Table 2. *Selected geometric parameters* (Å, °)

	(I)	(II)	(III)
C(1)—C(2)	1.380 (4)	1.368 (10)	1.382 (5)
C(2)—C(3)	1.388 (5)	1.393 (11)	1.394 (7)
C(3)—C(4)	1.376 (5)	1.386 (12)	1.370 (8)
C(4)—C(5)	1.378 (5)	1.365 (12)	1.378 (7)
C(5)—C(6)	1.387 (4)	1.385 (10)	1.381 (5)
C(6)—C(1)	1.389 (4)	1.384 (10)	1.379 (5)
C(1)—N(1)	1.422 (4)	1.429 (8)	1.437 (4)
C(7)—N(1)	1.352 (4)	1.342 (9)	1.471 (5)
C(7)—O(7)	1.221 (4)	1.220 (8)	-
C(8)—O(8)	-	-	1.226 (4)
C(8)—N(1)	-	-	1.358 (4)
C(7)—C(8)	1.515 (4)	1.511 (9)	-
C(8)—C(9)	1.513 (4)	1.509 (9)	1.505 (5)
C(9)—C(10)	1.524 (4)	1.521 (10)	1.531 (5)
C(10)—C(11)	1.511 (4)	1.474 (11)	1.484 (5)
C(11)—C(12)	1.498 (4)	1.498 (10)	-
C(11)—O(111)	-	-	1.244 (4)
C(11)—O(112)	-	-	1.299 (4)
C(12)—O(121)	1.220 (4)	1.232 (9)	-
C(12)—O(122)	1.287 (4)	1.316 (9)	-
C(1)—N(1)—C(7)	126.0 (3)	127.6 (7)	115.6 (4)
C(8)—N(1)—C(1)	-	-	123.5 (4)
C(7)—N(1)—C(8)	-	-	120.7 (4)
C(2)—C(1)—N(1)	117.9 (3)	117.0 (8)	119.6 (5)
C(6)—C(1)—N(1)	121.6 (3)	122.1 (6)	119.3 (4)
C(6)—C(1)—C(2)	120.5 (3)	120.9 (7)	120.8 (5)
C(3)—C(2)—C(1)	119.4 (4)	119.7 (8)	118.8 (6)
C(4)—C(3)—C(2)	120.3 (4)	119.6 (9)	120.4 (6)
C(3)—C(4)—C(5)	120.3 (3)	119.9 (8)	120.4 (6)
C(6)—C(5)—C(4)	120.0 (4)	120.9 (9)	119.9 (6)
C(5)—C(6)—C(1)	119.5 (3)	118.9 (8)	119.8 (5)
O(7)—C(7)—N(1)	123.5 (3)	123.0 (7)	-
C(8)—C(7)—N(1)	114.0 (3)	115.7 (6)	-
C(8)—C(7)—O(7)	122.4 (3)	121.3 (7)	-
C(7)—C(8)—C(9)	113.1 (3)	112.3 (6)	-
N(1)—C(8)—O(8)	-	-	120.8 (5)
C(9)—C(8)—O(8)	-	-	122.0 (4)
C(9)—C(8)—N(1)	-	-	117.2 (4)
C(8)—C(9)—C(10)	112.1 (3)	109.7 (6)	110.4 (4)
C(11)—C(10)—C(9)	111.6 (3)	111.3 (7)	112.6 (4)
C(12)—C(11)—C(10)	114.4 (3)	114.4 (7)	-
O(112)—C(11)—O(111)	-	-	122.1 (5)
C(10)—C(11)—O(111)	-	-	122.4 (4)
C(10)—C(11)—O(112)	-	-	115.4 (4)
O(122)—C(12)—O(121)	123.1 (3)	120.3 (7)	-
C(11)—C(12)—O(121)	122.6 (3)	123.2 (8)	-
C(11)—C(12)—O(122)	114.3 (3)	116.5 (7)	-

As far as possible similar conditions were used for the data collection and refinement of all three structures. Standard reflections were recentred every 100 reflections and scanned every hour to check for crystal movement and stability. 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, alkyl and amino H atoms were fixed geometrically and refined with isotropic displacement parameters. The carboxylic H-atom coordinates for (I) and (III) were located from the difference Fourier syntheses. For (II), the carboxylic H-atom coordinates could not be located. All non-H atoms were refined with anisotropic displacement parameters. The high *R* factor found for (II) is associated with poor crystal quality and the fact that the crystal was present in the peracid and could not otherwise be obtained by recrystallization.

