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Three-Dimensional Refinement of the Structure of Anthrone

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The crystal structure of anthrone has been refined on the basis of the disordered structure by two cycles of three-dimensional differential synthesis. The structure belongs to the space group $P2_1/a$ (C_{2h}^5). A molecule of anthrone is planar, and has no centre of symmetry, but on the basis of the disordered structure it possesses a centre of symmetry statistically.

Introduction

Anthrone crystallizes in the monoclinic system and belongs to the space group $P2_1/a$ (C_{2h}^5) (Srivastava, 1957). There are only two asymmetric molecules per unit cell. In the rotation photographs about the *b* axis, faint diffuse layer lines parallel to the regular layers were observed in positions corresponding to double the translation period of the *b* axis. The anomaly in the space group and the occurrence of diffuse layer lines were explained by assuming a disordered structure (Banerjee & Srivastava, 1960; Srivastava, 1961) in which the anthrone molecules were assumed to take two possible random orientations at 180° in the (010) plane while in the [010] direction there is no orientational disorder.

The crystal structure of anthrone has also been worked out and refined by successive cycles of two-dimensional difference synthesis (Srivastava, 1962). The value of

$$R = \frac{\Sigma||Fo|-|Fc||}{\Sigma|Fo|} \times 100$$

dropped to nearly 23% after the first cycle of difference synthesis from an initial value of 31%. A number of subsequent cycles of difference synthesis could not reduce *R* appreciably. To improve the agreement and derive coordinates to a higher degree of accuracy than had been obtained previously, the intensity data were completely revised and three-dimensional data for the reflexions *h*0*l* to *h*3*l* were collected, and the structure was refined by two cycles of three-dimensional differential synthesis.

Crystal data

Anthrone, $C_{14}H_{10}O$, molecular weight 194·22. Melting point, 154 °C. The molecule has no symmetry centre. Space group $P2_1/a$, with the following cell dimensions:

$$a=15{\cdot}80 \pm 0{\cdot}03,\ b=3{\cdot}998 \pm 0{\cdot}005,\ c=7{\cdot}86 \pm 0{\cdot}016\ \text{Å},$$
$$\beta=101°\ 40' \pm 10',\ Z=2,\ D_m=1{\cdot}332\ \text{g.cm}^{-3}\ \text{and}$$
$$D_x=1{\cdot}335\ \text{g.cm}^{-3}.\ \mu=7{\cdot}88\ \text{cm}^{-1}\ \text{for Cu}\ K\alpha$$

radiation.

Experimental

A set of six to nine equi-inclination Weissenberg photographs, with each exposure time related to the next roughly by a factor of three, were prepared for each layer line from the zero to the third layer, with the crystal mounted along the *b* axis. The maximum possible value of the *k* index was five for Cu $K\alpha$ radiation, but on account of the limitation of the camera design, reflexions only up to the third layer could be recorded in the *b* axis setting. A Nonius integrating Weissenberg camera as described by Wiebenga & Smits (1950) was used. The crystal was mounted along the needle axis (the *b* axis) and therefore the linear dimensions of the spots parallel to the rotation axis remained constant for various spots during the rotation of the crystal. Extension of the reflexion spots was therefore made to occur along the lateral direction only. With the use of the multiple film technique, a pack of three films was exposed, these being developed simultaneously with a film over which a set of standard spots had been exposed from the same crystal. Standard spots were recorded by taking oscillations from the crystal from one to nearly twenty oscillations in the usual way. Intensities were visually estimated and were later modified by the corresponding angle factors. Structure amplitudes were brought down to absolute scale by the method of Wilson (1942), an equal isotropic temperature factor being assumed for all the atoms. The value for the temperature factor thus obtained is 4·0 Å^2. For the carbon and oxygen atoms the atomic scattering factors of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955), and for the hydrogen atom those of McWeeny (1951), were used. After application of the isotropic temperature factor to the f values, the structure amplitudes and phases were computed from the coordinates obtained from the two-dimensional refinement of the structure (Srivastava, 1962).

