ently short, because they form the edges of the sodium coordination polyhedra.

Adjacent molecules related by a 2_1 screw parallel to the *a* axis are linked by a hydrogen bond between N(3) and O(4) ($\frac{1}{2}+X$, $\frac{1}{2}-Y$, $-Z$) (2·854 Å), and the molecules related by a translation along the *a* axis have a hydrogen bond between O(5′) and O(2) ($X+1$, Y, Z). These hydrogen bonds give rise to an infinite chain of nucleotide molecules parallel to the *a* axis. The nucleotide chains are stacked against the sodium coordination polyhedra (Fig. 3) as alternate channels in the structure.

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The Crystal and Molecular Structure of 5-Methylbenzfurazan-1-oxide

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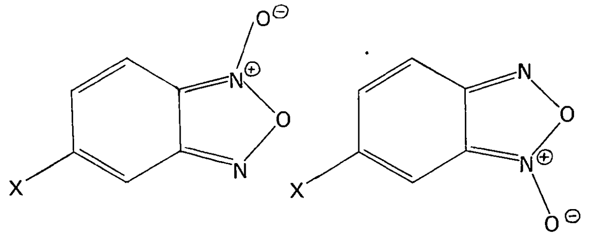
The crystal and molecular structure of 5-methylbenzfurazan-1-oxide, $C_7H_6N_2O_2$, has been determined. The compound crystallizes in monoclinic crystals with $a=4{\cdot}031$ (3), $b=15{\cdot}042$ (7), $c=11{\cdot}425$ (9) Å, $\beta=92{\cdot}82$ (7)°, in space group $P2_1/c\,(C_{2h}^5)$ with four molecules in the unit cell. Least-squares refinement of counter data gave a final conventional R factor of 0·059. Bond lengths were determined to an e.s.d. of 0·005 Å for the uncorrected values. Analysis was made of the rigid body motion, and libration corrections ranging from 0·003 to 0·009 Å were made.

Introduction

Earlier the structure of 5-chlorobenzfuroxan (=5-chlorobenzfurazan-1-oxide) was determined in this laboratory (Britton & Noland, 1962*a*, *b*). The question of interest at the time was whether this compound contained two *ortho*-nitroso groups or whether it was indeed the benzfuroxan. The structure was shown to be the benzfuroxan, but the bond lengths and angles were poorly determined, primarily because of the low melting point and high volatility of the compound. Shortly after the structure of the chlorobenzfuroxan was determined, a preliminary study (unpublished) of 5-methylbenzfuroxan was undertaken to see whether the methyl compound was the isomer similar to the chloro compound (first structure shown below), or whether it was the isomer opposite (second structure shown below).



However, this preliminary study, which did show them to be similar isomers, did not provide significantly more accurate molecular dimensions than were known for the chloro compound. We report here the completed structure determination, using counter data, for the methyl compound.

Experimental

The preparation of the compound was described earlier (Britton & Noland, 1962*b*). Crystals suitable for X-ray diffraction studies were grown by sublimation; the crystal used for the final intensity measurements was coated with a thin layer of Elmer's Glue-All to prevent sublimation in the X-ray beam. Crystals were examined by Weissenberg and precession photographs; systematic extinctions ($0k0$, $k=2n+1$; $h0l$, $l=2n+1$) indicate the space group to be $P2_1/c$ (C_{2h}^5) .The crystals grow as needles elongated along **a**; the crystal used for the intensity measurements was 0·32 × 0·32 mm in cross section and cut down to a length of 0·40 mm. Data were collected at room temperature with a 4-circle Hilger and Watts automatic diffractometer, using Zr-filtered Mo $K\alpha$ radiation ($\lambda=0{\cdot}7107$ Å). The cell dimensions are $a=4{\cdot}031(3)$, $b=15{\cdot}042(7)$, $c=11{\cdot}425(9)$ Å, $\beta=92{\cdot}82(7)^\circ$; they were obtained from the diffractometer measurements of the positions of forty reflections (ten planes, each measured in four different positions); the errors are e.s.d.'s from the least-squares refinement of the data. For four molecules per unit cell the molecular volume is 173·0 Å^3, which agrees well with the molecular volume of 5-chlorobenzfuroxan, 176 Å^3; the calculated density, 1·441(2) g.cm^{-3} agrees reasonably well with the experimental value of 1·430(5) g.cm^{-3} measured by flotation in potassium iodide solution.

A total of 1356 independent reflections were collected for θ between 0 and 26°. The distance from the source to the crystal was 28 cm, from the crystal to the aperture 20 cm; a 6·0 mm diameter circular aperture was used. The scan was one hundred 0·01° steps in θ and

