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The Crystal and Molecular Structure of the 2-Oxide of 4-Methyl-3-(p-bromophenyl)-1,2,5-oxadiazole

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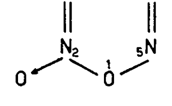
The 2-oxide of 4-methyl-3-(p-bromophenyl)-1,2,5-oxadiazole, BrC_6H_4–$(C_2N_2O_2)$–CH_3, is the isomer of lower melting point (88–89 °C), obtained by oxidation of methyl(p-bromophenyl)glyoxime. It crystallizes in the monoclinic system, space group $P2_1/n$, with four molecules in a cell having: $a_0 = 12{\cdot}720$, $b_0 = 10{\cdot}362$, $c_0 = 7{\cdot}415$ Å; $\beta = 97°54'$. A crystal structure analysis has been made, based on all the X-ray reflexions accessible to Cu $K\alpha$ radiation, measured with an automatic diffractometer. The molecule has the N-oxide structure (furoxan) and it is only approximately planar. A contact occurs between the bromine atoms and the extranuclear oxygen atoms of neighbouring molecules and this may explain the departure from strict planarity. An intramolecular contact takes place between the extranuclear oxygen atom of the furoxan group and a carbon atom of the phenyl ring; it may explain the chemical behaviour of this compound as compared with that of the higher melting isomer.

Following Wieland & Semper (1908), the term 'furoxan' is now generally accepted for those compounds which can be considered as oxidation products of α-dioximes and which are characterized by a $-C_2N_2O_2-$ nucleus. With the aryl-alkyl furoxans in particular, the existence of isomers with different chemical behaviour led to speculations about their structural formulae (*e.g.* Kaufman & Picard, 1959).

Recent neutron magnetic resonance and kinetics studies (Mallory & Cammarata, 1966) favour a positional isomerism of the following type.



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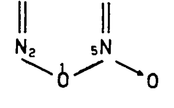

II

In order to solve this problem, we have undertaken the X-ray crystal structure analysis of the pair of isomers obtained from the oxidation of methyl-(*p*-bromophenyl)glyoxime (Ponzio, 1928*a*, *b*), having empirical formula $BrC_6H_4-(C_2N_2O_2)-CH_3$. As described in a preliminary communication (Calleri, Ferraris & Viterbo, 1967) we found that the isomerism is of type I,II.

In the present paper we report the crystal and molecular structure of the lower melting isomer, LMI, while the structure of the higher melting isomer, HMI, is the subject of the following paper.

Experimental

Crystal data

The compound prepared by Ponzio (1928*a*, *b*), was recrystallized from an aqueous-alcoholic solution as colourless prisms elongated in one direction, chosen as the *c* axis. The following crystal data were obtained: $C_9H_7BrN_2O_2$, $M=255{\cdot}06$, m.p.88–89 °C; monoclinic prismatic, $a_0=12{\cdot}7204\pm0{\cdot}0034$, $b_0=10{\cdot}3626\pm0{\cdot}0012$, $c_0=7{\cdot}4156\pm0{\cdot}0021$ Å, $\beta=97°54'\pm2'$; $V=986{\cdot}22$ Å^3. $D_m=1{\cdot}749$ g.cm^{-3} (by flotation), $Z=4$, $D_c=1{\cdot}750$ g.cm^{-3}, $F(000)=504$ e; space group $P2_1/n$ (C_{2h}^5); Cu $K\alpha$ radiation; preliminary single-crystal rotation and Weissenberg photographs and subsequent single crystal diffractometry; linear absorption coefficient $\mu=62{\cdot}0$ cm^{-1}.

The refinement of reciprocal unit-cell parameters has been done by a least-squares procedure using an adequate number of high θ values measured on a single-crystal diffractometer with Cu $K\alpha$ radiation ($\lambda\alpha_1=1{\cdot}54050$, $\lambda\alpha_2=1{\cdot}54434$ Å).

The forms exhibited by LMI crystals are, according to the above reported orientation of axes used throughout this paper: $m=\{110\}$, $n=\{320\}$, $a=\{100\}$, $c=\{\bar{1}01\}$ and $o=(\bar{1}11\}$ (Fig. 1). The predominant form is $\{110\}$ and there is distinct cleavage parallel to $\{\bar{1}01\}$.

To choose axes in accordance with space group $P2_1/c$ it would be sufficient to apply the transformation matrix $001/010/\bar{1}0\bar{1}$ to the description above.

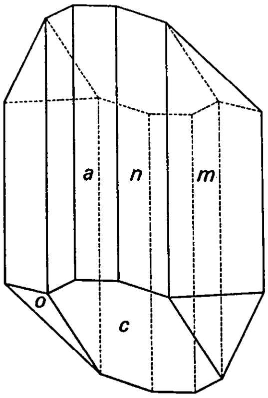

Fig. 1. Clinographic projection of a lower melting isomer crystal with all the forms found.

Intensity measurement

The intensities were measured with a General Electric automatic three-circle diffractometer, equipped with a Na(Tl)I scintillation counter (Cu $K\alpha$ radiation), employing the θ–2θ scanning method of integration with a scanning speed of 1° per minute; background was measured for a 0·5° interval on both sides of each peak. The crystal selected for the measurements (about 2·1 mm long and with a cross-section of about $0{\cdot}21\times0{\cdot}23$ mm^2) was mounted with its *c* axis parallel to the φ axis of the goniostat. The number of measured reflexions was 1914 (~97% of those accessible to Cu $K\alpha$ radiation). About 500 reflexions had a relative intensity, after subtraction of the background, of only 15–20 counts against the maximum value of 156000 counts for reflexion $\bar{2}02$; these latter reflexions, of low reproducibility, have been treated as 'unobserved', in fact they do not appear even on very long-exposure Weissenberg photographs.

The usual corrections were applied.

