Data collection: Enraf–Nonius (1977) CAD-4 diffractometer software. Cell refinement: Enraf–Nonius (1977) CAD-4 diffractometer software. Data reduction: *DATARED* (Pèpe, 1979). Program(s) used to solve structure: *MULTAN*80 (Main *et al.*, 1980). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *AME* (Software Systems, 1988).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1058). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cardiergue, H., Pèpe, G., Astier, J. P., Boistelle, R. & Fiard, J. F. (1993). *Acta Cryst.* C**49**, 1078–1080.

Enraf–Nonius (1977). *CAD-4 Operations Manual.* Enraf–Nonius, Delft, The Netherlands.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Universities of York, England, and Louvain, Belgium.

Pèpe, G. (1979). *DATARED. Programs for X-ray Diffraction Data Reduction.* CRMC2-CNRS, Marseille, France.

Pèpe, G., Pfefer, G., Boistelle, R. & Marchal, P. (1995). *Acta Cryst.* C**51**, 2671–2672.

Sheldrick, G. M. (1976). *SHELX*76. *Program for Crystal Structure Determination.* University of Cambridge, England.

Software Systems (1988). *AME, A Mouse Editor.* Software Systems, San Jose, CA 95126, USA.

Acta Cryst. (1996). C**52**, 1516–1520

Four ω-Phthalimidoaliphatic Peracids

N. Feeder* and W. Jones

Department of Chemistry, University of Cambridge, Lensfield Road, Cambridge CB2 1EW, England. E-mail: nf101@cus.cam.ac.uk

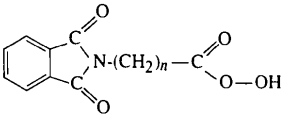
(*Received 26 October 1995; accepted 18 December 1995*)

Abstract

Hydrogen bonding between the peracid proton and phthalimido carbonyl O atom connect molecules into chains in the structures of 3-phthalimidoperoxypropanoic acid, $C_{11}H_9NO_5$, and 4-phthalimidoperoxybutanoic acid, $C_{12}H_{11}NO_5$. In contrast, the peracid function acts as both a hydrogen-bond donor and acceptor in the structures of 5-phthalimidoperoxypentanoic acid, $C_{13}H_{13}NO_5$, and 6-phthalimidoperoxyhexanoic acid, $C_{14}H_{15}NO_5$, resulting in a distinctly different chain motif.

Comment

Organic peracids have recently been recognized as potential low-temperature laundry bleaches. To have commercial utility, these materials are required to exhibit high solid-state thermal stability with respect to decomposition to the corresponding carboxylic acid. In order to probe the relationship between peracid crystal structure and this solid-state stability, the structures of the four homologues 3-phthalimidoperoxypropanoic acid, (I), 4-phthalimidoperoxybutanoic acid, (II), 5-phthalimidoperoxypentanoic acid, (III), and 6-phthalimidoperoxyhexanoic acid, (IV), have been determined. In particular, the influence of the alkyl chain length on the type of hydrogen-bonding adopted has been investigated (Feeder, 1992).



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

In all four title structures (Figs. 1–4), the phthalimido groups are close to being planar and have bond lengths and angles similar to those of both a series of ω-phthalimidoaliphatic acids (Feeder & Jones, 1996*a*) and a series of ω-phthalimidoaliphatic acid monohydrates (Feeder & Jones, 1994*a*) determined previously. The alkyl chains each adopt a regular all-*trans* conformation. The position of the peracid proton could not be determined in any of the four structures, although each of the peracid groups defined by the heavy C and O atoms is planar: (I) to 0.001 (4), (II) to 0.001 (1), (III) to 0.004 (4) and (IV) to 0.002 (2) Å. The bond lengths and angles associated with the peracid functions of compounds (II), (III) and (IV) are very similar to those reported previously for peroxypelargonic acid (Belitskus & Jeffrey, 1965), *o*-nitroperoxybenzoic acid (Sax, Beurskens & Chu, 1965), *p*-nitroperoxybenzoic acid (Kim, Chu & Jeffrey, 1970), a series of *p*-amidoperoxybenzoic acids (Feeder & Jones, 1996*b*) and two ω-saccharinperoxyaliphatic acids (Feeder & Jones, 1994*b*). The low O—O bond length in compound (I) (O111—O112) of 1.357 (7) Å is an artefact related to the high atomic displacement parameter (or disorder) of the O112 atom.

