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representing the amido group (C1, N1, C7, O7 and C8). The angles between the best planes for *A* and *B* are 40.0(1)° in (I) and 33.8(1)° in (II). The angle between the COO group and the amide group is similar for the two structures, *i.e.* 23.2(3)° for (I) and 31.5(1)° for (II).

The intermolecular hydrogen bonding for (I) and (II) is similar and results in ribbons, comparable with those found in one form of 6-oxo-6-(phenylamino)hexanoic acid (Feeder & Jones, 1994) and a *p*-amidobenzoic acid

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Two ω -Oxo- ω -(phenylamino) Aliphatic Acids

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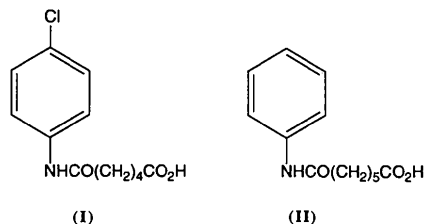
Abstract

The crystal structures of 6-(4-chlorophenylamino)-6-oxohexanoic acid, C₁₂H₁₄ClNO₃ (I), and 7-oxo-7-(phenylamino)heptanoic acid, C₁₃H₁₇NO₃ (II), have been determined. The hydrogen-bond networks found in both structures are very similar to that found in one form of 6-oxo-6-(phenylamino)hexanoic acid previously determined by us [Feeder & Jones (1994). *Acta Cryst.* **C50**, 816–820].

Comment

These structure determinations are part of a general study of the stability of organic peracids and peracid salts (Kariuki & Jones, 1990), and in particular of the role of hydrogen bonding in controlling the crystal structure of peracid and acid amides (Feeder & Jones, 1992).

The bond lengths and angles found are similar to those found for other ω -oxo- ω -(phenylamino) aliphatic acids (Feeder & Jones, 1994) and *p*-amidobenzoic acids (Feeder & Jones, 1993).



Each molecule is considered to contain two planar groups denoted as moiety *A*, the benzene ring [C1–C6, including Cl for (I)] and moiety *B*, the atoms

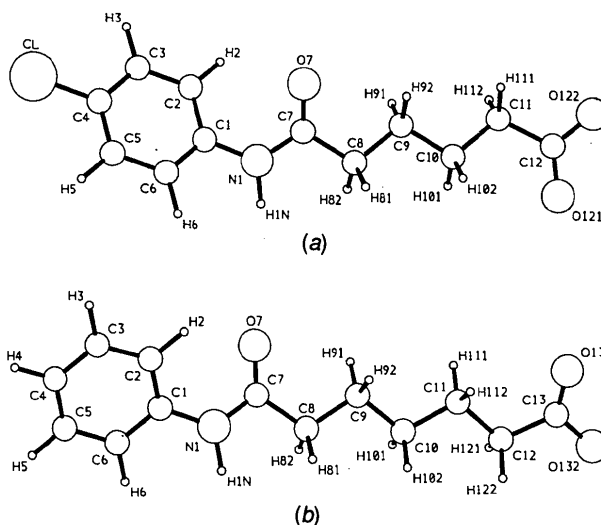


Fig. 1. Atom-numbering scheme for (a) (I) and (b) (II).

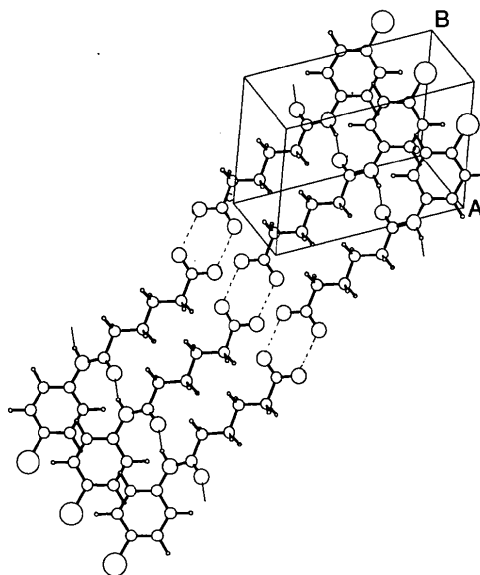


Fig. 2. Packing diagram for (I), viewed perpendicular to the plane of the benzene rings, demonstrating that a Cl-bounded ribbon-type structure is produced through intermolecular hydrogen bonding.

