

Fig. 2. Angles de liaisons ($^{\circ}$); les écarts types sont de l'ordre de 0,2 à 0,4 $^{\circ}$.

poursuivis en tenant compte de l'anisotropie de l'agitation thermique de tous les atomes non hydrogènes et en minimisant $\sum w(|KF_o| - |F_c|)^2$ où $w = 1/\sigma^2(F)$. Ils ont conduit pour les 1262 réflexions les plus intenses à un facteur d'accord pondéré final $R(w)$ de 0,046 ($R = 0,058$),*

* Les listes des facteurs de structure et des paramètres thermiques anisotropes ont été déposées au dépôt d'archives de la British Library Lending Division (Supplementary Publication No. SUP 35003: 10 pp.). On peut en obtenir des copies en s'adressant à: The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre.

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The Structure of *p*-Nitrophenoxyacetic Acid

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Abstract. $C_8H_7NO_5$, triclinic, $P\bar{1}$, $Z = 2$, $a = 8.283$ (1), $b = 6.960$ (3), $c = 7.553$ (3) Å, $\alpha = 95.712$ (19), $\beta = 108.528$ (21), $\gamma = 87.746$ (20) $^{\circ}$. The structure was determined by direct methods and refined to $R = 0.087$ by full-matrix least squares with anisotropic temperature factors. The molecules dimerize, about a centre of symmetry, as is usual with simple carboxylic acids, and

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Au Tableau 1 figurent les coordonnées finales ainsi que les facteurs d'agitation thermique équivalents des atomes numérotés conformément au schéma de la Fig. 2. Les longueurs de liaisons sont données dans le Tableau 2 et les angles sont portés sur la Fig. 2.

Discussion. Selon Pauling (1960), les rayons des atomes de carbone et d'azote, dans le cas d'une simple liaison covalente, sont respectivement égaux à 0,772 et 0,74 Å et prennent les valeurs 0,667 et 0,62 Å dans le cas d'une double liaison covalente. Ceci permet de prévoir des longueurs de liaisons carbone–azote plus courtes que les longueurs de liaisons carbone–carbone. La configuration (b) choisie est bien en accord avec cette prévision puisqu'elle nous donne d'une part: $N(7)-C(6) = 1,337$, $N(7)-C(8) = 1,321$ Å et, d'autre part $C(4)-C(5) = 1,367$ et $C(4)-C(9) = 1,376$ Å.

Un calcul de plan moyen a permis de vérifier la planéité de la partie de la molécule formée par les atomes N(1), C(2), N(3), C(4), C(5), C(6), C(7), C(8), C(9) et nous donne un angle dièdre de 56,2 $^{\circ}$ entre cette partie et le plan du phényle. Le plan formé par N(16), O(17), O(18) fait un angle de 5,4 $^{\circ}$ avec le plan du cycle alors que le groupement N(19), O(20), O(21) fait un angle relativement important de 35,9 $^{\circ}$.

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are linked by a pair of hydrogen bonds with $O \cdots O$ 2.59 (1) Å. The geometry of the carboxyl group shows that the packing of these groups is disordered.

Introduction. Crystals suitable for X-ray analysis were obtained by slow evaporation from a mixture of methanol and bromobenzene and also separately from

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chloroform. Accurate lattice constants were determined from high-angle reflections on Weissenberg photographs taken by the asymmetric film-loading method (Vijay Kumar & Rao, 1978). The data were collected using the multiple-film equi-inclination Weissenberg method. The intensities of 927 unique reflections from Cu *K* α radiation (1.5418 Å) were estimated visually and corrected for Lorentz, polarization and spot-extension factors. The reflections of different layers along the *c* axis were brought to a common scale by comparison with the cross-level photographs taken along the *b* axis. The *N*(*z*) test showed the crystal to be centrosymmetric. The overall temperature factor $B = 3.18 \text{ \AA}^2$ and the scale factor, used to place the intensities on an absolute scale, were obtained from a Wilson plot. An absorption correction was not applied. The size of the crystal used was $0.13 \times 0.19 \times 0.23 \text{ mm}$ and $\mu = 1.142 \text{ mm}^{-1}$.

