	+16.3
2	1	2	60.5	−61.6
2	1	3	31.7	+33.3
2	1	4	14.7	−15.9
2	1	5	6.6	−7.5
2	1	6	25.2	+27.0
2	1	7	5.9	−4.7
2	1	8	11.5	−11.7
2	1	9	1.5	−0.5
2	1	10	5.4	−5.2
2	1	11	1.7	+1.9
2	1	12	3.1	+2.4
2	1	14	5.7	−5.5
2	1	15	4.0	−3.5
2	1	16	3.7	−3.1
3	1	−17	3.2	+2.8
3	1	−16	7.7	−7.1
3	1	−15	7.3	+6.6
3	1	−14	6.1	−5.5
3	1	−13	3.6	−4.3
3	1	−11	2.9	−3.0
3	1	−10	15.8	+15.0
3	1	−9	16.2	+15.6
3	1	−8	48.1	+50.4
3	1	−7	19.9	+21.0
3	1	−6	10.8	+11.1
3	1	−5	7.4	−7.5
3	1	−4	23.5	−22.7
3	1	−3	14.2	−14.3
3	1	−2	37.2	+35.6
3	1	−1	13.5	+12.8
3	1	0	8.2	+8.4
3	1	1	8.0	+9.2
3	1	2	16.6	+19.4
3	1	3	23.0	−23.9
3	1	4	34.5	+36.7
3	1	5	3.9	−3.4
3	1	6	11.6	+12.1
3	1	7	10.1	+11.3
3	1	8	10.5	+11.3
3	1	9	10.2	+10.9
3	1	10	3.0	−3.3
3	1	11	3.0	+2.9
3	1	12	9.5	−8.8
3	1	13	3.3	−3.4
3	1	16	1.7	+1.4
4	1	−18	2.3	−2.0
4	1	−16	3.6	+3.6
4	1	−15	1.4	−1.9
4	1	−14	10.1	+10.4
4	1	−13	3.9	−4.3
4	1	−12	6.5	−5.9
4	1	−11	13.2	+12.7
4	1	−10	5.6	+6.0
4	1	−9	9.3	+8.4
4	1	−8	18.6	+18.1
4	1	−7	14.9	−15.5
4	1	−6	13.2	−12.6
4	1	−5	8.1	−8.7
4	1	−4	9.5	−9.3
4	1	−3	19.0	−17.8
4	1	−2	18.4	−17.3
4	1	−1	21.9	−21.0
4	1	0	22.3	+21.9
4	1	1	8.9	−9.8
4	1	2	23.1	+23.1
4	1	3	20.2	+20.2
4	1	4	30.0	−29.2
4	1	5	10.1	+9.9
4	1	6	12.8	−11.9
4	1	7	3.2	−3.1
4	1	8	1.8	+1.6
4	1	9	2.3	−2.2
4	1	10	6.2	−6.0
4	1	11	4.2	−4.0
4	1	12	1.9	−1.5
4	1	13	3.4	−3.0
4	1	14	1.0	−1.3
4	1	15	1.4	−1.2
5	1	−18	1.3	−1.1
5	1	−17	1.3	−1.0
5	1	−16	1.7	+2.0
5	1	−15	4.6	+4.1
5	1	−14	1.5	−0.7
5	1	−13	16.8	+16.3
5	1	−12	6.1	+6.4
5	1	−11	9.2	+8.1
5	1	−10	4.5	+2.6
5	1	−9	8.7	−8.1
5	1	−8	2.3	−1.7
5	1	−7	10.6	−9.0
5	1	−6	36.6	+36.9
5	1	−5	3.8	+3.6
5	1	−4	20.7	+21.3
5	1	−3	7.7	+6.0
5	1	−2	5.2	−4.1
5	1	−1	9.4	+7.6
5	1	0	37.9	+37.0
5	1	1	4.2	+3.6
5	1	2	13.8	+14.7
5	1	3	2.7	+3.1
5	1	4	1.7	+2.1
5	1	5	3.2	−2.3
5	1	6	8.0	+7.8
5	1	10	2.1	+1.8
5	1	11	3.1	+3.0
5	1	13	2.3	+2.5
5	1	14	2.9	−3.0
6	1	−19	2.4	+2.2
6	1	−17	2.9	+2.8
6	1	−16	2.1	−2.1
6	1	−15	3.3	+2.9
6	1	−14	5.4	+5.4
6	1	−13	4.9	−4.3
6	1	−12	10.3	+9.9
6	1	−11	4.3	−4.3
6	1	−10	3.0	−2.3
6	1	−9	1.2	−1.3
6	1	−8	6.2	−5.4
6	1	−7	19.0	−17.0
6	1	−6	13.7	−12.8
6	1	−5	3.7	+2.6
6	1	−4	1.5	−1.0
6	1	−3	20.6	+18.7
6	1	−2	15.6	+14.8
6	1	−1	9.5	+8.2
6	1	0	1.0	−1.4
6	1	1	9.8	−9.9
6	1	2	2.5	+2.2
6	1	3	11.9	−12.3
6	1	4	5.6	−5.8
6	1	5	1.5	−1.8
6	1	6	8.9	−8.3
6	1	8	3.8	−3.5
6	1	10	7.4	−6.9
7	1	−17	3.1	+2.2
7	1	−16	1.7	−1.2
7	1	−15	3.3	+2.7
7	1	−14	1.8	+2.7
7	1	−13	3.1	+2.1
7	1	−11	9.7	+8.0
7	1	−10	8.3	+8.2
7	1	−9	7.9	+6.1
7	1	−8	8.2	+8.7
7	1	−7	1.0	−0.6
7	1	−6	2.7	+2.7
7	1	−4	25.7	+28.3
7	1	−3	2.0	−2.2
7	1	−2	9.6	+9.7
7	1	−1	3.0	−3.2
7	1	0	13.8	−11.9
7	1	1	2.5	+2.7
7	1	2	18.3	+16.9
7	1	3	4.4	+4.6
7	1	4	16.2	+15.7
7	1	5	3.0	+3.1
7	1	7	1.6	−1.6
7	1	8	1.4	−1.6
7	1	10	3.7	−3.7
7	1	11	1.6	+1.3
7	1	12	1.0	−0.7
8	1	−19	1.2	+0.8
8	1	−16	1.5	−1.1
8	1	−15	1.7	+2.0
8	1	−14	1.7	−0.8
8	1	−12	1.5	−1.4
8	1	−11	8.6	−8.4
8	1	−10	4.3	−3.9
8	1	−9	1.4	−1.6
8	1	−8	11.2	+10.3
8	1	−7	1.3	−1.3
8	1	−6	15.9	+15.1
8	1	−4	6.8	−5.8
8	1	−3	11.0	+10.2
8	1	−2	10.3	−8.9
8	1	−1	4.9	−4.2
8	1	0	13.5	−12.6
8	1	1	9.1	−9.1
8	1	2	5.7	−5.0
8	1	6	6.8	−6.3
8	1	7	3.8	−3.1
8	1	8	1.7	+1.7
8	1	11	1.8	+1.6
9	1	−19	2.8	+2.1
9	1	−18	3.5	+4.1
9	1	−17	2.8	+1.9
9	1	−16	3.9	+4.8
9	1	−15	4.5	+4.1
9	1	−14	1.9	−1.6
9	1	−13	4.5	+4.0
9	1	−9	3.1	+2.8
9	1	−8	7.0	+7.3
9	1	−7	2.4	−2.9
9	1	−6	8.6	+8.8
9	1	−5	6.6	−7.3
9	1	−3	5.7	+5.8
9	1	−2	18.7	+18.8
9	1	−1	11.4	+11.6
9	1	0	14.7	+14.4
9	1	1	3.9	+4.0
9	1	2	7.3	−7.5
9	1	3	3.3	−4.2
9	1	4	4.0	−3.6
9	1	5	3.7	−4.8
9	1	6	2.4	+2.5
10	1	−19	1.0	−1.1
10	1	−15	2.0	−2.1
10	1	−14	2.5	−2.6
10	1	−12	4.9	+3.8
10	1	−10	1.2	+1.1