Three-dimensional differential syntheses

Before commencement of the three-dimensional work, the x and z coordinates were refined by the method of least squares, the revised $F(h0l)$ values being used. No significant change in the coordinates was indicated. With the same $F(h0l)$ values the x and z coordinates were then refined by a cycle of difference synthesis, and the y coordinates were similarly refined from the revised $F(hk0)$ values. Coordinates thus refined were then used for calculating the structure factors for all the planes lying within the Cu $K\alpha$ sphere of reflexion. Values of first and second differentials of electron densities at the assumed atomic positions were summed up, using only the observed reflexions, and the corrections for the atomic coordinates were computed. Corrections for the coordinates were appreciable and therefore another cycle of the differential synthesis was carried out, starting from the refined coordinates obtained in the first cycle of the synthesis. In the second cycle of the synthesis there was negligible change and therefore no further refinement was attempted. All the structure factors were recalculated at this stage and the R index came out to be 18% for the observed structure factors. Including the contributions of the eight hydrogen atoms of the anthrone molecule (leaving the two hydrogen atoms which are attached at the 9 and 10-positions) an R index of 17% was indicated. For this calculation the C–H distance was assumed to be 1·08 Å, along the diagonals of the benzene ring.

Table 1. *Atomic coordinates*

Atom	x	y	z
A'	2·146 Å	1·573 Å	3·069 Å
B'	0·869	1·056	2·676
C'	0·439	0·533	1·347
E	1·338	0·585	0·453
F	2·632	1·126	0·891
G	3·026	1·630	2·191
D	0·956	0·043	−0·994
O	1·663	0·095	−1·665

Table 2. *Bond lengths and valency angles*

Bond	Length	Average length of equivalent bonds
A′–B′	1·360 Å	1·368 Å
F–G	1·376	
B′–C′	1·412	1·400
E–F	1·389	
D–E	1·475	1·482
D′–C′	1·488	
G–A′	1·364	
E–C′	1·391	
D–O	1·109	

Bond angle	Angle	Average of equivalent angles
G–A′–B′	121° 21′	120° 28′
F–G–A′	119 34	
A′–B′–C′	120 08	120 26
E–F–G	120 45	
B′–C′–E	118 32	119 03
C′–E–F	119 34	
F–E–D	119 12	119 14
B′–C′–D′	119 16	
D–E–C′	121 12	121 42
E–C′–D′	122 12	
O–D–E	121 26	
C–D–E	116 36	
O–D–C	121 52	

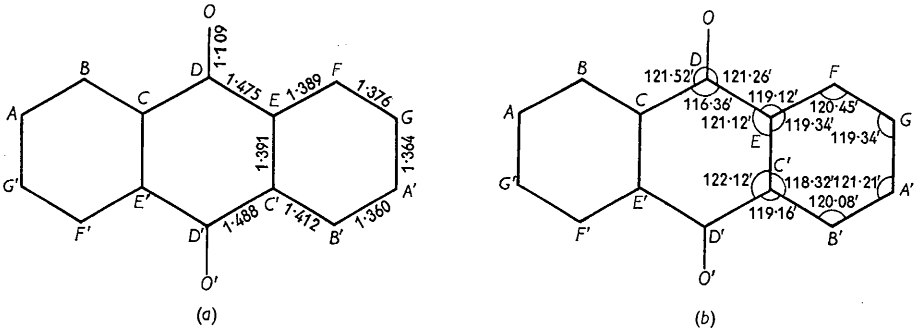

Fig. 1. (*a*) Bond lengths. Letter *O* stands for the group $\frac{1}{2}$(O + 2H) and other letters stand for the carbon atoms. (*b*) Bond angles.

Table 3. *Observed and calculated structure factor values for anthrone*

The observed structure factors are written under $|F_o|$.

Reflexions not observed on account of low intensity are marked (- -) in the $|F_o|$ column.

Some strong reflexions could not be observed owing to mechanical obstructions and measured intensities of some were very uncertain. In these cases the calculated values of structure factors have been used in the differential synthesis and their values are enclosed within small brackets under $|F_o|$.

Structure factors calculated from the final coordinates of Table 1 by means of the atomic f values of Berghuis *et al.* (1955) and of McWeeny (1951), modified by the application of the isotropic temperature factor are given in the column headed F_c.

Some reflexions have been calculated not from the final coordinates of Table 1 but from the coordinates which were used in the first cycle of differential synthesis. These calculated values are starred.