Determination and refinement of the structure

After determination of the coordinates of bromine atoms from projections of the Patterson function along [001] and [100], two successive three-dimensional electron-density syntheses indicated the positions of all the atoms (except the hydrogen atoms). The molecular configuration was clearly of the furazan *N*-oxide type, in fact a 2-oxide. The coordinates of the phenyl hydrogen atoms were obtained from a three-dimensional difference synthesis, computed at an advanced stage of the refinement. It was not possible to locate the hydrogen atoms of the methyl group since only a diffuse positive zone appears, in the difference synthesis, in the neighbourhood of the methyl carbon. The coordinates obtained from the second electron-density synthesis have been progressively refined by the least-squares method, using the full-matrix program of Busing, Martin & Levy (1962), with minor modifications. Initially only the scaling factor and the coordinates were refined with an overall temperature factor, tentatively assigned, B = 3.5 Å^2. The R value remained rather high (~0·32) until individual isotropic temperature factors were introduced, when it fell to ~0·15 and the vibrational parameters showed a rather marked differentiation. In these first cycles all the observed reflexions were given unit weight; unobserved reflexions were given zero weight throughout the refinement and they are not reported in Table 1 of amplitudes, except when $|F_c|\gg|F_o|$, in which case they are marked with an asterisk.

For later cycles the following weighting scheme was introduced:

$$w_{hkl}=A/[(3/|F_{\max}|)F_o^2+|F_o|+2|F_{\min}|] \qquad \text{if } |F_o|\ge2|F_{\min}|, \qquad (1)$$

$$w_{hkl}=aF_o^2 \qquad \text{if } |F_o|<2|F_{\min}|. \qquad (2)$$

Constants A and a were assigned such values as to have $0 < w_{hkl} \leq 1$ and to make the two weighting systems coincident when $|F_o| = 2|F_{\min}|$. $|F_{\max}|$ and $|F_{\min}|$ were chosen in such a way as to make the values of $w\Delta^2$ as constant as possible. The refinement was continued simultaneously varying the scaling factor and positional and anisotropic vibrational parameters (127 variables); the R value was reduced to 0·057. The coordinates of the four phenyl hydrogen atoms were introduced and refined at this stage, with a constant temperature factor $B = 4{\cdot}0$ Å^2 (139 variables), leading to $R = 0{\cdot}053$ (weighted $R = 0{\cdot}063$). During the last cycles a few reflexions (marked with an asterisk in Table 1) were assigned zero weight; these showed marked $|F_o - F_c|$ discrepancies and therefore they are probably affected by casual measurement errors. The final R value for all the reflexions reported in Table 1 is 0·073.

The computation of atomic scattering factors was done by linearly interpolating the values for neutral atoms recommended in *International Tables for X-ray Crystallography* (1962). The real part of the correction for the anomalous cattering of bromine ($\Delta f' = -1{\cdot}0$) was introduced. No absorption correction was used.

The final coordinates and vibrational anisotropic parameters are listed in Table 2, with their estimated standard deviations; the latter are the B_{ij} coefficients of the function:

$$\exp\left(-\tfrac{1}{4}\sum_{i=1}^{3}\sum_{j=1}^{3} B_{ij}h_ih_ja_i^*a_j^*\right). \qquad (3)$$

Table 1. *Observed structure amplitudes and calculated structure factors* (*e*)