10	1	−9	3.4	+3.5
10	1	−8	2.9	−2.8
10	1	−7	3.7	+4.2
10	1	−6	8.4	+8.0
10	1	−5	15.2	−14.2
10	1	−4	4.9	−4.0
10	1	−3	10.3	−10.9
10	1	−2	9.7	−9.6
10	1	−1	6.2	+6.1
10	1	0	1.2	+1.1
10	1	1	1.3	+0.6
10	1	2	4.9	−5.0
10	1	3	3.0	−2.9
10	1	5	1.4	+1.5
10	1	8	3.5	−3.4
11	1	−18	1.9	−1.1
11	1	−15	1.5	−2.0
11	1	−12	2.2	−1.8
11	1	−11	4.7	+4.3
11	1	−10	1.4	+1.6
11	1	−9	5.4	+4.5
11	1	−8	3.7	+4.5
11	1	−7	10.1	+9.0
11	1	−6	10.7	+10.6
11	1	−5	2.2	+1.9
11	1	−4	13.2	+12.9
11	1	−3	8.4	−7.8
11	1	−2	2.2	−2.5
11	1	−1	4.2	−5.2
11	1	0	3.3	−3.5
11	1	2	1.6	+1.7
11	1	3	2.0	+2.2
11	1	4	1.4	−1.3
11	1	5	2.2	+2.5
11	1	6	2.0	+1.5
12	1	−12	1.4	+1.0
12	1	−11	2.0	+2.4
12	1	−10	6.5	+5.2
12	1	−9	4.7	−4.1
12	1	−8	7.9	−7.0
12	1	−7	4.2	−3.7
12	1	−6	8.4	−8.0
12	1	−4	6.4	+5.6
12	1	−3	3.3	−3.8
12	1	−2	2.0	−2.2
12	1	1	1.5	+1.5
12	1	3	2.0	−1.7
12	1	4	4.0	−3.2
13	1	−15	2.2	+1.8
13	1	−14	4.9	+4.5
13	1	−13	4.7	+3.4
13	1	−12	6.8	+7.0
13	1	−11	3.6	+3.1
13	1	−6	2.0	−2.2
13	1	−3	2.0	−2.2
13	1	−2	6.2	+5.9
13	1	−1	3.3	−3.6
13	1	0	3.6	+3.2
13	1	1	1.5	+1.4
13	1	2	2.7	+2.3
13	1	3	3.3	+3.8
14	1	−15	2.0	+2.1
14	1	−14	1.9	+1.6
14	1	−13	1.5	−1.1
14	1	−12	1.3	−2.1
14	1	−11	3.7	−3.8
14	1	−10	3.1	−3.2
14	1	−7	2.0	−1.8
14	1	−6	2.0	−1.8
14	1	−1	1.3	−1.1
14	1	0	4.0	−4.2
0	2	0	14.6	+14.5
0	2	1	11.4	+11.9
0	2	2	31.1	+30.7
0	2	3	11.5	−12.2
0	2	4	5.9	+6.2
0	2	5	14.8	−14.2
0	2	6	14.1	−13.7
0	2	7	9.3	+9.6
0	2	8	1.0	−0.7
0	2	9	5.9	+6.2
0	2	11	7.0	−6.1
0	2	12	2.9	+3.1
0	2	13	2.8	+2.3
0	2	14	3.4	+3.9
0	2	15	5.1	+4.9
0	2	17	1.0	−1.0
1	2	−17	3.3	+3.0
1	2	−14	2.3	−1.9
1	2	−13	2.3	−2.4
1	2	−12	5.1	+5.5
1	2	−11	6.0	−6.6
1	2	−10	5.7	+6.6
1	2	−9	3.1	+2.4
1	2	−8	7.0	+7.4
1	2	−7	9.3	+8.3
1	2	−6	30.8	+32.7
1	2	−5	9.4	−10.1
1	2	−4	6.9	+7.2
1	2	−3	9.5	−10.9
1	2	−2	11.3	−12.1
1	2	−1	15.5	+14.9
1	2	0	4.0	+4.4
1	2	1	19.2	+18.9
1	2	2	24.3	−24.2
1	2	3	11.4	−13.1
1	2	4	10.0	−11.6
1	2	5	18.4	−19.5
1	2	6	3.2	−3.7
1	2	7	5.0	+5.3
1	2	8	9.3	−10.9
1	2	9	4.3	+4.2
1	2	10	3.3	+3.5
1	2	11	3.0	+3.4
1	2	14	8.4	−8.5
1	2	15	3.3	−2.7
1	2	16	4.4	−4.4
2	2	−16	2.7	−2.6
2	2	−14	1.9	−1.7
2	2	−13	3.1	+2.4
2	2	−12	1.9	+2.0
2	2	−11	6.8	−7.1
2	2	−10	3.0	−2.7
2	2	−9	9.3	−8.0
2	2	−8	11.7	+11.0
2	2	−7	19.6	+20.1
2	2	−6	10.8	+11.7
2	2	−5	22.9	+23.6
2	2	−4	2.7	−2.5
2	2	−3	18.7	−17.8
2	2	−2	26.5	+26.5
2	2	−1	25.8	−26.5
2	2	0	9.6	−9.5
2	2	1	4.8	−5.1
2	2	2	14.7	+15.9
2	2	3	5.0	−5.2
2	2	4	27.9	+27.4
2	2	5	1.5	−0.7
2	2	6	9.4	−9.9
2	2	7	4.0	+4.0
2	2	8	1.4	+0.8
2	2	9	11.2	+11.6
2	2	10	6.1	+6.6
2	2	11	4.4	+4.8
2	2	12	3.8	−4.1
2	2	13	7.0	−7.2
2	2	14	1.7	−1.8
2	2	15	1.0	−1.1
2	2	16	0.9	−0.7
3	2	−17	1.3	−1.7
3	2	−15	2.9	−3.3
3	2	−14	1.5	+2.3
3	2	−13	1.5	+1.2
3	2	−11	11.9	+11.1
3	2	−10	18.1	+17.9
3	2	−9	6.5	+5.7
3	2	−8	13.4	+13.2
3	2	−7	7.3	−8.3
3	2	−6	14.1	−12.0
3	2	−5	6.8	−7.8
3	2	−4	12.6	+13.6
3	2	−3	5.8	−7.4
3	2	−2	15.2	+15.3
3	2	−1	26.0	−27.3
3	2	0	17.8	−16.8
3	2	1	17.1	−16.6
3	2	2	17.2	−17.7
3	2	3	18.5	+19.7
3	2	4	23.3	−24.7
3	2	5	14.2	+15.3
3	2	6	2.7	+0.8
3	2	7	2.6	+2.8
3	2	8	7.2	+7.3
3	2	9	2.5	−2.1
3	2	10	10.1	−10.0
3	2	11	9.8	−9.6
3	2	12	6.5	−6.7
3	2	13	3.8	−3.8
4	2	−17	1.2	+1.1
4	2	−14	6.7	−6.3
4	2	−13	7.3	+7.2
4	2	−12	8.1	+9.0
4	2	−11	7.0	+7.3
4	2	−10	15.6	+14.8
4	2	−9	6.4	−6.4
4	2	−8	9.0	−8.8
4	2	−7	12.5	−11.3
4	2	−6	1.2	+1.0
4	2	−5	3.6	−3.6
4	2	−4	9.6	−8.5
4	2	−3	12.5	+11.1
4	2	−2	15.9	+15.5
4	2	−1	2.7	+2.1
4	2	0	30.7	+30.6
4	2	1	1.9	−1.3
4	2	2	11.4	−10.8
4	2	3	1.7	−1.8
4	2	4	22.4	+20.3
4	2	5	7.0	−6.8
4	2	6	13.5	+13.5
4	2	8	18.8	−17.9
4	2	9	3.0	−2.8
4	2	10	3.3	−3.1
4	2	11	2.5	+2.6
4	2	12	2.7	+2.4
4	2	13	3.1	+3.4
5	2	−17	1.0	+1.0

