Table 3. *Observed structure factors* (× 10) *for 5-bromo-benzfurazan-*1*-oxide*

H	K	FO	H	K	FO	H	K	FO	H	K	FO	H	K	FO	H	K	FO	H	K	FO
L=0			6	4	96	2	6	161	-3	1	1439	3	4	75	-6	1	132	2	3	379
			6	5	157	3	0	1088	-3	2	346	3	5	297	-6	2	460	2	5	210
0	2	773	7	1	305	3	1	373	-3	3	687	4	1	921	-6	3	77	3	2	121
0	4	605	7	2	133	3	2	953	-3	4	48	4	2	222	-6	4	271	3	3	339
0	6	129	7	3	131	3	3	134	-3	5	243	4	3	530	-6	5	91	3	4	116
1	1	461	7	4	100	3	4	382	-4	2	247	4	5	196	-7	1	51	3	5	169
1	2	232	7	5	80	3	6	198	-5	0	1052	5	2	475	-7	2	76	4	1	139
1	3	147	7	6	97	4	1	200	-5	1	226	5	3	103	-7	3	186	4	4	254
1	4	162	8	0	368	4	2	120	-5	2	823	5	4	253	-7	4	155	5	1	279
1	5	232	8	2	293	4	3	291	-5	4	199	5	6	92	-8	0	260	5	2	440
1	6	124	8	3	52	4	5	229	-6	1	178	6	2	488	-8	1	422	5	4	239
1	7	90	8	4	216	5	0	339	-6	3	134	6	4	271	-8	2	204	6	3	173
2	0	301	8	5	56	5	1	912	-7	1	500	6	6	82	-8	3	216	7	2	138
2	2	137	8	6	65	5	3	604	-7	3	351	7	3	186	-8	5	92	7	3	302
2	3	674	9	2	52	5	5	157	-7	4	109	7	5	131	-9	4	65	8	2	73
2	5	275	9	3	166	6	1	124	-7	5	115	-1	1	426	-9	5	63	9	1	107
2	6	67	10	0	93	6	2	149	-9	2	266	-1	2	236	-10	0	284	9	2	273
3	1	227	10	1	259	6	4	128				-1	3	402	-10	1	63	11	3	158
3	2	597	10	2	96	7	0	614	L=2			-1	4	175	-10	2	159	-1	1	1047
3	3	316	10	5	52	7	1	89				-1	5	143	-10	4	91	-1	4	140
3	4	281	11	3	40	7	2	464	0	2	288	-2	2	533	-11	3	91	-1	5	233
3	5	136	12	0	170	7	4	155	0	3	490	-2	3	149	-12	0	64	-2	1	153
3	6	148				8	1	97	0	4	200	-2	4	527	-12	1	91	-2	2	76
4	0	1293	L=1			8	3	107	0	5	287	-2	5	83	-12	2	63	-3	0	852
4	1	325				9	1	234	1	1	785	-3	1	113				-3	1	347
4	2	585	0	1	223	9	3	288	1	2	169	-3	2	203	L=3			-3	2	797
4	3	40	0	2	214	10	2	161	1	3	103	-3	3	329				-3	3	176
4	4	415	0	3	370	11	0	162	1	4	237	-3	5	122	0	1	222	-3	4	235
4	6	98	0	4	242	11	2	200	1	5	102	-3	6	92	0	2	344	-4	1	157
5	1	139	0	5	261	-1	2	1420	1	6	183	-4	0	384	0	4	226	-5	0	291
5	2	257	0	6	159	-1	3	189	2	1	686	-4	1	774	0	5	191	-5	1	559
5	3	237	1	0	92	-1	4	282	2	2	854	-4	2	278	0	6	111	-5	2	210
5	4	113	1	1	399	-1	6	163	2	3	149	-4	3	516	1	1	209	-5	3	307
5	5	199	1	2	56	-2	1	47	2	4	413	-4	5	176	1	2	731	-5	4	126
5	6	120	1	5	142	-2	2	259	2	5	75	-5	1	507	1	3	335	-7	1	185
6	0	216	2	1	310	-2	3	233	2	6	159	-5	2	103	1	4	156	-7	2	243
6	1	713	2	3	186	-2	4	187	3	1	185	-5	3	207	1	5	103	-8	1	100
6	2	59	2	4	386	-2	5	240	3	2	143	-5	5	156	1	6	132	-9	0	94
6	3	427	2	5	277	-3	0	584	3	3	183	-6	0	386	2	2	144	-9	1	181