l	10$|F_o|$	10F_c
	001	
1	250	283
2	213	-192
3	57	- 15
4	224	-225
5	52	- 46
6	33	- 10
7	27	- 11
8	--	12
9	31	- 12
10	--	3*
	201	
-10	49	- 39
- 9	16	8
- 8	--	7
- 7	29	- 49
- 6	107	99
- 5	43	56
- 4	42	48
- 3	130	134
- 2	177	-177
- 1	423	513
0	279	316
1	169	-191
2	173	157
3	319	-318
4	253	-274
5	54	55
6	39	51
7	39	- 30
8	56	- 55
9	--	6
	401	
-10	32	- 27
- 9	--	- 5
- 8	20	- 30
- 7	141	168
- 6	173	224
- 5	48	- 62
- 4	81	56
- 3	19	- 9
- 2	267	-296
- 1	152	-174
0	233	-217
1	173	149
2	65	72
3	63	- 46
4	--	9
5	--	- 10
6	35	- 40
7	86	- 66
8	--	- 6
9	--	3
	601	
-10	--	12*
- 9	28	- 22
- 8	--	27
- 7	148	142
- 6	57	-0.2
- 5	29	- 14
- 4	60	- 26
- 3	257	-318
- 2	54	- 51
- 1	130	111
0	36	- 7
1	318	-296
2	69	- 46
3	117	109
4	41	- 33
5	33	- 5
6	--	- 8
7	31	- 14
8	29	26
	801	
-10	--	19*
- 9	31	- 30
--8	37	- 26
- 7	--	- 42
- 6	--	- 1
- 5	54	63
- 4	96	-101
- 3	99	- 55
- 2	80	75
- 1	66	62
0	203	-205
	801	
1	268	-295
2	--	2
3	42	- 36
4	123	114
5	207	167
6	--	- 3
7	--	- 7
8	--	1
	1001	
- 9	26	- 32
- 8	--	- 12
- 7	34	- 4
- 6	50	- 19
- 5	--	- 49
- 4	52	48
- 3	--	14
- 2	71	- 73
- 1	34	43
0	55	- 58
1	44	42
2	--	- 13
3	--	- 9
4	210	193
5	64	61
6	41	- 34
7	--	2
	1201	
- 9	--	17*
- 8	32	31
- 7	24	- 15
- 6	111	-120
- 5	60	- 45
- 4	--	17
- 3	32	- 25
- 2	196	162
- 1	138	123
0	33	- 7
1	52	61
2	33	- 35
3	--	- 6
4	--	1
5	40	- 38
6	31	24
	1401	
- 8	21	11
7	--	- 12
- 6	38	- 34
- 5	38	- 1
- 4	28	- 47
- 3	128	122
- 2	192	207
- 1	--	- 32
0	35	- 15
1	32	- 16
2	83	- 69
3	35	- 18
4	--	- 13
5	--	- 1
	1601	
- 7	23	16
- 6	30	26
- 5	--	1
- 4	32	- 34
- 3	23	46
- 2	20	- 21
- 1	--	- 12
0	--	17
1	64	- 57
2	21	- 16
3	40	29
4	--	2
	1801	
- 6	--	19*
- 5	18	- 23
- 4	39	- 36
- 3	39	- 27
- 2	22	- 18
- 1	17	17
0	10	- 10
1	--	1
2	19	11
	2001	
- 4	--	14*
- 3	--	13*
- 2	19	- 17
- 1	46	- 36
0	--	1
	011	
1	(487)	-487*
2	58	- 33
3	184	-200
4	99	-100
5	80	49
6	13	- 11
7	23	- 4
8	--	- 6*
9	--	- 1*
	111	
- 9	--	8*
- 8	12	- 26
- 7	28	30
- 6	35	45
- 5	6	7
- 4	32	- 31
- 3	153	-153
2	27	15
- 1	51	29
0	(258)	258
1	(617)	617
2	121	-110
3	184	-217
4	54	- 34
5	20	- 4
6	--	- 2
7	48	- 53
8	--	- 5
9	--	10*
	211	
- 9	23	6
- 8	--	- 13
- 7	82	74
- 6	67	74
- 5	36	- 23
- 4	104	108
- 3	40	- 45
- 2	60	- 53
- 1	--	- 8
0	(569)	-569
1	168	-182
2	48	52
3	104	- 88
4	117	112
5	77	76
6	31	- 21
7	39	- 44
8	--	1
9	--	6
	311	
-10	--	- 13*
- 9	--	- 33*
- 8	32	48
- 7	81	89
- 6	--	- 6
- 5	43	33
- 4	21	31
- 3	83	- 60
- 2	90	- 85
- 1	100	- 80
0	347	348
1	72	119
2	28	- 16
3	16	17
4	133	-137
5	68	- 69
6	--	- 10
7	--	- 8
8	--	- 6
9	--	- 16*
	411	
- 9	--	3
- 8	28	36
