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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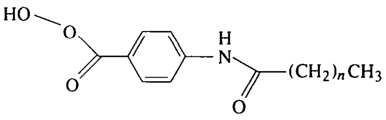
Abstract

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.

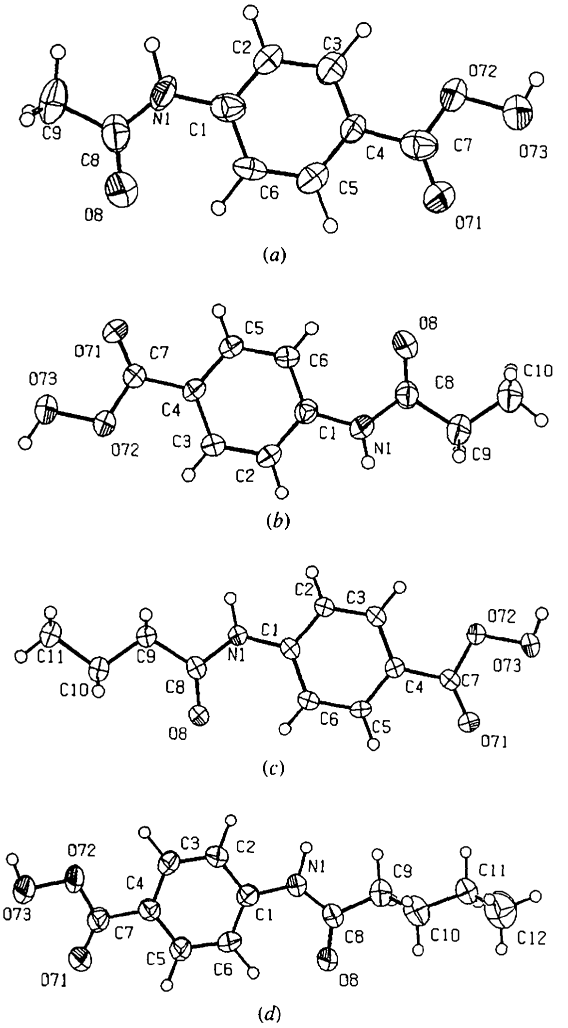

Fig. 1. Atomic numbering scheme for (*a*) (I), (*b*) (II), (*c*) (III) and (*d*) (IV). Ellipsoids are drawn at the 50% probability level.

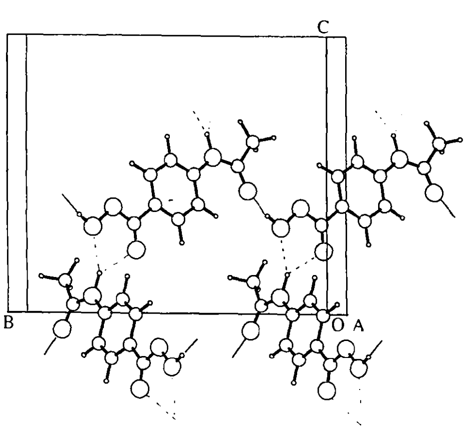

Fig. 2. A partial-packing diagram for (I), viewed at 10° to the *a* axis, illustrating that the intermolecular hydrogen bonding extends in three dimensions.

centred, as defined by Taylor, Kennard & Versichel (1984), involving both the peracid carbonyl and terminal peroxy O atoms as hydrogen-bond acceptors [(I) N(1)—H(1N)···O(71) 2.044, N(1)···O(71) 2.938, N(1)—H(1N)···O(73) 2.212, N(1)···O(73) 3.191 Å; (II) N(1)—H(1N)···O(71) 2.148, N(1)···O(71) 2.911, N(1)—H(1N)···O(73) 2.549, N(1)···O(73) 3.307 Å]. For both types, the complete hydrogen-bond arrangement involves interactions with components along each crystallographic axis and therefore gives rise to a three-dimensional network. This is illustrated by the partial packing diagram shown for (I) in Fig. 2.

