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# Structures of Five ω-Phthalimidoaliphatic Carboxylic Acids†

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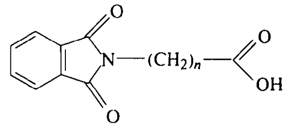
## Abstract

The crystal structures of five ω-phthalimidoaliphatic carboxylic acids are reported: 2-phthalimidoethanoic acid, $C_{10}H_7NO_4$ (I), 3-phthalimidopropanoic acid, $C_{11}H_9NO_4$ (II), 4-phthalimidobutanoic acid, $C_{12}H_{11}NO_4$ (III), 5-phthalimidopentanoic acid, $C_{13}H_{13}NO_4$ (IV), and 6-phthalimidohexanoic acid, $C_{14}H_{15}NO_4$ (V). Hydrogen bonding within the crystal structures of (I) and (II) produces chains of molecules. In (IV) carboxylic acid dimers are formed through hydrogen bonding between centrosymmetric pairs. The structures of (III) and (V) show a similar motif to (IV) except that the hydrogen bonds are between symmetry-independent molecules.

## Comment

As part of a general study on the stability of organic peracids and peracid salts (Kariuki & Jones, 1990), and in particular the role of hydrogen bonding in controlling the crystal structure of peracid and acid amides (Feeder & Jones, 1992), we report here the crystal structures of five ω-phthalimidoaliphatic carboxylic acids.



(I) $n = 1$
(II) $n = 2$
(III) $n = 3$
(IV) $n = 4$
(V) $n = 5$

The bond distances and angles observed for all five structures are similar to those reported for phthalimide (Matzat, 1972), *N*-(4-bromophenyl)phthalimide (Ribar, Stankovic & Halasi, 1976) and *N*-(4-iodophenyl)phthalimide (Ribar, Stankovic, Herak, Halasi & Djuric, 1974). In addition, this last structure is isostructural with (I).

The two symmetry-independent molecules in (III) and (V) have similar bond distances and angles, and differ only in their conformation. Also for these structures long C═O and short C—O bonds are observed suggesting a disordered model for the carboxyl groups (Leiserowitz, 1976). No such disorder is seen for (I), (II) and (IV).

The molecules of (I) and (II) contain two planar groups. The phthalimido moiety *A*, N(1),C(8),O(8),C(7),O(7),C(6)–C(1), is planar to within 0.052 Å for (I) and 0.042 Å for (II); the aliphatic acid group *B* in (I), H(101),O(101),C(10),O(100),C(9), is planar to within 0.007 Å. In (II) the aliphatic acid group *B*, H(111),O(111),C(11),O(110),C(10), is planar to within 0.089 Å and the group, N(1),C(9),C(10),C(11), is planar to within 0.093 Å. Table 4 gives details of the *AB* dihedral angles. Molecules of (III), (IV) and (V) on the other hand may be considered as consisting of three moieties. The phthalimido moiety *A* is again planar to within 0.030 and 0.044 Å for molecules 1 and 2 of (III), 0.023 Å for (IV), and 0.016 and 0.032 Å for molecules 1 and 2 of (V), respectively. The second group is the alkyl chain *C*, planar to within 0.003 Å for (IV), and 0.039 and 0.067 Å for molecules 1 and 2 of (V), respectively. The dihedral angles are summarized in Table 4 along with the angles made between these two groups and the COO group, *D*. The group consisting of the alkyl chain, N atom and carboxyl C atom is planar to within 0.404 and 0.416 Å for molecules 1 and 2 of (III), 0.133 Å for (IV) and 0.126 and 0.115 Å for molecules 1 and 2 of (V), respectively.

Fig. 2 is a *PLUTO* (Motherwell & Clegg, 1978) plot of a partial packing diagram for (I). The diagram illustrates that molecules related by a twofold-

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† It should be noted that the delay in publication of this paper was not due to problems with the structures, or to the slow response of the authors.

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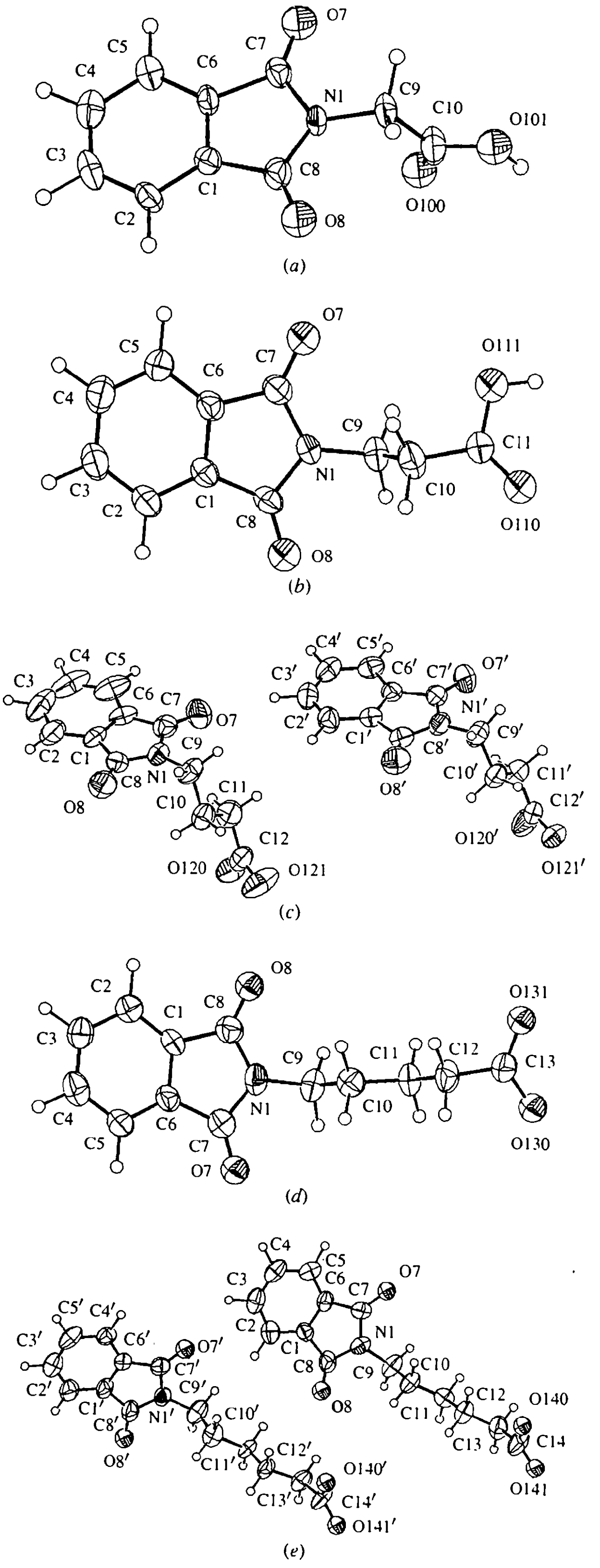