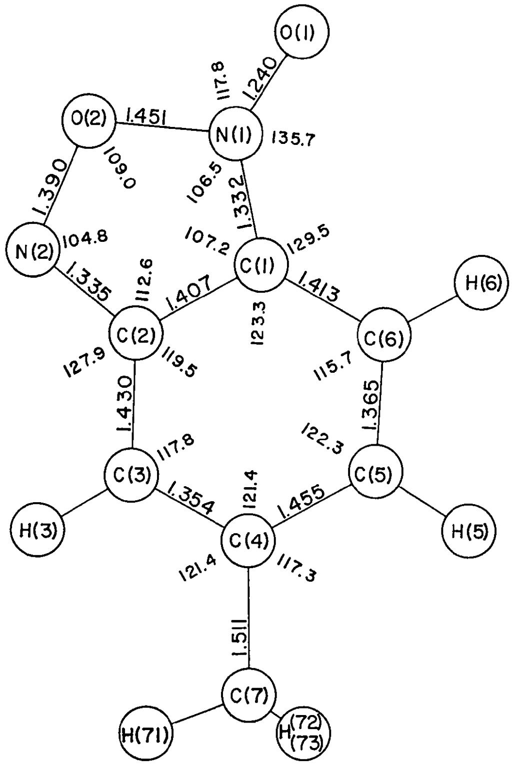


Fig. 1. Bond distances and angles, including corrections for rigid-body motions. The larger numbers are bond distances (Å); the smaller numbers are bond angles (°). Only one of the two sets of methyl hydrogen positions is shown. The other is obtained by 60° rotation about the C(4)–C(7) bond.

Table 1. *Positional and thermal parameters*

Anisotropic temperature factors are of the form $\exp[-\frac{1}{4}(B_{11}h^2a^{*2}+\ldots+2B_{12}hka^*b^*+\ldots)]$.

Estimated standard deviations for the final significant figures are given in parentheses.

The positional parameters are multiplied by 10^4.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
O(1)	6240 (8)	−299 (2)	1465 (3)	9·1 (2)	3·4 (1)	7·2 (2)	−0·9 (1)	0·7 (2)	−0·1 (1)
O(2)	4521 (7)	676 (2)	2855 (2)	6·7 (2)	5·1 (1)	5·1 (1)	−0·4 (1)	1·7 (1)	1·3 (1)
N(1)	6169 (8)	483 (2)	1792 (3)	5·4 (2)	4·1 (2)	4·9 (2)	−0·2 (1)	0·1 (2)	0·2 (1)
N(2)	4694 (9)	1582 (2)	3067 (3)	6·1 (2)	4·4 (2)	5·1 (2)	0·4 (2)	1·1 (2)	0·4 (1)
C(1)	7263 (9)	1254 (2)	1397 (3)	3·8 (2)	2·8 (2)	4·1 (2)	0·0 (1)	−0·1 (2)	0·5 (1)
C(2)	6342 (9)	1911 (2)	2185 (3)	3·8 (2)	4·0 (2)	3·4 (2)	0·3 (1)	0·6 (1)	0·4 (1)
C(3)	7192 (10)	2815 (2)	1976 (3)	4·4 (2)	3·7 (2)	4·0 (2)	0·5 (2)	0·2 (2)	−0·3 (1)
C(4)	8849 (9)	3001 (2)	1010 (3)	3·3 (2)	3·3 (2)	4·1 (2)	0·1 (1)	0·0 (1)	0·1 (1)
C(5)	9767 (10)	2306 (2)	212 (3)	4·5 (2)	4·0 (2)	3·7 (2)	0·0 (2)	0·6 (2)	0·2 (1)
C(6)	8993 (9)	1437 (2)	386 (3)	4·4 (2)	3·8 (2)	3·7 (2)	0·2 (2)	0·3 (2)	−0·3 (1)
C(7)	9788 (10)	3941 (2)	707 (4)	5·5 (2)	3·2 (2)	6·3 (2)	0·6 (2)	0·7 (2)	0·3 (2)

ω, from $-0{\cdot}50$ to $+0{\cdot}50°$ with respect to the calculated setting. The step time was two seconds for $0° < \theta \leq 20°$ and three seconds for $20° < \theta \leq 26°$; stationary background counts of 25 times the step time were taken at each end of the scan. The intensities of two check reflections were measured every 50 reflections; there was no trend with time in the intensities of these check reflections and the average deviation from the mean was about 1%.

The data were processed in the manner described by Corfield, Doedens & Ibers (1967); the value of 0·04 was used for p in the $\sigma(I)$ equation. Lorentz and polarization corrections were made; absorption corrections were not made ($\mu = 1{\cdot}2$ cm^{-1} for Mo $K\alpha$ radiation. The largest value of μr was 0·024.). The average value of σ $(F)/F$ was 0·054. The 702 reflections with $I > 2\sigma(I)$ were used in the refinement of the structure.

Solution and refinement

The y and z coordinates were originally found from film data using a $0kl$, sharpened Patterson projection.* The x coordinates were guessed from packing considerations and confirmed from $0kl$–$2kl$ film data. With this starting point and the diffractometer data the non-hydrogen atoms were refined by least-squares methods using isotropic thermal parameters to an r of 0·0618 and an R of 0·1179.† Further refinement with anisotropic thermal parameters for the non-hydrogen atoms gave r, 0·0346; R, 0·0853. A difference Fourier synthesis at this point showed eight peaks with heights between 0·3 and 0·5 e.Å^{-3}. One of these of height 0·3 e.Å^{-3} corresponded to no reasonable structural feature, but the others all could be interpreted as arising from hydrogen atoms. The ring hydrogens showed very clearly, but the methyl hydrogens appeared to be disordered. The hydrogen atoms were put in at idealized positions (tetrahedral or planar as appropriate) with C–H distances of 1·10 Å; two sets of half-hydrogen atoms, in each case with one atom in the plane of the ring, were used for the methyl hydrogens atoms. Without allowing the hydrogen parameters to vary, the remainder of the structure was further refined with anisotropic thermal parameters to r, 0·0100; $R = 0{\cdot}0590$. Since the R value (0·0590) is very near the estimated limit of the original data, futher refinement was regarded as unwarranted. The parameters resulting from this refinement are in Table 1, the hydrogen positions