Hydrogen bonds from the peracid proton to the amide carbonyl O atom are also formed in the structures of (III) and (IV) but this time between molecules related by translation along the *a* axis only [(III) O(73)—H(73)···O(8) 1.835, O(73)···O(8) 2.673 Å; (IV) O(73)—H(73)···O(8) 1.675, O(73)···O(8) 2.597 Å]. The amido proton is hydrogen bonded to a peracid carbonyl O atom between molecules related by a *b* glide in (III) and the twofold screw in (IV). No three-centred interaction is seen [(III) N(1)—H(1N)···O(71) 1.996, N(1)···O(71) 2.954 Å; (IV) N(1)—H(1N)···O(71) 1.949, N(1)···O(71) 3.023 Å]. Hence for these two structures the hydrogen-bond networks are two dimensional. This is illustrated by the partial packing diagrams shown in Figs. 3 and 4.

Each of the hydrogen-bond motifs found within these structures are very different to those found for a series of *p*-amidobenzoic acids (Feeder & Jones, 1993). In those structures carboxylic acid dimers were found to be linked through linear amide–amide hydrogen bonds to form ribbons or sheets.

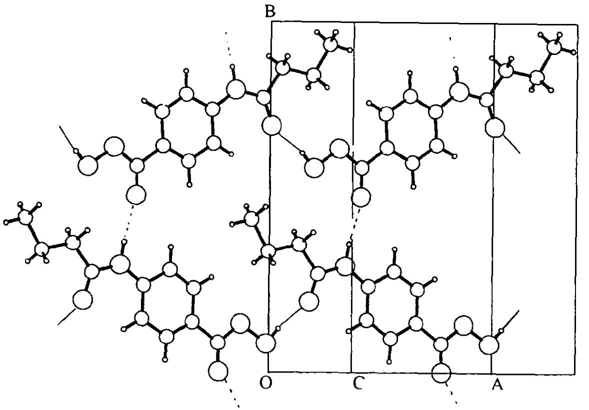

Fig. 3. A partial-packing diagram for (III), viewed at 30° to the *c* axis, illustrating that the intermolecular hydrogen bonding generates two-dimensional sheets.

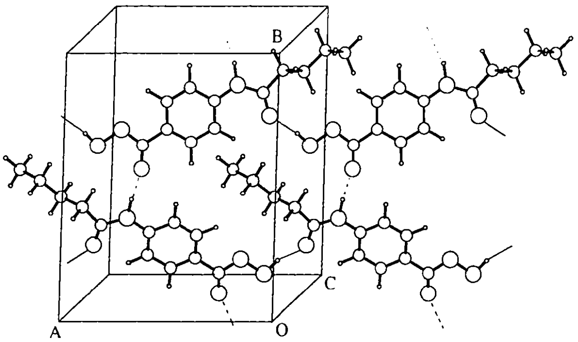

Fig. 4. A partial-packing diagram for (IV), viewed perpendicular to the plane of the benzene ring of the asymmetric unit, illustrating that the intermolecular hydrogen bonding again generates two-dimensional sheets despite the increased chain length.

Experimental

Compounds (I), (III) and (IV) were prepared by slow evaporation from ethanol. All compounds were provided by Interox plc.

Compound (I)

Crystal data

$C_9H_9NO_4$
M_r = 195.18
Monoclinic
Cc
a = 5.054 (1) Å
b = 14.747 (5) Å
c = 12.568 (3) Å
β = 92.88 (2)°
V = 935.5 (4) Å^3
Z = 4
D_x = 1.39 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 8–15°
μ = 0.10 mm^{-1}
T = 295 K
Rod
0.5 × 0.2 × 0.2 mm
White

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2930 measured reflections
2930 independent reflections
532 observed reflections [$F > 3\sigma(F)$]

θ_{max} = 30°
$h = -7 \rightarrow 7$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 17$
2 standard reflections monitored every 100 reflections intensity decay: none

Refinement

Refinement on F
R = 0.056
wR = 0.056
S = 1.184
532 reflections
132 parameters
No weights applied
$(\Delta/\sigma)_{max}$ = 0.226