Hydrogen bonding in all four structures links molecules into infinite chains. In the structures of (I) and (II), the hydrogen bond is between the peracid proton

© 1996 International Union of Crystallography
Printed in Great Britain – all rights reserved

Acta Crystallographica Section C
ISSN 0108-2701 © 1996

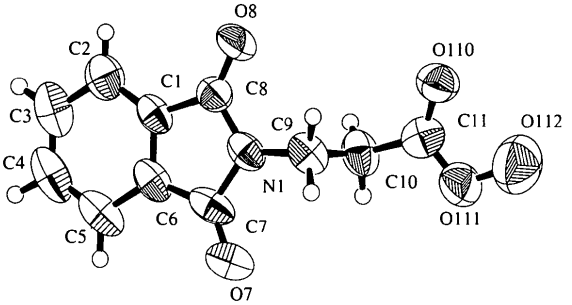


Fig. 1. *ORTEP*II (Johnson, 1976) plot of (I) showing ellipsoids at the 50% probability level.

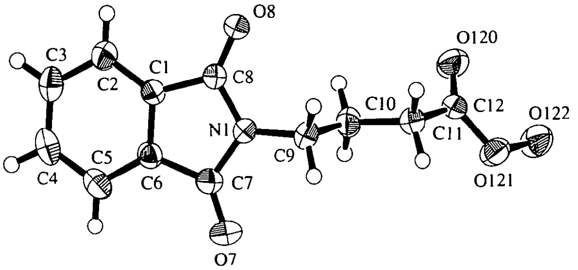


Fig. 2. *ORTEP*II (Johnson, 1976) plot of (II) showing ellipsoids at the 50% probability level.

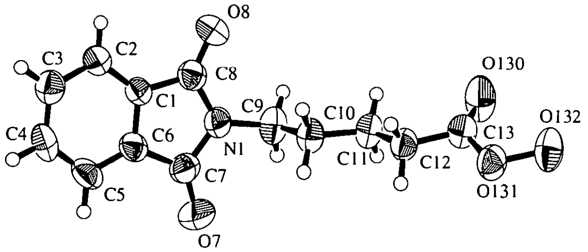


Fig. 3. *ORTEP*II (Johnson, 1976) plot of (III) showing ellipsoids at the 50% probability level.

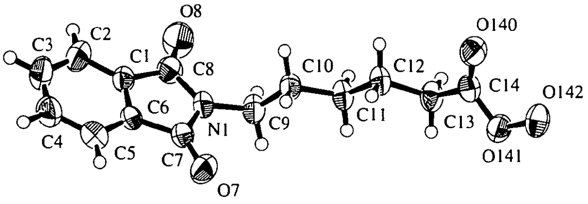


Fig. 4. *ORTEP*II (Johnson, 1976) plot of (IV) showing ellipsoids at the 50% probability level.

and a phthalimido carbonyl O atom. This is shown for compound (I) in Fig. 5, where adjacent molecules in the chain are related by the *c*-glide plane. In compound (II), there are again hydrogen bonds from the peracid proton to the phthalimido carbonyl O atom, but adjacent molecules in the chain are related by translation only.