Table 2. *Derived parameters for hydrogen atoms* ($\times 10^3$)

All atoms have $B = 5{\cdot}0$ Å^2.

	x	y	z
H(3)	651	335	260
H(5)	1113	246	−59
H(6)	969	91	−23
H(71)*	1112	395	−11
H(72)*	757	436	60
H(73)*	1145	422	142
H(74)*	897	441	138
H(75)*	864	413	−15
H(76)*	1253	399	67

* These atoms are half atoms to allow for the disorder in the methyl group.

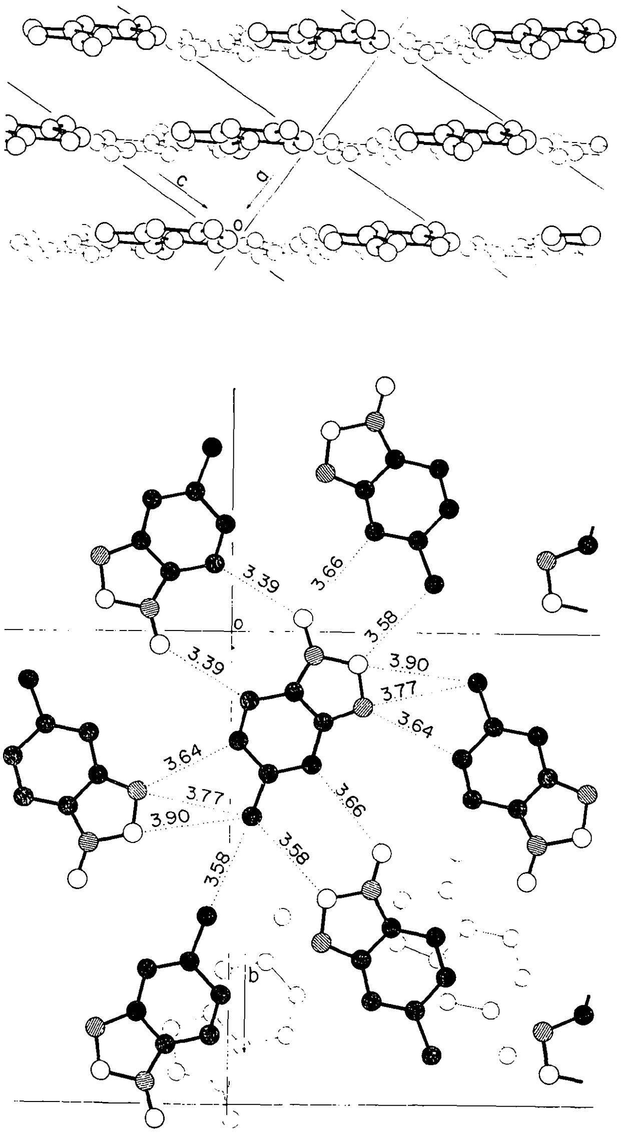


Fig. 2. The molecular packing. (*a*) Projection down **b**, (*b*) projection perpendicular to the (102) plane. Close intermolecular distances (uncorrected) in the (102) plane are shown in Å. Hydrogen atoms are omitted for clarity.

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* The programs used were *UMPREL* (Patterson maps), *UMLSTSQ* (Fourier syntheses and least squares), and *BADTEA* (distances, angles, and errors), all written by L. W. Finger, and *MGTL* (planar geometry and rigid-body motions) by Schomaker & Trueblood (1968), and local programs for the preliminary data handling. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962). No anomalous dispersion corrections were made.

† $r = \sum w(|F_o|^2 - |F_c|^2)^2 / \sum w|F_o|^4$; $R = \sum ||F_o| - |F_c|| / \sum |F_o|$. The numerator of r was the function minimized. The weights were $1/[\sigma(F^2)]^2$.

Table 3. *Observed and calculated structure factors* (× 10)

