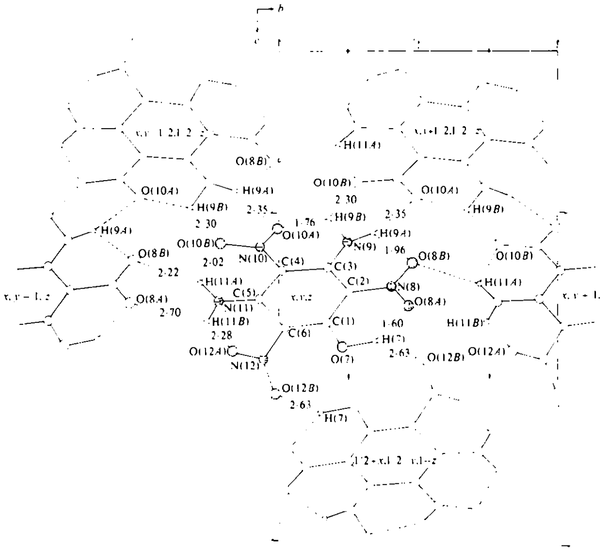

Fig. 2. Intermolecular packing viewed down **a**. All non-bonded distances less than 2·70 Å have been indicated by broken lines (e.s.d.'s are 0·06–0·07 Å). The intermolecular distances of 2·70 [H(11*B*)···O(8*A*)] and 2·63 Å [H(7)···O(12*B*)] are not hydrogen-bonded contacts. A number of the angles (°) associated with the intra- and intermolecular hydrogen contacts are: O(7)–H(7)···O(8*A*) = 143 (5), O(7)–H(7)···O(12*B*) = 110 (5), O(8*A*)···H(7)···O(12*B*) = 90 (3), N(9)–H(9*A*)···O(8*B*) = 128 (5), N(9)–H(9*A*)···O(10*A*)^{i} = 126 (6), O(10*A*)···H(9*A*)···O(8*B*) = 100 (2), N(9)–H(9*B*)···O(10*B*)^{i} = 120 (5), N(9)–H(9*B*)···O(10*A*) = 120 (5), O(10*B*)···H(9*B*)···O(10*A*) = 105 (3), N(11)–H(11*A*)···O(10*B*) = 117 (6), N(11)–H(11*A*)···O(8*B*)^{ii} = 148 (7), O(8*B*)···H(11*A*)···O(10*B*) = 96 (3), N(11)–H(11*B*)···O(12*A*) = 120 (6). Superscripts i and ii refer to molecules at $x, y + \frac{1}{2}, -z + \frac{1}{2}$ and $-x, y - \frac{1}{2}, -z + \frac{1}{2}$ respectively.

H(7) (—OH group) appear not to be associated with intermolecular hydrogen bonds, with O···H distances of 2·63 Å [to O(12*B*)] and 2·70 Å [to O(8*A*)] respectively.

Along **a**, molecules which are separated by the cell repeat have no C···C contacts less than 3·6 Å and there are no hydrogen bonds. The molecules are thus stacked in layers in which the normal to the benzene ring is inclined by 44° from **a**. The predicted length of the *a* axis of 4·73 Å, based on a 3·4 Å thick benzene ring angled at 44°, is close to the observed value of 4·97 Å.

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(*p*-Methylphenoxy)acetic Acid

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Abstract. $C_9H_{10}O_3$, monoclinic, $P2_1/c$, $Z = 4$, $a = 13.890$ (16), $b = 5.248$ (2), $c = 12.072$ (2) Å, $\beta = 103.00$ (13)°, $D_o = 1.326$, $D_c = 1.316$ Mg m^{-3}. The structure was solved by direct methods using Sayre's equation and refined to $R = 0.0865$ for 511 reflections by full-matrix least squares with anisotropic temperature factors. The molecules form dimers across centres of symmetry with O—H···O bonds of

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2·639 (11) Å and are held in the structure by normal van der Waals interactions.