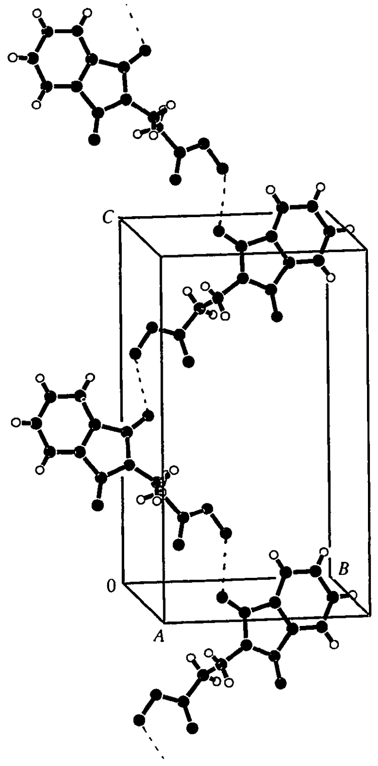


Fig. 5. Packing diagram for compound (I) demonstrating the chain motif formed through the peracid-to-phthalimido hydrogen bonds (O· · ·O contacts are shown as dashed lines).

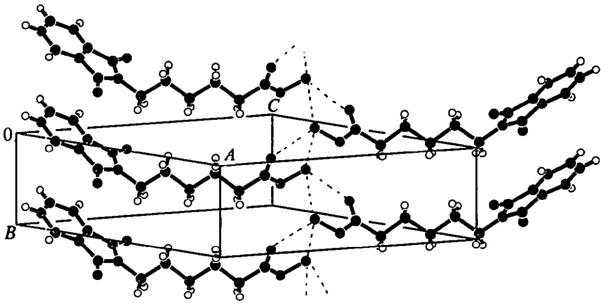


Fig. 6. Packing diagram for compound (IV) demonstrating the chain motif formed by the three-centre hydrogen bonds between peracid groups related by a twofold screw operation (O· · ·O contacts are shown as dashed lines).

For the structures of compounds (III) and (IV), the peracid function acts as both a hydrogen-bond acceptor and donor linking molecules by a twofold screw operation into chains, shown for compound (IV) in Fig. 6. It can be seen that, based on O· · ·O contacts, both the peracid carbonyl (O140) and terminal peroxy (O142) O atoms are the acceptor atoms for a three-centre hydrogen-bond with a peracid proton.

Experimental

Samples of compounds (I)–(IV) were provided by Solvay Interox.

Compound (I)

Crystal data

$C_{11}H_9NO_5$
$M_r = 235.19$
Monoclinic
$P2_1/c$
$a = 7.022\,(2)$ Å
$b = 9.532\,(2)$ Å
$c = 15.811\,(9)$ Å
$\beta = 99.03\,(3)°$
$V = 1045.1\,(7)$ Å^3
$Z = 4$
$D_x = 1.495$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 9–14°$
$\mu = 0.120$ mm^{-1}
$T = 295\,(2)$ K
Cuboid
0.40 × 0.40 × 0.40 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1912 measured reflections
1842 independent reflections
749 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0226$
$\theta_{\rm max} = 24.97°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 18$
2 standard reflections monitored every 100 reflections intensity decay: 2%

Refinement

Refinement on F^2
$R(F) = 0.0707$
$wR(F^2) = 0.2769$
$S = 1.001$
1795 reflections
158 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.1210P)^2 + 0.7913P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$

$\Delta\rho_{\rm max} = 0.388$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.367$ e Å^{-3}
Extinction correction: *SHELXL93* (Sheldrick, 1993)
Extinction coefficient: 0.005 (4)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (I)*