(Feeder & Jones, 1993). Fig. 2 illustrates one of these ribbons for the structure of (I). Two hydrogen-bond motifs are present. The first is the familiar carboxylic acid dimer motif (Leiserowitz, 1976). The second motif links dimers related by translation into ribbons through linear amide–amide interactions. The dihedral angles $A^{\wedge}B$ for the two structures are typical of this mode of hydrogen bonding in aromatic amides (Haisa, Kashino, Ueno, Shinozaki & Matsuzaki, 1980).

Experimental Compound (I)

Crystal data

$C_{12}H_{14}ClNO_3$
 $M_r = 255.69$
 Triclinic
 $P\bar{1}$
 $a = 4.863$ (5) Å
 $b = 9.963$ (3) Å
 $c = 14.024$ (3) Å
 $\alpha = 69.99$ (2)°
 $\beta = 76.70$ (4)°
 $\gamma = 80.28$ (4)°
 $V = 618.2$ (7) Å³
 $Z = 2$
 $D_x = 1.374$ Mg m⁻³

Data collection

Enraf–Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction:
 none
 2266 measured reflections
 2169 independent reflections
 1560 observed reflections
 $[I > 2\sigma(I)]$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.0432$
 $wR(F^2) = 0.1360$
 $S = 1.251$
 2169 reflections
 161 parameters
 $w = 1/[\sigma^2(F_o^2) + (0.0805P)^2 + 0.0909P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.751$
 $\Delta\rho_{\max} = 0.300$ e Å⁻³
 $\Delta\rho_{\min} = -0.355$ e Å⁻³

Compound (II)

Crystal data

$C_{13}H_{17}NO_3$
 $M_r = 235.28$

Mo $K\alpha$ radiation
 $\lambda = 0.71069$ Å
 Cell parameters from 25
 reflections
 $\theta = 9-15^\circ$
 $\mu = 0.305$ mm⁻¹
 $T = 293$ (2) K
 Plate-like
 $0.40 \times 0.40 \times 0.30$ mm
 Colourless

$R_{\text{int}} = 0.0092$
 $\theta_{\max} = 24.97^\circ$
 $h = -5 \rightarrow 5$
 $k = -11 \rightarrow 11$
 $l = 0 \rightarrow 16$
 2 standard reflections
 monitored every 100
 reflections
 intensity variation: none

Extinction correction:
 $F_c^* = kF_c[1 + (0.001x \times F_c^2 \lambda^3 / \sin 2\theta)]^{-1/4}$
 Extinction coefficient:
 0.0265 (56)
 Atomic scattering factors
 from *International Tables
 for Crystallography* (1992),
 Vol. C, Tables 4.2.6.8 and
 6.1.1.4)

Mo $K\alpha$ radiation
 $\lambda = 0.71069$ Å

Triclinic
 $P\bar{1}$
 $a = 5.001$ (1) Å
 $b = 7.972$ (7) Å
 $c = 16.843$ (2) Å
 $\alpha = 76.863$ (10)°
 $\beta = 80.667$ (10)°
 $\gamma = 76.14$ (5)°
 $V = 630.7$ (6) Å³
 $Z = 2$
 $D_x = 1.239$ Mg m⁻³

Data collection

Enraf–Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction:
 none
 2286 measured reflections
 2204 independent reflections
 1286 observed reflections
 $[I > 2\sigma(I)]$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.0468$
 $wR(F^2) = 0.1283$
 $S = 1.250$
 2204 reflections
 161 parameters
 $w = 1/[\sigma^2(F_o^2) + (0.0809P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

Cell parameters from 25
 reflections
 $\theta = 8-14^\circ$
 $\mu = 0.088$ mm⁻¹
 $T = 293$ (2) K
 Plate
 $0.50 \times 0.30 \times 0.10$ mm
 Colourless