The signs of 62 structure amplitudes were determined using Sayre's equation (Long, 1965), and the *E* map computed with these structure factors revealed the complete molecule ($R = 0.417$). Two cycles of full-matrix least-squares refinement with isotropic temperature factors reduced *R* to 0.198 and another five cycles of refinement led to $R = 0.144$. Refinement with anisotropic temperature factors for all non-hydrogen atoms reduced *R* to 0.107. The difference Fourier map revealed all the H atoms clearly but, for the H atom taking part in dimer formation, it showed two peaks having half the height of the other H peaks, separated by a distance of 1.28 Å. One more cycle of refinement including the H atom positions reduced *R* to 0.087. The final Fourier and difference Fourier maps did not show any unexplainable electron densities. The scattering-factor curves used in the structure factor

calculation were taken from *International Tables for X-ray Crystallography* (1962).

Discussion. The final atomic coordinates of all the atoms are given in Table 1.* Bond lengths and bond angles, which are shown in Fig. 1, are within the 3σ limits of other substituted phenoxyacetic acids (Smith, Kennard & White, 1976*a,b*). The slightly large values of the bond angles C(2)–C(1)–C(6) (121.3°) and C(3)–C(4)–C(5) (121.0°) may be due to the presence of NO₂ (a strong electron-withdrawing group) and O respectively. Similar effects have been reported in substituted benzene derivatives (Domenicano, Vaciago

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34948 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic positional parameters ($\times 10^4$) with e.s.d.'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>
O(1)	729 (8)	278 (10)	2771 (9)
O(2)	2392 (8)	1681 (10)	1653 (8)
N(1)	2019 (8)	1157 (9)	2971 (9)
C(1)	3174 (9)	1651 (11)	4807 (12)
C(2)	2752 (10)	1162 (12)	6325 (13)
C(3)	3835 (9)	1640 (12)	8066 (12)
C(4)	5389 (9)	2544 (11)	8305 (11)
C(5)	5823 (9)	2982 (11)	6808 (12)
C(6)	4717 (9)	2524 (11)	5032 (12)
O(3)	6368 (7)	2937 (8)	10103 (8)
C(7)	7976 (11)	3773 (13)	10447 (13)
C(8)	8804 (9)	4279 (11)	12473 (12)
O(4)	8117 (7)	3815 (9)	13621 (8)
O(5)	10208 (7)	5166 (8)	12909 (8)
H(1)	1636	667	5928
H(2)	3401	1374	8866
H(3)	6878	3161	6770
H(4)	4961	2594	3670
H(5)	8857	2970	10109
H(6)	7984	4872	9892

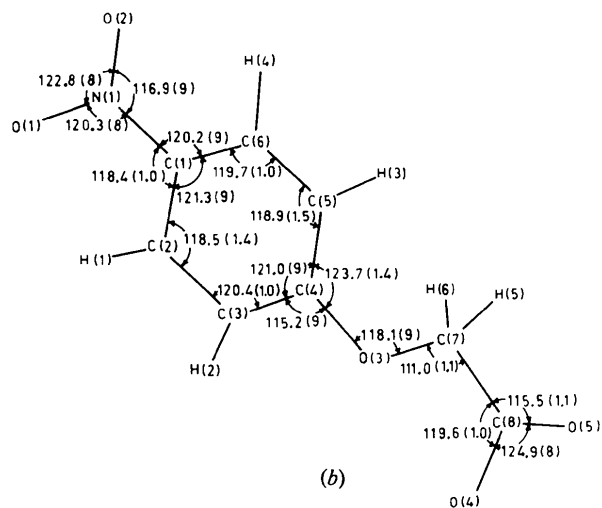
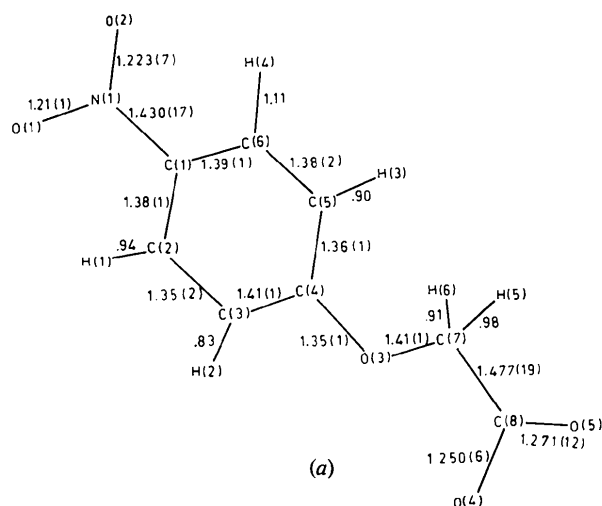


Fig. 1. (a) Bond lengths (Å) and (b) bond angles ($^\circ$) in *p*-nitrophenoxyacetic acid, with e.s.d.'s in parentheses.