Waals distances except the N(2)· · ·X distances of 3·14 and 3·20 Å in the bromo and chloro compounds, respectively; these should be compared with the expected van der Waals distances of 3·45 and 3·30 Å, respectively. The distance in the chloro compound, by itself, might not be convincing, but the distance in the bromo compound is significantly shorter than the van der Waals distance and moreover is shorter than the distance in the chloro compound, which would not be expected in the absence of any interaction.

Table 4. *Interatomic distances between molecules*

Distance	Atoms	$BrC_6H_3N_2O_2$	$ClC_6H_3N_2O_2$
a	X–N(2)	3·14 Å	3·20 Å
b	X–O(2)	3·69	3·66
c	X–O(1)	3·92	3·74
d	X–C(6)	4·15	4·05
e	C(5)–O(2)	3·58	3·59
f	C(5)–O(1)	3·99	3·96
g	C(3)–O(1)	3·64	3·53
h	N(2)–O(1)	4·20	3·87

It appears to us that this specific acid–base interaction, N· · ·X, provides a likely explanation for the difference in packing between the halogen compounds and the corresponding methyl compound. What is surprising about the situation is that the melting points of these three compounds, Br, 69; Cl, 48; CH_3, 98°C, taken by themselves would seem to indicate stronger interactions in the methyl compound than in the other two. We can offer no explanation for this anomaly.

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The Crystal Structure of 5,6-Dichlorobenzfurazan-1-oxide

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5,6-Dichlorobenzfurazan-1-oxide is monoclinic with $a=11{\cdot}609$ (19), $b=8{\cdot}956$ (15), $c=7{\cdot}355$ (12) Å, and $\beta=96{\cdot}2$ (1)°, with four molecules in the unit cell. The measured density is 1·80 (2) g.cm^{-3}; the calculated density is 1·790 (5) g.cm^{-3}. The space group is $C2/c\,(C_{2h}^{6})$. The molecules are disordered with the pseudo-twofold axis of the molecule approximately aligned with the twofold axis in the crystal. The disorder precludes any accurate determination of the molecular parameters. The molecules lie in sheets parallel to the $(10\bar{1})$ plane, and, within the sheets, in rows parallel to **b**. The O· · ·Cl contacts within the rows are approximately normal, near 3·2 Å. There is one short intermolecular distance in the structure, an O· · ·C distance of 3·16 or 3·33 Å depending on the disorder.

Introduction

Several years ago two of us (JH and LMT) undertook to determine the structure of 5,6-dichlorobenzfuroxan (= 5,6-dichlorobenzfurazan-1-oxide) in order to determine the configuration of the benzfuroxan ring. This work was set aside when the completed structure determination of 5-chlorobenzfuroxan (Britton & Noland,

1962) answered the same question. With the later recognition that there was a short intermolecular N···Cl contact in the 5-chlorobenzfuroxan, it was decided that the packing and intermolecular contacts of the dichloro compound were of sufficient interest to justify the completion of the structure determination. [See also the introductions to the two preceding papers (Britton & Noland, 1972; Britton, Hardgrove, Hegstrom & Nelson, 1972)].