Table 2 (*cont.*)

H	K	L	F(OBS)	F(CAL)
5	2	-15	3.4	+3.1
5	2	-14	13.2	+12.9
5	2	-12	15.5	+15.0
5	2	-11	4.2	-4.6
5	2	-10	10.9	-10.6
5	2	-9	3.4	-3.4
5	2	-8	4.5	-3.8
5	2	-6	2.6	+3.4
5	2	-5	10.5	-10.4
5	2	-4	18.5	-17.5
5	2	-3	6.3	-6.2
5	2	-2	1.3	-1.3
5	2	-1	2.1	+2.1
5	2	0	12.4	+12.1
5	2	1	5.7	-6.8
5	2	2	5.4	+5.6
5	2	4	2.9	+3.1
5	2	5	8.4	+8.7
5	2	6	19.9	-20.5
5	2	7	1.9	-2.5
5	2	8	18.5	-18.8
5	2	9	4.3	-4.3
5	2	10	2.3	-2.4
5	2	11	1.4	-1.6
5	2	13	2.0	-1.9
6	2	-17	4.7	+4.4
6	2	-16	2.8	+3.3
6	2	-15	1.7	+1.7
6	2	-14	2.2	-1.4
6	2	-12	16.7	-15.6
6	2	-11	2.4	+2.4
6	2	-10	7.3	+7.5
6	2	-9	7.4	-8.6
6	2	-8	11.6	+11.6
6	2	-6	4.8	+4.1
6	2	-5	10.3	+9.6
6	2	-4	19.6	+18.8
6	2	-3	1.3	+1.4
6	2	-2	6.1	-4.9
6	2	-1	3.6	+3.2
6	2	0	2.2	+1.6
6	2	1	5.0	-4.6
6	2	2	1.8	+1.9
6	2	3	5.5	-5.1
6	2	4	11.9	-11.5
6	2	5	3.7	-4.1
6	2	6	15.9	+14.5
6	2	8	6.8	+6.5
6	2	9	5.4	+4.9
6	2	10	6.8	-6.5
6	2	11	1.1	+1.3
7	2	-19	1.8	+1.9
7	2	-18	1.6	+2.4
7	2	-17	5.7	+5.6
7	2	-16	3.2	+3.3
7	2	-15	2.7	+2.7
7	2	-14	5.1	-4.6
7	2	-13	4.9	-4.9
7	2	-11	11.0	-10.9
7	2	-10	9.3	+9.7
7	2	-9	12.0	-12.3
7	2	-6	3.3	+3.7
7	2	-5	13.3	+11.8
7	2	-4	1.4	+1.4
7	2	-3	3.3	+2.4
7	2	-2	12.0	-11.0
7	2	-1	1.2	+0.3
7	2	0	1.2	+1.6
7	2	1	1.6	+1.4
7	2	2	4.3	-3.7
7	2	3	10.9	-11.3
7	2	4	15.0	-14.1
7	2	5	13.1	-12.4
7	2	6	3.5	-3.2
7	2	7	1.7	-1.2
7	2	8	2.2	-2.0
7	2	9	4.2	+4.5
7	2	10	3.9	-4.0
7	2	11	3.5	+3.7
8	2	-17	2.9	-3.5
8	2	-16	2.6	-2.2
8	2	-15	3.9	+3.7
8	2	-14	4.8	+5.1
8	2	-13	2.2	+1.8
8	2	-12	1.7	-1.1
8	2	-11	6.9	+6.3
8	2	-10	7.3	-7.3
8	2	-9	6.9	+6.0
8	2	-8	16.1	+16.7
8	2	-7	7.0	-7.8
8	2	-6	5.5	+6.0
8	2	-5	2.6	-2.2
8	2	-4	6.1	-5.5
8	2	-3	7.9	-7.6
8	2	-2	4.2	+4.2
8	2	-1	7.0	-7.5
8	2	1	10.3	+10.6
8	2	2	9.1	+9.6
8	2	3	8.2	+8.0
8	2	4	1.8	+1.6
8	2	5	2.8	-2.6
8	2	6	5.3	-5.0
8	2	7	3.8	-3.8
8	2	8	5.6	+4.8
9	2	-17	2.1	-2.6
9	2	-16	3.6	-3.8
9	2	-15	1.7	-2.1
9	2	-11	3.6	+4.1
9	2	-10	3.2	+3.0
9	2	-9	2.4	+3.0
9	2	-8	7.6	+8.3
9	2	-7	10.7	-11.2
9	2	-6	2.2	+3.0
9	2	-5	8.3	-8.5
9	2	-3	4.6	+4.3
9	2	-2	6.1	-6.1
9	2	-1	1.3	-1.3
9	2	0	15.2	-15.2
9	2	1	7.2	-8.3
9	2	2	6.7	-6.7
9	2	3	1.8	-2.0
9	2	5	3.3	+2.6
9	2	6	1.6	-1.6
9	2	7	1.3	+0.6
9	2	9	2.0	-2.2
10	2	-18	1.3	+1.7
10	2	-15	1.6	-2.2
10	2	-13	2.9	+2.5
10	2	-12	3.0	+3.1
10	2	-11	1.4	+0.8
10	2	-10	4.7	-3.8
10	2	-9	2.9	+2.7
10	2	-8	5.9	-5.6
10	2	-7	3.0	-2.8
10	2	-6	7.1	+8.3
10	2	-5	4.2	-4.2
10	2	-4	5.7	+5.9
10	2	-3	7.2	+7.7
10	2	-2	6.4	+6.9
10	2	0	1.8	+1.2
10	2	1	6.9	-7.2
10	2	2	5.4	-5.0
10	2	3	3.0	+2.7
10	2	5	1.5	+1.7
10	2	7	1.6	-1.4
11	2	-17	1.7	-1.2
11	2	-16	2.5	+2.0
11	2	-14	1.5	+1.3
11	2	-13	2.0	+2.5
11	2	-12	2.7	+2.2
11	2	-11	2.8	-1.7
11	2	-9	2.6	-1.7
11	2	-8	4.8	-4.8
11	2	-5	5.8	-5.7
11	2	-3	8.2	-8.2
11	2	-2	4.8	-5.1
11	2	-1	1.7	-2.5
11	2	0	2.5	-2.4
11	2	1	2.0	+2.1
11	2	2	2.9	-2.6
11	2	3	1.6	+1.4
11	2	4	1.3	-1.2
12	2	-15	1.0	-1.4
12	2	-13	1.4	-1.3
12	2	-12	1.5	+1.5
12	2	-11	3.2	+3.2
12	2	-10	1.7	+1.9
12	2	-9	3.1	+3.0
12	2	-7	2.0	+2.3
12	2	-5	3.3	-3.7
12	2	-3	3.3	-3.6
12	2	-1	2.0	+1.9
12	2	0	3.4	+3.7
12	2	1	2.0	-2.0
13	2	-14	2.0	+1.9
13	2	-13	2.1	-1.7
13	2	-12	1.8	-2.2
13	2	-11	1.8	-1.3
13	2	-9	2.0	-2.0
13	2	-7	3.4	-3.2
13	2	-6	4.6	-4.4
13	2	-5	3.1	-2.9
13	2	-4	2.9	-2.6
14	2	-13	1.4	-1.5
14	2	-11	2.5	-2.5
14	2	-8	1.4	-1.8
14	2	-7	1.7	+1.9
14	2	-6	2.5	+2.0
14	2	-5	1.5	+1.4
14	2	-4	1.7	+2.2
14	2	-2	1.4	-1.0
15	2	-10	1.7	-2.2
15	2	-9	2.1	-1.7
15	2	-8	2.1	-2.3
15	2	-7	1.4	-0.8
15	2	-6	1.7	+1.3
0	3	1	6.2	-6.8
0	3	2	1.2	-1.8
0	3	3	7.2	-7.7
0	3	4	13.0	-14.4
0	3	5	10.8	+11.4
0	3	6	8.3	-9.1
0	3	7	3.2	+3.5
0	3	8	5.1	-5.9
0	3	9	6.9	-5.9
0	3	10	3.1	-3.3
0	3	12	3.7	-4.0
0	3	13	3.1	+3.7
0	3	14	4.4	-4.6
1	3	-16	1.1	-1.0
1	3	-15	1.9	-2.1
1	3	-14	5.2	-5.6
1	3	-11	1.5	+1.7
1	3	-10	6.4	+5.2
1	3	-8	5.7	-5.5
1	3	-7	3.0	-2.6