H	K	L	FO	FC
0	0	2	10.1	9.3
0	0	4	29.9	-27.3
0	0	6	27.0	-24.6
0	0	8	11.9	-10.8
0	1	1	46.7	49.2
0	1	2	171.5	-188.6
0	1	3	16.9	16.8
0	1	4	48.3	-45.6
0	1	5	16.9	-15.0
0	1	6*	8.2	-11.7
0	1	7	11.2	-11.3
0	2	0	69.6	-79.9
0	2	1*	11.6	-15.2
0	2	2	60.0	-57.8
0	2	3	66.1	-66.7
0	2	4	29.3	26.3
0	2	5	31.2	-30.1
0	2	8	4.3	4.9
0	3	1	64.5	-61.8
0	3	2	22.1	-21.4
0	3	3	25.8	-25.2
0	3	4	11.6	-10.9
0	3	5	23.3	23.1
0	3	6	3.1	-2.9
0	3	7	13.0	14.0
0	4	0	84.3	-94.6
0	4	1	7.2	-6.7
0	4	2	43.8	-42.5
0	4	3	37.7	36.0
0	4	4	18.2	16.9
0	4	5	20.3	19.0
0	4	6	12.3	13.1
0	4	8*	6.3	8.8
0	5	1	10.6	-10.0
0	5	2	63.9	70.4
0	5	3	13.9	14.5
0	5	4	42.0	40.5
0	5	5	4.0	-3.5
0	5	6	10.8	11.5
0	5	7	3.8	-4.0
0	6	0	71.3	76.0
0	6	1	19.0	17.9
0	6	2	34.1	34.9
0	6	3	30.0	27.2
0	6	4	10.2	-9.9
0	6	6	9.8	-10.6
0	6	7*	4.2	2.5
0	6	8*	6.0	-8.0
0	7	1	47.4	46.3
0	7	2	30.6	-32.2
0	7	4	8.8	-10.1
0	7	5	10.4	-9.7
0	7	6	7.2	-6.6
0	7	7	4.3	-4.3
0	8	0	25.4	-25.0
0	8	1	34.5	-33.7
0	8	2	9.9	9.5
0	8	3	26.4	-25.3
0	8	5	12.9	-15.4
0	8	6	3.1	-3.0
0	9	1	27.2	-30.0
0	9	2*	5.2	-2.9
0	9	3	5.9	-5.6
0	9	4	14.0	-13.6
0	9	5	6.7	7.7
0	9	6	4.8	-3.9
0	10	0	23.4	-22.3
0	10	1	4.1	-3.3
0	10	2	10.5	-10.3
0	10	3	18.1	17.4
0	10	5	3.4	3.2
0	10	6	6.4	7.1
0	11	2	15.1	14.3
0	11	4	9.3	8.8
0	12	0	12.1	11.3
0	12	1	5.5	3.9
0	12	2	4.8	5.5
0	12	3	7.0	7.1
0	13	1	10.4	11.0
1	0	1	33.1	-30.8
-1	0	1	27.9	-28.8
1	0	3	44.4	-40.6
-1	0	3	4.8	3.4
1	0	5	15.7	-15.6
-1	0	5	29.1	28.0
1	0	7	4.7	5.5
-1	0	7	17.3	18.0
1	0	9	5.8	7.5
1	1	0	23.3	30.1
1	1	1*	14.9	-8.6
-1	1	1	25.3	26.1
1	1	2	79.7	-75.7
-1	1	2	75.5	-85.4
-1	1	3	31.6	29.4
1	1	4	25.0	-24.3
-1	1	4	60.5	56.4
1	1	5	29.4	26.6
-1	1	5	16.1	15.8
-1	1	6	18.1	18.4
1	1	7	12.7	13.7
-1	1	7	8.5	-7.4
1	1	8*	4.9	6.5
-1	1	8	3.3	3.9
-1	1	9	8.1	-7.2
1	2	0	48.2	-54.8
-1	2	1	4.0	5.1
1	2	2	25.5	-24.5
-1	2	2	94.6	91.2
1	2	3	19.7	19.8
1	2	4	34.6	31.5
-1	2	4*	33.0	65.9
1	2	5	12.1	11.7
-1	2	5	6.4	-7.3
1	2	6	31.0	32.7
-1	2	6	15.0	-14.1
1	2	7	7.7	-7.4
-1	2	7	9.8	-9.1
1	2	8*	3.7	6.7
-1	2	8	11.6	-12.3
1	3	0	73.8	80.8
1	3	1	7.9	-7.4
-1	3	1	11.7	12.2
1	3	2	72.7	74.0
-1	3	2	5.6	-5.7
1	3	3	10.5	10.2
-1	3	3	18.4	19.5
1	3	4	60.6	57.5
-1	3	4	46.9	-47.5
1	3	6*	6.3	-0.9
-1	3	6	39.1	-40.2
1	3	8*	4.1	-8.0
1	4	0	56.9	60.1
1	4	1	29.7	29.7
-1	4	1	17.5	17.9
1	4	2	29.7	27.0
-1	4	2	70.8	-71.4
1	4	3	35.0	34.5
-1	4	3	13.2	-12.9
1	4	4	30.1	-27.5
-1	4	4	19.0	-24.8
1	4	5	9.2	8.2
-1	4	5	25.2	-25.1
1	4	6	17.2	-18.7
-1	4	6*	1.0	6.2
1	4	7	3.9	-3.0
-1	4	7	9.1	-8.6
1	4	8*	3.4	-5.4
-1	4	8	8.1	9.0
1	5	1	27.6	28.6
-1	5	1	51.4	53.6
1	5	2	29.4	-28.3
-1	5	2	10.8	9.5
-1	5	3	37.2	-36.4
1	5	4	11.6	-11.2
-1	5	4	13.4	13.5
1	5	5	28.1	-27.9
-1	5	5	10.9	-10.6
1	5	6	4.3	-3.9
-1	5	6*	3.6	6.1
1	5	7	9.9	-10.6
-1	5	7	11.7	11.4
1	6	0	5.9	5.4
1	6	1	40.1	-39.1
-1	6	1	5.0	-4.4
1	6	2	6.0	-5.2
-1	6	2	20.8	-22.3
1	6	3	37.7	-39.5
-1	6	3	14.8	15.4
1	6	4	11.2	-11.4
1	6	5	13.9	-13.5
-1	6	5	22.0	25.0
1	6	6	9.5	-10.0
1	6	7	4.8	5.5
-1	6	7*	4.0	9.6
1	7	0	33.7	-35.6
1	7	1	24.9	-24.9
-1	7	1	15.0	15.3
1	7	2	31.1	-31.7
-1	7	2	13.1	12.7
-1	7	3	21.2	22.5
1	7	4	22.6	-21.3
-1	7	4	15.0	15.2
1	7	5	9.2	9.3
1	7	6	4.0	3.6
-1	7	6	15.2	15.1
1	7	7	5.6	6.2
1	8	0	30.3	-30.4
1	8	2	6.3	-6.8
-1	8	2	30.6	32.0
1	8	4	18.5	17.8
-1	8	4	14.3	15.2
-1	8	5*	4.6	2.7
1	8	6	13.5	14.7
1	9	0	3.0	3.2
1	9	1	24.6	-24.1
1	9	2	22.5	22.0
-1	9	2	5.5	6.6
1	9	3	10.3	8.1
-1	9	3	21.5	20.6
1	9	4	16.3	15.8
-1	9	4	12.2	-13.2
1	9	5	8.6	7.8
-1	9	5	2.4	-2.3
-1	9	6*	6.1	-10.0
1	10	1	18.4	16.7
1	10	2	9.4	9.6
1	10	3	22.8	22.1
-1	10	3	6.2	-5.5
-1	10	5	13.4	-14.6
1	10	6	5.0	-5.4
1	11	0	7.9	7.8
1	11	1	9.5	9.9