$R_{\text{int}} = 0.0150$
 $\theta_{\max} = 24.97^\circ$
 $h = -5 \rightarrow 5$
 $k = -9 \rightarrow 9$
 $l = 0 \rightarrow 20$
 2 standard reflections
 monitored every 100
 reflections
 intensity variation: none
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.272$ e Å⁻³
 $\Delta\rho_{\min} = -0.196$ 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 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$				
Compound (I)	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Cl	0.3794 (2)	0.85543 (8)	0.11253 (6)	0.1011 (4)
N1	0.6621 (3)	0.3153 (2)	0.43434 (13)	0.0429 (4)
O122	0.2039 (3)	-0.3811 (2)	0.98004 (13)	0.0719 (6)
C10	0.5113 (4)	-0.1116 (2)	0.7417 (2)	0.0482 (6)
O7	0.2251 (3)	0.2392 (2)	0.49571 (12)	0.0609 (5)
C1	0.5906 (4)	0.4438 (2)	0.35716 (15)	0.0386 (5)
C9	0.3884 (4)	0.0246 (2)	0.6693 (2)	0.0495 (6)
C2	0.3451 (5)	0.5310 (2)	0.3754 (2)	0.0512 (6)
C7	0.4780 (4)	0.2231 (2)	0.49938 (15)	0.0416 (5)
C11	0.2925 (5)	-0.1779 (3)	0.8352 (2)	0.0579 (6)
C3	0.2804 (5)	0.6566 (2)	0.3002 (2)	0.0602 (6)
O121	0.6400 (3)	-0.3709 (2)	0.89223 (13)	0.0725 (6)
C6	0.7712 (5)	0.4853 (3)	0.2634 (2)	0.0568 (6)
C4	0.4623 (6)	0.6958 (2)	0.2071 (2)	0.0582 (6)
C12	0.3928 (4)	-0.3186 (2)	0.9063 (2)	0.0498 (6)
C8	0.6044 (4)	0.0943 (2)	0.5763 (2)	0.0472 (6)
C5	0.7074 (6)	0.6120 (3)	0.1881 (2)	0.0677 (7)
Compound (II)				
N1	0.2955 (3)	0.6751 (2)	0.12533 (11)	0.0514 (5)
O7	0.7357 (3)	0.7008 (2)	0.07703 (10)	0.0704 (5)
C9	0.6174 (4)	0.7993 (3)	-0.08988 (13)	0.0536 (6)
C7	0.4954 (4)	0.7163 (3)	0.06487 (13)	0.0504 (6)
O132	1.0005 (3)	0.9233 (2)	-0.40256 (10)	0.0759 (6)
C10	0.4995 (4)	0.8977 (3)	-0.16814 (13)	0.0559 (6)
O131	0.6569 (3)	1.1107 (3)	-0.46215 (10)	0.0800 (6)
C12	0.5795 (4)	1.0108 (3)	-0.32006 (13)	0.0581 (6)

C8	0.3950 (4)	0.7889 (3)	-0.01781 (13)	0.0595 (7)
C11	0.7066 (4)	0.9000 (3)	-0.24448 (13)	0.0542 (6)
C1	0.3199 (4)	0.6129 (3)	0.20974 (13)	0.0477 (5)
C2	0.5536 (5)	0.4992 (3)	0.2388 (2)	0.0610 (7)
C13	0.7660 (5)	1.0094 (3)	-0.39804 (13)	0.0546 (6)
C4	0.3365 (6)	0.4897 (4)	0.3763 (2)	0.0809 (9)
C6	0.0973 (5)	0.6655 (3)	0.26476 (15)	0.0641 (7)
C5	0.1047 (5)	0.6039 (4)	0.3476 (2)	0.0759 (8)
C3	0.5590 (6)	0.4386 (4)	0.3220 (2)	0.0775 (8)

Table 2. Selected geometric parameters (Å, °)