1	3	-6	17.4	-15.4
1	3	-5	9.4	-8.6
1	3	-4	5.2	+4.9
1	3	-3	5.2	-5.8
1	3	-2	18.0	+17.9
1	3	0	9.4	-9.2
1	3	1	14.4	-15.0
1	3	2	24.3	-24.9
1	3	3	10.4	-10.8
1	3	4	3.6	-4.0
1	3	5	4.5	+4.0
1	3	6	9.8	+9.5
1	3	7	3.1	+2.8
1	3	8	1.3	+1.0
1	3	10	3.3	-3.0
1	3	11	1.5	-1.6
1	3	13	3.5	-3.3
2	3	-17	1.0	+1.0
2	3	-15	2.7	+2.5
2	3	-14	2.6	+2.7
2	3	-12	2.8	+3.0
2	3	-11	3.7	-3.9
2	3	-10	5.3	+5.2
2	3	-8	4.0	+5.1
2	3	-7	5.9	+5.0
2	3	-6	4.3	+4.4
2	3	-5	2.0	-2.3
2	3	-4	25.1	+25.4
2	3	-3	12.7	-13.4
2	3	-2	1.4	-1.0
2	3	-1	5.8	-5.1
2	3	0	21.1	-19.3
2	3	1	2.1	+2.2
2	3	2	1.7	+1.7
2	3	3	1.1	-0.8
2	3	4	17.4	-18.1
2	3	6	9.0	-9.7
2	3	7	5.0	+6.0
2	3	8	3.1	+3.4
2	3	10	4.6	-4.7
2	3	11	4.7	-4.7
2	3	12	1.4	-0.9
2	3	13	1.4	-0.7
2	3	14	2.1	-2.2
3	3	-16	1.3	+0.7
3	3	-14	2.5	+2.6
3	3	-11	2.2	+2.2
3	3	-8	2.8	-2.3
3	3	-7	5.7	-5.2
3	3	-6	6.3	-6.7
3	3	-5	2.3	+2.0
3	3	-4	16.5	-17.6
3	3	-3	8.5	-8.0
3	3	-2	9.1	-8.5
3	3	-1	16.7	-17.5
3	3	0	9.7	+9.0
3	3	2	13.5	+11.9
3	3	4	3.7	+3.5
3	3	5	4.5	+5.0
3	3	6	3.9	-3.4
3	3	7	7.1	+7.0
3	3	8	6.8	-7.2
3	3	9	8.5	-8.6
3	3	10	7.3	-7.6
3	3	11	4.8	-4.6
3	3	12	1.5	-1.8
3	3	14	2.6	+2.7
4	3	-17	2.1	-1.8
4	3	-16	1.2	-1.0
4	3	-14	3.6	+4.4
4	3	-12	9.1	+9.3
4	3	-11	1.8	-2.3
4	3	-9	5.7	+5.4
4	3	-8	9.5	+9.5
4	3	-7	1.9	+1.8
4	3	-6	2.5	-1.7
4	3	-5	8.6	-8.6
4	3	-4	14.1	-12.4
4	3	-3	10.9	-9.8
4	3	-2	12.1	+12.4
4	3	-1	11.5	-10.5
4	3	0	11.5	-10.3
4	3	1	2.4	-1.9
4	3	2	2.3	-1.8
4	3	3	6.4	+6.8
4	3	4	8.1	+9.5
4	3	5	8.8	+9.2
4	3	6	21.9	-21.4
4	3	8	8.5	-8.7
4	3	9	1.7	-2.0
4	3	10	1.4	+1.6
4	3	11	3.4	-3.1
4	3	12	5.6	-5.8
4	3	13	4.6	-4.7
5	3	-17	2.9	+2.7
5	3	-15	5.4	+5.4
5	3	-14	1.5	+1.8
5	3	-12	5.3	-5.0
5	3	-11	14.9	-14.6
5	3	-10	4.9	-5.3
5	3	-9	11.7	-11.2
5	3	-8	2.1	-1.8
5	3	-7	2.2	+2.2
5	3	-6	1.2	-0.5
5	3	-3	7.2	+5.6
5	3	-2	4.3	-4.1
5	3	-1	4.6	+4.1
5	3	1	2.3	+2.9
5	3	2	4.3	+5.3
5	3	3	2.4	-2.5
5	3	4	3.0	-3.3
5	3	5	23.1	-23.5
5	3	6	7.8	-8.4
5	3	7	7.0	-7.4
5	3	8	3.3	-2.9
5	3	9	11.3	+11.1
5	3	10	2.8	+2.5
5	3	11	3.7	+4.3
6	3	-18	1.1	+1.6
6	3	-17	3.1	+3.3
6	3	-16	3.8	+3.8
6	3	-14	2.2	-2.0
6	3	-13	2.9	+2.5
6	3	-12	9.5	+9.8
6	3	-11	6.7	-7.0
6	3	-10	9.2	+9.1
6	3	-9	12.7	-12.7
6	3	-8	13.2	-13.2
6	3	-7	1.5	+1.3
6	3	-1	6.6	+6.6
6	3	0	9.5	+9.9
6	3	2	13.9	-13.7
6	3	3	9.3	-10.3
6	3	4	5.3	-4.9
6	3	5	5.0	-5.6
6	3	7	3.0	+2.6
6	3	8	15.7	-15.6
6	3	10	3.0	-2.7
7	3	-18	1.7	+2.0
7	3	-17	1.5	-1.4
7	3	-16	5.6	-5.6
7	3	-15	6.3	-6.8
7	3	-14	7.4	-8.2
7	3	-12	3.3	-3.6
7	3	-11	9.2	+10.0
7	3	-10	1.6	+1.8
7	3	-9	2.3	-2.5
7	3	-8	8.0	+9.2
7	3	-7	6.5	-7.0
7	3	-6	2.7	+2.7
7	3	-4	1.8	-2.0
7	3	0	10.0	-11.2
7	3	1	9.2	-10.0
7	3	2	10.9	-12.2
7	3	3	1.7	-1.7
7	3	4	1.7	+2.0
7	3	5	8.1	+8.1
7	3	6	7.8	+8.4
7	3	8	3.4	+4.0
7	3	9	1.3	-1.8
7	3	10	1.1	-0.9
8	3	-17	2.2	+1.7
8	3	-13	3.5	-3.5
8	3	-12	7.6	-8.3
8	3	-10	9.3	+9.8
8	3	-9	7.5	-7.5
8	3	-8	11.5	+12.3
8	3	-7	3.1	-2.8
8	3	-6	4.7	-3.9
8	3	-5	6.1	+6.0
8	3	-4	1.8	+2.8
8	3	-3	1.8	-1.7
8	3	-2	8.7	-8.4
8	3	-1	4.4	-5.3
8	3	0	9.3	-10.0
8	3	1	1.8	-1.8
8	3	3	2.4	-2.5
8	3	4	8.0	-7.3
8	3	5	3.0	-3.1
8	3	9	2.5	+1.6
9	3	-17	1.4	-1.8
9	3	-16	1.9	+1.8
9	3	-15	1.7	+1.8
9	3	-14	1.5	+1.7
9	3	-12	1.7	+1.7
9	3	-11	1.9	+2.6
9	3	-10	4.2	-4.3
9	3	-9	3.9	+4.5
9	3	-8	6.1	-7.2
9	3	-7	5.5	-6.5
9	3	-5	6.4	-7.5
9	3	-3	3.5	-4.0
9	3	-1	1.9	-1.9
9	3	0	2.2	+1.8
9	3	1	3.4	+3.3
9	3	3	1.6	+1.7
9	3	8	1.7	-1.6
10	3	-17	2.2	-2.6
10	3	-15	1.2	+1.4
10	3	-14	2.0	+1.7
10	3	-13	1.5	+1.8
10	3	-12	1.5	+1.7
10	3	-10	2.9	-2.6
10	3	-8	4.6	+5.6
10	3	-7	6.1	-5.8
10	3	-5	4.5	-4.5
10	3	-4	5.8	-6.1
10	3	-3	3.2	-3.3
10	3	-1	1.9	-2.4
10	3	0	5.5	-5.4
10	3	1	3.0	+2.9
10	3	2	3.4	-3.3
10	3	5	2.4	-2.5
10	3	6	3.1	-3.0
11	3	-16	2.5	+1.6
11	3	-14	1.3	+1.1
11	3	-13	1.5	-1.2
11	3	-12	4.0	-3.8
11	3	-11	4.3	-4.5
11	3	-10	1.6	-2.5
11	3	-7	4.0	+4.3