-1	11	1	19.4	-20.0
1	11	2	10.3	9.1
-1	11	2	3.6	-4.3
-1	11	3	10.7	-10.5
-1	11	4*	13.0	-6.0
1	11	5*	5.5	-9.3
-1	11	5	3.7	-4.6
1	12	0	9.1	8.7
1	12	1	8.8	-7.9
-1	12	1	3.6	-3.1
-1	12	2	11.6	-11.8
1	12	3	10.2	-9.1
-1	12	3	3.4	4.3
1	13	0	9.2	-9.0
2	0	0*	16.2	10.6
2	0	2	55.2	53.3
-2	0	2	202.1	-233.9
2	0	4	66.9	66.0
-2	0	4	42.8	-45.9
2	0	6	29.4	25.9
2	0	8	3.7	3.5
2	1	0*	99.9	154.3
2	1	1	11.8	-13.0
-2	1	1	61.3	-55.7
2	1	2	83.7	80.9
-2	1	2	77.0	-69.3
2	1	3	35.0	33.1
-2	1	3	19.8	-20.6
2	1	4	13.9	12.7
-2	1	4	61.0	64.0
2	1	5	27.6	26.3
-2	1	5	11.1	-10.7
2	1	6	11.2	-10.5
-2	1	6	22.2	22.9
2	1	8*	7.2	-10.7
2	2	0	10.7	10.2
2	2	1	94.0	109.5
-2	2	1	26.4	-27.9
2	2	2	25.0	-23.4
-2	2	2	51.9	49.6
2	2	3	38.8	36.9
-2	2	3	18.9	17.2
2	2	4	12.4	-13.7
-2	2	4	52.0	50.3
2	2	5	13.7	-13.0
-2	2	5	16.3	16.4
2	2	6	11.0	-11.7
-2	2	6	10.4	-10.2
2	2	7	13.1	-14.1
-2	2	7	15.2	14.5
2	3	0	9.3	9.3
2	3	1	16.2	-13.8
-2	3	1	88.6	89.1
2	3	2	40.6	40.1
-2	3	2	3.3	4.5
2	3	3	65.5	-64.5
-2	3	3	37.1	33.0
2	3	4	12.5	-12.2
2	3	5	27.6	-26.1
-2	3	5	21.0	20.0
2	3	6	6.8	-7.5
2	3	7	6.6	-7.0
-2	3	8*	3.4	5.2
2	4	0	30.2	32.1
2	4	1	87.8	-83.8
-2	4	1	2 .9	21.4
2	4	2	49.5	-47.2
-2	4	2	51.1	50.2
2	4	4	37.0	-38.6
-2	4	4	25.4	31.2
2	4	5	8.6	9.2
-2	4	3	10.6	-10.0
2	4	6	12.1	-12.9
2	4	7	7.3	8.5
-2	4	7	10.9	-9.4
2	4	8	2.3	2.2
2	5	0	84.1	-93.8
2	5	1	5.7	5.4
-2	5	1	7.1	-6.2
2	5	2	41.7	-45.5
-2	5	2	31.6	31.6
2	5	3	24.0	22.9
2	5	4	7.4	-7.5
-2	5	4	19.6	-19.1
-2	5	5	4.8	-5.5
2	5	6	14.0	15.9
-2	5	6	24.1	-21.6
2	5	8*	6.2	9.8
2	6	1	25.4	-25.3
-2	6	1	15.7	16.8
2	6	2	24.3	26.2
-2	6	2	53.2	-57.8
2	6	4	33.6	32.1
-2	6	4	37.7	-38.1
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11	6	4*	2.9	4.9
11	7	0	8.9	9.6
11	7	1	6.4	5.5
11	7	2	9.2	8.9
-11	7	4	5.3	-5.3
11	8	0	9.1	8.8
11	8	1	3.1	-2.8
-11	8	1	5.3	5.1
-11	8	2	10.3	-10.1
-11	8	3	4.1	4.5
11	9	0	4.6	-4.6
11	9	1	3.8	-3.3
-11	9	2	2.4	2.2
12	0	0	6.5	6.6
12	0	2	9.4	-10.8
12	0	4	5.3	-6.3
-12	0	4	8.2	7.8
12	1	0	8.7	-8.8
12	1	1	7.4	6.0
-12	1	1	4.1	3.3
12	1	2	8.0	-6.8
-12	1	2	4.8	4.8
12	1	3	7.3	-8.0
-12	1	3	6.6	6.1
12	1	5	3.7	-3.7
-12	1	5	3.7	3.2
12	2	1	16.0	-14.3
-12	2	1	5.2	5.8
-12	2	2	6.1	-5.5
-12	2	3	10.1	-10.3
12	2	4	4.5	3.4
-12	3	1	14.1	-13.4
12	3	5	8.0	8.9
-12	3	3	6.7	-7.6
-12	3	5	9.5	-8.4
12	4	1	13.4	12.4
-12	4	1	7.0	-6.7
12	4	2	4.4	4.6
-12	4	2	2.2	-2.4
12	4	3	3.0	2.7
12	4	4	3.7	4.6
12	5	0	9.7	10.0
12	5	1	4.0	3.4
12	5	2	7.8	7.4
-12	5	3	8.1	6.5
12	6	0	3.9	3.4
-12	6	1	7.3	-7.5
12	6	2	3.3	-3.4
-12	6	2	6.4	5.9
12	6	3	3.4	-3.8
12	7	0	3.0	-3.9
12	7	1	10.4	-9.5
-12	7	1	3.9	2.4
12	7	2	6.6	-7.0
-12	7	3	6.3	6.5
12	8	0	5.4	-6.3
-12	8	1	7.9	7.7
-12	8	2	2.4	-2.5
13	0	1	3.4	-2.8
-13	0	1	26.6	-25.4
-13	0	3	6.2	-6.6
13	1	1	10.9	11.2
-13	1	2	10.9	-10.5
13	1	3	6.2	5.6
-13	1	3	13.1	13.6
13	2	0	7.5	8.2
13	2	1	7.8	6.1
-13	2	1	3.2	3.6
-13	2	4	7.7	6.5
13	3	0	8.1	8.0
13	3	1	9.5	8.6
-13	3	1	5.3	-4.6
-13	3	2	8.9	9.2
-13	3	4	8.0	7.5
13	4	0	5.3	-4.4
-13	4	1	9.1	9.6
13	4	2	3.5	-3.6
-13	4	3	9.8	10.7
-13	4	4	6.7	-7.3
13	5	1	5.5	-4.8
13	5	3	8.4	-8.1
-13	5	5	6.1	-5.6
13	6	0	3.1	-3.5
13	6	1	6.8	-6.0
-13	6	1	5.2	-5.7
13	6	2	4.4	-2.9
13	7	0	4.9	-5.7
-13	7	2	4.0	-4.4
14	0	0	6.8	7.9
-14	0	2	2.9	2.9
14	1	1	3.9	3.9
-14	1	2	6.1	-4.7
-14	1	3	4.4	4.4
14	2	1	4.3	2.7
-14	2	1	2.5	-2.3
-14	2	3	5.5	-4.9
14	3	0	3.4	-4.3
14	3	1	6.9	-6.9
-14	3	1	3.9	-3.8
14	3	3	2.9	-2.7
14	4	0	5.6	-5.2
-14	4	1	9.4	9.1
14	5	0	4.5	-2.8
14	5	2	5.7	4.8
-14	5	2	4.9	3.8
14	6	0	4.5	4.0
15	0	1	3.6	-3.6
-15	0	1*	4.9	2.0
15	1	1	6.7	-5.5
-15	1	1	6.2	6.4
15	2	0	5.9	-6.0
-15	2	1	2.3	-2.3
-15	3	1	3.2	3.6
16	0	0	2.8	-3.0
16	1	0	3.8	3.6