Compound (I)			
C1—C4	1.742 (2)	C9—C8	1.511 (3)
N1—C7	1.343 (3)	C2—C3	1.376 (3)
N1—C1	1.416 (3)	C7—C8	1.510 (3)
O122—C12	1.272 (3)	C11—C12	1.494 (3)
C10—C9	1.513 (3)	C3—C4	1.370 (4)
C10—C11	1.513 (3)	O121—C12	1.227 (3)
O7—C7	1.224 (3)	C6—C5	1.383 (3)
C1—C6	1.375 (3)	C4—C5	1.364 (4)
C1—C2	1.380 (3)		
C7—N1—C1	125.4 (2)	C4—C3—C2	119.7 (2)
C9—C10—C11	112.1 (2)	C1—C6—C5	120.5 (2)
C6—C1—C2	119.1 (2)	C5—C4—C3	120.7 (2)
C6—C1—N1	119.9 (2)	C5—C4—C1	119.9 (2)
C2—C1—N1	121.1 (2)	C3—C4—C1	119.4 (2)
C10—C9—C8	113.1 (2)	O121—C12—O122	122.7 (2)
C3—C2—C1	120.4 (2)	O121—C12—C11	121.7 (2)
O7—C7—N1	122.8 (2)	O122—C12—C11	115.5 (2)
O7—C7—C8	121.5 (2)	C7—C8—C9	113.3 (2)
N1—C7—C8	115.6 (2)	C4—C5—C6	119.5 (2)
C12—C11—C10	115.1 (2)		
O121...O122 ⁱ	2.617	N1...O7 ⁱⁱ	2.958

Symmetry codes: (i) 1 - x, -y - 1, 2 - z; (ii) 1 + x, y, z.

Compound (II)			
N1—C7	1.349 (3)	C12—C13	1.483 (3)
N1—C1	1.410 (3)	C12—C11	1.512 (3)
O7—C7	1.224 (2)	C1—C6	1.376 (3)
C9—C10	1.504 (3)	C1—C2	1.379 (3)
C9—C8	1.509 (3)	C2—C3	1.378 (4)
C7—C8	1.499 (3)	C4—C3	1.364 (4)
O132—C13	1.209 (3)	C4—C5	1.372 (4)
C10—C11	1.514 (3)	C6—C5	1.374 (3)
O131—C13	1.304 (3)		
C7—N1—C1	127.8 (2)	C6—C1—N1	118.0 (2)
C10—C9—C8	111.8 (2)	C2—C1—N1	122.8 (2)
O7—C7—N1	122.9 (2)	C3—C2—C1	119.6 (3)
O7—C7—C8	123.0 (2)	O132—C13—O131	122.2 (2)
N1—C7—C8	114.1 (2)	O132—C13—C12	123.5 (2)
C9—C10—C11	114.9 (2)	O131—C13—C12	114.3 (2)
C13—C12—C11	115.1 (2)	C3—C4—C5	119.6 (3)
C7—C8—C9	115.8 (2)	C5—C6—C1	120.7 (2)
C12—C11—C10	112.2 (2)	C6—C5—C4	120.0 (3)
C6—C1—C2	119.2 (2)	C4—C3—C2	121.0 (2)
O131...O132 ⁱ	2.644	N1...O7 ⁱⁱ	2.988

Symmetry codes: (i) 2 - x, 2 - y, -1 - z; (ii) x - 1, y, z.

The coordinates of the carboxylic acid H atoms could not be located in sensible positions. All other H atoms were fixed geometrically.

Data collection, cell refinement and data reduction: *CAD-4 Software* (Enraf-Nonius, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1994). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1070). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two Nitrogen-Containing Analogues of Chorismic Acid

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Abstract

The cyclohexane rings in both the title molecules, methyl 3-[(*tert*-butyloxycarbonyl)amino]-4-hydroxy-1-cyclohexanecarboxylate (3), C₁₃H₂₃NO₅, and methyl 4-[(*tert*-butyloxycarbonyl)amino]-3-hydroxy-1-cyclohexanecarboxylate (6), C₁₃H₂₃NO₅, have regular chair forms. The urethane side chains are extended, adopting the type b *trans-trans* conformation, as is generally observed for urethanes containing a secondary N atom. In the crystals, the *trans* planar NH—CO moieties of the urethane side chains and the hydroxyl substituents participate in extensive hydrogen bonding.