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

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71729 (39 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1018]

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2-Phthalimidoethanoic Acid Monohydrate (I) and 6-Phthalimidohexanoic Acid Monohydrate (II)

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Abstract

The hydrogen-bond networks of two ω-phthalimidoaliphatic carboxylic acid monohydrates, $C_{10}H_7NO_4.H_2O$ (I) and $C_{14}H_{15}NO_4.H_2O$ (II), have been investigated.

Comment

As part of a general study of the stability of organic peracids and peracid salts (Kariuki & Jones, 1990),

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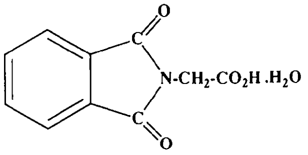
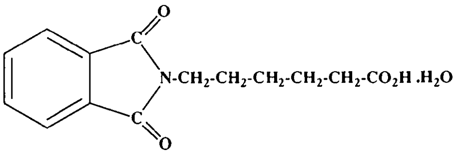
and in particular of the role of hydrogen bonding in controlling the crystal structure of peracid and acid amides (Feeder & Jones, 1992), we report here the structures for two ω-phthalimidoaliphatic carboxylic acid monohydrates.

The bond angles and distances for both structues are similar to those reported for phthalimide-related derivatives (Eckhart, 1972; Ribar, Stankovic & Halasi, 1976; Ribar, Stankovic, Herak, Halasi & Djuric, 1974; Feeder & Jones, 1993). The ionic form reported for 5-halogeno-2-phthalimido benzoic acid monohydrates (Mayer & Pratt, 1963) is not observed for either structure. The molecular geometries of (I) and (II) differ. For (I) there are two planar groups, the phthalimido group, *A*, planar to within 0.028 Å, and the aliphatic acid group, *B*, planar to within 0.009 Å. The dihedral angle *A*(I)*B*(I) is 82.3°. (II) can be thought of as consisting of three groups. The phthalimido group, *A*, is planar to within 0.029 Å, the alkyl chain, *C*, is planar to within 0.030 Å, and the dihedral angle *A*(II)*C*(II) is 69.0°. The carboxyl group (COOH), *D*, is planar to within 0.251 Å; the dihedral angles with *A* and *C* are: *A*(II)*D*(II) 71.6° and *C*(II)*D*(II) 138.5°.



(I)



(II)

Similar hydrogen bonding is exhibited in both compounds, although the packing arrangements are different (see Figs. 2 and 3). For (I), the O···O distances suggest that water acts as a hydrogen-bond donor to the phthalimido groups, thereby linking molecules related by translation along the *a* axis into chains (O···O=C 2.990, O···O=C 2.972 Å). In addition, chains related by the twofold screw axis are connected *via* acid-proton to water O-atom hydrogen bonds (O···O 2.572 Å). The result of this is, therefore, the development of sheets within the crystal. These sheets are then linked by hydrogen bonding through water proton to acid carbonyl interactions (O···O=C 2.985 Å). The inability to locate the water protons is likely to result from disorder required for this arrangement of in-plane and inter-plane hydrogen bonds. In (II), the water protons are again