Fig. 1. Atom-numbering scheme for (*a*) (I), (*b*) (II), (*c*) (III), (*d*) (IV) and (*e*) (V). Ellipsoids are drawn at the 50% probability level.

screw axis running parallel to the *a* axis are hydrogen bonded to form chains, through acid proton to phthalimido carbonyl contacts [O(101)—H(101)···O(7) 1.853, O(101)···O(7) 2.697 Å]. These chains are held together by van der Waals forces in an identical arrangement to that reported for *N*-(4-iodophenyl)phthalimide (Ribar, Stankovic, Herak, Halasi & Djuric, 1974). This seems to suggest that the phthalimido groups dominate the structure of (I) and the iodo compound.

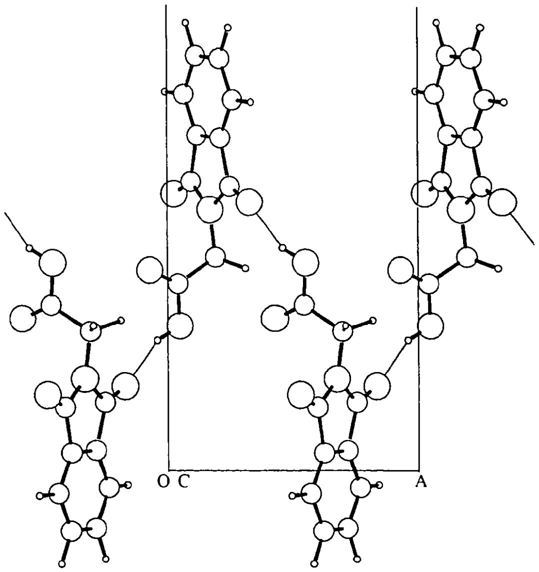

Fig. 2. A partial-packing diagram showing the chains of molecules generated by hydrogen bonding for (I) as viewed down the *c* axis.

Fig. 3 illustrates the hydrogen-bonding network seen in the structure of (II). As for (I), chains of molecules are generated by the hydrogen bonding of an acid proton to a phthalimido carbonyl group [O(111)—H(111)···O(8) 1.822, O(111)···O(7) 2.689 Å], this time linking molecules related by a *c* glide. The chains are stacked along the *b* axis, held together by some short C—H···O contacts involving the other two carbonyl groups, to form sheets.

Hydrogen bonding in the structures of (III), (IV) and (V) generates carboxylic acid dimers, a familiar motif observed for such functional groups (Leiserowitz, 1976). In the structures of (III) and (V) the dimers are formed between the symmetry-independent molecules [(III) O(121)···O(120′) 2.612, O(120)···O(121′) 2.598 Å; (V) O(140)···O(141′) 2.627, O(141)···O(140′) 2.607 Å]. These dimers are held together by van der Waals forces and some short C—H···O contacts involving the phthalimido carbonyl group. The packing arrangements for these dimers are shown in Figs. 4 and 5. The molecules connected through hydrogen bonding in the structure of (IV) are related by a centre of symmetry [O(130)···O(131) 2.620 Å]. The dimers are again held together by van der Waals forces and some short C—H···O contacts involving the phthalimido carbonyl group, in a manner similar to that observed for (V).

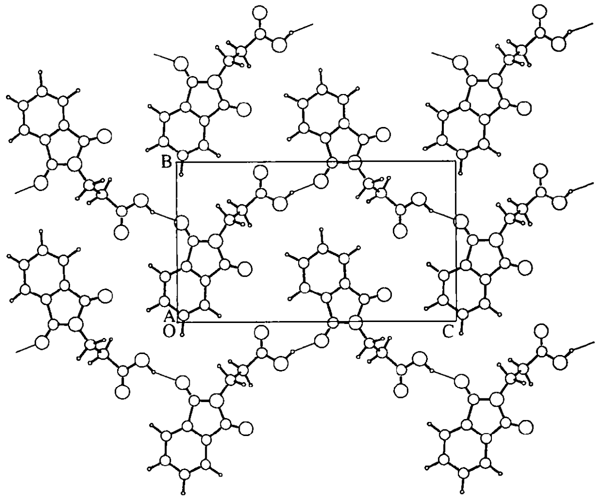

Fig. 3. Packing diagram showing the chains of molecules generated by hydrogen bonding for (II) as viewed down the *a* axis.

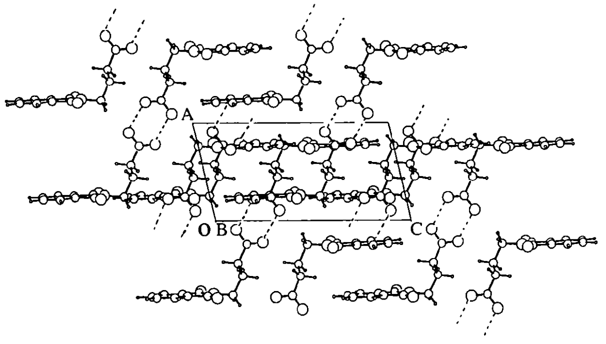

Fig. 4. Packing of the hydrogen-bonded dimers in (III).