Table 2. *Final fractional coordinates and vibrational parameters* (Å²) *with below, the significant figures of the standard deviations*

	x/a_0	y/b_0	z/c_0	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C(1)	0·4232	0·3110	0·1539	4·98	3·31	4·89	−0·52	0·18	0·37
	4	4	8	25	19	24	16	20	16
C(2)	0·5311	0·3113	0·1999	4·62	4·18	6·93	0·61	0·35	−0·88
	5	5	9	25	22	33	18	24	20
C(3)	0·5813	0·4248	0·2587	3·15	4·29	7·46	0·01	0·33	−0·14
	4	5	9	20	21	35	16	22	20
C(4)	0·5258	0·5386	0·2734	3·40	3·84	4·05	−0·46	0·36	0·69
	4	4	7	20	18	20	14	16	16
C(5)	0·4164	0·5342	0·2251	3·78	3·79	8·94	0·33	0·69	0·61
	4	5	10	23	20	39	18	24	24
C(6)	0·3645	0·4214	0·1671	3·84	4·70	7·65	−0·92	0·58	0·13
	5	5	10	23	24	36	18	22	24
C(7)	0·5784	0·6594	0·3394	3·63	3·68	4·75	−0·24	0·48	0·42
	4	4	7	20	17	22	16	18	18
C(8)	0·5370	0·7854	0·3635	4·69	3·99	3·86	−0·24	0·52	−0·56
	4	5	7	22	19	21	18	18	18
C(9)	0·4259	0·8345	0·3339	5·44	4·13	9·17	1·42	−0·55	−0·90
	5	5	11	29	22	41	20	28	26
N(1)	0·6138	0·8640	0·4266	5·77	3·96	6·29	−0·05	1·01	−0·24
	4	4	7	23	19	25	16	20	16
N(2)	0·6825	0·6676	0·3831	3·73	3·84	6·60	−0·39	0·05	0·50
	3	4	7	18	17	25	16	16	16
O(1)	0·7063	0·7977	0·4426	5·12	4·77	7·47	−1·14	−0·12	−0·48
	3	4	7	18	16	24	14	16	16
O(2)	0·7579	0·5941	0·3900	3·75	5·40	11·87	0·30	−0·05	0·11
	3	4	7	16	19	38	16	18	22
Br	0·35354	0·15662	0·06662	6·63	4·32	5·62	−1·54	0·52	−0·26
	5	6	9	4	3	4	2	2	2
H(2)	0·574	0·234	0·180		B=4·0 Å²				
	5	7	9						
H(3)	0·653	0·435	0·266		B=4·0				
	5	7	9						
H(5)	0·374	0·598	0·230		B=4·0				
	6	7	10						
H(6)	0·285	0·416	0·144		B=4·0				
	5	7	9						

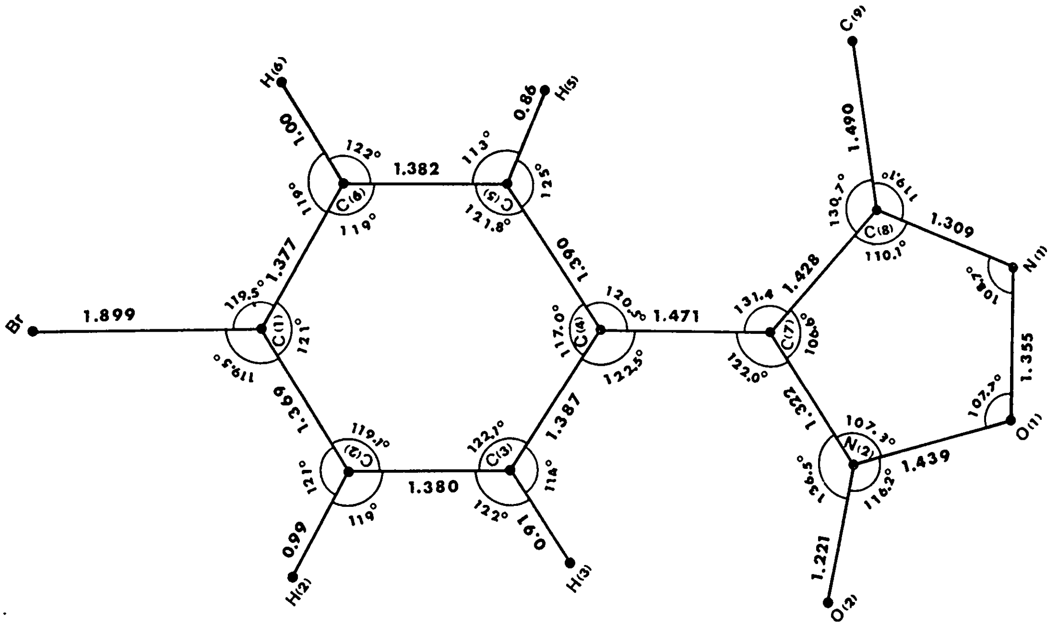

Fig. 2. Molecular bonds and angles.