Experimental

5,6-Dichlorobenzfuroxan was prepared by hypochlorite oxidation of 4,5-dichloro-2-nitroaniline. Since then much the same preparation has been described by Mallory, Manatt & Wood (1965). Roughly cubic crystals 0·6 to 0·8 mm on an edge were examined by precession photographs using Mo $K\alpha$ radiation ($\lambda=0{\cdot}7107$ Å) and were found to be monoclinic with $a=11{\cdot}609(19)$, $b=8{\cdot}956(15)$, $c=7{\cdot}355(12)$ Å, and $\beta=96{\cdot}2(1)^\circ$. The errors are the one part in 600 we estimate to be the limit of this method. The calculated density of 1·790(5) g.cm^{-3} for $Z=4$ agrees well with the experimental density of 1·80(2) g.cm^{-3} determined by flotation. Systematic extinctions (hkl, $h+k=2n+1$; $h0l$, $l=2n+1$) show the space group to be either $Cc\,(C_s^4)$ or $C2/c\,(C_{2h}^6)$. The eventual structure determination suggests that the latter is correct; this is discussed at length below.

Intensity data were collected using precession photographs of $0kl$–$4kl$, $h0l$–$h3l$, hhl, and $h,2h,l$. The intensities were measured by visual comparison with an intensity standard, and put on a common scale by comparison of all reflections common to two or more films. There were 482 independent reflections of measurable intensity. These were corrected for Lorentz and polarization factors* but not for absorption. The linear absorption coefficient for Mo $K\alpha$ radiation is 8·0 cm^{-1} so that μR is approximately 0·3. At the time the data were collected absorption corrections were not routinely made under these circumstances and the lack of an accurate record of the size and shape of the crystal precludes making them now.

Solution and refinement

The structure was solved approximately from examination of a three-dimensional Patterson map, which appeared to show that a molecule with twofold symmetry lay along the twofold axis of the crystal (space group $C2/c$). This required the molecules to be disordered, and was slightly surprising since there was nothing in the photographs, either in terms of abnormal streaks or spot shapes or of unusually rapid decline in intensity that had suggested disorder. The other possibility was that the molecules were ordered in space group Cc but happened by coincidence to have the pseudo-twofold axis of the molecule approximately parallel to **b**. Refinement was tried, more-or-less successfully, in both space groups, refining individual atomic parameters, but the choice between the two alternatives was not clear. The problem was set aside again at this point to wait for the accurate determination of the molecular dimensions of a model compound and the availability of a group refinement program.

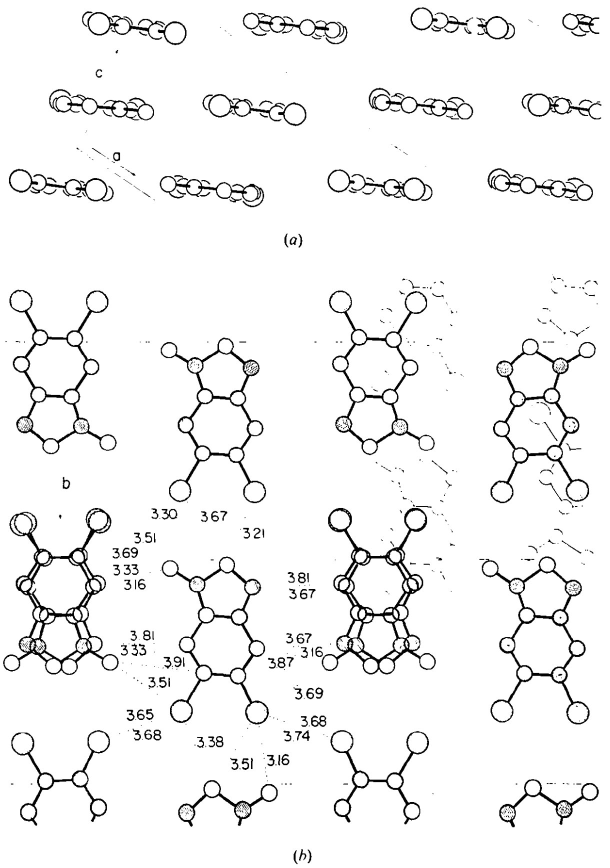

Fig. 1. The crystal structure of 5,6-dichlorobenzfurazan-1-oxide. (*a*) Projection along **b**. Only one of the disordered molecules is shown at each position. (*b*) Projection perpendicular to the planes of molecules apparent in the top projection, *i.e.* perpendicular to $(10\bar{1})$. Short intermolecular distances are given in Å. To the left and right of the central molecule both of the disordered orientations are shown superimposed. For the other molecules only one of the two orientations is shown. In the upper right-hand corner some molecules in the next layer are also shown.