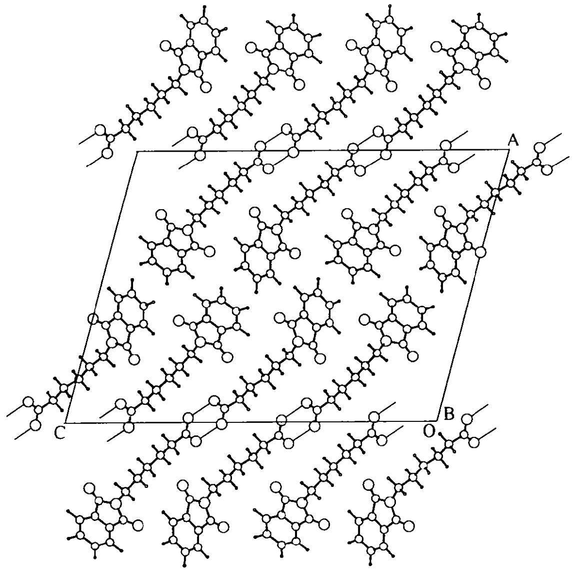

Fig. 5. Packing of the hydrogen-bonded dimers in (V).

## Experimental

The compounds were provided by Interox plc and the crystals were obtained from ethanol solutions.

### Compound (I)

*Crystal data*

$C_{10}H_7NO_4$
$M_r = 205.1$
Orthorhombic
*Pbca*
$a = 7.573\ (2)$ Å
$b = 21.152\ (5)$ Å
$c = 11.756\ (2)$ Å
$V = 1883\ (1)$ Å$^3$
$Z = 8$
$D_x = 1.45$ Mg m$^{-3}$
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10$–$15°$
$\mu = 0.114$ mm$^{-1}$
$T = 295$ K
Rod
0.4 × 0.2 × 0.2 mm
White

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3150 measured reflections
3150 independent reflections
907 observed reflections $[F > 3\sigma(F)]$
$\theta_{max} = 30°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 29$
$l = 0 \rightarrow 16$
2 standard reflections monitored every 100 reflections
intensity decay: none

*Refinement*

Refinement on $F$
$R = 0.066$
$wR = 0.066$
$S = 1.949$
907 reflections
142 parameters
Unit weights applied
$(\Delta/\sigma)_{max} = 0.696$
$\Delta\rho_{max} = 0.30$ e Å$^{-3}$
$\Delta\rho_{min} = -0.32$ e Å$^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

### Compound (II)

*Crystal data*

$C_{11}H_9NO_4$
$M_r = 219.1$
Monoclinic
$P2_1/c$
$a = 7.079\ (4)$ Å
$b = 8.949\ (5)$ Å
$c = 15.893\ (7)$ Å
$\beta = 98.84\ (4)°$
$V = 995\ (1)$ Å$^3$
$Z = 4$
$D_x = 1.46$ Mg m$^{-3}$
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 8$–$14°$
$\mu = 0.113$ mm$^{-1}$
$T = 295$ K
Plate
0.3 × 0.3 × 0.1 mm
White

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1951 measured reflections
1886 independent reflections
1067 observed reflections $[F > 3\sigma(F)]$

$R_{\rm int} = 0.01$
$\theta_{\rm max} = 25°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 18$
2 standard reflections monitored every 100 reflections intensity decay: none

*Refinement*

Refinement on $F$
$R = 0.046$
$wR = 0.046$
$S = 0.756$
1067 reflections
152 parameters
Unit weights applied

$(\Delta/\sigma)_{\rm max} = 0.072$
$\Delta\rho_{\rm max} = 0.16$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.20$ e Å$^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

**Compound (III)**

*Crystal data*

$C_{12}H_{11}NO_4$
$M_r = 233.1$
Triclinic
$P\overline{1}$
$a = 7.336$ (6) Å
$b = 10.950$ (3) Å
$c = 14.693$ (5) Å
$\alpha = 100.79$ (3)°
$\beta = 104.50$ (5)°
$\gamma = 83.53$ (6)°
$V = 1119$ (1) Å$^3$
$Z = 4$
$D_x = 1.38$ Mg m$^{-3}$

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 8$–$12°$
$\mu = 0.105$ mm$^{-1}$
$T = 295$ K
Rod
0.4 × 0.2 × 0.15 mm
White

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4112 measured reflections
3944 independent reflections
1771 observed reflections $[F > 3\sigma(F)]$

$R_{\rm int} = 0.03$
$\theta_{\rm max} = 25°$
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = 0 \rightarrow 17$
2 standard reflections monitored every 100 reflections intensity decay: none

*Refinement*

Refinement on $F$
$R = 0.064$
$wR = 0.064$
$S = 1.115$
1771 reflections
315 parameters
Unit weights applied
$(\Delta/\sigma)_{\rm max} = 0.136$

$\Delta\rho_{\rm max} = 0.28$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.23$ e Å$^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

**Compound (IV)**

*Crystal data*

$C_{13}H_{13}NO_4$
$M_r = 247.1$
Monoclinic
$P2_1/n$
$a = 6.574$ (4) Å
$b = 4.899$ (2) Å
$c = 37.413$ (13) Å
$\beta = 95.18$ (3)°
$V = 1200$ (1) Å$^3$
$Z = 4$
$D_x = 1.37$ Mg m$^{-3}$

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 9$–$14°$
$\mu = 0.102$ mm$^{-1}$
$T = 295$ K
Plate
0.5 × 0.3 × 0.1 mm
White

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2388 measured reflections
2365 independent reflections
891 observed reflections $[F > 3\sigma(F)]$

$R_{\rm int} = 0.05$
$\theta_{\rm max} = 25°$
$h = -7 \rightarrow 7$
$k = 0 \rightarrow 5$
$l = 0 \rightarrow 44$
2 standard reflections monitored every 100 reflections intensity decay: none

*Refinement*

Refinement on $F$
$R = 0.072$
$wR = 0.072$
$S = 1.595$
891 reflections
168 parameters
Unit weights applied

$(\Delta/\sigma)_{\rm max} = 0.075$
$\Delta\rho_{\rm max} = 0.26$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.25$ e Å$^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

**Compound (V)**

*Crystal data*

$C_{14}H_{15}NO_4$
$M_r = 261.1$
Monoclinic
$P2_1/c$
$a = 21.055$ (8) Å
$b = 4.662$ (1) Å
$c = 28.067$ (9) Å
$\beta = 105.22$ (3)°
$V = 2658$ Å$^3$
$Z = 8$
$D_x = 1.30$ Mg m$^{-3}$

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 8$–$12°$
$\mu = 0.058$ mm$^{-1}$
$T = 295$ K
Rod
0.3 × 0.1 × 0.1 mm
White