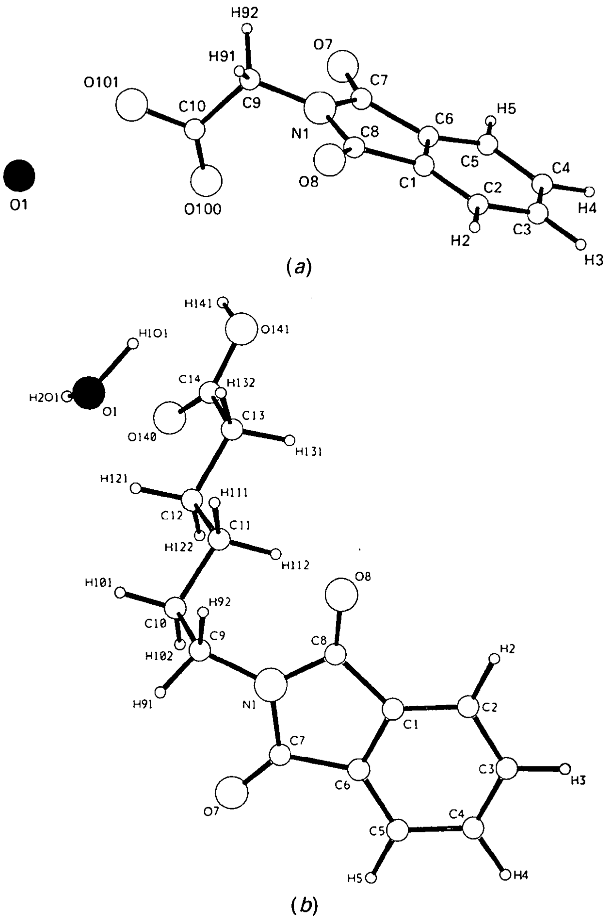


Fig. 1. Atom-numbering scheme for (*a*) (I) and (*b*) (II).

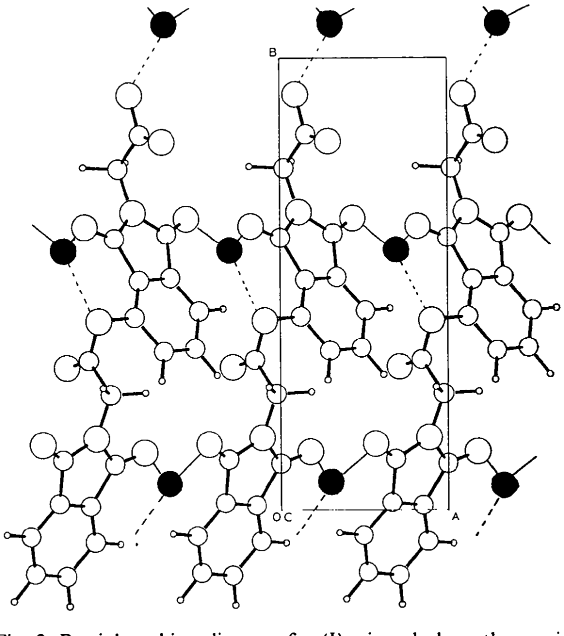


Fig. 2. Partial packing diagram for (I), viewed along the *c* axis.

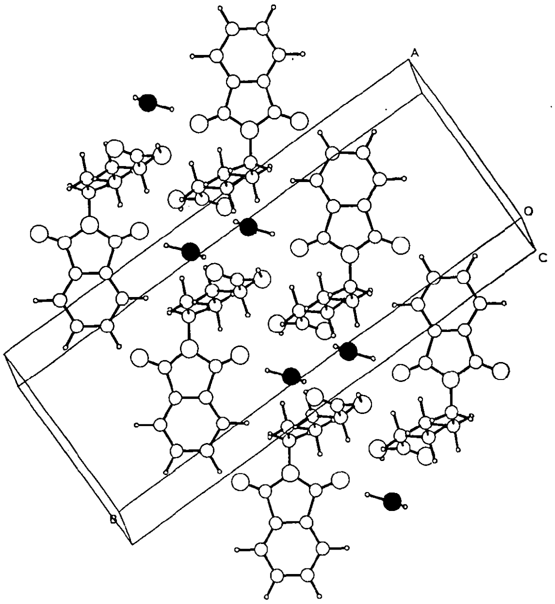


Fig. 3. Packing diagram for (II), viewed perpendicular to the phthalimido ring.