11	3	-4	2.2	-2.6
11	3	-3	3.7	-4.1
11	3	-2	2.2	-1.8
11	3	-1	2.0	-2.1
11	3	0	3.3	+3.8
11	3	1	1.1	+0.9
11	3	2	2.0	+2.1
11	3	4	3.1	-3.0
12	3	-15	2.0	-2.0
12	3	-9	2.7	-2.4
12	3	-7	3.6	-3.9
12	3	-5	2.2	-2.6
12	3	-4	1.5	-1.8
12	3	-1	2.6	-2.4
12	3	0	2.2	-1.7
12	3	1	1.7	-1.9
12	3	2	2.9	-3.1
13	3	-13	1.5	-1.2
13	3	-12	1.4	+1.3
13	3	-8	2.2	-3.3
13	3	-7	3.4	-3.9
13	3	-6	1.2	-1.1
13	3	-4	2.0	+2.6
13	3	-3	2.5	+3.1
13	3	-2	1.1	+1.0
14	3	-11	1.7	-0.9
14	3	-10	2.5	-2.8
14	3	-9	1.0	-1.0
14	3	-8	1.7	-1.1
14	3	-7	0.9	-1.5
14	3	-6	1.0	+0.8
14	3	-5	2.0	-2.5
14	3	-4	1.0	-1.6
0	4	0	7.7	-7.8
0	4	1	5.5	-5.6
0	4	2	12.6	-14.0
0	4	3	1.0	-0.7
0	4	4	5.2	-5.5
0	4	5	7.9	-7.7
0	4	6	1.4	+1.5
0	4	7	3.5	-2.6
0	4	8	3.8	-3.6
0	4	11	3.2	-3.4
0	4	12	1.7	-2.1
1	4	-14	2.9	+3.2
1	4	-13	3.1	+3.0
1	4	-12	7.8	+8.9
1	4	-11	2.3	+1.9
1	4	-9	2.6	-2.7
1	4	-8	6.1	-6.4
1	4	-7	1.4	-1.5
1	4	-6	3.0	+3.3
1	4	-5	4.4	-4.7
1	4	-4	5.7	+6.1
1	4	-3	3.5	-3.9
1	4	-2	4.0	+5.0
1	4	-1	1.9	-1.6
1	4	0	6.8	+5.7
1	4	1	1.6	-1.6
1	4	2	4.6	-4.8
1	4	3	6.6	+8.1
1	4	4	5.3	-6.0
1	4	5	7.8	+8.8
1	4	6	6.3	-6.5
1	4	7	2.4	-2.3
1	4	8	8.8	-9.9
1	4	9	5.5	-5.7
1	4	12	1.8	+1.7
1	4	13	1.8	+2.0
1	4	14	1.7	-1.4
2	4	-15	3.0	-3.0
2	4	-12	3.8	-3.8
2	4	-11	1.9	+2.6
2	4	-10	4.3	-3.5
2	4	-9	4.0	-3.5
2	4	-8	2.1	-2.1
2	4	-7	2.8	+2.3
2	4	-6	1.4	-1.4
2	4	-4	10.2	-9.5
2	4	-3	17.1	-16.9
2	4	-2	21.2	-21.7
2	4	-1	14.2	-13.9
2	4	0	0.9	+0.8
2	4	1	3.4	-4.0
2	4	2	2.8	+2.4
2	4	3	5.3	+4.7
2	4	4	13.3	-13.3
2	4	5	7.2	+7.0
2	4	6	1.7	-2.4
2	4	7	4.5	-5.3
2	4	9	3.1	-3.3
2	4	10	1.9	-2.0
2	4	13	4.3	-3.7
2	4	14	3.7	-3.2
3	4	-16	1.7	+2.1
3	4	-13	5.4	-5.1
3	4	-12	1.7	+0.9
3	4	-11	3.4	-3.5
3	4	-10	3.4	+3.8
3	4	-9	1.7	-1.5
3	4	-8	1.8	+1.8
3	4	-7	1.8	+1.8
3	4	-6	1.4	+2.3
3	4	-5	5.7	+5.1
3	4	-4	6.6	+6.9
3	4	-3	6.2	-5.2
3	4	-2	2.1	-2.6
3	4	0	4.7	-4.9
3	4	1	1.7	-2.4
3	4	2	1.7	+2.5
3	4	3	4.7	-4.7
3	4	4	6.3	-6.2
3	4	5	5.7	+5.7
3	4	6	7.6	-8.4
3	4	7	3.5	+3.8
3	4	8	2.4	-2.3
3	4	10	1.8	-1.6
3	4	13	2.5	-1.9
4	4	-16	1.7	-1.7
4	4	-15	1.3	+1.0
4	4	-14	2.7	+2.4
4	4	-13	4.9	-5.1
4	4	-12	3.6	+3.1
4	4	-10	1.7	-1.8
4	4	-9	1.8	+2.2
4	4	-8	11.4	-11.3
4	4	-7	17.2	-17.6
4	4	-6	19.3	-20.4
4	4	-5	10.8	-9.8
4	4	-4	1.6	-1.3
4	4	-3	12.6	+11.5
4	4	-1	5.1	+5.0
4	4	0	6.5	-6.7
4	4	1	3.5	-3.9
4	4	2	8.2	+8.9
4	4	3	7.4	-8.3
4	4	4	3.1	-2.6
4	4	5	3.9	-4.8
4	4	6	12.5	-11.9
4	4	8	3.1	-3.3
4	4	9	3.1	-3.1
4	4	10	4.8	-4.9
4	4	11	2.5	-2.3
5	4	-15	1.3	+1.7
5	4	-12	3.5	+3.4
5	4	-11	3.5	-3.9
5	4	-10	6.7	+7.0
5	4	-9	2.2	+2.2
5	4	-8	3.6	+4.4
5	4	-7	1.6	-1.7
5	4	-6	2.5	-2.4
5	4	-5	5.1	-5.6
5	4	-4	6.4	-6.1
5	4	-3	1.4	-1.4
5	4	-2	2.6	+3.0
5	4	0	4.6	+4.7
5	4	1	5.5	+5.0
5	4	2	5.6	-5.0
5	4	3	4.3	-4.1
5	4	4	4.0	-3.1
5	4	5	2.7	-3.2
5	4	6	3.8	-3.6
5	4	7	5.1	+5.5
5	4	8	3.5	-3.7
5	4	10	2.2	-2.0
5	4	11	1.0	-1.4
6	4	-16	2.2	-2.4
6	4	-14	4.8	-5.0
6	4	-13	1.3	-1.0
6	4	-12	4.2	-4.3
6	4	-11	13.4	-12.5
6	4	-10	7.7	-7.6
6	4	-9	7.0	-5.9
6	4	-8	3.0	+2.7
6	4	-7	5.7	+5.5
6	4	-6	4.3	+4.2
6	4	-4	5.0	-4.7
6	4	-3	1.8	+2.1
6	4	-1	3.3	+3.3
6	4	0	14.0	-12.6
6	4	1	9.5	-9.7
6	4	2	15.4	-15.6
6	4	3	7.3	-7.8
6	4	4	1.8	+2.1
6	4	5	1.7	-1.9
6	4	7	1.7	-1.9
6	4	8	1.2	+1.6
6	4	9	2.1	+2.1
6	4	10	2.4	+2.5
7	4	-15	1.7	-1.2
7	4	-14	2.0	+1.9
7	4	-13	2.1	+2.2
7	4	-11	1.8	-2.0
7	4	-10	2.6	+2.8
7	4	-9	7.8	-7.4
7	4	-5	3.5	+2.6
7	4	-4	5.3	+5.4
7	4	-3	2.7	+2.7
7	4	-2	5.7	-5.2
7	4	-1	4.8	-3.8
7	4	0	7.2	-6.8
7	4	1	5.5	-5.4
7	4	3	2.3	+2.1
7	4	4	1.8	-1.9
7	4	6	2.0	-1.8
7	4	8	2.2	-2.1
7	4	9	2.2	+2.5
8	4	-15	2.9	-2.5
8	4	-14	3.5	-3.8
8	4	-11	3.2	+3.0
8	4	-9	1.7	+1.3
8	4	-8	4.0	-3.7
8	4	-6	5.5	+4.8
8	4	-5	6.3	-6.3
8	4	-4	5.5	-5.6
8	4	-3	14.2	-14.4
8	4	-2	14.8	-15.2
8	4	-1	3.8	-3.4
8	4	1	6.8	+6.6
8	4	8	1.6	-1.1
9	4	-13	1.4	+1.2
9	4	-12	2.1	+2.1
9	4	-10	1.3	+0.8
9	4	-9	2.6	-2.6