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
5192 measured reflections
5095 independent reflections
1242 observed reflections $[F > 3\sigma(F)]$

$R_{\rm int} = 0.05$
$\theta_{\rm max} = 25°$
$h = -25 \rightarrow 25$
$k = 0 \rightarrow 4$
$l = 0 \rightarrow 33$
2 standard reflections monitored every 100 reflections intensity decay: none

### *Refinement*

Refinement on $F$
$R = 0.080$
$wR = 0.080$
$S = 1.835$
1242 reflections
354 parameters
Unit weights applied

$(\Delta/\sigma)_{\rm max} = 0.207$
$\Delta\rho_{\rm max} = 0.34$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.29$ e Å$^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å$^2$) for (I), (II), (III), (IV) and (V)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $U_{\rm eq}$ |
|---|---|---|---|---|
| (I) | | | | |
| N(1) | 0.1722 (7) | 0.3691 (2) | 0.8743 (5) | 0.033 (3) |
| O(7) | 0.3301 (7) | 0.3796 (2) | 1.0414 (4) | 0.047 (3) |
| O(8) | 0.0274 (7) | 0.3915 (2) | 0.7065 (4) | 0.053 (3) |
| O(100) | −0.0713 (9) | 0.2837 (2) | 0.9588 (6) | 0.106 (5) |
| O(101) | 0.0488 (8) | 0.2057 (2) | 0.8624 (5) | 0.058 (3) |
| C(1) | 0.1202 (8) | 0.4741 (3) | 0.8380 (5) | 0.031 (3) |
| C(2) | 0.0675 (8) | 0.5318 (3) | 0.7946 (6) | 0.039 (4) |
| C(3) | 0.1160 (9) | 0.5852 (3) | 0.8547 (6) | 0.045 (4) |
| C(4) | 0.2120 (9) | 0.5813 (3) | 0.9543 (6) | 0.045 (4) |
| C(5) | 0.2635 (9) | 0.5235 (3) | 0.9995 (6) | 0.039 (3) |
| C(6) | 0.2145 (8) | 0.4706 (3) | 0.9385 (5) | 0.033 (4) |
| C(7) | 0.2508 (9) | 0.4021 (3) | 0.9641 (6) | 0.033 (3) |
| C(8) | 0.0953 (9) | 0.4089 (3) | 0.7937 (7) | 0.038 (4) |
| C(9) | 0.1944 (9) | 0.3021 (2) | 0.8544 (6) | 0.038 (3) |
| C(10) | 0.0419 (10) | 0.2646 (3) | 0.8991 (7) | 0.048 (4) |
| (II) | | | | |
| N(1) | 0.6850 (4) | −0.0063 (3) | 0.6410 (2) | 0.058 (2) |
| O(7) | 0.6635 (4) | 0.1691 (3) | 0.7448 (2) | 0.061 (2) |
| O(8) | 0.7333 (4) | −0.1208 (3) | 0.5174 (2) | 0.054 (2) |
| O(110) | 0.8496 (5) | −0.4359 (3) | 0.8034 (2) | 0.071 (2) |
| O(111) | 0.7693 (4) | −0.2365 (3) | 0.8718 (2) | 0.057 (2) |
| C(1) | 0.7412 (5) | 0.1509 (4) | 0.5334 (2) | 0.037 (2) |
| C(2) | 0.7735 (5) | 0.2131 (4) | 0.4572 (2) | 0.047 (2) |
| C(3) | 0.7766 (6) | 0.3674 (5) | 0.4533 (2) | 0.052 (3) |
| C(4) | 0.7520 (6) | 0.4561 (5) | 0.5222 (3) | 0.055 (3) |
| C(5) | 0.7207 (6) | 0.3923 (4) | 0.5987 (2) | 0.050 (2) |
| C(6) | 0.7168 (5) | 0.2387 (4) | 0.6022 (2) | 0.039 (2) |
| C(7) | 0.6846 (5) | 0.1393 (4) | 0.6729 (2) | 0.041 (2) |
| C(8) | 0.7223 (5) | −0.0081 (4) | 0.5582 (2) | 0.038 (2) |
| C(9) | 0.6596 (5) | −0.1376 (4) | 0.6916 (2) | 0.044 (2) |
| C(10) | 0.8488 (6) | −0.1974 (5) | 0.7360 (2) | 0.054 (3) |
| C(11) | 0.8229 (5) | −0.3047 (4) | 0.8054 (2) | 0.043 (2) |
| (III) Molecule 1 | | | | |
| N(1) | 0.2521 (7) | 0.7805 (5) | 0.4609 (3) | 0.053 (4) |
| O(7) | 0.2763 (8) | 0.5674 (5) | 0.4347 (4) | 0.090 (4) |
| O(8) | 0.2180 (8) | 0.9846 (5) | 0.4396 (4) | 0.087 (4) |
| O(120) | 0.8990 (7) | 0.7298 (5) | 0.6632 (3) | 0.078 (3) |
| O(121) | 0.7746 (7) | 0.8063 (7) | 0.7837 (4) | 0.114 (5) |
| C(1) | 0.2338 (9) | 0.8146 (7) | 0.3107 (4) | 0.055 (4) |
| C(2) | 0.2128 (11) | 0.8674 (9) | 0.2306 (5) | 0.087 (6) |
| C(3) | 0.2148 (13) | 0.7847 (13) | 0.1461 (7) | 0.114 (8) |
| C(4) | 0.2356 (13) | 0.6593 (13) | 0.1452 (7) | 0.104 (8) |
| C(5) | 0.2539 (11) | 0.6058 (9) | 0.2257 (6) | 0.082 (6) |
| C(6) | 0.2528 (9) | 0.6888 (7) | 0.3079 (5) | 0.051 (4) |
| C(7) | 0.2639 (9) | 0.6644 (7) | 0.4057 (5) | 0.058 (5) |
| C(8) | 0.2331 (9) | 0.8751 (6) | 0.4093 (5) | 0.055 (5) |
| C(9) | 0.2430 (10) | 0.8035 (7) | 0.5612 (4) | 0.068 (5) |
| C(10) | 0.4402 (10) | 0.8378 (6) | 0.6298 (5) | 0.064 (5) |
| C(11) | 0.5636 (11) | 0.7233 (7) | 0.6341 (5) | 0.075 (5) |
| C(12) | 0.7580 (11) | 0.7553 (8) | 0.6995 (5) | 0.073 (6) |
| (III) Molecule 2 | | | | |
| N(1′) | 0.7419 (7) | 0.7135 (5) | 1.0976 (4) | 0.057 (4) |
| O(7′) | 0.7156 (8) | 0.9220 (5) | 1.1608 (4) | 0.084 (4) |
| O(8′) | 0.7812 (8) | 0.5000 (5) | 1.0826 (3) | 0.083 (4) |
| O(120′) | 0.0962 (7) | 0.8429 (5) | 0.9075 (3) | 0.082 (4) |
| O(121′) | 0.2207 (6) | 0.7625 (4) | 0.7854 (3) | 0.063 (3) |
| C(1′) | 0.7615 (8) | 0.6323 (6) | 1.2327 (4) | 0.046 (4) |