For the group refinement the intramolecular dimensions of the light atoms were assigned to be the same as those in 5-methylbenzfuroxan [without libration corrections (see the preceding paper Britton & Noland, 1972)], except that the molecule was made exactly planar, and the chlorine atoms were placed so that the C–Cl distance was 1·705 Å, the Cl···Cl distance was 3·12 Å, and the C_2Cl_2 group had $2/m$ symmetry. These chlorine distances are based on the results of Rudman (1971).

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* The programs used were the same as those used in the two preceding papers; R and r have the same definitions.

Group refinement was carried out treating the entire molecule as a single group with isotropic thermal parameters for all the atoms. The first attempt was refinement in $C2/c$ with the molecule aligned so that the twofold axis of the C_2Cl_2 atom cluster coincided with the twofold axis of the space group. Although this should not have been an ambiguous starting point, since the molecule as a whole does not have twofold symmetry, nevertheless it produced a singular matrix. For the next attempt the initial values for the orientational angles of the group were changed randomly by about 2°; this time refinement proceeded smoothly, converging with r=0·120, R=0·183.* Next, refinement was carried out in Cc using the same starting parameters as were used for the convergent refinement in $C2/c$. This refinement converged with r=0·139, R=0·195. Since there are two fewer parameters in Cc (the x and z coordinates of the group center may be arbitrarily fixed) than in $C2/c$, the lower r value in $C2/c$ strongly supports the choice of the centric space group and disorder.

Although, as mentioned above, refinement could be carried out with individual atom parameters, and indeed with individual atoms and anisotropic thermal parameters in $C2/c$, refinement could be reached with r=0·067, R= 0·135, nevertheless we are reporting only the results of the group refinement in $C2/c$. The nearness of the atomic positions in the two disordered positions (as can be seen in Fig. 1) clearly makes any anisotropic thermal parameters meaningless as well as giving average atomic positions that would lead to molecular dimensions surely less accurate than those in the model. The final group parameters are given in Table 1, the atomic parameters from the group refinement in Table 2, and the observed structure factors in Table 3.

Table 1. *Group parameters*

The origin of the group is halfway between atoms C(5) and C(6). The angles are in radians. δ, ε and η are angles that bring about alignment (except for translation) of an internal coordinate system within the group with a fixed external system. See La Placa & Ibers (1965) for the definitions.

x	y	z
0·5073 (8)	0·5131 (7)	0·2396 (11)
δ	ε	η
1·625 (3)	2·769 (5)	3·107 (4)

Table 2. *Positional parameters* ($\times 10^3$) *and temperature factors for the atoms*

	x	y	z	B
Cl(4)	628 (1)	688 (1)	331 (2)	3·4 (3)
Cl(5)	376 (1)	670 (1)	134 (2)	4·4 (4)
O(1)	343 (1)	36 (1)	120 (3)	5·6 (6)
O(2)	528 (1)	20 (1)	265 (3)	4·8 (6)
N(1)	429 (1)	105 (1)	186 (2)	4·8 (8)
N(2)	615 (1)	117 (1)	332 (2)	4·9 (6)
C(1)	459 (1)	248 (1)	207 (2)	3·1 (7)
C(2)	572 (1)	252 (1)	295 (2)	3·4 (8)
C(3)	626 (1)	393 (1)	335 (2)	5·1 (12)
C(4)	566 (1)	517 (1)	285 (1)	2·4 (8)
C(5)	449 (1)	509 (1)	194 (1)	3·0 (9)
C(6)	395 (1)	377 (1)	154 (1)	2·4 (8)