- 7	86	95
- 6	59	- 76
- 5	73	- 78
- 4	50	- 42
	411	
- 3	162	-205
- 2	--	6
- 1	--	26
0	99	- 84
1	18	24
2	--	- 18
3	52	- 47
4	67	65
5	--	- 16
6	44	- 48
7	--	9
8	26	28
9	6	4
	511	
- 9	--	- 18*
- 8	22	32
- 7	40	18
- 6	103	108
- 5	62	75
- 4	121	-138
- 3	27	- 14
- 2	--	9
- 1	58	- 42
0	197	-218
1	149	-168
2	120	133
3	29	- 8
4	23	- 40
5	41	22
6	19	- 16
7	--	- 6
8	20	- 18
	611	
- 9	17	- 8
- 8	22	27
- 7	26	- 50
- 6	111	-122
- 5	--	- 2
- 4	86	- 69
- 3	--	- 5
- 2	154	185
- 1	62	80
0	141	-151
1	124	-110
2	44	35
3	--	- 26
4	77	54
5	55	57
6	--	2
7	29	34
8	--	6
	711	
- 9	10	- 13
- 8	21	- 33
- 7	28	43
- 6	98	100
- 5	--	- 34
- 4	50	- 29
- 3	55	- 32
- 2	101	- 96
- 1	30	- 21
0	136	-148
1	66	- 64
2	91	- 82
3	41	48
4	149	164
5	28	22
6	--	- 23
7	--	- 9
8	12	- 8
	811	
- 9	--	- 22*
- 8	--	0
- 7	40	- 38
- 6	--	- 7
- 5	--	0
- 4	--	5
- 3	86	77
- 2	68	45
- 1	77	- 86
0	105	-102
1	100	112
2	67	51
3	52	41
	811	
4	136	142
5	--	- 5
6	46	- 36
7	--	0
8	--	- 24
	911	
- 9	--	1*
- 8	24	- 13
- 7	22	- 12
- 6	23	- 70
- 5	16	21
- 4	26	37
- 3	100	-138
- 2	21	21
- 1	52	47
0	--	11
1	89	- 85
2	80	- 94
3	84	60
4	63	63
5	33	33
6	44	29
7	24	- 23
	1011	
- 9	--	17*
- 8	25	32
- 7	--	- 5
- 6	25	- 40
- 5	--	- 9
- 4	--	8
- 3	--	5
- 2	67	72
- 1	--	16
0	64	44
1	88	102
2	--	- 10
3	46	53
4	10	- 5
5	88	- 99
6	8	- 12
7	--	- 8
	1111	
- 9	--	- 1*
- 8	--	- 7
- 7	44	- 46
- 6	61	- 45
- 5	--	- 5
- 4	--	- 25
- 3	91	91
- 2	91	100
- 1	--	- 18
0	--	16
1	27	- 27
2	--	- 11
3	--	- 21
4	--	6
5	71	73
6	--	10
7	--	- 18
	1211	
- 9	--	20*
- 8	--	6*
- 7	26	- 37
- 6	--	- 10
- 5	15	35
- 4	--	- 8
- 3	99	100
- 2	93	109
- 1	42	- 56
0	--	- 3
1	22	- 27
2	26	- 24
3	--	4
4	58	- 50
5	11	- 20
6	--	12
	1311	
- 8	15	26
- 7	--	1
- 6	33	- 24
- 5	63	- 69
- 4	11	16
- 3	104	111
	1311	
- 2	47	21
- 1	47	44
0	23	14
1	23	- 28
2	--	- 8
3	--	- 15
4	--	10
5	9	- 8
6	--	- 10
	1411	
- 8	--	9*
- 7	--	- 4*
- 6	31	45
- 5	23	18
- 4	23	20
- 3	49	71
- 2	63	- 74
- 1	93	- 82
0	--	- 5
1	36	- 52
2	--	3
3	29	30
4	--	- 3
5	--	- 4
	1511	
- 8	--	- 16*
- 7	15	18
- 6	--	- 8
- 5	30	- 28
- 4	13	- 6
- 3	--	- 11
- 2	71	65
- 1	72	70
0	25	- 40
1	22	- 18
2	--	0
3	--	- 9
4	21	- 19
5	--	- 26
	1611	
- 7	--	- 10*
- 6	--	11*
- 5	--	- 27
- 4	--	- 2
- 3	32	- 28
- 2	58	- 67
- 1	--	5
0	--	2*
1	--	- 2
2	39	36
3	--	10
4	--	- 18
	1711	
- 6	--	0
- 5	--	- 11*
- 4	21	- 22
- 3	--	- 6
- 2	--	28
- 1	--	- 28
0	--	2*
1	--	12
2	22	- 22
3	--	- 1
	1811	
- 6	--	- 12*
- 5	16	- 22
- 4	--	15
- 3	--	2
- 2	--	3
- 1	--	- 2
0	--	- 4
1	--	10
2	--	7
	1911	
- 5	--	11*
- 4	--	- 2*
- 3	17	- 14
- 2	30	- 36
- 1	14	- 11
0	--	13*
1	--	- 9