| C(2′) | 0.7781 (10) | 0.5561 (6) | 1.2993 (5) | 0.063 (5) |
| C(3′) | 0.778 (10) | 0.6120 (7) | 1.3911 (5) | 0.069 (5) |
| C(4′) | 0.7670 (9) | 0.7397 (7) | 1.4163 (5) | 0.063 (5) |
| C(5′) | 0.7473 (9) | 0.8167 (6) | 1.3494 (5) | 0.056 (4) |
| C(6′) | 0.7453 (8) | 0.7595 (6) | 1.2570 (4) | 0.048 (4) |
| C(7′) | 0.7306 (9) | 0.8150 (7) | 1.1706 (5) | 0.055 (5) |
| C(8′) | 0.7624 (9) | 0.6013 (7) | 1.1298 (5) | 0.058 (5) |
| C(9′) | 0.7526 (10) | 0.7228 (8) | 1.0008 (5) | 0.073 (5) |
| C(10′) | 0.5583 (10) | 0.7071 (7) | 0.9290 (5) | 0.064 (5) |
| C(11′) | 0.4324 (10) | 0.8222 (7) | 0.9377 (5) | 0.067 (5) |
| C(12′) | 0.2392 (10) | 0.8061 (6) | 0.8708 (5) | 0.058 (5) |
| (IV) | | | | |
| N(1) | 0.3410 (10) | 0.1679 (16) | 0.1404 (2) | 0.051 (4) |
| O(7) | 0.6175 (9) | 0.2728 (17) | 0.1095 (2) | 0.089 (5) |
| O(8) | 0.1130 (9) | 0.1597 (15) | 0.1837 (2) | 0.071 (4) |
| O(130) | −0.2698 (9) | −0.3469 (16) | −0.0046 (2) | 0.077 (4) |
| O(131) | −0.4921 (9) | −0.2720 (16) | 0.0345 (2) | 0.083 (5) |
| C(1) | 0.4080 (10) | 0.4553 (17) | 0.1884 (2) | 0.040 (5) |
| C(2) | 0.4029 (12) | 0.6068 (19) | 0.2195 (2) | 0.054 (6) |
| C(3) | 0.5637 (12) | 0.7890 (19) | 0.2277 (2) | 0.052 (5) |
| C(4) | 0.7141 (12) | 0.828 (2) | 0.2048 (2) | 0.057 (5) |
| C(5) | 0.7183 (12) | 0.673 (2) | 0.1736 (2) | 0.056 (6) |
| C(6) | 0.5618 (11) | 0.4928 (18) | 0.1659 (2) | 0.043 (5) |
| C(7) | 0.5236 (12) | 0.301 (2) | 0.1350 (2) | 0.053 (6) |
| C(8) | 0.2648 (12) | 0.2483 (19) | 0.1722 (2) | 0.052 (5) |
| C(9) | 0.2468 (12) | −0.0358 (19) | 0.1160 (2) | 0.054 (5) |
| C(10) | 0.0865 (12) | 0.0904 (19) | 0.0882 (2) | 0.050 (5) |
| C(11) | −0.0342 (11) | −0.1266 (18) | 0.0670 (2) | 0.047 (5) |
| C(12) | −0.1921 (12) | −0.0043 (19) | 0.0397 (2) | 0.052 (5) |
| C(13) | −0.3267 (12) | −0.217 (2) | 0.0218 (2) | 0.013 (6) |
| (V) Molecule 1 | | | | |
| N(1) | 0.2954 (6) | 0.004 (3) | 0.4315 (4) | 0.054 (8) |
| O(8) | 0.2312 (5) | 0.202 (3) | 0.3600 (4) | 0.085 (8) |
| O(7) | 0.3823 (5) | −0.120 (3) | 0.4984 (4) | 0.073 (13) |
| O(140) | 0.0781 (7) | 0.078 (4) | 0.6148 (6) | 0.142 (13) |
| O(141) | −0.0062 (7) | −0.154 (4) | 0.5791 (5) | 0.165 (15) |
| C(6) | 0.3933 (7) | 0.222 (3) | 0.4347 (5) | 0.054 (10) |
| C(5) | 0.4564 (7) | 0.314 (4) | 0.4450 (6) | 0.058 (11) |
| C(4) | 0.4733 (8) | 0.507 (4) | 0.4136 (7) | 0.068 (10) |
| C(3) | 0.4277 (9) | 0.605 (4) | 0.3723 (7) | 0.077 (12) |
| C(2) | 0.3631 (8) | 0.507 (4) | 0.3600 (6) | 0.064 (12) |
| C(1) | 0.3470 (7) | 0.316 (4) | 0.3921 (5) | 0.049 (10) |
| C(8) | 0.2820 (8) | 0.180 (4) | 0.3889 (5) | 0.058 (11) |
| C(7) | 0.3588 (6) | 0.012 (4) | 0.4597 (5) | 0.050 (10) |
| C(9) | 0.2428 (8) | −0.182 (4) | 0.4439 (6) | 0.073 (12) |
| C(10) | 0.2058 (7) | −0.007 (4) | 0.4748 (5) | 0.058 (10) |
| C(11) | 0.1613 (7) | −0.199 (3) | 0.4943 (5) | 0.049 (9) |
| C(12) | 0.1277 (7) | −0.037 (4) | 0.5283 (6) | 0.072 (12) |
| C(13) | 0.0829 (7) | −0.232 (4) | 0.5478 (6) | 0.073 (12) |
| C(14) | 0.0488 (10) | −0.069 (5) | 0.5819 (6) | 0.085 (15) |
| (V) Molecule 2 | | | | |
| N(1′) | 0.2966 (6) | 0.898 (3) | 0.1935 (5) | 0.054 (8) |
| O(8′) | 0.2385 (5) | 0.937 (3) | 0.1109 (4) | 0.086 (9) |
| O(7′) | 0.3741 (6) | 0.952 (3) | 0.2675 (4) | 0.083 (9) |
| O(140′) | 0.0680 (7) | 0.888 (4) | 0.3647 (6) | 0.169 (16) |
| O(141′) | −0.0159 (6) | 0.670 (4) | 0.3270 (5) | 0.148 (14) |
| C(6′) | 0.3807 (7) | 1.211 (3) | 0.1931 (5) | 0.041 (9) |
| C(5′) | 0.4356 (7) | 1.375 (3) | 0.2044 (5) | 0.052 (10) |
| C(4′) | 0.4494 (8) | 1.543 (4) | 0.1673 (7) | 0.071 (13) |
| C(3′) | 0.4078 (8) | 1.525 (4) | 0.1198 (6) | 0.068 (13) |
| C(2′) | 0.3525 (7) | 1.353 (4) | 0.1076 (6) | 0.072 (11) |
| C(1′) | 0.3395 (7) | 1.202 (4) | 0.1458 (5) | 0.058 (11) |
| C(8′) | 0.2853 (8) | 0.996 (4) | 0.1457 (6) | 0.059 (11) |
| C(7′) | 0.3539 (7) | 1.011 (4) | 0.2238 (6) | 0.059 (11) |
| C(9′) | 0.2517 (10) | 0.665 (5) | 0.2100 (8) | 0.072 (12) |
| C(10′) | 0.2046 (7) | 0.844 (3) | 0.2329 (6) | 0.067 (11) |
| C(11′) | 0.1627 (6) | 0.633 (3) | 0.2519 (5) | 0.049 (9) |
| C(12′) | 0.1192 (7) | 0.784 (4) | 0.2786 (5) | 0.063 (11) |
| C(13′) | 0.0796 (7) | 0.583 (4) | 0.3010 (6) | 0.066 (12) |
| C(14′) | 0.0414 (8) | 0.727 (4) | 0.3318 (6) | 0.072 (13) |