K	L	FO	FC
H=0			
0	0		3120
0	12	65	-69
0	10	61	65
0	8	113	-116
0	6	183	183
0	4	55	55
0	2	412	-422
1	11	35	-35
1	10	70	-70
1	9	88	-86
1	7	260	266
1	6	91	-91
1	4	211	-211
1	3	161	-190
1	2	547	-554
1	1	185	182
2	11	55	-59
2	10	81	-83
2	9	35	-37
2	8	30	-3
2	7	176	-176
2	6	88	86
2	4	262	-257
2	3	377	387
2	2	220	-220
2	1	524	-532
2	0	160	-155
3	12	35	34
3	11	113	111
3	10	91	96
3	9	55	-50
3	8	113	-113
3	6	160	158
3	5	37	-35
3	4	261	-253
3	3	464	-465
3	2	58	54
3	1	133	131
4	10	40	46
4	9	96	98
4	8	35	45
4	7	55	55
4	6	83	81
4	5	35	39
4	3	158	158
4	2	111	-105
4	1	105	-106
4	0	98	-95
5	13	34	39
5	12	81	-80
5	11	37	-39
5	10	74	78
5	7	58	-59
5	6	89	-86
5	5	121	121
5	4	151	151
5	3	65	64
5	2	101	103
5	1	103	103
6	13	30	-12
6	12	32	-10
6	10	35	-17
6	9	41	45
6	7	32	-27
6	5	207	-207
6	4	101	-98
6	3	232	246
6	2	113	-106
6	1	110	106
6	0	35	-39
7	12	35	-20
7	11	37	-37
7	8	73	75
7	7	30	-32
7	6	37	-37
7	5	65	-63
7	4	30	32
7	3	116	-116
7	2	187	190
7	1	181	172
8	12	30	-1
8	10	83	-83
8	9	101	105
8	8	40	37
8	7	68	-66
8	6	57	48
8	5	68	-71
8	4	63	-65
8	2	322	312
8	1	156	-151
8	0	123	-126
9	11	35	-34
9	9	45	48
9	8	83	-91
9	7	58	58
9	6	66	-66
9	5	48	45
9	4	45	-45
9	3	32	27
9	2	96	101
9	1	29	-29
10	10	34	32
10	9	88	-90
10	8	32	29
10	6	125	-128
10	4	59	-56
10	3	118	-121
10	2	66	65
10	1	185	192
10	0	80	-85
11	9	59	51
11	8	110	108
11	7	80	-81
11	6	60	-70
11	5	35	20
11	4	53	58
11	2	113	-118
11	1	178	-183
12	9	32	25
12	7	45	50
12	5	35	29
12	4	101	96
12	3	98	108
12	2	41	22
12	1	121	123
12	0	155	-160
13	8	45	45
13	5	93	-95
13	4	44	-56
13	3	197	207
13	2	106	115
14	5	35	-41
14	4	64	61
14	3	46	-41
14	1	78	74
15	7	30	-25
15	4	46	-46
15	2	55	61
16	6	37	-29
16	5	43	-41
16	2	32	-17
18	3	35	-22
18	1	44	-35
H=1			
0	-10	96	-96
0	-8	86	86
0	-6	271	-272
0	-4	320	307
0	-2	307	298
0	0	242	-257
0	2	1590	1756
0	4	261	-260
0	6	40	-35
0	8	60	66
1	-12	30	17
1	-10	48	50
1	-7	171	-171
1	-6	123	126
1	-5	362	365
1	-4	148	136
1	-3	30	35
1	-2	221	212
1	-1	362	-392
1	0	560	590
1	1	93	98
1	2	415	-415
1	3	241	243
1	4	138	-136
1	6	91	-90
1	8	30	-37
1	9	108	105
1	11	37	-37
1	12	71	-75
2	-11	68	-68
2	-9	133	133
2	-8	125	-126
2	-5	121	118
2	-4	83	86
2	-3	25	34
2	-2	96	-96
2	-1	349	-359
2	0	447	-469
2	1	630	649
2	2	90	-81
2	3	212	-222
2	4	27	-12
2	5	110	113
2	6	177	-170
2	8	45	45
2	9	121	-118
2	10	37	-30
2	11	40	-39
3	-12	48	-41
3	-10	45	-50
3	-9	151	153
3	-8	88	-93
3	-7	96	-100
3	-6	160	153
3	-5	66	-55
3	-4	156	-158
3	-3	83	86
3	-2	315	302
3	-1	293	-292
3	0	121	-126
3	1	49	-48
3	3	106	105
3	5	272	-273
3	6	50	-51
3	7	69	-68
3	8	91	90
3	10	59	-60
3	12	59	59
3	13	59	55
4	-12	30	-24
4	-11	46	50
4	-10	83	85
4	-8	68	70
4	-7	135	-126
4	-5	116	-111
4	-4	182	182
4	-3	43	-37
4	-2	105	-106
4	0	93	-91
4	1	46	50
4	2	49	-40
4	3	141	-141
4	5	111	116
4	6	71	70
4	7	50	50
4	11	50	49
5	-10	138	133
5	-9	76	-75