Table 2 (*cont.*)

H	K	L	F(OBS)	F(CAL)
9	4	-8	5.2	+5.4
9	4	-7	3.5	-2.9
9	4	-6	1.5	-1.6
9	4	-5	1.9	-1.5
9	4	-4	5.4	-5.5
9	4	-3	2.9	-3.1
9	4	-1	1.8	+1.5
9	4	0	3.2	-3.2
9	4	1	1.4	+1.6
9	4	4	2.7	+2.5
9	4	6	2.7	-2.4
10	4	-15	1.0	-0.5
10	4	-10	1.3	+0.7
10	4	-9	2.4	-2.7
10	4	-8	6.9	-8.0
10	4	-7	2.9	-3.2
10	4	-6	10.1	-11.0
10	4	-5	1.8	-1.8
10	4	-4	3.8	+4.0
10	4	-2	3.3	+3.4
10	4	-1	1.6	-1.4
10	4	0	2.9	-2.6
10	4	2	1.0	-0.9
10	4	4	1.2	-1.3
11	4	-14	1.2	-2.0
11	4	-11	1.0	-0.7
11	4	-9	1.8	-1.5
11	4	-7	2.9	-3.5
11	4	-6	2.0	+2.5
11	4	-1	1.7	+1.8
11	4	2	4.2	-4.9
12	4	-12	1.8	-2.1
12	4	-11	1.0	-1.6
12	4	-10	2.0	-2.5
12	4	-9	2.7	-2.4
12	4	-7	2.0	-1.7
12	4	-6	2.9	-2.9
12	4	-4	2.2	-2.4
13	4	-7	1.1	-0.7
13	4	-6	1.2	+0.8
0	5	1	10.1	+10.9
0	5	2	5.4	-5.3
0	5	3	11.6	+13.6
0	5	4	5.4	+5.5
0	5	5	4.2	+5.2
0	5	8	2.8	-3.6
0	5	9	4.5	-4.8
0	5	11	1.8	-1.6
0	5	12	1.0	-1.4
0	5	13	1.0	-0.7
1	5	-12	1.0	-1.5
1	5	-11	2.7	-2.4
1	5	-10	3.4	-3.5
1	5	-9	2.0	-2.0
1	5	-6	5.6	-5.4
1	5	-5	6.4	-6.2
1	5	-4	5.6	-5.5
1	5	-3	2.2	+1.9
1	5	-2	1.4	-1.2
1	5	0	10.0	-9.3
1	5	1	9.0	-10.5
1	5	2	11.0	-12.7
1	5	3	2.2	+2.3
1	5	4	2.9	-2.6
1	5	5	1.4	+0.4
1	5	6	3.3	-2.9
1	5	7	4.8	-5.6
1	5	8	3.3	-3.0
1	5	12	0.8	-1.1
2	5	-14	2.5	+2.3
2	5	-11	2.0	-1.9
2	5	-9	3.3	-3.1
2	5	-8	4.9	-4.1
2	5	-6	2.2	-1.9
2	5	-5	3.6	-3.4
2	5	-4	3.3	-2.9
2	5	-3	3.6	+2.4
2	5	-2	6.4	+6.8
2	5	-1	5.5	+6.0
2	5	0	16.0	+17.6
2	5	1	2.9	-2.3
2	5	2	3.3	-2.5
2	5	4	12.5	-12.6
2	5	6	4.4	-4.5
2	5	7	2.9	+1.9
2	5	8	2.9	-2.1
2	5	9	4.2	+3.9
2	5	10	1.6	+1.4
2	5	11	0.8	-0.5
2	5	12	1.4	+1.0
3	5	-13	2.9	-3.0
3	5	-10	3.1	-3.0
3	5	-8	3.4	-3.4
3	5	-7	1.4	-2.1
3	5	-6	3.6	-3.0
3	5	-5	4.6	-4.5
3	5	-4	11.1	-11.0
3	5	-3	7.9	-8.8
3	5	-2	11.1	-11.2
3	5	2	1.4	-1.0
3	5	3	3.8	-4.2
3	5	4	7.9	-7.2
3	5	5	3.1	+2.0
3	5	8	1.4	-1.3
3	5	9	3.8	-3.4
3	5	10	4.2	-3.7
4	5	-13	3.3	-2.7
4	5	-12	1.4	+1.2
4	5	-10	2.9	+2.7
4	5	-9	4.4	-4.8
4	5	-6	2.7	+2.4
4	5	-5	9.6	+9.7
4	5	-4	6.6	+6.3
4	5	-3	1.4	+1.4
4	5	-2	4.2	-3.9
4	5	-1	5.3	-5.5
4	5	0	4.9	-5.0
4	5	1	3.3	-2.8
4	5	5	3.8	+3.5
4	5	6	2.2	+1.9
4	5	7	1.4	+1.6
4	5	8	1.7	-1.3
4	5	10	2.9	-2.8
5	5	-13	2.5	-2.0
5	5	-11	5.9	-5.6
5	5	-10	4.3	-4.5
5	5	-9	4.0	-3.6
5	5	-8	5.7	-6.7
5	5	-7	3.3	-3.2
5	5	-6	4.3	-4.3
5	5	-3	2.0	+2.2
5	5	-2	3.4	-2.9
5	5	0	5.9	-6.3
5	5	2	2.2	-2.0
5	5	3	2.2	-3.1
5	5	4	3.3	-3.0
5	5	5	3.7	-4.5
5	5	6	5.7	-5.7
5	5	7	3.6	-3.6
6	5	-8	6.1	+6.0
6	5	-7	2.7	-2.8
6	5	-6	1.5	+1.7
6	5	-5	1.4	-1.5
6	5	-4	1.4	-1.3
6	5	-3	3.7	+3.5
6	5	-2	2.7	-2.2
6	5	-1	3.4	-3.2
6	5	1	1.7	-1.8
6	5	2	2.9	+2.7
6	5	3	4.4	+4.2
6	5	4	2.2	-1.8
6	5	5	1.2	+0.8
6	5	6	3.6	-2.9
6	5	8	2.0	+1.7
7	5	-14	3.1	-3.7
7	5	-8	3.1	-2.6
7	5	-7	1.1	-0.8
7	5	-4	3.6	-2.7
7	5	-3	3.6	-2.3
7	5	-2	7.6	-7.8
7	5	-1	3.8	-3.9
7	5	0	5.3	-5.7
7	5	2	4.9	-4.6
7	5	5	2.7	+1.8
7	5	7	1.5	+1.1
8	5	-13	1.6	-1.2
8	5	-12	3.3	+3.0
8	5	-9	2.5	+2.7
8	5	-8	2.5	+2.3
8	5	-5	6.3	-6.4
8	5	-4	5.2	-5.0
8	5	-3	1.6	-2.2
8	5	-1	5.6	+5.4
8	5	3	2.0	-1.4
8	5	4	2.9	+2.1
8	5	5	4.2	+2.7
9	5	-13	1.4	-1.0
9	5	-12	2.2	-2.6
9	5	-11	1.0	-1.3
9	5	-8	2.2	-2.8
9	5	-7	1.7	-2.5
9	5	-6	6.8	-7.7
9	5	-5	4.3	-5.5
10	5	-12	2.0	+1.9
10	5	-11	2.2	-1.9
10	5	-9	4.7	-5.1
10	5	-8	3.9	-3.8
10	5	-7	3.1	-3.2
10	5	-6	2.2	+2.5
10	5	-5	2.2	+2.1
10	5	-4	2.0	+2.1
10	5	-3	3.9	+3.0
10	5	-1	2.2	+2.1
10	5	0	2.0	+1.5
10	5	1	2.2	+1.9
0	6	0	2.4	-2.4
0	6	1	4.0	+3.2
0	6	3	1.5	-1.7
0	6	4	4.9	-5.0
0	6	5	3.9	-4.4
0	6	6	7.0	-7.2
0	6	7	1.5	-1.2
1	6	-8	5.3	-4.0
1	6	-7	5.9	-5.4
1	6	-6	6.0	-5.5
1	6	-5	3.1	-2.9
1	6	-3	2.0	+2.1
1	6	-2	4.0	+4.2
1	6	-1	6.6	-6.5
1	6	0	3.6	+4.1
1	6	1	3.6	+3.3
1	6	2	3.6	+3.5
1	6	3	10.0	+10.2
1	6	6	3.6	-3.8
1	6	8	2.0	-1.6
1	6	9	1.7	-1.2
2	6	-11	2.2	-1.8
2	6	-10	2.7	-2.8
2	6	-9	3.6	-3.0
2	6	-8	3.1	-2.7
2	6	-5	1.4	+1.3
2	6	-4	3.3	-2.3
2	6	-2	2.0	-1.6
2	6	0	6.5	-6.6
2	6	1	2.9	-2.6
2	6	2	8.4	-8.3
2	6	3	2.9	-3.2
2	6	8	3.1	-2.1
3	6	-11	3.6	-3.1
3	6	-10	2.5	-2.4
3	6	-9	2.5	-2.4
3	6	-7	3.7	+3.3
3	6	-5	2.2	+1.9
3	6	-4	1.2	+1.1
3	6	-3	3.1	+2.4
3	6	-2	3.6	+3.2
3	6	-1	3.3	+2.9
3	6	1	5.8	-5.5
3	6	2	1.6	-1.8
3	6	5	1.9	+1.9
3	6	8	1.5	-1.2
4	6	-9	2.5	-1.9
4	6	-8	3.1	-3.4
4	6	-6	2.9	-2.1
4	6	-4	1.7	-1.7
4	6	-3	2.7	-2.4
4	6	-2	4.8	-4.9
4	6	-1	3.3	-3.5
4	6	0	3.3	-3.7
4	6	1	1.4	+1.2
4	6	2	3.1	-2.6
4	6	3	3.8	+3.4
4	6	7	2.7	-2.7
5	6	-10	2.2	+2.1
5	6	-8	3.3	+3.3
5	6	-7	1.4	-1.3
5	6	-5	0.8	+0.8
5	6	-4	3.6	-3.0
5	6	0	3.1	+2.5
5	6	1	2.0	+1.9
5	6	4	2.2	-1.6
5	6	6	1.5	+1.8
6	6	-6	3.8	-3.9
6	6	-5	2.2	-2.0
6	6	-4	4.6	-4.6
6	6	-3	1.5	-1.6
6	6	-2	1.2	-1.2
6	6	-1	1.5	+0.9
6	6	2	3.4	-3.0
6	6	3	2.0	-1.8
6	6	4	4.8	-4.2
7	6	-10	1.7	-1.2
7	6	-6	3.6	+3.4
7	6	-2	2.5	-1.5
7	6	1	1.7	-1.5
7	6	2	1.3	+1.3
7	6	3	1.7	+1.4
8	6	-9	2.5	-2.1
8	6	-8	2.7	-2.5
8	6	-7	2.9	-2.5
8	6	-6	3.6	-3.1
8	6	-4	2.0	-1.5
8	6	-2	2.0	-1.8
8	6	-1	2.5	-1.7
8	6	0	2.0	-1.8
9	6	-7	1.8	-2.0
9	6	-6	1.4	-1.0
0	7	1	1.8	+1.6
0	7	2	1.6	+1.6
0	7	3	1.8	+1.6
0	7	5	2.5	+2.5
0	7	6	3.0	+2.6
1	7	-5	3.6	+2.9
1	7	-3	2.2	+2.1
1	7	-1	1.4	+1.0
1	7	0	1.4	-1.2
1	7	1	2.0	+1.5
1	7	3	1.7	-2.2
2	7	-6	1.7	-0.9
2	7	-4	1.0	+1.0
2	7	-3	2.0	+1.5
2	7	-2	2.9	+3.0
2	7	-1	2.2	+2.0
2	7	3	1.3	+0.4
3	7	-5	0.9	-0.7
3	7	-4	1.0	-1.1
3	7	-2	2.0	-1.4
3	7	0	2.5	-2.3
3	7	1	2.5	+1.9
3	7	2	3.1	-2.8
4	7	-6	1.4	+1.6
4	7	-5	1.4	+1.9
4	7	-4	1.0	-1.1
4	7	-3	1.7	-2.5
4	7	-2	1.4	-1.4
4	7	-1	0.9	-1.0
4	7	2	1.7	+1.0