Introduction. Crystals suitable for X-ray diffraction work were grown by evaporation of a saturated solution of the compound in a mixture of xylene and CCl_4. Accurate values for the cell parameters were derived by a least-squares fit of the θ values of high-angle reflections measured on zero-layer Weissenberg photographs. The data were collected using the multiple-film equi-inclination Weissenberg method. Intensities of 580 unique reflections [from Cu $K\alpha$ radiation ($\lambda = 1{\cdot}5418$ Å)] were estimated visually by comparison with a graded intensity scale prepared from the same crystal. These were corrected for Lorentz, polarization and spot-extension factors. No absorption correction was applied; the size of the crystal used was $0{\cdot}10 \times 0{\cdot}15 \times 0{\cdot}20$ mm and $\mu = 0{\cdot}835$ mm^{-1}. The overall temperature factor ($B = 2{\cdot}80$ Å^2) and scale factor used to put the intensities on an absolute scale were obtained from a Wilson plot.

The signs of 120 reflections were determined using Sayre's equation, with the help of Long's (1965) program run on an EC 1030 computer. A three-dimensional E map computed with these 120 reflections revealed eight atoms of the molecule and the subsequent Fourier synthesis gave all the 12 non-H atoms of the molecule. The R value was 0·394 when all the 12 atoms were used for structure-factor calculations. Three cycles of full-matrix least-squares refinement of the positions, scale and isotropic temperature factors were made for all the non-H atoms using *ORGLS* (Busing & Levy, 1962). The R value reduced to 0·104. The final refinement of all the atomic parameters with anisotropic temperature factors lowered the R factor to 0·0865. The atomic scattering factors used in the structure factor calculations were taken from *International Tables for X-ray Crystallography* (1962). The positions of the H atoms were fixed from a three-dimensional difference Fourier map.

Table 1. *Fractional atomic positional parameters* ($\times 10^4$) *and equivalent isotropic B values with e.s.d.'s in parentheses*

The B_{eq} values were calculated using the expression:

$$B_{eq} = \tfrac{4}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + 2\beta_{13}ac\cos\beta).$$

	x	y	z	B_{eq} (Å^2)
C(1)	1323 (7)	−1656 (24)	1399 (8)	2·9 (4)
C(2)	1304 (8)	−1066 (28)	240 (9)	4·2 (4)
C(3)	1960 (8)	665 (26)	−67 (8)	3·3 (4)
C(4)	2631 (7)	1952 (24)	774 (8)	2·5 (4)
C(5)	2689 (7)	1494 (24)	1954 (8)	2·9 (4)
C(6)	2007 (8)	−317 (24)	2227 (8)	2·3 (4)
C(7)	652 (8)	−3616 (28)	1744 (10)	4·7 (4)
C(8)	3847 (8)	5307 (24)	1232 (8)	2·9 (4)
C(9)	4326 (7)	7223 (25)	608 (10)	2·7 (4)
O(1)	3220 (5)	3674 (16)	372 (5)	3·1 (2)
O(2)	4171 (5)	7439 (17)	−477 (5)	3·3 (2)
O(3)	4879 (5)	8883 (17)	1273 (6)	3·4 (2)
H(C7)	800	−5300	1800	
H′(C7)	100	−3000	2000	
H″(C7)	400	−4800	1100	
H(C2)	604	−1950	−332	
H(C3)	1906	1033	−840	
H(C5)	3181	2442	2559	
H(C6)	2011	−746	2994	
H(C8)	4300	4000	1670	
H′(C8)	3430	6000	1700	
H(O3)	5354	10722	873	

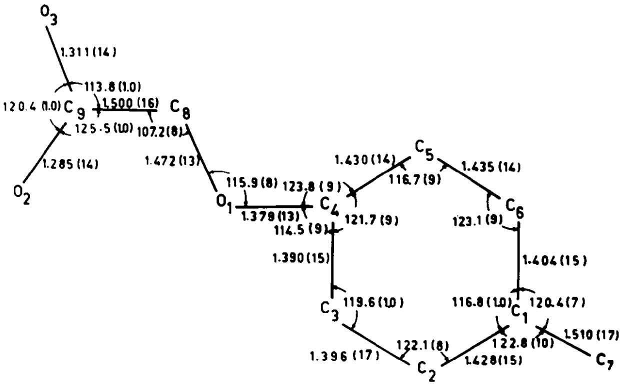

Fig. 1. Molecular geometry of (*p*-methylphenoxy)acetic acid; bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses.