Table 2. *Comparison of bond lengths (Å) for (I), (II), (III), (IV) and (V)*

| | (I) | (II) | (III) | |
|---|---|---|---|---|
| | | | Molecule 1 | Molecule 2 |
| C(1)—C(2) | 1.383 (9) | 1.384 (7) | 1.374 (11) | 1.375 (10) |
| C(2)—C(3) | 1.382 (9) | 1.383 (7) | 1.396 (13) | 1.372 (11) |
| C(3)—C(4) | 1.380 (11) | 1.385 (7) | 1.362 (16) | 1.376 (11) |
| C(4)—C(5) | 1.389 (9) | 1.392 (7) | 1.387 (15) | 1.383 (11) |
| C(5)—C(6) | 1.379 (8) | 1.376 (7) | 1.370 (11) | 1.381 (10) |
| C(6)—C(1) | 1.382 (8) | 1.378 (7) | 1.362 (11) | 1.371 (10) |
| C(7)—C(6) | 1.505 (9) | 1.478 (7) | 1.491 (11) | 1.482 (10) |
| C(7)—N(1) | 1.399 (10) | 1.399 (6) | 1.381 (8) | 1.404 (10) |
| C(7)—O(7) | 1.189 (9) | 1.205 (6) | 1.203 (9) | 1.197 (9) |
| C(8)—C(1) | 1.485 (8) | 1.480 (7) | 1.477 (9) | 1.487 (9) |
| C(8)—N(1) | 1.396 (10) | 1.381 (6) | 1.370 (10) | 1.377 (10) |
| C(8)—O(8) | 1.204 (9) | 1.215 (6) | 1.201 (9) | 1.207 (10) |
| C(9)—N(1) | 1.445 (8) | 1.451 (6) | 1.464 (9) | 1.466 (10) |
| C(9)—C(10) | 1.497 (11) | 1.514 (7) | 1.582 (9) | 1.552 (9) |
| C(10)—C(11) | | 1.495 (7) | 1.464 (11) | 1.482 (11) |
| C(11)—C(12) | | | 1.543 (10) | 1.517 (10) |
| C(10)—O(100) | 1.179 (10) | | | |
| C(10)—O(101) | 1.320 (9) | | | |
| O(101)—H(101) | 0.88 (9) | | | |
| C(11)—O(110) | | 1.191 (6) | | |
| C(11)—O(111) | | 1.324 (6) | | |
| O(111)—H(111) | | 0.93 (5) | | |
| C(12)—O(120) | | | 1.260 (10) | 1.289 (10) |
| C(12)—O(121) | | | 1.240 (9) | 1.234 (9) |

| | (IV) | (V) | |
|---|---|---|---|
| | | Molecule 1 | Molecule 2 |
| C(1)—C(2) | 1.382 (12) | 1.37 (2) | 1.37 (2) |
| C(2)—C(3) | 1.396 (12) | 1.39 (2) | 1.38 (2) |
| C(3)—C(4) | 1.378 (11) | 1.37 (2) | 1.39 (2) |
| C(4)—C(5) | 1.396 (12) | 1.37 (2) | 1.40 (2) |
| C(5)—C(6) | 1.368 (12) | 1.35 (2) | 1.35 (2) |
| C(6)—C(1) | 1.386 (11) | 1.40 (2) | 1.38 (2) |
| C(7)—C(6) | 1.494 (13) | 1.50 (2) | 1.48 (2) |
| C(7)—N(1) | 1.396 (11) | 1.36 (2) | 1.38 (2) |
| C(7)—O(7) | 1.190 (11) | 1.23 (2) | 1.22 (2) |
| C(8)—C(1) | 1.476 (11) | 1.49 (2) | 1.49 (2) |
| C(8)—N(1) | 1.388 (11) | 1.42 (2) | 1.38 (2) |
| C(8)—O(8) | 1.203 (11) | 1.17 (2) | 1.22 (2) |
| C(9)—N(1) | 1.453 (11) | 1.52 (2) | 1.59 (2) |
| C(9)—C(10) | 1.542 (11) | 1.55 (2) | 1.56 (2) |
| C(10)—C(11) | 1.508 (12) | 1.50 (2) | 1.51 (2) |
| C(11)—C(12) | 1.514 (12) | 1.53 (2) | 1.50 (2) |
| C(12)—C(13) | 1.486 (13) | 1.51 (2) | 1.50 (2) |
| C(13)—C(14) | | 1.54 (2) | 1.49 (2) |
| C(13)—O(130) | 1.262 (11) | | |
| C(13)—O(131) | 1.254 (10) | | |
| C(14)—O(140) | | 1.18 (2) | 1.21 (2) |
| C(14)—O(141) | | 1.21 (2) | 1.21 (2) |