$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (II)

Crystal data

$C_{10}H_{11}NO_4$
M_r = 209.20
Monoclinic
Cc
a = 4.8853 (10) Å
b = 15.680 (4) Å
c = 12.793 (3) Å
β = 92.21 (2)°
V = 979.3 (4) Å^3
Z = 4
D_x = 1.419 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 11–15°
μ = 0.111 mm^{-1}
T = 296 (2) K
Rod
0.5 × 0.2 × 0.2 mm
White

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1480 measured reflections
1480 independent reflections
882 observed reflections [$I > 2\sigma(I)$]

θ_{max} = 30°
$h = -6 \rightarrow 6$
$k = 0 \rightarrow 22$
$l = 0 \rightarrow 17$
2 standard reflections monitored every 100 reflections intensity decay: 0.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0412
$wR(F^2)$ = 0.1180
S = 0.977
1480 reflections
142 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.100P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = −0.152
$\Delta\rho_{max}$ = 0.164 e Å^{-3}
$\Delta\rho_{min}$ = −0.162 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)

Compound (III)

Crystal data

$C_{11}H_{13}NO_4$
M_r = 223.2
Orthorhombic
Pbcm
a = 10.750 (3) Å
b = 14.477 (6) Å
c = 6.875 (3) Å
V = 1069.9 (7) Å^3
Z = 4
D_x = 1.39 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 8–15°
μ = 0.11 mm^{-1}
T = 295 K
Cuboid
0.2 × 0.2 × 0.2 mm
White transparent

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1826 measured reflections
1826 independent reflections
791 observed reflections [$F > 3\sigma(F)$]

θ_{max} = 30°
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 9$
2 standard reflections monitored every 100 reflections intensity decay: none

Refinement

Refinement on F
$R = 0.054$
$wR = 0.054$
$S = 0.88$
791 reflections
105 parameters
No weights applied
$(\Delta/\sigma)_{\max} = 0.120$
$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (IV)

Crystal data

$C_{12}H_{15}NO_4$
$M_r = 237.26$
Monoclinic
$P2_1/c$
$a = 10.729$ (5) Å
$b = 14.026$ (7) Å
$c = 8.654$ (3) Å
$\beta = 111.59$ (3)°
$V = 1211$ Å^3
$Z = 4$
$D_x = 1.30$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 0.092$ mm^{-1}
$T = 295$ K
Rod
0.4 × 0.2 × 0.2 mm
White transparent

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2238 measured reflections
2238 independent reflections
842 observed reflections [$F > 3\sigma(F)$]
$\theta_{\max} = 25°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 16$
$l = -10 \rightarrow 10$
2 standard reflections monitored every 100 reflections intensity decay: none

Refinement

Refinement on F
$R = 0.068$
$wR = 0.068$
$S = 1.286$
842 reflections
164 parameters
No weights applied
$(\Delta/\sigma)_{\max} = 0.22$
$\Delta\rho_{\max} = 0.46$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ 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)*