Discussion. The final atomic coordinates of all the atoms are given in Table 1.* Bond lengths and bond angles are given in Fig. 1, with e.s.d.'s in parentheses. The individual bond lengths of the aromatic ring deviate significantly from the generally accepted value of 1·398 Å for the benzene ring; the deviations are, however, comparable with those observed in (2-chlorophenoxy)acetic acid (Chandrasekhar & Pattabhi, 1977). The equations of the least-squares planes through C(1) to C(6) and through C(8), C(9), O(2) and O(3) are: (1) $0{\cdot}685X - 0{\cdot}728Y - 0{\cdot}003Z = 1{\cdot}616$; (2) $0{\cdot}796X - 0{\cdot}604Y + 0{\cdot}027Z = 2{\cdot}359$. The aromatic ring is planar within experimental error; the carbon atom of the methyl group is nearly in the benzene plane. The angle between these two planes is 9·7°. This is comparable in the order of magnitude with the angles of 7·0 and 6·6° in (2-chlorophenoxy)acetic acid (Chandrasekhar & Pattabhi, 1977), 4·1° in (2,4,5-trichlorophenoxy)acetic acid (Graham, Kennard & White, 1976) and 5·5° in (*p*-nitrophenoxy)acetic acid (Vijay Kumar & Rao, 1980). It is, however, significantly different from 85·2° found in (2,4-dichlorophenoxy)-

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36063 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

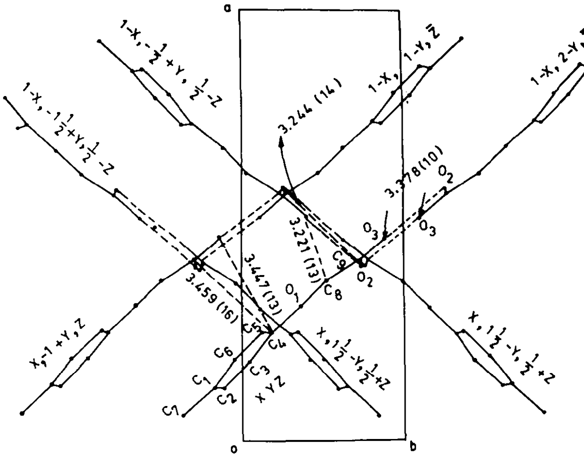

Fig. 2. Projection of the structure of (*p*-methylphenoxy)acetic acid on the (001) plane. Short contacts of less than 3·5 Å are shown.

acetic acid (Smith, Kennard & White, 1976) and 90° in 3-indolylacetic acid (Karle, Britts & Gum, 1964). In the COOH group, the differences in the two C—O bond lengths and the values of the C—C—O angles are similar to those observed in other substituted phenoxyacetic acids, where double- and single-bond characters distinctly exist, but it is different from the case of (*p*-nitrophenoxy)acetic acid where occupational disorder of the H atom of the COOH group exists, leading to near equality in C—O bond lengths and C—C—O angles.

The carboxylic group takes part in dimer formation across centres of symmetry situated halfway along the *x* axis. Fig. 2 shows the packing arrangement of these dimers consisting of two stacks of hydrogen-bonded molecule pairs related to each other by the glide plane *c* (0$\frac{1}{4}$0).

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Structure of 6-Nitro-2,3-dihydroxyquinoxaline* Hydrate

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Abstract. $C_8H_5N_3O_4.H_2O$, monoclinic, $P2_1/c$, a = 7·489 (1), b = 11·949 (2), c = 12·066 (2) Å, β = 120·43 (2)°, Z = 4. 732 independent and non-zero reflexions were measured on a single-crystal diffractometer. The structure was solved by direct methods and refined by full-matrix least squares (173 parameters) to a final R = 0·052. The compound exists in crystals in the keto form only (the average C—O bond distance is 1·229 Å). The carbon ring is aromatic (average C—C bond distance of 1·386 Å). The molecules are linked by a three-dimensional set of strong N—H···O and O—H···O hydrogen bonds.

Introduction. Structure investigations of substituted quinoxalines have been undertaken in our department (Stępień, Grabowski, Cygler & Wajsman, 1976; Grabowski, Stępień, Cygler & Wajsman, 1977; Stępień, 1977). The results presented here are a continuation of this work. Crystals of 6-nitro-2,3-dihydroxyquinoxaline hydrate were obtained from the Center for Research and Development of Dyes in Zgierz, Poland.

The light-yellow crystals were recrystallized from ethanol at room temperature. The crystals used for the X-ray measurements had average dimensions of 0·2 × 0·2 × 0·3 mm. The intensities were measured on a Syntex $P2_1$ diffractometer, by the θ–2θ scan method,

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* Alternative name: 6-nitro-2,3(1*H*,4*H*)-quinoxalinedione.

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