involved in linking organic molecules into chains; however, this time the connections are from one water proton to one phthalimido carbonyl (O—H···O=C 1.534, O···O=C 2.745 Å) and one water proton to one carboxyl carbonyl (O—H···O=C 1.865, O···O=C 2.770 Å). The organic molecules are related by a [101] translation. Chains related by an inversion centre are connected by carboxyl-proton to water O-atom hydrogen bonding (O—H···O 1.374, O···O 2.595 Å). In this way the hydrogen bonding extends to form a 'block'. Blocks related by the c glide are then held together, not by hydrogen bonding, but by π–π stacking of the rings.

Experimental

Crystals of compound (I) were obtained by slow evaporation from water/ethanol at room temperature. Crystals of compound (II) were obtained by slow evaporation from aqueous acetic acid at room temperature.

Compound (I)

Crystal data

$C_{10}H_7NO_4.H_2O$
$M_r = 223.1$
Monoclinic
$P2_1/c$
$a = 10.000\ (2)$ Å
$b = 13.788\ (3)$ Å
$c = 14.216\ (3)$ Å
$\beta = 149.20\ (2)°$
$V = 1004$ Å^3
$Z = 4$
$D_x = 1.48$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 9$–$14°$
$\mu = 0.11$ mm^{-1}
$T = 295$ K
Plate-like
0.3 × 0.3 × 0.1 mm
White, transparent

Data collection

CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3062 measured reflections
2914 independent reflections
1383 observed reflections $[F > 3\sigma(F)]$
$R_{\rm int} = 0.012$

$\theta_{\rm max} = 30°$
$h = -14 \rightarrow 14$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 19$
2 standard reflections monitored every 100 reflections intensity variation: none

Refinement

Refinement on F
$R = 0.060$
$wR = 0.060$
1383 reflections
147 parameters
Only H-atom U's refined
Unit weights applied

$(\Delta/\sigma)_{\rm max} = 0.002$
$\Delta\rho_{\rm max} = 0.38$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.20$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (II)

Crystal data

$C_{14}H_{15}NO_4.H_2O$
$M_r = 279.1$
Monoclinic
$P2_1/c$
$a = 8.771\ (3)$ Å
$b = 22.553\ (5)$ Å
$c = 7.257\ (2)$ Å
$\beta = 99.33\ (6)°$
$V = 1417$ Å^3
$Z = 4$
$D_x = 1.31$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 8$–$12°$
$\mu = 0.09$ mm^{-1}
$T = 295$ K
Rod-like
0.5 × 0.1 × 0.1 mm
White, transparent

Data collection

CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4362 measured reflections
4126 independent reflections
1734 observed reflections $[F > 3\sigma(F)]$
$R_{\rm int} = 0.031$

$\theta_{\rm max} = 30°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 31$
$l = -10 \rightarrow 10$
2 standard reflections monitored every 100 reflections intensity variation: none

Refinement

Refinement on F
$R = 0.054$
$wR = 0.054$
1734 reflections
199 parameters
Unit weights applied
$(\Delta/\sigma)_{\rm max} = 0.005$

$\Delta\rho_{\rm max} = 0.18$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.20$ e Å^{-3}
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)