Results and discussion

The structure is shown in Fig. 1. The molecules lie approximately in planes parallel to (10$\bar{1}$). The average distance between layers is 3·19 Å. The shortest interatomic distances between layers are one at 3·21 Å and six between 3·35 and 3·40 Å; these are calculated considering both possible alignments of the disordered molecules. The short interatomic distances between molecules within the plane are shown in the Figure. Two of the molecules are shown in both possible orientations, the rest only in one. The molecules lie in rows parallel to **b**, but the O···Cl contacts between molecules in the

Table 3. *Observed structure factors* ($\times$ 10) *for* 5,6-*dichlorobenzfurazan*-1-*oxide*

H	K	FO
L=0		
2	0	1087
4	0	1403
6	0	204
8	0	327
10	0	128
12	0	235
16	0	71
1	1	283
3	1	237
5	1	321
9	1	97
13	1	54
15	1	66
0	2	97
2	2	207
4	2	452
6	2	130
8	2	232
10	2	74
12	2	66
1	3	1018
3	3	862
5	3	163
7	3	472
9	3	209
11	3	92
0	4	107
2	4	398
4	4	510
1	5	508
3	5	207
5	5	423
0	6	513
4	6	286
6	6	156
2	8	186
4	8	199
8	8	194
1	9	107
3	9	79
0	10	105
2	10	61
1	11	66
0	12	64
L=1		
1	1	304
3	1	551
5	1	107
7	1	395
11	1	115
-3	1	403
-5	1	403
-7	1	194
-9	1	278
-13	1	69
0	2	1117
2	2	355
4	2	434
6	2	199
8	2	54
10	2	140
-2	2	673
-4	2	395
-6	2	217
-8	2	173
-12	2	102
3	3	694
5	3	61
7	3	217
9	3	107
11	3	79
-1	3	895
-3	3	106
-5	3	298
-9	3	161
-11	3	77
0	4	426
2	4	403
4	4	508
-2	4	184
-4	4	518
1	5	153
3	5	224
5	5	115
-1	5	656
-5	5	184
0	6	431
2	6	138
4	6	171
6	6	122
-2	6	273
-4	6	196
-6	6	107
1	7	242
3	7	355
7	7	191
-1	7	763
-7	7	66
0	8	163
2	8	115
-2	8	117
-4	8	115
-8	8	84
1	9	89
3	9	102
-1	9	281
-9	9	87
0	10	56
2	10	138
4	10	133
-4	10	151
-1	11	43
0	12	102
-2	12	92
L=2		
0	0	1393
2	0	1082
4	0	561
6	0	342
10	0	309
12	0	235
14	0	79
-2	0	2566
-6	0	860
-10	0	158
-12	0	138
-14	0	110
1	1	125
3	1	385
5	1	13
7	1	133
9	1	66
11	1	94
13	1	59
-1	1	276
-3	1	54
-5	1	230
-7	1	158
-9	1	38
-13	1	64
2	2	411
4	2	296
6	2	253
8	2	186
10	2	125
-2	2	74
-4	2	207
-6	2	296
-10	2	163
1	3	605
3	3	556
5	3	434
7	3	390
9	3	79
11	3	82
-11	3	79
-1	3	987
-3	3	995
-5	3	561
-9	3	362
0	4	273
2	4	503
4	4	140
-2	4	87
-4	4	367
1	5	71
3	5	421
5	5	133
-1	5	485
-3	5	209
-5	5	273
0	6	138
2	6	276
6	6	291
-2	6	304
-4	6	133
-6	6	184
7	7	122
0	8	94
2	8	217
8	8	69
-2	8	26
-4	8	173
-8	8	99
1	9	41
3	9	54
-1	9	122
-9	9	66
0	10	61
2	10	31
4	10	79
-2	10	87
1	11	54
-1	11	113
0	12	38