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	U_{eq}
(I)				
C(1)	0.0	0.1106 (8)	0.0	0.048 (7)
C(2)	−0.137 (3)	0.0457 (8)	0.0508 (11)	0.057 (7)
C(3)	−0.330 (3)	−0.0058 (9)	−0.0028 (10)	0.059 (7)
C(4)	−0.382 (3)	0.0102 (8)	−0.1099 (10)	0.076 (6)
C(5)	−0.248 (3)	0.0766 (8)	−0.1602 (10)	0.067 (9)
C(6)	−0.056 (3)	0.1272 (8)	−0.1069 (9)	0.053 (7)
C(7)	−0.577 (3)	−0.0422 (8)	−0.1767 (11)	0.055 (7)
C(8)	0.347 (3)	0.2286 (8)	0.0304 (11)	0.055 (7)
C(9)	0.538 (3)	0.2652 (7)	0.1173 (12)	0.075 (6)
N(1)	0.197 (2)	0.1598 (6)	0.0620 (10)	0.051 (6)
O(8)	0.328 (3)	0.2615 (7)	−0.0576 (10)	0.086 (6)
O(71)	−0.616 (3)	−0.0336 (6)	−0.2698 (9)	0.083 (6)
O(72)	−0.705 (2)	−0.1005 (6)	−0.1189 (8)	0.069 (5)
O(73)	−0.888 (3)	−0.1519 (7)	−0.1883 (10)	0.081 (6)
(II)				
C(1)	−0.2745 (8)	0.1055 (2)	0.2044 (3)	0.0333 (6)†
C(2)	−0.1250 (9)	0.0424 (2)	0.1551 (3)	0.0439 (8)†
C(3)	0.0675 (8)	0.0050 (2)	0.2113 (3)	0.0433 (8)†
C(4)	0.1129	0.0092 (2)	0.3178	0.0344 (7)†
C(5)	−0.0356 (9)	0.0721 (2)	0.3665 (3)	0.0405 (7)†
C(6)	−0.2273 (9)	0.1200 (2)	0.3104 (3)	0.0425 (8)†
C(7)	0.3122 (8)	−0.0411 (2)	0.3820 (3)	0.0356 (7)†
C(8)	−0.6231 (9)	0.2169 (2)	0.1626 (4)	0.0404 (7)†
C(9)	−0.8002 (10)	0.2455 (3)	0.0687 (4)	0.0527 (9)†
C(10)	−0.9830 (12)	0.3201 (3)	0.0904 (5)	0.0647 (11)†
N(1)	−0.4648 (8)	0.1496 (2)	0.1406 (3)	0.0409 (6)†
O(8)	−0.6192 (9)	0.2515 (2)	0.2481 (3)	0.0586 (7)†
O(71)	0.3549 (9)	−0.0337 (2)	0.4741 (3)	0.0533 (7)†
O(72)	0.4405 (9)	−0.0974 (2)	0.3227 (3)	0.0574 (8)†
O(73)	0.6335 (9)	−0.1479 (2)	0.3863 (3)	0.0618 (8)†
(III)				
C(1)	0.3513 (4)	0.2451 (3)	1/4	0.033 (2)
C(2)	0.4663 (4)	0.2887 (3)	1/4	0.037 (3)
C(3)	0.5740 (4)	0.2377 (3)	1/4	0.037 (3)
C(4)	0.5685 (4)	0.1416 (3)	1/4	0.034 (3)
C(5)	0.4537 (4)	0.0984 (3)	1/4	0.044 (3)
C(6)	0.3445 (4)	0.1497 (3)	1/4	0.046 (3)
C(7)	0.6819 (4)	0.0832 (3)	1/4	0.038 (3)
C(8)	0.1234 (4)	0.2809 (3)	1/4	0.039 (3)
C(9)	0.0409 (4)	0.3661 (3)	1/4	0.048 (3)
C(10)	−0.0963 (4)	0.3463 (3)	1/4	0.048 (3)
C(11)	−0.1717 (5)	0.4363 (3)	1/4	0.108 (3)
N(1)	0.2460 (3)	0.3035 (3)	1/4	0.040 (2)
O(8)	0.0856 (3)	0.2016 (2)	1/4	0.055 (2)
O(71)	0.6854 (3)	0.0011 (2)	1/4	0.061 (3)
O(72)	0.7832 (3)	0.1379 (2)	1/4	0.051 (3)
O(73)	0.8958 (3)	0.0823 (3)	1/4	0.057 (3)
(IV)				
C(1)	0.3509 (6)	0.7856 (5)	0.2302 (9)	0.044 (5)
C(2)	0.4755 (7)	0.8283 (5)	0.2668 (10)	0.058 (5)
C(3)	0.5801 (7)	0.7787 (5)	0.2534 (9)	0.053 (5)
C(4)	0.5640 (7)	0.6841 (5)	0.1997 (9)	0.045 (4)
C(5)	0.4404 (7)	0.6412 (5)	0.1643 (10)	0.053 (5)
C(6)	0.3349 (7)	0.6914 (5)	0.1794 (10)	0.052 (5)
C(7)	0.6723 (7)	0.6266 (5)	0.1818 (11)	0.057 (5)
C(8)	0.1269 (8)	0.8139 (5)	0.2437 (11)	0.100 (6)
C(9)	0.0457 (8)	0.8880 (6)	0.2898 (14)	0.089 (7)
C(10)	−0.0827 (9)	0.9045 (6)	0.1633 (16)	0.103 (8)
C(11)	−0.1721 (8)	0.9747 (5)	0.2138 (13)	0.081 (6)
C(12)	−0.3002 (10)	0.9909 (7)	0.0695 (13)	0.099 (8)
N(1)	0.2493 (5)	0.8411 (4)	0.2523 (8)	0.055 (4)
O(8)	0.0854 (5)	0.7331 (4)	0.2073 (9)	0.100 (4)
O(71)	0.6657 (5)	0.5440 (4)	0.1438 (7)	0.070 (4)
O(72)	0.7801 (5)	0.6809 (4)	0.2082 (9)	0.097 (4)
O(73)	0.8847 (6)	0.6236 (5)	0.1864 (12)	0.134 (6)