As regards the reported e.s.d.'s we draw attention to the following considerations concerning the fact that we have neglected the absorption correction. Some computations, based on experimental data, considering crystals of different size, made by Srivastava & Lingafelter (1966) and in a similar way by Werner (1964), have shown that the absorption effect, generally, increases the values of the e.s.d.'s and of R; only in some unfavourable cases does it affect the positional parameters to an extent greater than the σ computed with a full-matrix least-squares program.

Looking at the value of μ and at the size of our crystal, on the basis of the results obtained by the above mentioned authors*, we can say that the order of magnitude of our e.s.d.'s for the positional parameters is correct and that the e.s.d.'s of the thermal parameters can be considered correct within a factor of about 2.

Description and discussion of the structure

A view of the molecule in its plane is shown in Fig. 2, which also gives the numbering of the atoms used in this paper.

The best mean plane through all the atoms (except hydrogen atoms) is given in fractional coordinates and referred to the crystallographic axes, by the equation:

$$3{\cdot}0277x+2{\cdot}5098y-7{\cdot}1474z=0{\cdot}9868\,. \tag{4}$$

In the first column of Table 3 are reported the distances of the atoms from plane (4); assuming a mean standard deviation of 0·008 Å for these distances and applying the χ^2 test, the molecules deviate significantly from strict planarity. On the basis of the t test (Cruickshank & Robertson, 1953), the C(1) and C(2) atoms are significantly, and C(6) and N(2) possibly, out of plane (4). We therefore calculated the mean planes for the bromophenyl and methylfuroxan moieties, which have respectively the equations:

$$3{\cdot}0882x+2{\cdot}5056y-7{\cdot}1441z=1{\cdot}0079\,, \tag{5}$$

$$3{\cdot}0593x+2{\cdot}5732y-7{\cdot}1337z=1{\cdot}0616\,. \tag{6}$$

Table 3. *Distances* (Å) *of the molecular atoms from the reported planes*

	I	II	III	IV
Br	−0·000	−0·000	0·040	—
C(1)	0·025	0·022	−0·002	—
C(2)	0·026	0·016	0·001	—
C(3)	0·009	−0·004	−0·000	—
C(4)	−0·003	−0·012	0·001	—
C(5)	−0·005	−0·008	−0·005	—
C(6)	0·020	0·020	0·005	—
C(7)	0·006	—	0·026	−0·016

Table 3 (*cont.*)

	I	II	III	IV
C(8)	−0·012	—	0·023	−0·009
C(9)	−0·011	—	0·030	0·007
N(1)	0·009	—	0·056	0·004
N(2)	−0·016	—	0·006	−0·011
O(1)	0·010	—	0·050	0·005
O(2)	−0·011	—	0·004	−0·003
H(2)	−0·051	−0·065	−0·087	—
H(3)	−0·181	−0·199	−0·188	—
H(5)	−0·002	−0·002	0·006	—
H(6)	0·109	0·115	0·092	—

The distances of atoms from planes (5) and (6) are listed in columns II and IV of Table 3. The methylfuroxan group comes out to be significantly planar, while the bromophenyl group is significantly non planar: atoms C(2) and C(6) are out of plane (5). In column III of Table 3 are reported the distances of the atoms of the phenyl ring from their own mean plane of equation:

$$3{\cdot}0173x+2{\cdot}3918y-7{\cdot}1729z=0{\cdot}9143\,. \tag{7}$$

The atoms of the phenyl group are seen to be closely coplanar. In the same column III we report also the distances of the other atoms from plane (7); the most significant deviations are those of the Br, N(1) and O(1) atoms, and an examination of all the deviations shows that the bromine atom and the furoxan group are slightly above the plane of the phenyl ring.

A general view of molecular packing is shown in Fig. 3. The molecules are slightly inclined ($\sim$14°) to the (100) plane. Two molecules, related by a centre of symmetry, are so arranged that the phenyl group of one nearly faces the furoxan ring of the other (the distance between the two rings is 3·58 Å) and so as to superpose the least number of atoms; in fact, only atoms C(2), C(4) and C(6) of one molecule superpose respectively on atoms C(8), C(4) and N(2) of the other. Between the above molecules there are no unduly short intermolecular contacts and this is in agreement with the easy $\{\bar{1}01\}$ cleavage. The only remarkable intermolecular contact is that of 3·08 Å between the bromine atom of one molecule and oxygen O(2) at $x-\frac{1}{2},\frac{1}{2}-y,z-\frac{1}{2}$. This may possibly explain the departure from strict planarity of the molecule.

Inspection of bond lengths and angles of a single molecule, reported in Table 4, shows, first, that the difference between the shortest and longest C–C bond in the phenyl group is only possibly significant and that the individual bond-lengths do not differ significantly from their mean (1·381 Å). Bond C(4)–C(7) presents 15% of double-bond character (Pauling, 1960) so that there is a certain degree of resonance between the phenyl and furoxan rings. As regards this latter ring, the C(7)–C(8) bond has about 33% of double-bond character, in agreement with the lengthening of the two C=N bonds; the difference between bonds C(7)–N(2) and C(8)–N(1) is possibly significant The difference between N(1)–O(1) and N(2)–O(1) is highly

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* We have in mind essentially the case IIω quoted in Table 1 of Srivastava & Lingafelter's paper, but we point out that our μ value is considerably smaller.