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

	x	y	z	U_{eq}
(I)				
N(1)	0.1114 (6)	0.6606 (2)	0.1229 (4)	0.039
O(1)	0.3049 (8)	1.0766 (2)	0.3099 (5)	0.076
O(7)	−0.1762 (7)	0.6294 (3)	0.0692 (5)	0.077
O(8)	0.4329 (6)	0.6469 (2)	0.2052 (4)	0.059
O(100)	0.2868 (6)	0.8177 (2)	0.3341 (4)	0.049
O(101)	0.0936 (7)	0.9209 (2)	0.1179 (4)	0.055
C(1)	0.3359 (7)	0.5167 (3)	0.2580 (5)	0.035
C(2)	0.5089 (9)	0.4423 (3)	0.3456 (6)	0.046
C(3)	0.4845 (10)	0.3601 (3)	0.3876 (6)	0.052
C(4)	0.2947 (10)	0.3537 (3)	0.3418 (6)	0.060
C(5)	0.1213 (10)	0.4296 (3)	0.2531 (6)	0.056
C(6)	0.1488 (8)	0.5107 (3)	0.2143 (5)	0.039
C(7)	0.0008 (8)	0.6036 (3)	0.1266 (6)	0.047
C(8)	0.3135 (8)	0.6131 (3)	0.1985 (5)	0.038
C(9)	0.0247 (8)	0.7576 (3)	0.0477 (6)	0.041
C(10)	0.1544 (8)	0.8336 (3)	0.1865 (6)	0.040
(II)				
N(1)	0.4831 (3)	0.3120 (1)	0.6665 (4)	0.048
O(1)	0.1219 (4)	0.5530 (2)	0.4847 (4)	0.080
O(7)	0.3648 (3)	0.2205 (1)	0.6499 (4)	0.073
O(8)	0.6633 (3)	0.3858 (1)	0.6764 (4)	0.069
O(140)	0.0923 (3)	0.4556 (1)	−0.1605 (4)	0.079
O(141)	0.2629 (3)	0.5255 (1)	−0.1839 (4)	0.081
C(1)	0.7288 (4)	0.2819 (2)	0.6400 (4)	0.048
C(2)	0.8844 (4)	0.2785 (2)	0.6287 (5)	0.061
C(3)	0.9458 (4)	0.2222 (2)	0.6148 (5)	0.071
C(4)	0.8559 (5)	0.1723 (2)	0.6115 (5)	0.070
C(5)	0.6995 (4)	0.1757 (2)	0.6229 (5)	0.057
C(6)	0.6387 (4)	0.2316 (1)	0.6358 (4)	0.047
C(7)	0.4783 (4)	0.2504 (2)	0.6503 (4)	0.049
C(8)	0.6292 (4)	0.3338 (2)	0.6615 (4)	0.049
C(9)	0.3494 (4)	0.3484 (2)	0.6894 (5)	0.059
C(10)	0.2522 (4)	0.3651 (2)	0.5033 (5)	0.056
C(11)	0.3303 (4)	0.4088 (2)	0.3893 (5)	0.052
C(12)	0.2279 (4)	0.4237 (1)	0.2050 (5)	0.050
C(13)	0.2988 (4)	0.4701 (2)	0.0943 (5)	0.055
C(14)	0.2060 (4)	0.4824 (2)	−0.0944 (5)	0.053

Table 2. *Selected geometric parameters* (Å, °)