L=3		
1	1	372
3	1	77
5	1	400
7	1	151
9	1	122
13	1	38
-5	1	296
-7	1	163
-9	1	173
-11	1	115
0	2	46
2	2	395
4	2	77
6	2	138
8	2	125
10	2	66
-2	2	551
-4	2	255
-6	2	281
-10	2	102
1	3	439
3	3	212
5	3	592
7	3	158
-1	3	120
-3	3	283
-5	3	191
-7	3	181
-11	3	82
0	4	161
2	4	436
4	4	105
-2	4	296
-4	4	48
1	5	171
5	5	163
-1	5	191
-3	5	222
-5	5	71
2	6	176
6	6	128
-2	6	278
-4	6	107
-6	6	138
1	7	286
3	7	265
7	7	92
-1	7	319
-3	7	444
-7	7	255
0	8	51
2	8	64
4	8	151
8	8	105
-2	8	153
1	9	107
3	9	41
-1	9	117
-3	9	133
0	10	51
2	10	158
-2	10	77
1	11	74
L=4		
0	0	413
2	0	480
4	0	189
6	0	71
8	0	263
10	0	99
12	0	82
14	0	82
-2	0	977
-4	0	1094
-6	0	171
-8	0	309
-14	0	51
1	1	232
3	1	112
5	1	122
9	1	112
13	1	66
-3	1	212
-7	1	151
-9	1	41
0	2	219
2	2	283
4	2	133
6	2	224
10	2	69
-2	2	79
-4	2	54
-6	2	153
-8	2	120
-10	2	61
1	3	474
3	3	227
5	3	467
9	3	105
-1	3	128
-3	3	418
-7	3	296
-11	3	166
0	4	342
2	4	153
4	4	227
-2	4	107
-4	4	79
1	5	337
3	5	166
5	5	77
-1	5	79
-3	5	237
-5	5	64
0	6	186
2	6	92
4	6	224
6	6	69
-2	6	56
-4	6	156
-6	6	128
7	7	79
0	8	151
2	8	71
4	8	130
-2	8	28
1	9	94
-3	9	43
2	10	84
-2	10	66
1	11	59
-1	11	41
L=5		
1	1	128
3	1	227
5	1	181
7	1	89
9	1	38
-1	1	171
-3	1	69
-7	1	173
-11	1	107
0	2	245
4	2	120
8	2	94
-2	2	61
-4	2	314
-8	2	130
1	3	151
3	3	97
5	3	156
-1	3	166
-3	3	99
-7	3	71
0	4	332
2	4	26
4	4	286
-4	4	207
3	5	102
-1	5	74
-3	5	99
-5	5	115
0	6	120
2	6	28
4	6	99
-2	6	77
-4	6	204
1	7	237
7	7	97
-1	7	105
-3	7	202
-7	7	94
0	8	51
2	8	77
4	8	56
-4	8	56
1	9	61
-1	9	43
-3	9	64
0	10	107
4	10	87
L=6		
0	0	332
4	0	92
6	0	153
8	0	128
12	0	74
-2	0	74
-4	0	541
-6	0	391
-8	0	138
-10	0	84
1	1	72
3	1	66
7	1	99
-1	1	105
-3	1	56
-5	1	130
-9	1	61
0	2	148
2	2	51
4	2	166
8	2	74
-2	2	79
-4	2	71
-8	2	69
3	3	316
5	3	89
7	3	99
-1	3	250
-5	3	138
0	4	360
2	4	133
4	4	179
-2	4	163
1	5	110
3	5	43
-1	5	115
-3	5	69
-5	5	153
0	6	89
2	6	130
4	6	77
6	6	77
-2	6	66
-4	6	56
0	8	56
2	8	77
4	8	99
-2	8	69
-6	8	79
1	9	43
0	10	28
L=7		
1	1	66
3	1	120
7	1	38
-1	1	87
-3	1	51
-5	1	38
-9	1	54
0	2	43
2	2	59
-2	2	94
-4	2	59
3	3	117
-1	3	94
0	4	61
2	4	158
4	4	71
-2	4	158
1	5	43
-5	5	66
0	6	48
2	6	31
-2	6	59
-4	6	59
-6	6	56
-1	7	138
-7	7	56
2	8	51
-2	8	31
L=8		
2	0	59
4	0	97
6	0	99
-2	0	168
-6	0	214
-8	0	97
1	1	48
-1	1	48
-3	1	38
-5	1	38
-7	1	38
2	2	94
-2	2	77
1	3	110
3	3	99
-1	3	43
-3	3	110
0	4	46
2	4	105
-2	4	66
-4	4	64
-1	5	56
-5	5	64
2	6	43
-2	6	56
L=9		
0	4	54
1	1	38
2	2	46
1	3	92
2	4	51
-2	4	31

