these sheets into a three-dimensional network by hydrogen bonds. The hydrogen bonds themselves form infinite chains in the [100] directions. This network and the atomic numbering are depicted in Fig. 2. Relevant data on the geometries of the acetate ions, the water molecules, the hydrogen bonds and Ca coordinations are collected in Table 2. Only one of the two crystallograpically independent water molecules coordinates to Ca in this polymorph of $Ca(OAc)_2.H_2O$ as opposed to two in the previously studied monohydrate. The carboxyl geometry and Ca–carboxyl interactions are comparable with the results of Einspahr & Bugg (1981). The present bond distances, bond angles and planarity of the acetate ions lie well within the ranges reported there. The two interaction modes found in our study are common to acetate–calcium interactions. For both interaction modes different geometries have been reported previously. The bidentate mode has Ca—O distances between 2·4 and 2·8 Å and Ca—O—C angles between 80 and 100°. The interactions of Ca(1) with O(3) and O(4), Ca(1) with O(5^{i}) and O(6^{i}) and of Ca(2) with O(1) and O(2) are also found in the present structure. The unidentate mode has Ca—O distances between 2·2 and 2·6 Å and Ca—O—C angles between 100 and 180°. All non-bidentate Ca—O interactions are unidentate. The distances and angles lie within the ranges reported. For Ca, coordination numbers ranging from six to nine have been reported but the most commonly occurring coordination numbers are seven and eight, the numbers also found in this study. The slight tendency for the higher coordination numbers to form larger Ca—O distances is also reflected in this study (Table 2). The Ca ions have a closed-shell electronic configuration (p^6). Such ions allow a wide range in angular distortion in the polyhedra formed by the surrounding oxygen atoms. Ca(1) is coordinated by eight acetate O atoms. The coordination can be described as a distorted monocapped pentagonal bipyramid. Ca(2) is coordinated by six acetate O atoms and one water O atom. This coordination can be described as a distorted pentagonal bipyramid. These two coordination types are also found in the previously reported polymorph of calcium acetate monohydrate (Klop, Schouten, van der Sluis & Spek, 1984) and in the structure of the related calcium hydrogen triacetate monohydrate (Klop & Spek, 1984).

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p-Nitrophenoxyacetamide

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Abstract. $C_8H_8N_2O_4$, $M_r = 197{\cdot}2$, monoclinic, $P2_1/c$, $a = 7{\cdot}818$ (2), $b = 7{\cdot}589$ (2), $c = 14{\cdot}790$ (2) Å, $\beta = 98{\cdot}43$ (2)°, $V = 868{\cdot}02$ Å^3, $Z = 4$, $D_m = 1{\cdot}506$ (3), $D_x = 1{\cdot}501$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}79$ mm^{-1}, $F(000) = 408$, $T = 426$ (1) K, final $R = 0{\cdot}0507$ for 798 observed reflections [$I \geq 2\sigma(I)$]. The molecules are hydrogen bonded: N—H···O = 2·886 (3), N—H = 0·97 (7), H—O = 1·92 (6) Å, N—H···O angle = 175·5 (8)°.

Introduction. The title compound is known for its auxion and hypoglycemic activity. Its crystal and molecular structure determination was undertaken as part of a programme to establish structure–activity

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relationships in the substituted phenoxyacetic acids and amides.