Table 3. (*cont.*)

l	$10\vert F_o\vert$	$10F_c$
	2011	
-3	--	-1*
-2	--	-16*
-1	--	-1*
	021	
1	15	5
2	51	-70
3	80	-92
4	48	58
5	24	28
6	33	-27
7	32	21
8	17	13
9	--	-11*
	121	
-9	--	-10*
-8	27	34
-7	68	77
-6	--	-3
-5	--	-2
-4	42	-25
-3	63	59
-2	130	89
-1	99	-99
0	105	105
1	120	-77
2	242	-202
3	29	38
4	22	-15
5	--	-24
6	36	-46
7	30	-27
8	--	16
9	--	1*
	221	
-9	--	7*
-8	42	60
-7	28	24
-6	35	-19
-5	54	21
-4	65	-51
-3	184	-181
-2	94	-95
-1	12	-46
0	21	-10
1	273	286
2	174	166
3	31	-41
4	--	-17
5	51	-48
6	40	-36
7	24	8
8	--	-11
	321	
-9	--	11*
-8	42	53
-7	--	-9
-6	--	1
-5	--	6
-4	75	-63
-3	34	29
-2	23	13
-1	50	43
0	134	-126
1	310	-338
2	8	-1
3	--	16
4	--	-15
5	49	39
6	--	8
7	--	18
8	--	-5
	421	
-9	--	-10*
-8	14	11
-7	--	4
-6	--	-16
-5	17	-25
-4	54	-57
-3	--	20
-2	94	86
-1	83	-95
0	38	-31
1	189	187
2	27	2
3	--	-1
4	29	8
5	66	-66
6	--	-4
7	--	20
8	--	-1
	521	
-9	14	23
-8	6	-7
-7	--	-3
-6	18	26
-5	86	-77
-4	68	-44
-3	27	16
-2	26	-9
-1	75	-50
0	251	-188
1	28	-24
2	30	18
3	39	22
4	142	175
5	35	51
6	--	-14
7	--	-11
8	--	6
	621	
-9	19	-33
-8	--	-12
-7	--	-2
-6	33	34
-5	77	81
-4	20	17
-3	--	-17
-2	21	30
-1	166	-138
0	64	-74
1	34	-47
2	--	-9
3	114	158
4	57	68
5	40	-33
6	--	6
7	--	-12
8	17	-24
	721	
-9	9	6
-8	--	0
-7	--	10
-6	65	-63
-5	81	-58
-4	--	22
-3	30	-27
-2	26	11
-1	37	20
0	37	45
1	14	20
2	--	-23
3	151	168
4	54	59
5	36	-39
6	--	7
7	21	-21
	821	
-9	--	6*
-8	--	-7
-7	30	-27
-6	51	68
-5	59	62
-4	28	-37
-3	--	5
-2	39	-22
-1	75	-54
0	42	40
1	--	-18
2	--	1
3	52	66
4	--	11
5	--	-6
6	--	-12
7	23	-33
	921	
-9	--	5*
-8	--	-1*
-7	29	-27
-6	55	-62
-5	--	12
-4	--	-12
-3	73	52
-2	113	96
-1	37	9
0	--	2
1	25	-22
2	62	82
3	38	37
4	41	-62
5	22	33
6	28	-20
7	--	-37
	1021	
-8	18	-21
-7	34	-40
-6	--	-5
-5	--	-21
-4	44	41
-3	87	93
-2	--	-20
-1	--	11
0	36	45
1	59	-52
2	90	-80
3	37	-30
4	--	-10
5	16	19
6	16	19
	1121	
-8	--	9*
-7	--	2*
-6	--	10
-5	--	-12
-4	50	30
-3	100	94
-2	6	-7
-1	26	-16
0	--	-11
1	--	-6
2	27	42
3	17	-21
4	--	14
5	52	-52
6	35	-74
	1221	
-8	--	-16
-7	12	-14
-6	14	-10
-5	12	-12
-4	49	63
-3	46	47
-2	--	-18
-1	13	-10
0	38	-30
1	63	-57
2	--	-8
3	--	2
4	--	-16
5	24	28
	1321	
-8	--	20*
-7	--	10*
-6	--	-3*
-5	--	2
-4	40	41
-3	--	-14
-2	--	-9
-1	--	19
0	46	-58
1	--	-9
2	12	12
3	--	3
4	28	-27
5	34	-79
	1421	
-7	--	22
-6	--	-2
-5	39	-46
-4	33	-25
-3	--	-8
-2	--	-1
-1	20	-15
0	24	7
1	--	8
2	24	30
3	--	-11
4	17	-24
	1521	
-7	--	0
-6	--	11*
-5	--	14