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

	x	y	z	$U_{\rm eq}$
O7	0.7323 (6)	−0.0754 (5)	0.5163 (3)	0.0855 (15)
O8	0.6521 (7)	0.1869 (5)	0.7469 (2)	0.0830 (14)
O110	0.7614 (7)	−0.2222 (5)	0.8737 (3)	0.0827 (14)
O111	0.8585 (7)	−0.3778 (6)	0.7891 (3)	0.0914 (15)
O112	0.8343 (10)	−0.4636 (6)	0.8551 (4)	0.144 (2)
N1	0.6808 (6)	0.0280 (6)	0.6422 (3)	0.0620 (13)
C1	0.7099 (7)	0.2590 (8)	0.6057 (3)	0.061 (2)
C2	0.7133 (8)	0.4038 (8)	0.6032 (4)	0.079 (2)
C3	0.7458 (9)	0.4638 (9)	0.5287 (5)	0.094 (2)
C4	0.7725 (10)	0.3865 (11)	0.4600 (5)	0.098 (3)
C5	0.7710 (9)	0.2413 (10)	0.4604 (4)	0.084 (2)
C6	0.7363 (8)	0.1803 (8)	0.5360 (4)	0.066 (2)
C7	0.7194 (8)	0.0296 (9)	0.5579 (4)	0.071 (2)
C8	0.6763 (7)	0.1640 (6)	0.6753 (4)	0.0590 (15)
C9	0.6548 (8)	−0.0992 (7)	0.6904 (4)	0.069 (2)
C10	0.8448 (9)	−0.1515 (7)	0.7385 (4)	0.076 (2)
C11	0.8163 (8)	−0.2546 (9)	0.8063 (5)	0.073 (2)

Compound (II)

Crystal data

$C_{12}H_{11}NO_5$
$M_r = 249.22$
Triclinic
$P\bar{1}$
$a = 7.968\,(2)$ Å
$b = 8.463\,(2)$ Å
$c = 9.4295\,(10)$ Å
$\alpha = 85.473\,(10)°$
$\beta = 113.31\,(2)°$
$\gamma = 105.37\,(2)°$
$V = 562.8\,(2)$ Å^3
$Z = 2$
$D_x = 1.471$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10–15°$
$\mu = 0.116$ mm^{-1}
$T = 295\,(2)$ K
Rod
0.40 × 0.20 × 0.20 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3456 measured reflections
3271 independent reflections
1980 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0177$

$\theta_{\rm max} = 29.97°$
$h = -11 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = 0 \rightarrow 13$
2 standard reflections monitored every 100 reflections intensity decay: 3% (correction applied)

Refinement

Refinement on F^2
$R(F) = 0.0439$
$wR(F^2) = 0.1378$
$S = 1.000$
3228 reflections
168 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.0642P)^2 + 0.1133P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.411$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.233$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 2. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (II)*

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

	x	y	z	$U_{\rm eq}$
N1	0.3329 (2)	0.1899 (2)	0.25571 (15)	0.0356 (3)
O7	0.0764 (2)	−0.0320 (2)	0.2142 (2)	0.0608 (4)
O8	0.5945 (2)	0.40087 (15)	0.36936 (14)	0.0449 (3)
O120	0.1664 (2)	0.6657 (2)	−0.1408 (2)	0.0556 (4)
O121	−0.0196 (2)	0.4812 (2)	−0.33407 (15)	0.0477 (3)
O122	−0.1174 (2)	0.6093 (2)	−0.4016 (2)	0.0560 (4)
C1	0.4492 (2)	0.2078 (2)	0.5202 (2)	0.0331 (3)
C2	0.5553 (3)	0.2478 (2)	0.6742 (2)	0.0412 (4)
C3	0.4968 (3)	0.1519 (3)	0.7807 (2)	0.0482 (5)
C4	0.3387 (3)	0.0211 (2)	0.7335 (2)	0.0497 (5)
C5	0.2326 (3)	−0.0207 (2)	0.5781 (2)	0.0455 (4)
C6	0.2909 (2)	0.0753 (2)	0.4727 (2)	0.0354 (4)
C7	0.2123 (2)	0.0633 (2)	0.3012 (2)	0.0393 (4)
C8	0.4751 (2)	0.2826 (2)	0.3799 (2)	0.0333 (3)
C9	0.3121 (3)	0.2199 (2)	0.0961 (2)	0.0387 (4)
C10	0.2160 (3)	0.3559 (2)	0.0275 (2)	0.0404 (4)
C11	0.2149 (2)	0.3957 (2)	−0.1323 (2)	0.0382 (4)
C12	0.1236 (2)	0.5332 (2)	−0.1986 (2)	0.0360 (4)

Compound (III)