Experimental. 2 g of *p*-nitrophenoxy acetic acid chloride was mixed with excess of ammonium carbonate and, after the first vigorous reaction had subsided, the mixture was heated for half an hour. After cooling, the ammonium chloride and carbonate were removed by washing with water, giving a residue of *p*-nitrophenoxyacetamide. Pale yellow crystals were obtained by slow evaporation of a saturated solution in methanol at room temperature. A crystal of dimensions $0{\cdot}40 \times 0{\cdot}15 \times 0{\cdot}53$ mm was mounted on a Nonius CAD-4 diffractometer and the cell parameters were refined by the least-squares method from the measurements of 24 well-centred reflections lying in the θ range 16–44°. Density measured by flotation method using a mixture of carbon tetrachloride and heptane. Data collected at room temperature in the ω–2θ scan mode with graphite-monochromatized Mo $K\alpha$ radiation, in the θ range from 0 to 60° with index range $h\,0\rightarrow8$, $k\,0\rightarrow8$, $l \pm 16$ and backgrounds measured for 1/6 total scan angle on either side of Bragg peak. 798 unique reflections were collected with $I > 2{\cdot}5\sigma(I)$ (1500 total) and used for structure determination. Three standard reflections, monitored every hour, did not show any systematic change. The intensities were corrected for Lorentz and polarization effects, but not for absorption.

The structure was solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and refined by full-matrix least-squares method using *SHELX*76 (Sheldrick, 1976). Refinement with anisotropic temperature factors for all non-hydrogen atoms reduced the R value to 0·0621. All H atoms were located by a different Fourier synthesis and their inclusion in the refinement led to an R value of 0·0507. The function minimized during the least-squares refinement was $(|F_o| - |F_c|)^2$, with weights shown to be satisfactory by a weight analysis, $(\Delta/\sigma)_{max} = 0{\cdot}068$. Max. and min. heights in the final difference Fourier synthesis were 0·28 and −0·19 e Å^{-3} respectively. No correction for secondary extinction. The atomic scattering factors given by Cromer & Mann (1968) were used for non-hydrogen atoms, while for H atoms those of Stewart, Davidson & Simpson (1965) were used.

Discussion. Final positional coordinates of the atoms and their equivalent temperature factors (U_{eq}) are given in Table 1.* Bond lengths and bond angles are given in Table 2. The molecular structure and numbering scheme are shown in Fig. 1 (*PLUTO*; Motherwell & Clegg, 1978).

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43734 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic fractional parameters of non-hydrogen* (×10[4]) *and hydrogen atoms* (×10[3]) *and their equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$U_{eq} = \textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	U_{eq}/U_{iso}(Å^2)
C(1)	2585 (7)	478 (7)	9829 (4)	0·0416 (20)
C(2)	2641 (8)	−1300 (8)	9589 (4)	0·0525 (23)
C(3)	2110 (8)	−2557 (8)	10167 (4)	0·0520 (23)
C(4)	1567 (7)	−2050 (7)	10964 (4)	0·0428 (20)
C(5)	1507 (7)	−296 (8)	11215 (4)	0·0452 (20)
C(6)	2033 (8)	957 (8)	10641 (4)	0·0453 (21)
C(7)	3643 (9)	1405 (9)	8474 (4)	0·0505 (24)
C(8)	4280 (8)	2985 (8)	8056 (4)	0·0460 (22)
N(1)	1005 (7)	−3356 (7)	11556 (4)	0·0589 (21)
N(2)	4032 (8)	4535 (7)	8409 (4)	0·0638 (23)
O(1)	1006 (8)	−4903 (7)	11332 (4)	0·1006 (27)
O(2)	531 (7)	−2910 (6)	12273 (3)	0·0784 (20)
O(3)	3016 (5)	1823 (5)	9307 (3)	0·0539 (16)
O(4)	5012 (6)	2804 (5)	7379 (3)	0·0638 (17)
H(2)	307 (6)	−164 (7)	896 (3)	0·015 (5)
H(5)	95 (7)	6 (8)	1186 (4)	0·020 (5)
H(3)	217 (8)	−408 (8)	1008 (4)	0·027 (7)
H(6)	198 (7)	227 (7)	1076 (4)	0·018 (6)
H(10)	355 (8)	448 (9)	905 (5)	0·032 (8)
H(101)	441 (9)	558 (9)	812 (4)	0·030 (8)
H(71)	449 (8)	47 (8)	847 (4)	0·023 (7)
H(72)	285 (8)	82 (9)	809 (4)	0·027 (8)