-4	--	-1
-3	--	-8
-2	--	16
-1	47	-54
0	40	-37
1	--	16
2	--	-12
3	--	-4
4	--	-20
5	--	4
	1621	
-6	--	-21
-5	24	-35
-4	--	-11
-3	10	-14
-2	--	5
-1	45	60
0	31	21
1	--	-7
2	--	2
3	15	-18
4	--	-13
	1721	
-5	--	13*
-4	--	3*
-3	--	4
-2	36	-33
-1	32	-36
0	--	16
1	--	-2
2	--	15
	1821	
-4	--	10*
-3	22	-28
-2	--	-1
-1	12	16
0	--	-11
1	--	9
	031	
1	43	-13
2	25	30
3	18	21
4	32	19
5	35	-38
6	14	-14
7	--	2
8	20	-44
	131	
-8	15	28
-7	--	-7
-6	18	-26
-5	36	-29
-4	45	-29
-3	--	1
-2	56	-66
-1	36	-30
0	--	34
1	--	-10
2	--	11
3	--	-16
4	29	-37
5	--	-4
6	--	0
7	20	21
8	--	24
	231	
-8	--	4
-7	--	3
-6	25	30
-5	28	-38
-4	58	-59
-3	14	21
-2	104	99
-1	--	-16
0	40	-53
1	54	51
2	107	-57
3	52	-34
4	21	14
5	28	-31
6	14	16
7	--	-5
	331	
-8	--	-2*
-7	--	-8*
-6	9	-6
-5	33	-36
-4	--	9
-3	--	0
-2	83	-77
-1	44	-89
0	56	-64
1	45	50
2	123	129
3	82	48
4	28	16
5	--	-4
6	--	1
7	--	6*
	431	
-8	--	-8*
-7	--	14
-6	9	-13
-5	--	-10
-4	--	9
-3	46	49
-2	15	21
-1	92	-82
0	13	17
1	112	-91
2	124	-113
3	102	105
4	26	33
5	--	-3
6	27	27
7	10	-6
	531	
-8	--	-6
-7	--	17
-6	--	-5
-5	--	-15
-4	21	13
-3	19	-12
-2	48	-22
-1	48	-44
0	--	-15
1	94	84
2	118	113
3	77	91
4	--	7
5	34	-40
6	25	-28
7	8	-11
	631	
-8	--	12*
-7	--	-21
-6	--	12
-5	--	16
-4	49	-54
-3	--	1
-2	37	-32
-1	--	-6
0	--	8
1	80	-82
2	77	70
3	66	72
4	21	-25
5	32	32
6	--	-3
7	--	-26
	731	
-8	--	-24*
-7	--	-5
-6	--	-9
-5	36	44
-4	52	40
-3	46	30
-2	27	29
-1	--	13
0	--	-4
1	15	-14
2	36	29
3	--	28
4	28	-16
5	14	-13
6	20	-22
	831	
-7	11	-13
-6	--	1
-5	55	-84
-4	--	-4
-3	62	70
-2	--	-18
-1	51	38
0	24	26
1	--	5
2	19	20
3	26	-38
4	--	6
5	--	10
6	--	-18
	931	
-7	--	-25
-6	--	19
-5	52	57
-4	76	73
-3	49	53
-2	--	-16
-1	51	-26
0	46	-51
	931	
1	21	-28
2	--	-10
3	29	-19
4	--	10
5	--	10
	1031	
-7	--	8*
-6	17	-35
-5	15	-22
-4	59	91
-3	--	12
-2	--	0
-1	40	38
0	26	-37
1	33	-30
2	--	3
3	31	20
4	--	-2
5	7	-8
	1131	
-7	--	-2*
-6	--	-2*
-5	18	18
-4	35	44
-3	--	-3
-2	30	-30
-1	33	-31
0	21	-21
1	--	8
2	18	-20
3	29	-32
4	--	-8
5	--	-4
	1231	
-6	--	-5*
-5	--	22
-4	--	-2
-3	--	-20
-2	18	29
-1	15	-21
0	25	-33
1	18	16
2	41	41
3	--	1
4	17	-21
	1331	
-6	--	-24*
-5	--	-17*
-4	11	-10
-3	12	-8
-2	--	13
-1	--	-14
0	--	-5
1	--	8
2	24	-21
3	19	-21
	1431	
-5	--	-9*
-4	--	-1*
-3	--	12
-2	--	-18*
-1	--	1*
0	--	17*
1	--	4*
2	--	8*
3	--	-18
	1531	
-5	--	-2*
-4	--	-25
-3	15	-8
-2	--	-3
-1	--	-3
0	15	18
1	9	5
2	--	1
	1631	
-4	--	-17*
-3	--	-17
-2	--	-4*
-1	--	6*
0	22	-32