& Coulson, 1975). The average C—C bond length in the benzene ring is 1.38 Å and the average C—H length is 0.94 Å. The C(7)—C(8) bond is 1.48 Å, which is shorter than the 1.517 Å reported for the 2,4-di- and 2,4,5-trichlorophenoxyacetic acids (Smith *et al.*, 1976*a*, *b*), but a similar shortening of the C(7)—C(8) bond has been observed in 2-chlorophenoxyacetic acid (Chandrasekhar & Pattabhi, 1977). The nearly equal lengths of the two C—O bonds [1.271 (12) and 1.250 (6) Å] and the smaller difference in the C—C—O angles [115.5 (1.1) and 119.6 (1.0)°] in the carboxyl group

may be attributed to the disorder of the carboxyl group. Similar disorder has been observed in the carboxyl group of β -chloro-*cis*-cinnamic acid (Filippakis, Leiserowitz, Rabinovich & Schmidt, 1972).

The equations of the least-squares planes (1) through O(1), O(2) and N(1), (2) through C(1) to C(6), and (3) through C(7), C(8), O(4) and O(5), are

$$\begin{aligned} (1) & 0.5032X - 0.8612Y - 0.0718Z = 0.1779, \\ (2) & 0.4457X - 0.8944Y - 0.0370Z = 0.1785, \\ (3) & -0.4641X + 0.8840Y - 0.0563Z = 0.6450. \end{aligned}$$

The N(1) atom is in the plane of the benzene ring and the unsymmetrical nitro group is strictly planar; the dihedral angle between these two planes is 4.3°. The planar acetic acid group makes an angle of 5.5° with the ring plane, which is very similar to those of many substituted phenoxyacetic acids (Smith *et al.*, 1976*b*). The packing of these dimeric molecules is shown in Fig. 2, along with principal intermolecular distances.

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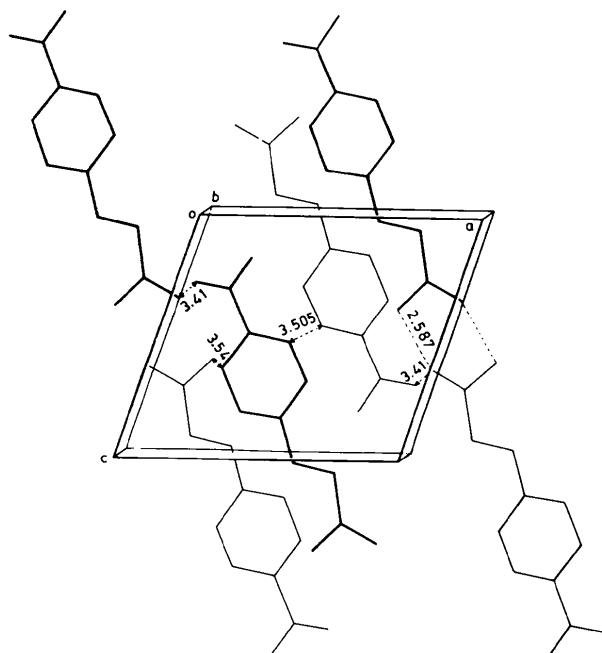


Fig. 2. Packing of the molecules in the unit cell viewed perpendicular to the *ac* plane, showing some intermolecular distances (Å). E.s.d.'s in the distances are 0.01 Å.

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cis-5-*tert*-Butyl-1,3,2-dioxathiane 2-Oxide

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Abstract. C₇H₁₄O₃S, monoclinic, *P*2₁/*b*, *Z* = 4, *a* = 6.106 (4), *b* = 7.743 (4), *c* = 18.968 (7) Å, γ = 99.46 (5)°, *D*_c = 1.338 Mg m⁻³ at 123 K. The sulfite ring adopts a chair conformation with the *tert*-butyl group in equatorial and the S=O group in axial

position. The conformation confirms the interpretation of IR and NMR spectra.

Introduction. Substituted cyclic sulfites are very well suited for the study of the influence of the anomeric

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