Table 2. *Bond lengths* (Å) *and angles* (°), *with e.s.d.'s in parentheses*

C(1)–C(2)	1·398 (8)	C(4)–N(1)	1·433 (7)
C(2)–C(3)	1·385 (8)	N(1)–O(1)	1·220 (7)
C(3)–C(4)	1·366 (8)	N(1)–O(2)	1·221 (6)
C(4)–C(5)	1·385 (8)	C(1)–O(3)	1·353 (6)
C(5)–C(6)	1·377 (8)	O(3)–C(7)	1·426 (6)
C(6)–C(1)	1·382 (7)	C(7)–C(8)	1·469 (8)
C(8)–O(4)	1·231 (7)	C(8)–N(2)	1·313 (8)
C(2)–H(2)	1·07 (5)	C(3)–H(3)	1·17 (6)
C(5)–H(5)	1·14 (5)	C(6)–H(6)	1·01 (5)
C(7)–H(71)	0·97 (6)	C(7)–H(72)	0·89 (6)
N(2)–H(10)	1·07 (7)	N(2)–H(101)	0·97 (7)

C(1)–C(2)–C(3)	118·9 (6)	C(3)–C(4)–N(1)	119·6 (5)
C(2)–C(3)–C(4)	119·9 (6)	C(5)–C(4)–N(1)	118·4 (5)
C(3)–C(4)–C(5)	122·0 (5)	C(4)–N(1)–O(1)	119·1 (6)
C(4)–C(5)–C(6)	118·3 (5)	C(4)–N(1)–O(2)	119·9 (5)
C(5)–C(6)–C(1)	120·9 (6)	O(2)–N(1)–O(1)	121·0 (6)
C(6)–C(1)–C(2)	120·1 (5)	C(2)–C(1)–O(3)	124·3 (5)
C(6)–C(1)–O(3)	115·7 (5)	C(1)–O(3)–C(7)	118·2 (5)
O(3)–C(7)–C(8)	111·3 (5)	C(7)–C(8)–O(4)	118·5 (6)
C(7)–C(8)–N(2)	119·0 (5)	N(2)–C(8)–O(4)	122·5 (6)

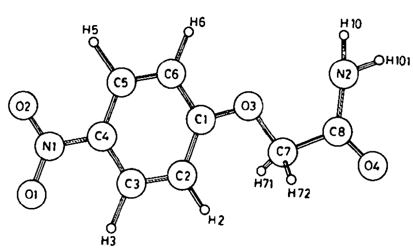

Fig. 1. Structure of PNPA, showing the numbering of the atoms.

The bond lengths and bond angles of the molecule are comparable with those found in *o*-chlorophenoxyacetamide (2CLPA) (Lakshmi Rao, Rao & Padmanabhan, 1987), 2-methyl-4-chlorophenoxyacetamide (2M4CLPA) (Lakshmi Rao, Seshadri & Rao, 1987), *p*-chlorophenoxyacetamide (4CLPA) (Krishnasamy, 1983) and *o*-cresoxyacetamide (2MPA) (Krishnasamy, 1983). In the nitro group of the molecule, the bonds N(1)—O(1) and N(1)—O(2) (Fig. 1) are comparable to the N—O lengths 1·210 (1) and 1·223 (7) Å in *p*-nitrophenoxyacetic acid (PNP) (Vijay Kumar & Rao, 1980) and 1·224 and 1·219 Å in *p*-nitrobenzoic acid (Colapietro & Domenicano, 1977) respectively. The NO_2 group is symmetrical about the bond C(4)—N(1), while the N atom is out of the benzene plane by 0·021 Å. The distances O(1)—C(3) and O(2)—C(5) are equal. The dihedral angle between the plane of the benzene ring and the plane of the nitro group is only 1·6 (1)°. This shows that the symmetrical nitro group has not undergone any significant rotation about C(4)—N(1). The two substituent groups NO_2 and O(3) (the electron-withdrawing groups) have produced small deformations of the skeletal geometry of the ring. This is reflected by slightly larger values of the angles in the benzene ring at the sites of substitution C(4) and C(1). These changes are similar to those observed in PNP.