Description of the structure

The final coordinates of the asymmetric unit of the structure of anthrone are given in Table 1, the origin being at the symmetry centre. Bond lengths and angles are given in Table 2. Fig. 1(*a*) and (*b*) shows these bond lengths and angles. Figs. 2, 3, and 4 show the structure viewed in the [010], [100] and [001] directions respectively. Values of the observed and calculated structure amplitudes along with their phases are given in Table 3. The intermolecular distances are greater than 3·3 Å and they are given in Fig. 2.

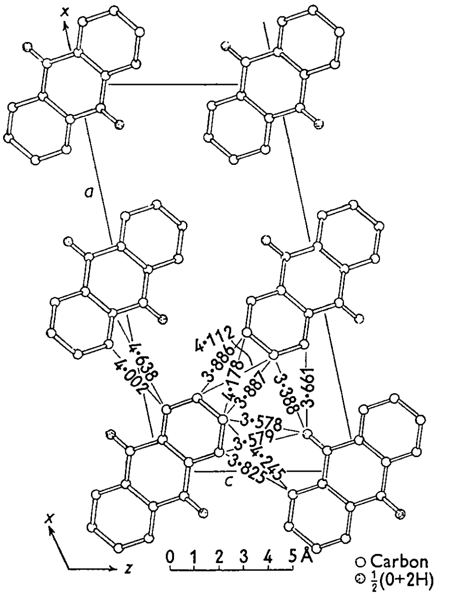

Fig. 2. Projection of the anthrone molecule structure in the [010] direction.

The equation of the molecular plane with reference to the crystallographic axes is

$$y = 0{\cdot}3340x + 0{\cdot}2823z\,.$$

The molecule is tilted about the line joining the 9 and 10 positions, at an angle of 25° 59′ with the (010) plane. The direction joining the 9 and 10 positions makes an angle of 2° 04′ with the (010) plane.

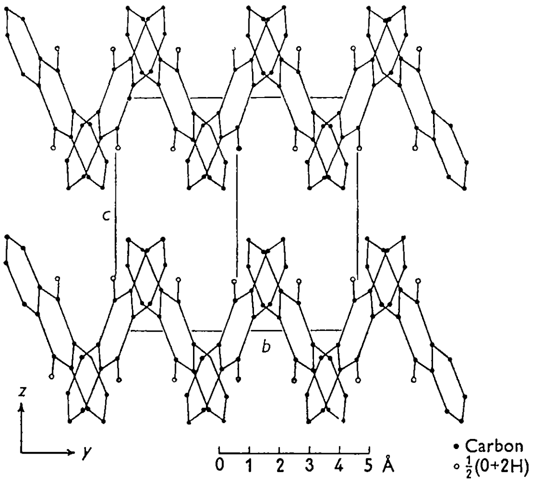

Fig. 3. Projection of the structure on (100).

The standard deviations of the coordinates have been calculated by the formula of Cruickshank (1949):

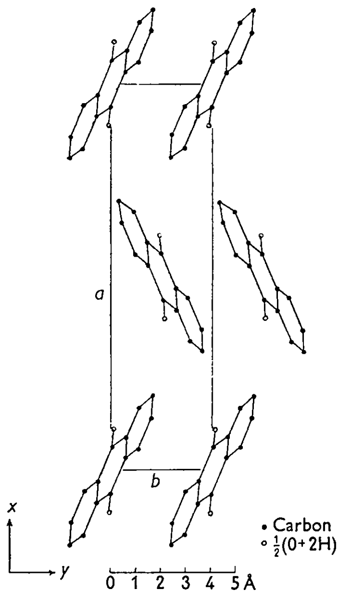

Fig. 4. Projection of the structure on (001).