5	-8	103	-100
5	-6	43	-44
5	-5	66	-68
5	-4	65	68
5	-3	287	282
5	-2	98	-98
5	-1	70	60
5	0	125	-126
5	3	73	71
5	4	45	45
5	5	32	40
5	6	83	80
5	7	40	32
5	8	45	-45
5	9	37	-39
6	-9	46	39
6	-7	51	-44
6	-5	35	24
6	-3	231	223
6	-2	103	-98
6	-1	297	-291
6	0	177	-161
6	1	106	-103
6	4	30	27
6	5	121	125
6	6	51	-55
6	7	86	-88
7	-10	45	45
7	-8	37	-40
7	-7	50	-34
7	-6	138	-135
7	-5	30	-35
7	-3	116	-108
7	-1	85	-86
7	1	190	176
7	2	65	-64
7	3	51	48
7	4	148	148
7	5	61	-59
7	6	37	39
7	8	35	-34
8	-8	133	-135
8	-7	125	-123
8	-6	75	75
8	-5	81	81
8	-4	91	86
8	-3	123	125
8	-2	64	-63
8	-1	64	64
8	0	267	253
8	2	83	-81
8	3	156	-156
8	4	206	201
8	6	45	-41
8	11	55	43
9	-11	40	27
9	-9	56	-60
9	-8	48	56
9	-7	136	131
9	-6	83	81
9	-2	66	69
9	-1	35	22
9	0	60	-55
9	1	88	-90
9	2	30	30
9	3	73	69
9	4	35	32
9	5	45	27
9	8	56	-59
9	9	43	44
9	10	63	-68
10	-11	37	29
10	-8	29	27
10	-7	108	111
10	-5	55	-58
10	-4	80	-86
10	-2	60	-64
10	-1	158	153
10	0	153	151
10	1	143	-143
10	2	86	-86
10	3	106	103
10	4	50	-51
10	5	64	-70
10	8	96	-95
10	9	29	-25
10	10	48	40
10	11	41	-49
11	-7	60	56
11	-6	128	-126
11	-5	60	-65
11	-4	86	95
11	-2	30	-20
11	0	69	78
11	1	108	-111
11	2	79	-80
11	3	172	-172
11	4	98	-96
11	5	32	29
11	6	63	65
11	9	61	-65
11	10	51	53
12	-7	49	-40
12	-5	60	-65
12	-4	60	-60
12	-3	98	-98
12	-2	126	116
12	1	93	-88
12	2	131	-126
12	3	83	88
12	4	50	43
12	5	105	106
12	6	46	49
12	9	41	37
13	-9	30	11
13	-6	51	-53
13	-3	130	-131
13	-2	53	56
13	-1	237	237
13	0	118	-111
13	1	40	40
13	4	70	66
13	5	105	103
13	7	41	-43
14	-3	76	80
14	-1	44	32
14	1	79	-76
14	3	41	44
14	5	39	-35
14	6	60	61
15	-2	66	60
15	2	32	-15
15	4	65	65
15	6	40	-34
16	-4	48	-40
16	-3	40	34
16	4	37	-27
18	-1	34	29
H=2			
0	-12	48	-35
0	-10	34	35
0	-8	93	-91
0	-6	45	54
0	-4	260	-255
0	-2	247	238
0	0	227	237
0	2	63	-64
0	4	492	495
0	6	79	-74
0	12	37	35
1	-8	70	70
1	-7	60	60
1	-6	53	-48
1	-5	187	-182
1	-4	212	211
1	-3	217	217
1	-2	86	80
1	-1	55	55
1	0	88	91
1	1	230	-226
1	2	277	272
1	3	61	64
1	4	263	-262
1	5	116	116
1	8	37	-30
1	11	37	27
2	-11	35	35
2	-9	101	-103
2	-7	153	158
2	-6	85	-88
2	-1	64	70
2	0	40	43
2	1	111	-113
2	2	355	-350
2	3	335	332
2	5	76	-70
2	8	81	-75
2	11	51	-51
3	-12	43	-35
3	-10	70	-75
3	-8	53	-39
3	-7	128	136
3	-6	30	-39
3	-5	83	-88
3	-4	125	126
3	-3	83	-81
3	-2	93	-93
3	-1	83	88
3	0	86	83
3	1	39	38
3	3	126	-123
3	4	50	46
3	6	32	-35
3	7	106	-103
3	9	37	-35
3	10	37	34
4	-10	40	-37
4	-9	69	64
4	-8	138	146
4	-7	60	-56
4	-6	71	61
4	-5	91	-96
4	-4	55	-43
4	-3	98	-98
4	-2	143	138
4	0	40	-45
4	1	32	35
4	4	37	-30
4	5	90	-81
4	7	53	48
4	8	54	53
4	9	30	44
4	10	30	-27
5	-12	32	18
5	-8	138	136
5	-7	60	-63
5	-6	73	-75
5	-5	46	41
5	-4	44	-40
5	-3	51	-45
5	-1	262	267
5	2	81	-81
5	3	50	-51
6	-7	111	106
6	-5	35	-35
6	-4	65	-66
6	-2	60	-56
6	-1	78	79
6	0	35	-40
6	1	193	-192
6	2	55	-58
6	6	44	32
6	7	51	43
6	9	30	-30