Table 3. *Comparison of bond angles (°) for (I), (II), (III), (IV) and (V)*

| | (I) | (II) | (III) | |
|---|---|---|---|---|
| | | | Molecule 1 | Molecule 2 |
| C(7)—N(1)—C(8) | 112.8 (5) | 111.2 (4) | 112.3 (6) | 111.9 (6) |
| C(9)—N(1)—C(7) | 124.3 (6) | 123.1 (4) | 125.2 (7) | 125.1 (7) |
| C(9)—N(1)—C(8) | 122.0 (7) | 125.6 (4) | 122.2 (7) | 122.6 (7) |
| C(8)—C(1)—C(6) | 108.5 (6) | 107.8 (4) | 108.4 (7) | 108.2 (7) |
| C(2)—C(1)—C(6) | 120.8 (7) | 121.5 (4) | 122.1 (8) | 121.2 (7) |
| C(2)—C(1)—C(8) | 130.8 (7) | 130.6 (4) | 129.5 (8) | 130.5 (7) |
| C(3)—C(2)—C(1) | 117.1 (7) | 116.5 (5) | 116.1 (10) | 117.6 (7) |
| C(2)—C(3)—C(4) | 121.6 (7) | 122.2 (5) | 120.7 (10) | 121.4 (7) |
| C(3)—C(4)—C(5) | 121.7 (7) | 120.8 (5) | 123.3 (10) | 121.3 (8) |
| C(4)—C(5)—C(6) | 116.0 (7) | 116.8 (5) | 115.0 (9) | 116.9 (6) |
| C(1)—C(6)—C(7) | 108.4 (6) | 108.2 (4) | 107.8 (6) | 108.4 (7) |
| C(5)—C(6)—C(7) | 128.8 (7) | 129.6 (4) | 129.3 (9) | 129.9 (7) |
| C(5)—C(6)—C(1) | 122.7 (6) | 122.2 (4) | 122.9 (9) | 121.6 (7) |
| C(6)—C(7)—N(1) | 104.6 (6) | 106.0 (4) | 105.4 (7) | 105.4 (6) |
| O(7)—C(7)—N(1) | 126.2 (6) | 123.9 (4) | 124.4 (8) | 124.5 (8) |
| O(7)—C(7)—C(6) | 129.1 (7) | 130.1 (3) | 130.2 (7) | 130.1 (7) |
| C(1)—C(8)—N(1) | 105.6 (7) | 106.7 (4) | 106.1 (7) | 106.0 (7) |
| O(8)—C(8)—N(1) | 124.9 (7) | 123.4 (4) | 126.0 (8) | 125.4 (7) |
| O(8)—C(8)—C(1) | 129.5 (7) | 130.0 (4) | 128.0 (7) | 128.5 (7) |
| C(10)—C(9)—N(1) | 111.9 (6) | 111.7 (4) | 111.6 (6) | 111.7 (7) |
| C(9)—C(10)—C(11) | | 111.8 (4) | 108.0 (7) | 111.3 (7) |
| C(10)—C(11)—C(12) | | | 108.8 (7) | 111.7 (7) |
| C(9)—C(10)—O(101) | 110.8 (7) | | | |
| O(100)—C(10)—C(9) | 126.1 (7) | | | |
| O(100)—C(10)—O(101) | 123.1 (8) | | | |
| H(101)—O(101)—C(10) | 108 (6) | | | |
| C(10)—C(11)—O(111) | | 112.0 (4) | | |
| O(110)—C(11)—C(10) | | 125.1 (5) | | |
| O(110)—C(11)—O(111) | | 122.9 (5) | | |
| H(111)—O(111)—C(11) | | 105 (3) | | |
| C(11)—C(12)—O(121) | | | 121.5 (9) | 121.6 (8) |
| C(11)—C(12)—O(120) | | | 117.1 (8) | 116.3 (7) |
| O(120)—C(12)—O(121) | | | 121.3 (8) | 122.1 (7) |

| | (IV) | (V) | |
|---|---|---|---|
| | | Molecule 1 | Molecule 2 |
| C(8)—N(1)—C(7) | 112.0 (8) | 115 (1) | 112 (1) |
| C(9)—N(1)—C(7) | 123.4 (8) | 123 (2) | 125 (2) |
| C(9)—N(1)—C(8) | 124.5 (8) | 122 (1) | 123 (2) |
| C(8)—C(1)—C(6) | 108.3 (8) | 110 (1) | 108 (1) |
| C(2)—C(1)—C(6) | 121.0 (8) | 122 (1) | 122 (2) |
| C(2)—C(1)—C(8) | 130.6 (8) | 128 (2) | 130 (2) |
| C(3)—C(2)—C(1) | 116.9 (8) | 116 (2) | 116 (2) |
| C(2)—C(3)—C(4) | 121.6 (9) | 121 (2) | 123 (2) |
| C(3)—C(4)—C(5) | 120.9 (9) | 121 (2) | 119 (2) |
| C(4)—C(5)—C(6) | 117.2 (8) | 119 (2) | 118 (2) |
| C(1)—C(6)—C(7) | 107.9 (8) | 107 (1) | 108 (2) |
| C(5)—C(6)—C(7) | 129.9 (8) | 133 (2) | 131 (2) |
| C(5)—C(6)—C(1) | 122.2 (9) | 121 (2) | 122 (2) |
| C(6)—C(7)—N(1) | 105.4 (8) | 106 (1) | 107 (2) |
| O(7)—C(7)—N(1) | 125.4 (8) | 127 (2) | 123 (2) |
| O(7)—C(7)—C(6) | 129.0 (9) | 127 (1) | 130 (2) |
| C(1)—C(8)—N(1) | 106.3 (8) | 103 (1) | 106 (1) |
| O(8)—C(8)—N(1) | 125.4 (8) | 126 (2) | 127 (2) |
| O(8)—C(8)—C(1) | 128.3 (8) | 132 (2) | 127 (2) |
| C(10)—C(9)—N(1) | 112.0 (7) | 109 (1) | 104 (2) |
| C(9)—C(10)—C(11) | 111.5 (7) | 110 (1) | 107 (2) |
| C(10)—C(11)—C(12) | 111.8 (8) | 112 (1) | 111 (1) |
| C(13)—C(12)—C(11) | 111.8 (8) | 111 (1) | 113 (1) |
| C(14)—C(13)—C(12) | | 111 (2) | 114 (2) |
| O(131)—C(13)—C(12) | 118.8 (8) | | |
| C(12)—C(13)—O(130) | 120.0 (8) | | |
| O(131)—C(13)—O(130) | 121.2 (8) | | |
| O(141)—C(14)—C(13) | | 113 (2) | 121 (2) |
| O(140)—C(14)—C(13) | | 122 (2) | 121 (2) |
| O(140)—C(14)—O(141) | | 123 (2) | 118 (2) |