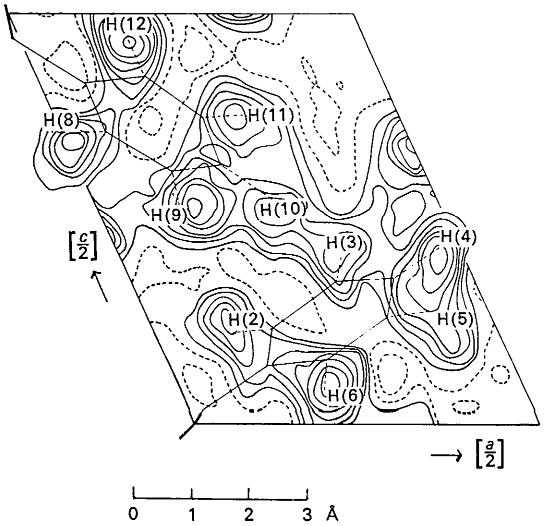

Fig. 3. Difference projection along **b**, showing approximate positions of hydrogen atoms. Contours are at intervals of 0·2 e.Å^{-2}.

plitudes for the non-zero planes. The structure factors for the 579 unobserved planes were also calculated and found to have very small values. These were not used at all in the refinement, nor in the calculation of the R value.

Description of the structure

Four molecules of *trans*-azobenzene are arranged in the unit cell with space group symmetry $P2_1/a$, the centres of each molecule lying on a centre of symmetry of the lattice (Fig. 4). There are thus two non-equivalent pairs, and one-half of each kind of azobenzene molecule comprises one asymmetric unit. The crystal is isostructural with stilbene (Robertson & Woodward, 1937) and tolane (Robertson & Woodward, 1938). There are only van der Waals forces between the molecules, so it is believed that this disposition is adopted by molecules of this shape for economic packing causes. No property of the nitrogen atoms or azo group is responsible, as the next homologue, *p*-azotoluene (Brown, 1966) crystallizes with all its molecules in crystallographically equivalent positions. The bond-lengths and inter-bond angles are listed in Table 3, and also shown in Fig. 5.

The mean C–C length in the benzene ring (1·380 Å) compares very favourably with the mean value found in numerous other compounds. The mean C–N length (1·452 Å) is in good agreement, too; published values range from 1·41 to 1·47 Å. One of the N=N lengths (1·243 Å) is in accord with Pauling's predicted value of 1·23 Å, while the other (1·172 Å) is low. The angles of the benzene ring are all within a few minutes of 120°,

Table 3. *Bond lengths and inter-bond angles*

C(1)—C(2)	1·384 Å	C(1)—C(2)—C(3)	119°24′
C(2)—C(3)	1·390	C(2)—C(3)—C(4)	120 27
C(3)—C(4)	1·368	C(3)—C(4)—C(5)	119 53
C(4)—C(5)	1·389	C(4)—C(5)—C(6)	120 18
C(5)—C(6)	1·379	C(5)—C(6)—C(1)	119 34
C(6)—C(1)	1·385	C(6)—C(1)—C(2)	120 20
C(1)—N(1)	1·433	C(6)—C(1)—N(1)	124 7
N(1) -N(1′)	1·243	C(2)—C(1)—N(1)	115 29
		C(1)—N(1) -N(1′)	113 33
C(7)—C(8)	1·373	C(7)—C(8)—C(9)	119 55
C(8)—C(9)	1·373	C(8)—C(9)—C(10)	120 24
C(9)—C(10)	1·365	C(9)—C(10)-C(11)	119 52
C(10)-C(11)	1·385	C(10)-C(11)-C(12)	120 13
C(11)-C(12)	1·375	C(11)-C(12)-C(7)	119 16
C(12)-C(7)	1·383	C(12)-C(7)—C(8)	120 20
C(7)—N(2)	1·472	C(12)-C(7)—N(2)	126 57
N(2) -N(2′)	1·172	C(8)—C(7)—N(2)	112 43
		C(7)—N(2) -N(2′)	111 4

C–H (assumed) 1·08 Å.
Mean standard deviation for bond lengths, 0·0030 Å.
Mean standard deviation for angles, 14′.

but those between the C–N bond and the ring appear to be distorted by steric repulsion between C(6) and N(1′) and between C(12) and N(2′). This repulsive effect also increases the angle at the nitrogen atom from a normal tetrahedral value to ~113°. If this system were unstrained, the distance between C(6) and N(1′) would be 2·53 Å, which is too close for non-bonded atoms; the distortion increases the distance to 2·73 Å in one molecule and 2·75 Å between C(12) and N(2′) in the other.

Each benzene ring is planar; that belonging to the molecule centred on (0,0,0) has the equation

$$0{\cdot}4545x' + 0{\cdot}4107y - 0{\cdot}7904z' = 0{\cdot}1627$$

and that belonging to the molecule centred on $(0,0,\frac{1}{2})$

$$0{\cdot}4693x' - 0{\cdot}4671y + 0{\cdot}7494z' = 3{\cdot}7143$$

where $x' = x + z\cos\beta$ and $z' = z\sin\beta$. The average displacement of an atom from the mean plane of the ring is 0·0053 Å.