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

Table 2. *Comparison of bond lengths and angles (Å, °)*

C(1)—C(2)	1.359 (16)	1.394 (5)	1.388 (8)	1.391 (10)
C(2)—C(3)	1.383 (16)	1.380 (5)	1.373 (8)	1.361 (11)
C(3)—C(4)	1.378 (12)	1.390 (4)	1.393 (8)	1.395 (11)
C(4)—C(5)	1.363 (15)	1.386 (5)	1.384 (8)	1.385 (11)
C(5)—C(6)	1.372 (15)	1.380 (5)	1.389 (8)	1.379 (11)
C(6)—C(1)	1.381 (13)	1.386 (4)	1.384 (8)	1.383 (11)
C(7)—C(4)	1.480 (16)	1.478 (4)	1.484 (8)	1.468 (11)
C(7)—O(71)	1.184 (14)	1.194 (4)	1.191 (7)	1.199 (10)
C(7)—O(72)	1.316 (15)	1.335 (4)	1.345 (7)	1.333 (10)
O(72)—O(73)	1.454 (12)	1.455 (3)	1.454 (6)	1.447 (9)
O(73)—H(73)	0.84 (9)	0.98 (8)	0.85 (7)	0.95 (12)
C(1)—N(1)	1.429 (14)	1.396 (4)	1.412 (7)	1.409 (10)
C(8)—N(1)	1.341 (14)	1.345 (4)	1.358 (7)	1.343 (10)
C(8)—O(8)	1.207 (14)	1.220 (5)	1.218 (8)	1.216 (10)
C(8)—C(9)	1.519 (16)	1.521 (5)	1.518 (8)	1.502 (12)