Table 4. *Dihedral angles (°) of moieties of (I), (II), (III), (IV) and (V)*

| | *AB* | *AC* | *AD* | *CD* |
|---|---|---|---|---|
| (I) | 87.5 | | | |
| (II) | 8.2 | | | |
| (III) (1) | | 112.6 | 92.2 | 121.5 |
| (2) | | 114.5 | 83.2 | 132.2 |
| (IV) | | 91.2 | 8.9 | 83.2 |
| (V) (1) | | 86.6 | 63.7 | 135.5 |
| (2) | | 96.4 | 120.6 | 53.8 |

The structures were solved by direct methods using *SHELXS*86 (Sheldrick, 1985), and refined by full-matrix least-squares analysis using *SHELX*76 (Sheldrick, 1976). The ring and alkyl H atoms were fixed geometrically and refined with isotropic displacement parameters. The carboxylic H-atom coordinates were located from the difference Fourier synthesis maps for (I) and (II) but could not be located for (III), (IV) or (V). All non-H atoms were refined with anisotropic displacement parameters. The slightly high *R* values for (IV) and (V) are attributed to the difficulty of obtaining high-quality crystals for these two compounds.

We acknowledge support from the SERC (studentship for NF). We are grateful to Interox plc (and in particular Dr A. James) for the provision of samples and numerous discussions.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1020). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# Structures of Four *p*-Amidoperbenzoic Acids†

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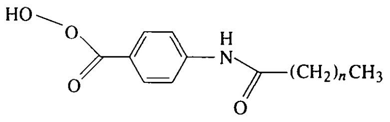
## Abstract

The crystal structures of *p*-acetamidoperbenzoic acid, $C_9H_9NO_4$ (I), *p*-propanamidoperbenzoic acid, $C_{10}H_{11}NO_4$ (II), *p*-butanamidoperbenzoic acid, $C_{11}H_{13}NO_4$ (III), and *p*-pentanamidoperbenzoic acid, $C_{12}H_{15}NO_4$ (IV) have been determined. Extensive hydrogen-bond networks are observed within each structure. The hydrogen-bond motifs forming these networks are very different to those observed in the structures of comparable *p*-amidobenzoic acids previously investigated by us [Feeder & Jones (1993). *Acta Cryst.* B**49**, 541–546].

## Comment

As part of a general study on the stability of organic peracids and peracid salts (Kariuki & Jones, 1990), and in particular the role of hydrogen bonding in controlling the crystal structure of peracid and acid amides (Feeder & Jones, 1992), we report here the crystal structures of the compounds (I)–(IV).



(I) $n = 0$
(II) $n = 1$
(III) $n = 2$
(IV) $n = 3$

The final heavy-atom fractional coordinates and equivalent isotropic temperature factors are listed in Table 1. The atom numbering schemes are shown in Figs. 1(*a*)–1(*d*), and a comparison of bond lengths and angles is given in Table 2.

The bond lengths and angles found for compounds (I)–(IV) are similar to those found for a series of *p*-amidobenzoic acids investigated by us (Feeder & Jones, 1993). The peroxy O—O bond lengths are not significantly different from those found for peroxypelargonic acid [O—O 1.442 (12) Å; Belitskus & Jeffrey, 1965], *o*-nitroperoxybenzoic acid [O—O 1.478 (7) Å; Sax, Beurskens & Chu, 1965] and *p*-nitroperoxybenzoic acid [O—O 1.48 (2) Å; Kim, Chu & Jeffrey, 1970].

Similarities are found in the structures of (I), (II) and (IV) in that the molecular geometries can be described by three planar groups – moiety *A* the benzene ring, C(1)–(C6), moiety *B* the amido group, C(1),N(1),C(8),O(8),C(9), and moiety *C* the peracid group, O(71),C(7),O(72),O(73),H(73). The planarity of these groups and the dihedral angles are given in Table 3. Structure solution revealed that for (III) each of the heavy atoms in the asymmetric unit lies on special positions ($z$ = 0.25), and as a result the molecule is planar.

Extensive hydrogen-bond networks are formed within each of the four structures and in each case constructed from similar motifs. (I) and (II) are isostructural and the networks involve two types of hydrogen bond. Firstly, each molecule is hydrogen bonded to two others to form a chain by the interaction of a peracid proton with an amido carbonyl O atom between molecules related by translation in both **a** and **b** (C centring) [(I) O(73)—H(73)···O(8) 1.759, O(73)···O(8) 2.573 Å; (II) O(73)—H(73)···O(8) 1.747, O(73)···O(8) 2.690 Å]. Each molecule in the chain is further hydrogen bonded to other molecules by the interaction of an amido proton with a peracid group between molecules related by a *c* glide. This interaction is three-

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† It should be noted that the delay in publication of this paper was not due to problems with the structures, or to the slow response of the authors.

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