$$\sigma(x) = \frac{\dfrac{2\pi}{V}\sqrt{\left[\sum_h\sum_k\sum_l\left\{h^2(F_o-F_c)^2\right\}\right]}}{\dfrac{4\pi^2}{Va}\sum_h\sum_k\sum_l\left[h^2F_o(hkl)\cos\left\{2\pi\left(\dfrac{hx}{a}+\dfrac{ky}{b}+\dfrac{lz}{c}\right)\right\}\right]}$$

and similar expressions for $\sigma(y)$ and $\sigma(z)$, where V is the unit cell volume and the other symbols have their usual meanings. Accidentally absent reflexions have not been used in the summation. The standard deviations for each atom were calculated separately, and are given in Table 4 as values after averaging.

Table 4. *Standard deviations*

Atom	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$
Carbon	0·012 Å	0·017 Å	0·014 Å
Oxygen	0·016	0·037	0·018

Discussion

From the observed systematic extinctions the space group for anthrone was fixed as $P2_1/a$ although there were only two asymmetric molecules per unit cell. Diffuse layer lines were found to be interleaved with the regular layer lines in the rotation photographs about the *b* axis, owing to the disorder in the structure. Diffuseness extends in the directions perpendicular to the *b* axis, indicating that there is disorder over the plane perpendicular to the axis. There is no extension of the diffuseness parallel to the *b* axis, a more or less perfect order along that direction being thus indicated. About the centre, the anthrone molecule is assumed to take two possible orientations at 180° in its own plane in a random manner. This would consequently impart a molecular centre of symmetry in the statistical sense. This disorder is confined to the orientation of the molecule in the plane perpendicular to the *b* axis. For the molecules lying in the column parallel to the *b* axis there is no orientational disorder. Many structures have been worked out where a similar type of disorder had to be postulated. Structures where the molecules were assumed to take possible orientations in the molecular plane, and showed diffuse layer lines in the rotation photographs, are 9-chloro-10-bromoanthracene (Hospital, 1961), 9-bromo-10-methylanthracene (Prat, 1961) and Wolffram's red salt (Craven & Hall, 1961). Diffuse layer lines in the rotation photographs have also been observed in form II of $Na_2P_4O_{11}$ (Gryder, Donnay & Ondik, 1958) and in the crystal lattices of the molecular complexes of 4,4′-dinitrobiphenyl with 4-chlorobiphenyl and 4,4′-diiodobiphenyl (James & Saunder, 1948). Some of the structures presented a similar type of space group difficulty, although diffuse layer lines were not observed in those cases. On the basis of the disordered structure better agreement was obtained with the experimental results, and the anomaly of the space group was also removed. From the absence of the diffuse layers it was concluded that the disorder was homogeneous and was present throughout the whole structure. Examples of such structures are *p*-chlorobromobenzene (Hendricks, 1933; Klug, 1947); 2-amino-4-methyl-6-chloropyrimidine (Clews & Cochran, 1948); 1-bromo-4-chlorocyclohexane (Hassel & Vihovde, 1953); 1,2-dichlorotetramethylbenzene (Tulinsky & White, 1958); diindenyliron (Trotter, 1958); *N*-oxyphenazine (Curti, Riganti & Locchi, 1961; azulene (Robertson, Shearer, Sim & Watson, 1962); and *p*-chloronitrobenzene (Mak & Trotter, 1962). In acepleiadylene (Hanson, 1960) on account of the statistical overlapping, C–C bond length ranged from 1·27 to 1·52 Å. Iron(II) fluoride tetrahydrate investigated by Penfold & Taylor (1960) presents a more interesting case where twelve sites were available for water and fluorine and in the disordered structure each site was found to be occupied by $\frac{1}{3}O+\frac{1}{6}F$. In the case of cycl(3,2,2)azine one is not able to distinguish whether the molecules are oriented at all orientations in their planes or rotating in their own plane freely on account of the thermal motion (Hanson, 1961).

On account of the overlapping of the two orientations of anthrone molecules in the crystal structure, the bond length C=O and the bond angle *CDE* are 1·109 Å and 116° 36′ respectively, thus differing considerably from their corresponding normal values (1·22 Å and 120°). The average length of the bond *DE* and *D′C′* is 1·482 Å which lies well between the normal C–C distances for aliphatic and aromatic structures. All other bond lengths of this molecule are quite near to the standard value of 1·38 Å for the benzene ring and the valency angles are in the neighbourhood of 120°.

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