During the course of the preliminary investigation it was noted in a periodical review of the literature that Pepinsky, Jona & Shirane (1956) had observed the appearance at 176 °K. of a peak in the dielectric constant of methyl ammonium alum, the occurrence of the peak being accompanied by a sharp maximum in the loss factor; this effect is presumably similar to that reported by Griffiths & Powell (1952).

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An X-ray crystallographic study of 1-(5′-bromo-2′-hydroxy-4′-methoxyphenyl)-3-oxo-isoindolo-1-en.

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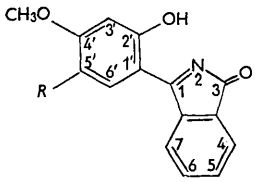
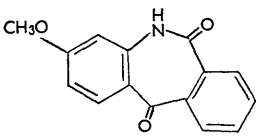
Lamchen (in preparation) has described the synthesis of a red crystalline compound which he has formulated as 1-(2′-hydroxy-4′-methoxyphenyl)-3-oxo-isoindolo-1-en (I*a*). An alternative structure (II), with the same empirical formula ($C_{15}H_{11}NO_3$), could be suggested for this compound and the present study was undertaken to decide between these alternatives. To enable a direct structure determination by the 'heavy-atom method', a bromo-derivative was prepared in which the bromine atom was substituted on to the phenyl group at one of the positions 3′-, 5′- or 6′-; on chemical grounds, the 5′-position (structure I*b*) seemed most probable and has been verified by the X-ray analysis.



(I*a*): R = H
(I*b*): R = Br



(II)

The crystals grow from a chloroform–petroleum–ether solution in the form of very fine, deep-red needles which are frequently twinned. X-ray oscillation and Weissenberg photographs showed them to be triclinic, with one very short unit-cell dimension parallel to the needle axis.

Crystal data

$C_{15}H_{10}NO_3Br$, $M = 332{\cdot}2$,
$a = 3{\cdot}96 \pm 0{\cdot}01$, $b = 11{\cdot}91 \pm 0{\cdot}03$, $c = 13{\cdot}53 \pm 0{\cdot}04$ Å,
(λ(Cu $K\alpha$) = 1·5418 Å)
$\alpha = 100°\ 46' \pm 20'$, $\beta = 91°\ 40' \pm 20'$, $\gamma = 91°\ 20' \pm 20'$,
$U = 625{\cdot}6$ Å^3, $D_m = 1{\cdot}76$ g.cm.$^{-3}$, $Z = 2$, $D_c = 1{\cdot}763$ g.cm.$^{-3}$,
$F(000) = 332$.

The 0*kl* intensity data were measured by visual estimation, from a series of multiple-film Weissenberg photographs. Of the 417 non-equivalent reflections within the Cu $K\alpha$ limit, 339 were recorded. A statistical survey of the intensity data gave an $N(z)$ distribution approximating more closely to the theoretical curve for a non-centrosymmetric projection (Howells, Phillips & Rogers, 1950), suggesting the space group $P1$. However, when allowance is made for the predominant heavy bromine atoms, as suggested by Sim (1958), the results seem to favour the centrosymmetric space group $P\bar{1}$.

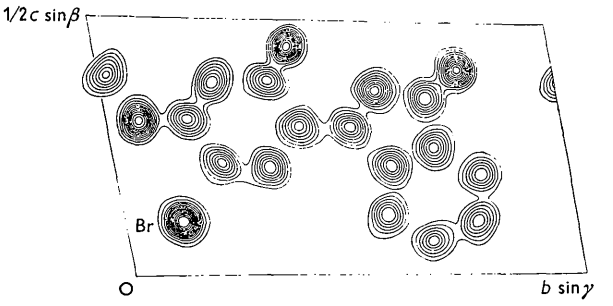


Fig. 1. The electron-density projection normal to the *a*-axis. The first contour is at 2 e.Å^{-2} and the contour interval is 1 e.Å^{-2}, except over the bromine atom where the interval is 5 e.Å^{-2}.

(y, z) parameters for all the atoms in the molecule were derived from an electron-density projection calculated with the phases of the bromine contributions to the structure factors. A set of 0*kl* structure factors based on these atomic positions showed satisfactory agreement with the observed structure amplitudes (the reliability factor $R = \Sigma|F_o - F_c| / \Sigma|F_o| = 0{\cdot}17$). The final electron-density distribution is shown in Fig. 1 where a clearly resolved projection of the atomic maxima corresponding to a molecule of structure I*b* can be seen. The average peak height over the carbon atoms is 8·8 e.Å^{-2} and over the oxygen atoms is 12·3 e.Å^{-2}, while the peak height of the one nitrogen atom is 11·0 e.Å^{-2}.

The present two-dimensional analysis confirms the proposed structure (I*b*). Packing considerations suggest that the molecules are linked by a system of O–H · · · N bonds approximately 2·8 Å in length; a complete analysis is required to investigate this hydrogen bonding and to provide details of the molecular geometry.

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