Table 4. *Bond lengths and bond angles with the significant figures of the standard deviations in brackets*

Br—C(1)	1·899 (4) Å	C(7)-N(2)	1·322 (6) Å
C(1)-C(2)	1·369 (8)	C(8)-N(1)	1·309 (7)
C(2)-C(3)	1·380 (8)	N(1)-O(1)	1·355 (6)
C(3)-C(4)	1·387 (7)	N(2)-O(1)	1·439 (6)
C(4)-C(5)	1·390 (7)	N(2)-O(2)	1·221 (6)
C(5)-C(6)	1·382 (8)	C(2)-H(2)	0·99 (7)
C(6)-C(1)	1·377 (7)	C(3)-H(3)	0·91 (7)
C(4)-C(7)	1·471 (6)	C(5)-H(5)	0·86 (7)
C(7)-C(8)	1·428 (7)	C(6)-H(6)	1·00 (7)
C(8)-C(9)	1·490 (8)		
Br—C(1)-C(2)	119·5 °(3)	C(9)-C(8)-N(1)	119·1 °(5)
Br—C(1)-C(6)	119·5 (4)	C(8)-N(1)-O(1)	108·2 (4)
C(2)-C(1)-C(6)	121·0 (5)	C(7)-N(2)-O(1)	107·3 (4)
C(1)-C(2)-C(3)	119·1 (5)	C(7)-N(2)-O(2)	136·5 (4)
C(2)-C(3)-C(4)	122·1 (5)	O(1)-N(2)-O(2)	116·2 (4)
C(3)-C(4)-C(5)	117·0 (4)	N(1)-O(1)-N(2)	107·7 (4)
C(3)-C(4)-C(7)	122·5 (5)	H(2)-C(2)-C(1)	121 °(4)
C(5)-C(4)-C(7)	120·5 (4)	H(2)-C(2)-C(3)	119 (4)
C(4)-C(5)-C(6)	121·8 (5)	H(3)-C(3)-C(2)	122 (4)
C(5)-C(6)-C(1)	119·0 (5)	H(3)-C(3)-C(4)	114 (4)
C(4)-C(7)-C(8)	131·4 (5)	H(5)-C(5)-C(4)	125 (4)
C(4)-C(7)-N(2)	122·0 (4)	H(5)-C(5)-C(6)	113 (4)
C(8)-C(7)-N(2)	106·6 (4)	H(6)-C(6)-C(5)	122 (4)
C(7)-C(8)-C(9)	130·7 (5)	H(6)-C(6)-C(1)	119 (4)
C(7)-C(8)-N(1)	110·1 (4)		

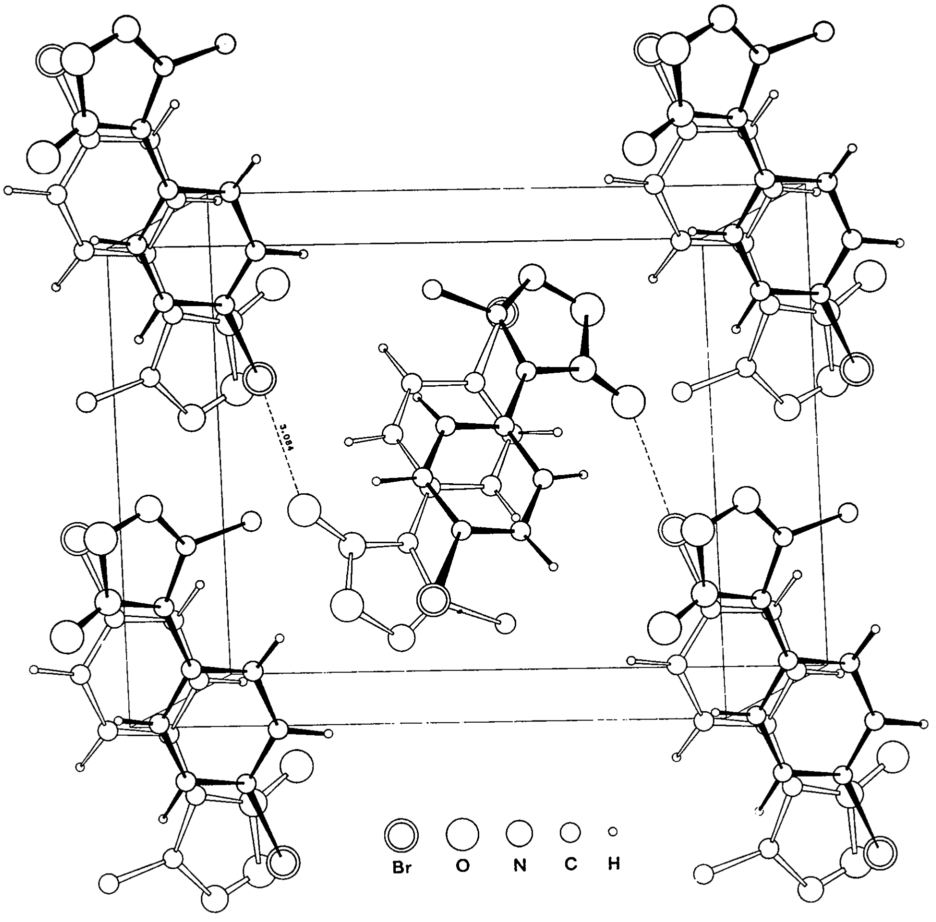

Fig. 3. Clinographic projection, along the *z* axis, of the content of one unit cell (*y* axis vertical).

significant and N(2)–O(2) has a very high double-bond character.

Finally, the shortening of bond C(8)–C(9) with respect to a single-bond value may be ascribed to an effect of hyperconjugation of the methyl group.

Two significant intramolecular contacts are those between O(2) and H(3) (2·24 ± 0·07 Å) and between C(9) and H(5) (2·63 ± 0·07 Å); they are probably related to some of the characteristics of stability of the lower melting isomer with respect to the higher melting isomer.