Crystal data

$C_{13}H_{13}NO_5$
$M_r = 263.24$
Monoclinic
$P2_1$
$a = 6.7773\ (10)$ Å
$b = 4.7222\ (10)$ Å
$c = 19.935\ (4)$ Å
$\beta = 98.22\ (2)^\circ$
$V = 631.5\ (2)$ Å^3
$Z = 2$
$D_x = 1.385$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 8$–15°
$\mu = 0.108$ mm^{-1}
$T = 295\ (2)$ K
Plate
0.40 × 0.40 × 0.20 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2277 measured reflections
2214 independent reflections
1145 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.1128$
$\theta_{\rm max} = 24.99^\circ$
$h = -8 \rightarrow 7$
$k = -5 \rightarrow 5$
$l = 0 \rightarrow 23$
2 standard reflections monitored every 100 reflections
intensity decay: 1%

Refinement

Refinement on F^2
$R(F) = 0.0611$
$wR(F^2) = 0.2020$
$S = 1.014$
2181 reflections
178 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.1032P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.004$
$\Delta\rho_{\rm max} = 0.270$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.193$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.067 (15)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (III)*

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

	x	y	z	$U_{\rm eq}$
N1	0.9699 (6)	−0.1681 (11)	0.2068 (2)	0.0571 (13)
O8	1.1739 (6)	−0.1546 (11)	0.1239 (2)	0.0784 (14)
O7	0.7138 (6)	−0.0776 (14)	0.2678 (2)	0.095 (2)
O130	1.7588 (7)	−0.5699 (12)	0.4191 (2)	0.097 (2)
O131	1.7438 (6)	−0.2359 (9)	0.4971 (2)	0.0700 (13)
O132	1.9133 (6)	−0.3809 (11)	0.5341 (2)	0.0884 (15)
C6	0.7335 (7)	0.1531 (13)	0.1609 (3)	0.0546 (15)
C5	0.5736 (7)	0.3306 (16)	0.1466 (3)	0.068 (2)
C4	0.5570 (8)	0.4880 (15)	0.0868 (3)	0.069 (2)
C3	0.6959 (8)	0.4597 (15)	0.0436 (3)	0.067 (2)
C2	0.8574 (8)	0.2807 (13)	0.0578 (3)	0.060 (2)
C1	0.8733 (7)	0.1271 (13)	0.1172 (3)	0.0478 (13)
C8	1.0252 (8)	−0.0747 (14)	0.1460 (3)	0.058 (2)
C7	0.7948 (8)	−0.0348 (15)	0.2196 (3)	0.062 (2)
C9	1.0874 (9)	−0.3570 (15)	0.2546 (3)	0.065 (2)
C10	1.2326 (8)	−0.1932 (13)	0.3051 (3)	0.060 (2)
C11	1.3787 (8)	−0.3814 (14)	0.3491 (3)	0.061 (2)
C12	1.5112 (7)	−0.2081 (14)	0.4006 (3)	0.061 (2)
C13	1.6832 (9)	−0.3686 (15)	0.4379 (3)	0.0578 (15)

Compound (IV)

Crystal data

$C_{14}H_{15}NO_5$
$M_r = 277.27$
Monoclinic
$P2_1/c$
$a = 19.028\ (5)$ Å
$b = 4.5724\ (10)$ Å
$c = 15.517\ (6)$ Å
$\beta = 90.16\ (3)^\circ$
$V = 1350.0\ (7)$ Å^3
$Z = 4$
$D_x = 1.364$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 8$–13°
$\mu = 0.104$ mm^{-1}
$T = 295\ (2)$ K
Plate
0.40 × 0.40 × 0.20 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2496 measured reflections
2391 independent reflections
885 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0370$

$\theta_{\rm max} = 24.97^\circ$
$h = -22 \rightarrow 22$
$k = 0 \rightarrow 5$
$l = 0 \rightarrow 18$
2 standard reflections monitored every 100 reflections
intensity decay: 3% (correction applied)