The equation of the plane through C(1), N(1) and (0,0,0) is

$$0{\cdot}5400x' + 0{\cdot}1289y - 0{\cdot}8318z' = 0,$$

which makes an angle of 17°6′ with the plane of the C(1)–C(6) ring. The equation of the plane through C(7), N(2) and $(0,0,\frac{1}{2})$ is

$$0{\cdot}4581x' - 0{\cdot}5553y + 0{\cdot}6942z' = 3{\cdot}4212$$

and this makes an angle of 5°57′ with the plane of the C(7)–C(12) ring.

These equations lead to results slightly different from those in the previously published structure (de Lange, Robertson & Woodward, 1939), the figures from which are given in brackets below. The angle between the N(1)=N(1′) bond and the plane of the C(1)–C(6) ring (*i.e.* the complement of the angle between the bond and the plane-normal) is 15°22′ (15·1°), while the angle of rotation of the ring from the position of a flat molecule is 17°7′ (17·4°). The angle between the N(2)=N(2′) bond and the plane of the C(7)–C(12) benzene ring is 5°45′ (1·1°), and the angle of rotation of this ring from a flat molecule is 6°0′ (1·8°).

The inclination of the benzene rings is such that neighbouring molecules along **b** lie in planes whose perpendicular distance apart is 2·35 Å for those molecules centred on (0,0,0), and 2·70 Å for those centred on $(0.0,\frac{1}{2})$. Measurement of the electron density map (Fig. 1) indicates that layers of the first kind of molecule [*i.e.* those centred on (0,0,0)] occupy only 14/30ths of the unit-cell volume, while those of the second kind, centred on $(0,0,\frac{1}{2})$, occupy 16/30ths.

The intermolecular distances are of the usual order of magnitude; there are about 50 non-hydrogen contacts per molecule within the range 3·63 to 4·00 Å. There seem to be no particular differences between the van der Waals contacts of the two non-equivalent molecules, or between the different parts of the same molecule.

Disorder in atomic positions

Examination of the temperature factors in Table 1 shows that the diagonal B_{ij} values for atoms C(7)–C(12) and N(2) are appreciably larger than those for the corresponding atoms C(1)–C(6) and N(1) in the other

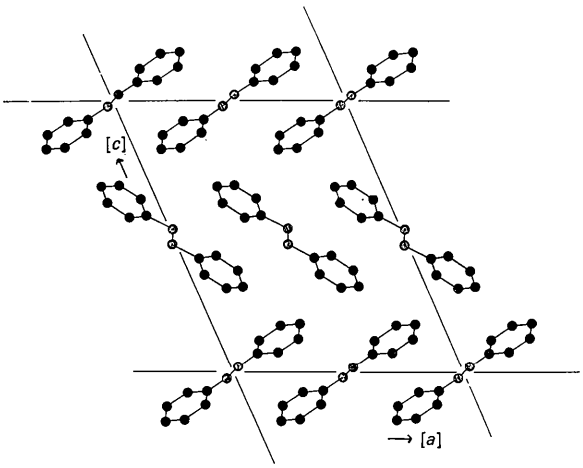

Fig. 4. Diagram of the contents of one unit cell. Molecules of the two kinds occupy separate layers parallel to (001).

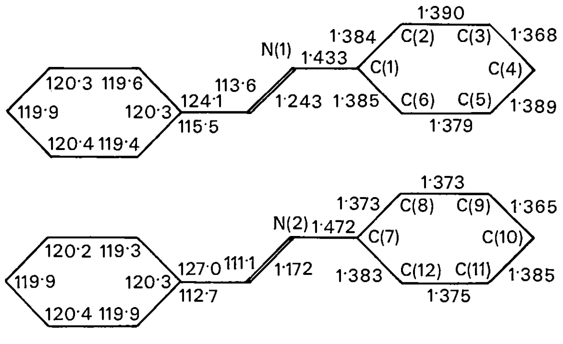

Fig. 5. Bond lengths and inter-bond angles in the two non-equivalent molecules. (Each molecule possesses a centre of symmetry.)

kind of molecule. The mean isotropic *B*'s for the corresponding hydrogen atoms, as well as the standard deviations of the corresponding atomic coordinates are also larger in the molecules centred on $(0,0,\frac{1}{2})$ than those centred on (0,0,0). The explanation of this could be that as the molecule on $(0,0,\frac{1}{2})$ occupies more space in the unit cell, it also possesses greater freedom of movement, and therefore higher vibration amplitudes. But since the determination of the crystal structure of *p*-azotoluene (Brown, following paper), it would appear that a more probable explanation is that for atoms C(7)–C(12) and N(2) there are two or more closely similar positions in which the molecules are able to lie, and that the higher temperature factors are an expression of this disorder. There has been no attempt in the course of this work to resolve the vibration ellipsoids into equivalent spheres as was done in the case of *p*-azotoluene, so it is not known how many configurations in space the molecule may adopt, or what proportions of the whole lie in each. Neither is it known whether the same proportion adopt each configuration in different crystalline specimens.

The stepwise configuration of the azobenzene molecule allows the possibility of the sense of the step being 'up' or 'down' with respect to the axis of symmetry, and as the two halves of the molecules (average coordinates as determined by the refinement process) lie in planes which are only 0·15 Å apart, an interchange of the sense of the step of the molecule would involve changes in atomic coordinates of approximately this amount, which is well within the range of the vibration amplitudes indicated in Table 1.

If disorder is the correct explanation of the anomalous thermal parameters for the molecule centred on $(0,0,\frac{1}{2})$, then this is also the explanation of the differences in bond-lengths and inter-bond angles between the two molecules. There are no grounds for expecting the two crystallographically non-equivalent molecules to possess different internal dimensions, but if the coordinates determined for one of them are averages of two or more molecular positions, then the bond-lengths and angles calculated from the mean may differ quite considerably from those of each molecule singly, as in the case of *p*-azotoluene (Brown, 1966). The dimensions of the average molecule centred on $(0,0,\frac{1}{2})$ may not therefore be taken as those of a single molecule and should be disregarded in favour of those calculated for the molecule centred on (0,0,0).

Correction of bond-lengths for thermal vibrations

The least-squares method described by Cruickshank (1956) for analysing the rigid-body vibrations of a molecule has been written as a computer program by Bujosa & Cruickshank (1961). Half of the molecule of azobenzene centred on (0,0,0) was treated as a rigid body; the other molecule, centred on $(0,0,\frac{1}{2})$ was not considered for the reasons of disorder mentioned in the preceding paragraph. The program calculates the magnitudes and directions of the principal moments of inertia of the half-molecule, and transforms the atomic coordinates and thermal parameters to these inertial axes. The translational and rotational components are then solved and a calculated set of temperature factors obtained for each atom. Then the observed values of *B* along the line from the atom to the origin are worked out and compared with the calculated values. These *B*(obs) and *B*(cal) values shown in Table 4 indicate the closeness of the rigid-body approximation as far as in-plane vibrations only are concerned. The program also computes the corrections to be applied to the atomic coordinates to allow for the angular oscillations; the revised values of the coordinates are also given in Table 4.

Table 4. *Corrected coordinates and radial B values*

	x/a	y/b	z/c	*B*(obs)	*B*(cal)
C(1)	0·1410	0·1341	0·0679	3·16	3·48
C(2)	0·1733	0·3461	0·1144	3·66	3·32
C(3)	0·2942	0·3907	0·1712	3·45	3·41
C(4)	0·3812	0·2308	0·1782	3·37	3·54
C(5)	0·3486	0·0211	0·1291	3·55	3·61
C(6)	0·2288	−0·0278	0·0742	3·89	3·62
N(1)	0·0145	0·1031	0·0114	3·32	3·18

From these revised coordinates, a corrected set of bond-lengths for the one molecule not subject to disorder is given in Table 5. The mean C–C length in the benzene ring is now 1·386 Å instead of 1·382 Å, and the mean increase taken over all the bond-lengths is 0·003 Å. The changes in angles are insignificant.

Table 5. *Bond lengths corrected for molecular vibrations*

C(1)–C(2)	1·388 Å
C(2)–C(3)	1·391
C(3)–C(4)	1·372
C(4)–C(5)	1·393
C(5)–C(6)	1·380
C(6)–C(1)	1·390
C(1)–N(1)	1·434
N(1)–N(1′)	1·247
e.s.d. (average)	0·003

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