(I)			
C(1)—C(2)	1.376 (7)	C(7)—O(7)	1.206 (7)
C(2)—C(3)	1.392 (8)	C(8)—C(1)	1.488 (8)
C(3)—C(4)	1.382 (9)	C(8)—N(1)	1.394 (7)
C(4)—C(5)	1.398 (8)	C(8)—O(8)	1.202 (7)
C(5)—C(6)	1.377 (7)	C(9)—N(1)	1.446 (7)
C(6)—C(1)	1.377 (7)	C(9)—C(10)	1.509 (7)
C(6)—C(7)	1.485 (7)	C(10)—O(100)	1.196 (7)
C(7)—N(1)	1.392 (7)	C(10)—O(101)	1.322 (6)
C(7)—N(1)—C(8)	112.2 (4)	C(5)—C(6)—C(1)	122.0 (5)
C(9)—N(1)—C(8)	123.9 (4)	C(6)—C(7)—N(1)	105.5 (4)
C(9)—N(1)—C(7)	123.9 (4)	O(7)—C(7)—N(1)	124.1 (5)
C(6)—C(1)—C(8)	108.4 (4)	O(7)—C(7)—C(6)	130.4 (5)
C(2)—C(1)—C(8)	129.7 (5)	C(1)—C(8)—N(1)	105.6 (4)
C(2)—C(1)—C(6)	121.9 (5)	O(8)—C(8)—N(1)	124.1 (5)
C(3)—C(2)—C(1)	116.4 (5)	O(8)—C(8)—C(1)	130.2 (5)
C(4)—C(3)—C(2)	121.6 (5)	C(10)—C(9)—N(1)	112.3 (4)
C(3)—C(4)—C(5)	121.1 (5)	O(101)—C(10)—C(9)	109.5 (4)
C(4)—C(5)—C(6)	116.9 (5)	O(100)—C(10)—C(9)	125.4 (5)
C(1)—C(6)—C(7)	108.3 (4)	O(100)—C(10)—O(101)	125.1 (5)
C(5)—C(6)—C(7)	129.7 (5)		
(II)			
C(1)—C(2)	1.383 (6)	C(9)—N(1)	1.462 (6)
C(2)—C(3)	1.389 (7)	C(9)—C(10)	1.522 (6)
C(3)—C(4)	1.372 (7)	C(10)—C(11)	1.520 (6)
C(4)—C(5)	1.389 (7)	C(11)—C(12)	1.523 (6)
C(5)—C(6)	1.377 (6)	C(12)—C(13)	1.514 (6)
C(6)—C(1)	1.380 (6)	C(13)—C(14)	1.501 (6)
C(6)—C(7)	1.489 (6)	C(14)—O(140)	1.198 (6)
C(7)—N(1)	1.395 (6)	C(14)—O(141)	1.312 (6)
C(7)—O(7)	1.202 (6)	O(141)—H(141)	1.247 (40)
C(8)—C(1)	1.484 (6)	H(1O1)—O(1)	1.227 (73)
C(8)—N(1)	1.379 (6)	H(2O1)—O(1)	0.953 (60)
C(8)—O(8)	1.211 (6)		
C(7)—N(1)—C(8)	111.6 (4)	O(7)—C(7)—C(6)	129.2 (4)
C(9)—N(1)—C(8)	124.7 (4)	C(1)—C(8)—N(1)	106.7 (4)
C(9)—N(1)—C(7)	123.7 (4)	O(8)—C(8)—N(1)	124.0 (4)
C(6)—C(1)—C(8)	107.9 (4)	O(8)—C(8)—C(1)	129.3 (4)
C(2)—C(1)—C(8)	130.8 (4)	C(10)—C(9)—N(1)	112.4 (4)
C(2)—C(1)—C(6)	121.3 (4)	C(9)—C(10)—C(11)	114.0 (4)
C(3)—C(2)—C(1)	116.9 (5)	C(10)—C(11)—C(12)	111.6 (4)
C(4)—C(3)—C(2)	121.5 (4)	C(13)—C(12)—C(11)	112.6 (4)
C(3)—C(4)—C(5)	121.5 (5)	C(14)—C(13)—C(12)	113.7 (4)
C(4)—C(5)—C(6)	116.9 (5)	O(141)—C(14)—C(13)	113.2 (4)
C(1)—C(6)—C(7)	107.9 (4)	O(140)—C(14)—C(13)	124.5 (4)
C(5)—C(6)—C(7)	130.3 (4)	O(140)—C(14)—O(141)	122.3 (4)
C(5)—C(6)—C(1)	121.8 (4)	H(141)—O(141)—C(14)	109.9 (17)
C(6)—C(7)—N(1)	105.9 (4)	H(2O1)—O(1)—H(1O1)	108.9 (42)
O(7)—C(7)—N(1)	124.9 (4)		

Standard reflections were recentred every 100 reflections and scanned every hour to check for crystal movement and stability. 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). For both (I) and (II), the ring and alkyl H atoms were fixed geometrically and refined with isotropic displacement parameters. For (II), the carboxylic acid and water H-atom coordinates were located from the difference Fourier synthesis, and refined with isotropic displacement parameters. The corresponding atoms for (I), however, could not be located. All non-H atoms were refined with anisotropic displacement parameters. Molecular graphics were pproduced using *PLUTO* (Motherwell & Clegg, 1978).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71714 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1031]

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