6	11	32	-13
7	-8	55	54
7	-7	40	-48
7	-6	70	-75
7	-5	83	-86
7	-4	130	-128
7	-3	32	-20
7	-1	101	-101
7	2	51	53
7	3	96	98
7	4	56	-53
7	6	54	58
8	-11	53	-51
8	-6	130	-131
8	-5	60	-55
8	-4	101	101
8	-2	79	76
8	-1	61	65
8	0	43	-41
8	1	58	59
8	2	118	111
8	4	40	-20
8	5	64	-70
8	6	90	90
8	10	30	16
9	-9	49	45
9	-7	71	-68
9	-6	75	88
9	-5	167	162
9	-4	51	51
9	-2	37	-34
9	0	46	45
9	7	41	45
9	8	32	-30
10	-6	35	20
10	-3	46	50
10	3	35	40
10	4	53	55
10	6	48	-49
11	-6	32	-35
11	-5	32	24
11	-4	35	34
11	1	45	-40
11	6	41	-37
11	7	51	-50
12	-6	40	-32
12	-4	30	27
12	-3	34	-32
12	0	49	-41
12	1	80	-79
12	2	49	37
12	3	43	40
13	-4	40	35
13	-2	34	-20
13	3	78	75
14	0	43	-37
14	1	54	60
14	2	37	-10
H=4			
0	-8	34	-25
0	2	32	40
0	4	32	27
1	-8	32	25
1	-5	44	-43
1	-3	34	35
1	-1	37	-45
1	0	70	81
2	-5	59	-59
2	-3	32	35
2	4	30	1
2	6	50	-64
2	7	32	45
3	-6	65	-55
3	-5	45	43
3	-4	30	15
3	7	35	-45
4	-4	81	74
4	-3	54	-55
4	6	34	22
5	-5	60	-59
5	-4	37	34
5	-3	37	40
5	-1	43	41
5	3	45	43
5	5	35	-24
5	6	30	-32
6	-5	35	29
6	-4	35	-16
6	-3	76	83
6	-2	30	-12
6	-1	35	-41
6	0	32	-45
7	-6	35	-30
7	-3	54	-59
7	-2	63	-63
7	5	30	12
8	0	50	60
9	-5	32	27
9	-3	37	-25
9	-2	37	40
9	-1	44	40
9	3	45	-54
9	4	46	45
9	5	83	60
9	6	35	-22
10	-8	37	25
10	-5	81	93
10	-3	51	-50
10	0	63	-48
10	1	106	103
10	2	121	120
10	3	37	-29
10	4	74	-70
10	6	75	-71
10	7	35	-41
10	10	50	-44
11	-6	44	35
11	-5	40	40
11	-4	85	-83
11	-2	48	48
11	-1	40	-39
11	3	32	-41
11	4	79	-75
11	5	106	-108
11	6	37	-40
11	8	35	37
12	-8	34	-35
12	-5	45	-43
12	-4	34	27
12	-3	46	-56
12	-2	75	-68
12	-1	118	-118
12	0	76	83
12	1	37	40
12	3	32	-34
12	4	63	-60
12	5	32	39
12	6	40	35
12	7	68	53
13	-8	22	-22
13	-6	45	44
13	-4	30	-40
13	-1	45	-51
13	1	165	163
13	2	66	-65
13	3	35	30
13	4	34	-6
14	-7	32	18
14	-6	30	11
14	-2	43	-39
14	-1	81	85
14	3	60	-55
15	-2	32	-25
15	0	56	60
H=3			
0	-10	40	-37
0	-8	30	30
0	-6	60	-60
0	-2	103	-101
0	0	98	103
0	2	108	101
0	4	32	32
0	6	96	100
1	-6	55	56
1	-5	76	73
1	-4	51	-50
1	-3	108	-111
1	-2	166	162
1	-1	49	43
1	0	30	29
1	1	34	27
1	3	91	-93
1	4	88	83
1	5	49	45
1	6	108	-110
1	7	48	50
2	-9	30	30
2	-7	98	-98
2	-5	101	96
2	0	35	-16
2	2	32	32
2	4	165	-162
2	5	135	135
2	7	30	-25
2	9	32	-20
3	-8	83	-83
3	-7	43	40
3	-5	73	71
3	-2	55	54
3	-1	49	-50
3	0	51	-40
3	3	61	63
3	5	91	-86
3	6	37	39
3	10	35	22
4	-8	43	-30
4	-7	35	40
4	-6	136	136
4	-5	73	-79
4	-4	68	64
4	-3	45	-40
4	-2	49	-49
4	-1	39	-44
4	0	49	53
5	-7	60	-55
5	-6	86	83
5	-3	55	58
5	-2	32	-20
5	1	131	138
5	4	55	-51
6	-6	35	-20
6	-5	123	123
6	-3	39	-37
6	-2	70	-70
6	-1	40	22
6	0	45	-49
6	1	32	-17
6	3	91	-93
7	-9	44	-34
7	-7	37	34
7	-6	40	34
7	-5	56	-59
7	-4	85	-88
7	-3	68	-78
7	-2	68	-64
7	0	35	39
7	1	66	-65
7	4	40	37
7	5	35	37
8	-9	49	-39
8	-8	44	-29
8	-4	76	-78
8	-2	101	93
8	4	35	25
9	-8	30	-10
9	-7	41	45
9	-5	51	-50
9	-4	78	71
9	-3	110	106
9	4	39	39
9	6	34	37