Refinement

Refinement on F^2
$R(F) = 0.0497$
$wR(F^2) = 0.1600$
$S = 0.914$
2335 reflections
187 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.0607P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.004$
$\Delta\rho_{\rm max} = 0.262$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.195$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 4. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (IV)*

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

	x	y	z	$U_{\rm eq}$
N1	0.2001 (2)	0.1213 (8)	0.7388 (2)	0.0450 (9)
O7	0.1195 (2)	0.2973 (8)	0.8371 (2)	0.0710 (11)
O8	0.2579 (2)	0.0208 (8)	0.6127 (2)	0.0850 (12)
O140	0.47793 (15)	0.2644 (8)	1.1141 (2)	0.0631 (10)
O141	0.41745 (14)	−0.0443 (7)	1.1994 (2)	0.0585 (9)
O142	0.45308 (15)	0.1065 (7)	1.2702 (2)	0.0658 (10)
C1	0.1582 (2)	0.3553 (10)	0.6183 (2)	0.0453 (11)
C2	0.1456 (2)	0.4564 (12)	0.5364 (3)	0.0659 (15)
C3	0.0905 (3)	0.6499 (12)	0.5248 (3)	0.073 (2)
C4	0.0491 (2)	0.7328 (12)	0.5924 (3)	0.0685 (14)
C5	0.0609 (2)	0.6259 (11)	0.6745 (3)	0.0588 (13)
C6	0.1165 (2)	0.4383 (10)	0.6857 (2)	0.0438 (12)
C7	0.1425 (2)	0.2853 (11)	0.7638 (3)	0.0488 (12)
C8	0.2124 (2)	0.1486 (11)	0.6512 (3)	0.0525 (13)
C9	0.2412 (2)	−0.0620 (10)	0.7971 (3)	0.0541 (13)
C10	0.2923 (2)	0.1105 (10)	0.8514 (2)	0.0495 (12)
C11	0.3303 (2)	−0.0827 (10)	0.9161 (2)	0.0491 (12)
C12	0.3732 (2)	0.0931 (10)	0.9809 (2)	0.0499 (12)
C13	0.4015 (2)	−0.1016 (11)	1.0521 (2)	0.0534 (13)
C14	0.4371 (2)	0.0659 (11)	1.1229 (3)	0.0487 (12)

Table 5. *Selected geometric parameters (Å, °) of the peracid groups in compounds (I)–(IV)*

	(I) (*n* = 11)	(II) (*n* = 12)	(III) (*n* = 13)	(IV) (*n* = 14)
O*n*0—C*n*	1.228 (7)	1.190 (2)	1.167 (7)	1.202 (5)
O*n*1—C*n*	1.252 (8)	1.340 (2)	1.348 (7)	1.345 (5)
O*n*1—O*n*2	1.357 (7)	1.461 (2)	1.446 (6)	1.462 (4)
C*n*—O*n*1—O*n*2	109.7 (5)	110.73 (13)	110.4 (4)	110.9 (3)
O*n*0—C*n*—O*n*1	122.7 (7)	124.9 (2)	124.5 (6)	124.3 (4)
O*n*0—C*n*—C(*n*−1)	123.6 (7)	126.6 (2)	126.9 (5)	126.4 (4)
O*n*1—C*n*—C(*n*−1)	113.6 (6)	108.47 (14)	108.5 (5)	109.3 (4)
O*n*2—O*n*1—C*n*—O*n*0	−0.2 (9)	−0.3 (2)	−0.8 (9)	−0.8 (5)
O*n*2—O*n*1—C*n*—C(*n*−1)	178.1 (5)	178.24 (13)	−178.7 (4)	177.9 (3)
C(*n*−2)—C(*n*−1)—C*n*—O*n*0	−73.2 (8)	55.1 (2)	25.0 (9)	−44.4 (6)
C(*n*−2)—C(*n*−1)—C*n*—O*n*1	108.6 (7)	−123.4 (2)	−157.2 (5)	136.9 (4)

The peroxy H atom was not located in any of the four title compounds. All other H atoms were placed at the expected positions, riding with U_{iso} refined for each. The terminal peroxy O atom (O112) of compound (I) was found to be disordered over two sites. Refinement of the atom at these sites with a range of occupancies significantly lowered the *R* value for the structure. The displacement parameters of the atoms at these sites, however, were found to be unacceptably high and hence it was concluded that the data were not of sufficient quality to model the disorder in this way. Finally, the atom position was refined starting at some mean of the two sites with full occupancy. This accounts for the high U_{eq} value for this atom and also for the slightly high *R* value reported.