The bond O(3)—C(7) is 1·426 (6) Å, which is similar to those reported in PNP [1·41 (1)], 2,4-dichlorophenoxyacetic acid (2,4D) (Smith, Kennard & White, 1976*a*) [1·423 (5)] and 2,4,5-trichlorophenoxyacetic acid (2,4,5T) (Smith, Kennard & White, 1976*b*) [1·417 (8) Å] *etc*. But the bond distance C(7)—C(8) is significantly shorter than the value of 1·520 (6) Å observed in 2,4,5T for C(7)—C(8). Similar shortening is observed in 2,4D and in PNP [1·477 (10)] and 2-chlorophenoxyacetic acid (1·41 Å) (Kennard & Smith, 1981).

The molecules are hydrogen bonded as shown in Fig. 2, with N—H···O bonds of length 2·886 (3) Å. The bonds are formed by both the nitrogen and the oxygen atoms of a molecule (x,y,z) taking part in bond formation with the oxygen atom $(1-x, \frac{1}{2}+y, \frac{3}{2}-z)$ and the nitrogen atom $(1-x, -\frac{1}{2}+y, \frac{3}{2}-z)$ of two molecules which are related by a translation **b**. The first molecule is related by the symmetry element $2_1[\frac{1}{2}y\frac{3}{4}]$ to the latter two molecules.

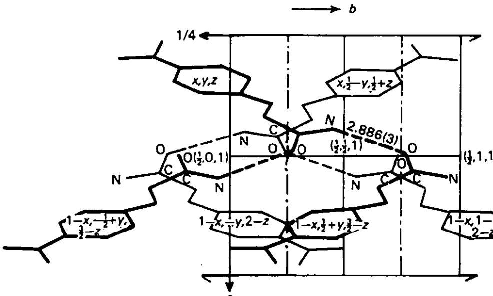

Fig. 2. Projection of the title compound in the *x*–*y* plane showing the N—H···O bond formation.

The hydrogen bonds are in two sets and run in zigzag fashion symmetrically across the centres of symmetry $(\frac{1}{2},0,1)$, $(\frac{1}{2},\frac{1}{2},1)$ and $(\frac{1}{2},1,1)$. The features of the hydrogen-bond network in this structure resemble those reported in monofluoroacetamide (Hughes & Small, 1962). In the latter case, however, both the hydrogen atoms of the amide are used in the network of hydrogen bonds running through the lattice. The N—H···O bonds of the present structure compare with those found in 2M4CLPA, 2CLPA, 4CLPA and 2MPA.

The temperature factors of the O atoms of the nitro group are larger than those of the O and N of the amide group. This shows that the thermal vibrations of the molecules are influenced by the hydrogen bonds. The dihedral angle between the planar benzene ring and the phenoxyacetamide group is 5·2 (3)°. This is comparable to those found in 2M4CLPA, 2CLPA, 4CLPA and 2MPA and acetic acids (Smith & Kennard, 1979), but is significantly different from the 85·2° observed in 2,4D. There are no significant short contacts other than the hydrogen bonds.