Table 4. *Results of rigid-body analysis of thermal parameters*

	Eigenvalues	Eigenvectors† $(\times 10^4)$		
Librational tensor, **L**	33·2 $(^\circ)^2$	−3360	−8551	3949
	15·1	−4492	5139	7339
	10·3	−8309	716	−5517
Translational tensor, **T**	0·0523 Å^2	7130	1822	−6771
		1271	9153	3822
		6900	−3533	6318
Symmetrized‡ screw tensor, **S**	$\begin{pmatrix} 46 & -118 & -2 \\ & -84 & -3 \\ & & 39 \end{pmatrix} \times 10^5$ rad. Å.			

† Direction cosines of the eigenvectors are relative to **a**, **b** and **c***. **L** is independent of the origin, **T** and **S** depend upon it and are given relative to the unique origin that symmetrizes **S**. This unique origin is about 0·90 Å from the centroid of the molecule, displaced along the three crystal axes by 0·35, 0·73 and −0·39 Å.

‡ **S** is expressed relative to the principal axes of **L** and to the unique origin given above.

in Table 2, and the observed and calculated structure factors in Table 3.*

A rigid body analysis was made after the method of Schomaker & Trueblood (1968). The rigid body tensors derived from the temperature factors are given in Table 4; the r.m.s. difference between observed and calculated B_{ij} is 0·25 Å^2.

Results

The bond distances and angles are given in Table 5, both uncorrected and corrected for libration. The corrected values are also shown in Fig. 1. Inspection of the bond distances suggests that of the following resonance forms that might be drawn for this molecule

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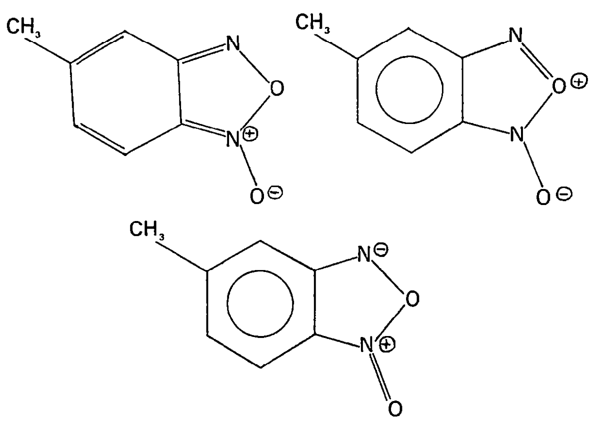
* In the last cycle of refinement no calculated shift was as large as 20% of the e.s.d. of the corresponding parameter. The standard deviation of an observation of unit weight was 1·36. Of the 654 reflections with $I \le 2\sigma(I)$, none have $F_c > 5\sigma(F)$, nine have $4\sigma(F) \le F_c < 5\sigma(F)$, and 31 have $3\sigma(F) \le F_c < 4\sigma(F)$.



the first makes the largest contribution. The C(3)–C(4) and C(5)–C(6) distances[*] are distincly shorter than the rest of the benzene ring distances and are only slightly larger than the C–C double bond distance. The two C–N distances are about halfway between single and double bond distances. On the other hand the inequality of the two ring N–O distances suggests an appreciable contribution from the second resonance form, and the N(1)–O(1) distance of 1·240 Å, when compared to the N–O distance in pyridine 1-oxide of 1·35 Å (Ulku, Huddle & Morrow, 1971) suggests an appreciable contribution from the third structure. The structure of benzotrifuroxan has also been determined accurately (Cady, Larson & Cromer, 1966; Maslen, 1968). The average dimensions in the furoxan ring in benzotrifuroxan are quite similar to those in our compound.

Table 5. *Bond distances and bond angles*

E.s.d.'s for the uncorrected values are given in parentheses. Since the hydrogen parameters were assumed, as discussed in the text, the distances and angles involving hydrogen are omitted from this table.

	Uncorrected	Corrected
N(1)–O(1)	1·235 (4) Å	1·240 Å
N(1)–O(2)	1·443 (4)	1·451
N(2)–O(2)	1·385 (4)	1·390
C(1)–N(1)	1·327 (5)	1·332
C(2)–N(2)	1·330 (5)	1·335
C(1)–C(2)	1·399 (5)	1·407
C(2)–C(3)	1·425 (5)	1·430
C(3)–C(4)	1·348 (5)	1·354
C(4)–C(5)	1·446 (5)	1·455
C(5)–C(6)	1·360 (5)	1·365
C(6)–C(1)	1·406 (5)	1·413
C(4)–C(7)	1·508 (5)	1·511
N(1)–O(2)–N(2)	108·9 (3)°	109·0°
O(1)–N(1)–O(2)	117·7 (3)	117·8
O(1)–N(1)–C(1)	135·8 (3)	135·7
O(2)–N(1)–C(1)	106·5 (3)	106·5
O(2)–N(2)–C(2)	104·9 (3)	104·8
N(1)–C(1)–C(2)	107·2 (3)	107·2
N(1)–C(1)–C(6)	129·6 (3)	129·5
C(2)–C(1)–C(6)	123·2 (3)	123·3
N(2)–C(2)–C(1)	112·5 (3)	112·6
N(2)–C(2)–C(3)	128·0 (3)	127·9
C(1)–C(2)–C(3)	119·6 (3)	119·5
C(2)–C(3)–C(4)	117·9 (3)	117·8
C(3)–C(4)–C(5)	121·3 (3)	121·4
C(3)–C(4)–C(7)	121·5 (3)	121·4
C(5)–C(4)–C(7)	117·3 (3)	117·3
C(4)–C(5)–C(6)	122·3 (3)	122·3
C(5)–C(6)–C(1)	115·8 (3)	115·7

The planarity of the molecule was tested by fitting least-squares planes through the benzene ring atoms and through all the non-hydrogen atoms. The results of the first calculation are given in Table 6. The atoms O(1), N(2), and C(7) are significantly out of the plane. The non-planarity of C(7), in particular, is surprising, but it does appear to be real. The least-squares plane has the equation $0·8426X - 0·1255Y + 0·5237Z - 0·0082 = 0$, where X, Y, and Z are coordinates (in Å) measured parallel to **a**, **b**, and **c***, respectively.