For all compounds, data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software* (*SETANG*); data reduction: *TEXSAN* (Molecular Structure Corporation, 1995); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *TEXSAN*; software used to prepare material for publication: *SHELXL*93.

The authors acknowledge support from the SERC (studentship for NF) and are grateful to Solvay Interox (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: HA1155). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Belitskus, D. & Jeffrey, G. A. (1965). *Acta Cryst.* **18**, 458–463.

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5. Enraf–Nonius, Delft, The Netherlands.

Feeder, N. (1992). PhD thesis, University of Cambridge, England.

Feeder, N. & Jones, W. (1994*a*). *Acta Cryst.* C**50**, 820–823.

Feeder, N. & Jones, W. (1994*b*). *Acta Cryst.* C**50**, 1347–1349.

Feeder, N. & Jones, W. (1996*a*). *Acta Cryst.* C**52**, 913–919.

Feeder, N. & Jones, W. (1996*b*). *Acta Cryst.* C**52**, 919–923.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kim, H. S., Chu, S.-C. & Jeffrey, G. A. (1970). *Acta Cryst.* B**26**, 896–900.

Molecular Structure Corporation (1995). *TEXSAN. Single Crystal Structure Analysis Software*. Version 1.7-1. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Sax, M., Beurskens, P. & Chu, S. (1965). *Acta Cryst.* **18**, 252–258.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Acta Cryst. (1996). C**52**, 1520–1523

(±)-2-Oxocyclododecaneacetic Acid: Structure and Hydrogen-Bonding Pattern of a Large-Ring γ-Keto Acid

HUGH W. THOMPSON, MARIE L. COTÉ AND ROGER A. LALANCETTE*

Carl A. Olson Memorial Laboratories, Department of Chemistry, Rutgers University, Newark, NJ 07102, USA

(*Received 3 January 1995; accepted 24 July 1995*)

Abstract

In the title compound, $C_{14}H_{24}O_3$, the ring adopts a typical 'square' [3333] cyclododecane conformation and enantiomeric pairs of molecules form centrosymmetric dimers across the cell corners by mutual hydrogen bonding of carboxy groups, O(2)···O(3)(−*x*, 2 − *y*, −*z*) [2.662 (4) Å]. The ketonic O(1) atom does not participate in the hydrogen bonding but has a close contact [2.487 (3) Å] with the ring H(12*B*)(*x* − 1, *y*, *z*) atom of an adjacent molecule. The observed carboxy C—O and C═O bond lengths and angles correspond to accepted values for a well ordered dimer.

Comment

The crystalline states of acetic and formic acids involve chains (catemers) created by repeating intermolecular O—H···O═C hydrogen bonds (Jones & Templeton, 1958; Nahringbauer, 1978). This pattern is otherwise unusual among carboxylic acids, which typically form hydrogen-bonded dimers (Leiserowitz, 1976). Our interest in the X-ray structures of simple keto-carboxylic acids concerns such hydrogen-bonding motifs, of which four are known. The commonest forms acid dimers without the involvement of the ketone. Less frequently, intermolecular carboxy-to-ketone hydrogen bonds repeat infinitely along one cell axis to yield a catemer. A third, rare arrangement is an internal hydrogen bond, and one instance is known of acid-to-ketone dimerization. We have previously referenced and discussed numerous examples (Thompson, Lalancette & Vanderhoff, 1992; Coté, Thompson & Lalancette, 1996).

© 1996 International Union of Crystallography
Printed in Great Britain – all rights reserved