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Structure of Garcinone B*

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Abstract. 5,9,11-Trihydroxy-3,3-dimethyl-10-(3-methyl-2-butenyl)-3*H*,12*H*-pyrano[3,2-*a*]xanthen-12-one monohydrate, $C_{23}H_{22}O_6.H_2O$, $M_r = 412{\cdot}4$, triclinic, $P\bar{1}$, $a = 8{\cdot}306$ (1), $b = 8{\cdot}543$ (1), $c = 14{\cdot}695$ (1) Å, $\alpha = 95{\cdot}12$ (1), $\beta = 101{\cdot}44$ (1), $\gamma = 86{\cdot}91$ (1)°, $V = 1017{\cdot}2$ (2) Å^3, $Z = 2$, $D_m = 1{\cdot}342$ (3) (flotation), $D_x = 1{\cdot}346$ Mg m^{-3}, Cu $K\alpha$ radiation, $\lambda = 1{\cdot}5418$ Å, $\mu = 0{\cdot}787$ mm^{-1}, $F(000) = 436$, $T = 294$ K, final $R = 0{\cdot}062$ for 2652 reflections [$I > 3\sigma(I)$]. The xanthone ring system is nearly planar. The dihedral angle between the xanthone ring plane and the plane of the isoprenyl side chain is 111·5 (2)°. The molecules are linked by hydrogen bonds *via* a molecule of water.

Introduction. Several naturally occurring xanthones isolated from plants have created considerable interest in terms of their biological importance and potential as drugs (Gopalakrishnan, Shankaranarayanan, Nazimudeen, Viswanathan & Kameswaran, 1980; Shankaranarayanan, Gopalakrishnan & Kameswaran, 1979). The title compound is one of the constituents of the mangosteen fruit hulls (*Garcinia mangostana*, Guttiferae), which have been in use as a folk medicine for the treatment of dysentery (Yates & Stout, 1958). The X-ray structural analysis of this compound was carried out in order to ascertain its conformation and molecular geometry.

Experimental. The fruit hulls after extraction with petroleum ether were further extracted with benzene. The benzene extract was chromatographed on silica gel (4:1 benzene/chloroform), further purified by prep. TLC to yield the title compound, which is identical in all respects, *viz* m.p., TLC and IR, to an authentic specimen. Recrystallization of the compound from methanol gave yellow needles. Crystal dimensions 0·11 × 0·07 × 0·10 mm. CAD-4 diffractometer, monochromated Cu $K\alpha$ radiation. Cell parameters from least-squares refinement of setting angles of 25 reflections (θ range 25–35°). Intensity data for $0 < \theta < 55°$, $\omega/2\theta$ scans, two check reflections for every 98 reflections did not vary significantly over the course of the data collection. Lp but no absorption correction, 3939 reflections (h 0→9, k −9→9, l −16→16) of which 2652 [$I > 3\sigma(I)$] used in calculations. Direct methods with *MULTAN*80 (Main, fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); an E map calculated from the set of phases with the highest figure of merit revealed the structure. Full-matrix least-squares refinement on F. Anisotropic temperature factors for C and O atoms, isotropic for H. H positions except those bound to the water molecule were revealed from a difference map. $w = 1{\cdot}032/\sigma^2(F_o) + 0{\cdot}003F_o^2$, final $R = 0{\cdot}062$, $wR = 0{\cdot}067$ for 2652 reflections. $R = 0{\cdot}072$ for all reflections. $S = 1{\cdot}51$; final ΔF map featureless, $(\Delta/\sigma)_{max} = 0{\cdot}51$, final $\Delta\rho$ excursions −0·23–0·32 e Å^{-3}. No corrections for secondary extinction, scattering factors as in *SHELX* (Sheldrick, 1976). Calculations of geometrical data and crystal packing were computed by the program *PARST* (Nardelli, 1983). Calculations performed on an IBM 370 computer.

Discussion. Final positional parameters of the non-H atoms are given in Table 1.‡ Bond distances and angles are given in Fig. 1. A perspective view of the molecule is in Fig. 2. The values of the bond lengths and angles are similar to those observed in other comparable systems (Yoshida, Tanaka, Ashida, Kakudo, Fukuyama & Katsube, 1979; Soderholm, Sonnerstam, Norrestam & Palm, 1976).

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‡ Lists of structure factors, anisotropic thermal parameters of the non-H atoms, hydrogen-bond distances and angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44048 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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