In Table 5 are reported the elements characterizing the vibrational ellipsoids, referred to their own principal axes, *i.e.* in the form:

$$\sum_{i=1}^{3} q_i \sum_{j=1}^{3} g_{ij} h_{ij} a_j^{*2} , \qquad (8)$$

where q_i are the three principal vibrational semi-axes (in Å²) and g_{ij} are their direction cosines with respect to the reciprocal unit-cell axes (j = 1,2,3 for a^*, b^*, c^* respectively). As can be seen from B_{ij} and root mean square deviation values, reported in Tables 2 and 5,

Table 5. *Parameters characterizing the vibrational ellipsoids referred to their own principal axes*

	i	q_i (Å²)	r.m.s.d. (Å)	g_{i1}	g_{i2}	g_{i3}
C(1)	1	1·125	0·239	−0·706	0·039	−0·801
	2	1·426	0·269	−0·678	0·272	0·583
	3	0·778	0·199	0·221	0·961	−0·133
C(2)	1	1·202	0·247	0·871	0·400	0·403
	2	1·856	0·307	0·165	0·311	−0·904
	3	0·904	0·214	−0·463	0·862	0·139
C(3)	1	1·070	0·233	0·002	0·999	0·044
	2	1·898	0·310	0·023	0·044	−0·986
	3	0·787	0·200	0·999	0·006	0·160
C(4)	1	0·881	0·211	−0·735	0·337	−0·683
	2	1·210	0·248	−0·261	0·659	0·662
	3	0·742	0·194	0·625	0·672	−0·307
C(5)	1	1·015	0·227	0·729	0·677	−0·003
	2	2·264	0·339	0·039	0·110	0·989
	3	0·858	0·209	−0·684	0·728	−0·147
C(6)	1	1·318	0·258	−0·543	0·836	−0·151
	2	1·931	0·313	−0·007	0·088	0·986
	3	0·813	0·203	0·840	0·541	0·074
C(7)	1	0·949	0·219	−0·784	0·557	−0·379
	2	1·237	0·250	−0·076	0·351	0·914
	3	0·833	0·205	0·616	0·752	−0·146
C(8)	1	1·108	0·237	0·297	0·657	−0·645
	2	1·192	0·246	−0·936	0·343	−0·205
	3	0·838	0·206	0·186	0·671	0·736
C(9)	1	1·406	0·267	0·795	0·464	0·496
	2	2·583	0·362	0·314	0·236	−0·867
	3	0·804	0·202	−0·519	0·854	−0·030
N(1)	1	1·420	0·268	0·932	0·013	−0·231
	2	1·587	0·284	−0·360	0·099	−0·968
	3	0·984	0·223	0·016	0·995	0·100
N(2)	1	0·997	0·225	−0·637	0·729	−0·337
	2	1·743	0·297	−0·163	0·192	0·936
	3	0·847	0·207	0·754	0·657	0·100
O(1)	1	1·502	0·276	−0·694	0·691	−0·296
	2	1·998	0·318	−0·280	0·004	−0·912
	3	0·909	0·215	0·664	0·723	0·282
O(2)	1	1·363	0·263	0·173	0·985	0·028
	2	3·054	0·393	−0·067	0·007	0·979
	3	0·915	0·215	−0·983	0·173	−0·201
Br	1	1·417	0·268	−0·122	0·174	−0·985
	2	1·866	0·307	−0·884	0·429	0·064
	3	0·881	0·211	0·452	0·886	0·163

the thermal vibration is rather high, in accordance with the relatively low melting point. An analysis of the orientation of the ellipsoids indicates that their longest axes are nearly normal to plane (4) of the molecule, while the longest component in the molecular plane has a tendency to be parallel to a bond. This does not apply to the atoms bound to three other (non-hydrogen) atoms; these have a smaller thermal vibration and their ellipsoids are essentially revolution ones. The magnitude and anisotropy of the thermal parameters for atoms C(9) and O(2) are remarkable.

A short comparison of the characteristics of the two isomers is reported at the end of the following paper on the structure of the higher melting isomer.

The computations were done by Olivetti ELEA 6001 and IBM 7090 (C.N.U.C.E., Pisa) computers.

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The Crystal and Molecular Structure of the 5-Oxide of 4-Methyl-3-(*p*-Bromophenyl)-1,2,5-Oxadiazole

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The 5-oxide of 4-methyl-3-(*p*-bromophenyl)-1,2,5-oxadiazole, BrC_6H_4–$(C_2N_2O_2)$–CH_3, is the isomer of higher melting point (108–109°C), obtained by oxidation of methyl(*p*-bromophenyl)glyoxime. It crystallizes in the monoclinic system, space group $P2_1/n$, with four molecules in a cell having: $a_0 = 12{\cdot}648$, $b_0 = 10{\cdot}136$, $c_0 = 7{\cdot}499$ Å; $\beta = 94°52'$. A crystal-structure analysis has been based on all the X-ray reflexions accessible with Cu $K\alpha$ radiation. The molecule, approximately planar, has the configuration of furazan 5-oxide: the two isomers in question are positional isomers. The methyl group is involved in two intramolecular contacts with the extranuclear oxygen atom of the furoxan nucleus and with a hydrogen atom of the phenyl group. The molecular structures of the two isomers described in this and the preceding paper are compared.

As reported in the preceding paper (Calleri, Ferraris & Viterbo, 1969), which describes the structure of the lower melting point isomer of methyl(*p*-bromophenyl)furoxan, we are studying the isomerism of aryl-alkyl furoxans and, as a part of our programme the structure of the higher melting point isomer (HMI) of methyl(*p*-bromophenyl)furoxan is reported in the present paper.

Experimental

Crystal data and intensity measurement

In this case also, the compound prepared by Ponzio (1928*a,b*), was recrystallized from an aqueous-alcoholic solution, yielding prismatic crystals elongated in a direction chosen as the *c* axis. The following crystal data were obtained: $C_9H_7BrN_2O_2$; $M = 255{\cdot}06$; m.p.