Table 6. *Planarity of the molecule*

The deviations are from the plane defined by the atoms C(1) to C(6).

	Deviation		Deviation
C(1)	0·002 Å	O(1)	−0·015 Å
C(2)	−0·002	O(2)	−0·007
C(3)	0·002	N(1)	−0·008
C(4)	−0·002	N(2)	−0·014
C(5)	0·002	C(7)	−0·027
C(6)	−0·002		

The crystal structure is shown in Fig. 2. Rather surprisingly, the molecules form sheets, which associate in parallel layers. The average distance between layers is 3·22 Å; the two shortest interatomic distances between layers are O(1)–C(6), 3·38 Å, and O(2)–C(1), 3·41 Å. These are uncorrected distances. The short intermolecular distances within a layer are shown in Fig. 2. Except for the $CH_3 \cdots CH_3$ distance of 3·58 Å, none of these distances is shorter than the expected van der Waals distance. It is not clear why the planar layers should form. The orientations of the molecules are such that there is a strong temptation to say that X···H–C (where X=O or N) hydrogen bonding is involved with the ring hydrogens in particular. However the distances O(1)–C(6), 3·39 Å; O(1)–C(3), 3·66 Å; N(2)–C(5), 3·64 Å are not particularly short and the corresponding X···H distances of 2·40, 2·57 and 2·59 Å are all too long to be considered hydrogen bonds according to the arguments of Donohue (1968).

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[*] The labels given to atoms correspond to those in Fig. 1 and not to the conventional numbering of the ring system in this compound, which would be C(4)–C(5) and C(6)–C(7) for the bonds referred to here.

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The Crystal Packing of 5-Bromo- and 5-Chlorobenzfurazan-1-oxide

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The packing of 5-bromobenzfurazan-1-oxide and 5-chlorobenzfurazan-1-oxide has been studied by refining single-crystal, film X-ray diffraction data treating the molecules as groups of known structure. Short intermolecular contacts, N···Br, 3·14 Å, and N···Cl, 3·20 Å, were found.

Introduction

Several years ago three of us (GLH, RH, and GVN) undertook the structure determination of 5-bromobenzfurazan-1-oxide to determine the configuration of the benzfuroxan ring. With the publication of the structure of 5-chlorobenzfurazan-1-oxide (Britton & Noland, 1962) the determination was set aside. After the determination of the structure of 5-methylbenzfurazan-1-oxide [see the preceding article (Britton & Noland, 1972)] the structure of the chloro compound was reexamined to see whether there was any obvious reason why the packing in the chloro and methyl compounds was different. A previously overlooked short intermolecular N···Cl distance was found; this is taken to indicate a weak acid-base interaction and presumably explains the difference in packing. It was then decided to continue with the determination of the structure of the bromo compound to a point where the intermolecular distances could be compared.

Experimental

The unit-cell dimensions and space group for 5-bromobenzfurazan-1-oxide were reported earlier (Britton & Noland, 1962). The results here are in agreement with those, $a=12{\cdot}67$ (2), $b=7{\cdot}35$ (1), $c=7{\cdot}79$ (1)Å, $\beta=90{\cdot}0$ (1)° and space group $P2_1/n\,(C^5_{2h})$. Crystals suitable for intensity measurements were grown from an ethanol solution. A crystal was cleaved to a roughly cubic shape and wedged in a thin-walled glass capillary tube, which was then filled with water and sealed. Weissenberg photographs of layers *hk*0–*hk*3 were collected and were measured by visual comparison with an intensity standard. There were 262 independent reflections of measurable intensity; these were corrected for Lorentz and polarization factors but not for absorption.*

In view of the limited amount of data and the lack of absorption corrections (the crystal dimensions had not been recorded when the data were collected in 1962) it was decided to carry out the refinement treating the entire molecule as a rigid group of known dimensions. The dimensions for the molecule were taken from those of the corresponding methyl compound (see the preceding paper; values before libration corrections were used) except that the methyl group with the carbon atom 1·508 Å from the ring was replaced with a bromine atom 1·870 Å from the ring, and the molecule was made exactly planar. The initial position and orientation of the molecule were chosen to correspond to the known structure of the chloro compound. Scale factors for the individual layers were treated as independent variables. Convergence was reached after four cycles of refinement with $r=0{\cdot}185$ and $R=0{\cdot}200$. The

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* The programs used were the same as those in the preceding paper (Britton & Noland, 1972), plus *GROUP*, a local modification of a group refinement program by R. J. Doedens based on *ORFLS*. R and r are defined as they were previously with the weights assigned according to: $w=(F_w/F_o)^4$ for $F_o>F_w$ and $w=1$ for $F_o\le F_w$. F_w was 10·0 for the bromo compound and 8·0 for the chloro compound for the F_o on an absolute scale.