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* For all of these refinements F_w was taken as 25 in the assignment of weights (for F_c on an absolute basis).

rows, 3·16, 3·21, 3·30 or 3·51 Å, are not unusually short compared with the normal van der Waals O···Cl distance of 3·20 Å. In the chloro- and bromobenzfuroxans short N···X distances were found. It was our expectation that similar short N···X distances would be found here, but the packing precludes any N···Cl contacts, and replaces them with normal O···Cl contacts. There is one unusually short intermolecular contact, O(1)···C(3)–3·16 Å or O(1)···C(6)–3·33 Å, depending on the disorder. However, these distances are similar to those Donohue (1968) has discussed in detail and rejected as O···H–C hydrogen bonds. In view of the additional uncertainty due to the disorder, there is no reason to regard this as other than an accidentally short distance. It should be mentioned that the refinement in the ordered, acentric arrangement still leaves this distance short, at 3·12 Å. Generally the packing is similar for both the ordered and disordered models; there are no special features in one that are not in the other as well, so that the packing does not appear to favor one arrangement over the other, and we are left accepting the evidence of the *r* values as the deciding factor in favor of disorder.

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The Crystal Structure of 5-Iodobenzfurazan-1-oxide

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5-Iodobenzfurazan-1-oxide is monoclinic with $a=10·413$ (17), $b=19·787$ (33), $c=7·596$ (13) Å, and $\beta=103·4$ (1)°, eight molecules in the unit cell, space group $P2_1/c$ (C^2_{5h}). The structure was solved using film data. Refinement using both film and counter data led to results with large standard deviations in the positional parameters. The final refinement of the diffractometer data, treating the molecules as rigid groups and allowing for anisotropic thermal motion only for the iodine atoms, led to a conventional *R* value of 0·134. The molecules lie in two independent chains parallel to **b**. In each chain there is a short O···I intermolecular contact of about 3·1 Å.

Introduction

With the recognition that short intermolecular N···X distances are found in crystalline 5-chloro- and 5-bromobenzfuroxan (see Britton, Hardgrove, Hegstrom & Nelson, 1972) and knowing that the O···I interaction in *p*-iodonitrosobenzene is strong enough that the molecules occur as loosely polymerized monomers in the crystal rather than as the dimers that are more common with nitroso compounds (Webster, 1956), we have determined the crystal structure of 5-iodobenzfuroxan (=5-iodobenzfurazan-1-oxide), primarily in order to determine the packing.

Experimental

5-Iodobenzfuroxan was prepared by J. B. Hanson and W. E. Noland by the oxidation of 4-iodo-2-nitroaniline with sodium hypochlorite in basic solution in ethanol at 0°C. The precipitate was recrystallized from an ethanol/water mixture and then sublimed to give crystals suitable for diffraction studies. The melting point of the sublimed crystals was 72 to 73°C; the infrared spectrum was very similar to that of 5-chlorobenzfuroxan; the analysis was: calculated for $IC_6H_3N_2O_2$; C, 27·50; H, 1·15; N, 10·69; found: C, 27·59; H, 1·23; N, 10·87.

The crystals were examined by Weissenberg and precession photographs and found to be monoclinic, with $a=10·413$ (17), $b=19·787$ (33), $c=7·596$ (13) Å and $\beta=103·4(1)$°. The errors are those we normally associate with the precession method. Mo $K\alpha$ radiation ($\lambda=0·7107$ Å) was used for all of the data collection, film or diffractometer. Assuming $Z=8$, we calculate a molecular volume of 190·3 Å^3, which compares well with the molecular volumes of the chloro and bromo analogs, 176·1 and 185·5 Å^3, respectively (Britton & Noland, 1962). Systematic extinctions ($h0l$, $l=2n+1$;