C(9)—C(10)		1.503 (6)	1.502 (8)	1.429 (13)
C(10)—C(11)			1.534 (9)	1.546 (13)
C(11)—C(12)				1.495 (14)
C(8)—N(1)—C(1)	127.8 (10)	129.9 (3)	129.4 (5)	128.7 (7)
C(2)—C(1)—N(1)	117.3 (10)	115.8 (3)	116.2 (5)	117.3 (7)
C(6)—C(1)—N(1)	122.9 (11)	124.9 (3)	123.7 (5)	124.0 (7)
C(6)—C(1)—C(2)	119.8 (11)	119.2 (3)	120.0 (5)	118.7 (7)
C(3)—C(2)—C(1)	121.4 (11)	120.4 (3)	120.5 (5)	120.9 (7)
C(2)—C(3)—C(4)	118.5 (11)	120.3 (3)	120.0 (5)	120.7 (8)
C(5)—C(4)—C(7)	116.3 (9)	118.4 (3)	118.4 (5)	117.9 (7)
C(3)—C(4)—C(7)	123.6 (9)	122.4 (3)	122.3 (5)	123.5 (8)
C(3)—C(4)—C(5)	120.1 (10)	119.2 (3)	119.3 (5)	118.5 (7)
C(6)—C(5)—C(4)	121.3 (11)	120.7 (3)	120.8 (5)	120.6 (8)
C(5)—C(6)—C(1)	118.9 (11)	120.2 (3)	119.3 (5)	120.5 (8)
C(72)—C(7)—C(4)	111.0 (10)	110.6 (3)	109.3 (5)	109.8 (7)
C(71)—C(7)—C(4)	125.3 (11)	125.6 (3)	126.5 (5)	126.2 (8)
C(71)—C(7)—O(72)	124 (1)	123.9 (3)	124.2 (5)	124.0 (8)
O(73)—O(72)—C(7)	109 (1)	110.5 (2)	110.4 (4)	109.1 (6)
O(73)—O(73)—O(72)	104 (7)	104 (4)	100 (5)	101 (7)
H(73)—O(73)—O(72)	104 (7)	104 (4)	99.7 (5)	101 (7)
C(8)—C(8)—N(1)	123.2 (3)	123.2 (3)	123.4 (5)	122.4 (8)
C(9)—C(8)—N(1)	113.9 (11)	112.3 (3)	111.8 (5)	116.1 (8)
C(9)—C(8)—O(8)	122.3 (11)	124.5 (3)	124.8 (5)	121.4 (8)
C(10)—C(9)—C(8)	113.9 (5)		114.8 (5)	113.5 (9)
C(9)—C(10)—C(11)			110.9 (5)	114.2 (10)
C(12)—C(11)—C(10)				109.6 (10)

Table 3. *Ring-plane geometry (Å, °) for (I), (II) and (IV)*

	Maximum deviations			Dihedral angles		
	*A**	*B*	*C*	*AB*	*AC*	*BC*
(I)	0.009	0.013	0.008	4.0	4.8	8.5
(II)	0.003	0.007	0.071	5.6	2.8	8.4
(IV)	0.007	0.037	0.102	6.0	7.3	1.9

* Moiety *A* comprises C(1)–C(6), moiety *B* C(1),N(1),C(8),O(8),C(9) and moiety *C* O(71),C(7),O(72),O(73),H(73).

In all four compounds, the ring, alkyl and amine H atoms were fixed geometrically, while the coordinates of H(73) were located from the difference Fourier map. All H atoms were refined with isotropic displacement parameters.

For all compounds, data collection: *CAD*-4 (Enraf–Nonius, 1989); cell refinement: *CAD*-4; data reduction: *CAD*-4; program used to solve structure: *SHELXS*86 (Sheldrick, 1990). For compounds (I), (III) and (IV), program used to refine structure: *SHELX*76 (Sheldrick, 1976). For compound (II), program used to refine structure: *SHELXL*93 (Sheldrick, 1993). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1075). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-(2-Aminophénylthio)-2-(2-thiényl)éthyl 2-Thiényl Cétone

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Abstract

The X-ray structure of the title compound, $C_{17}H_{15}NOS_3$, unambiguously establishes the nature of the unexpected and non-cyclized compound obtained by reacting 2-aminobenzenethiol with 1-(2-thienyl)-3-(2-thienyl)-2-propen-1-one. The molecule as a whole is not planar, the two thienyl rings, *P*1 and *P*2, and the phenyl ring, *P*3, are planar and make (*P*1, *P*2), (*P*1, *P*3) and (*P*2, *P*3) dihedral angles of 75.9 (2), 31.9 (2) and 70.7 (1)°, respectively. Atoms S1 and O exhibit a *Z* conformation around the C10—C14 bond.

Commentaire

Les benzodiazépines appartiennent à une série chimique très importante en thérapeutique utiliseés pour ses propriétés anxiolytiques. La recherche de nouveaux analogues a conduit l'un d'entre nous (JCL) à envisager la synthèse du composé 2,4-bis(2-thiényl)-2,3-dihydro-1,5-benzothiazépine dans lequel un atome de soufre a remplacé un groupement —NH— de la 1,5-diazépine.

L'étude radiocristallographique montre que le composé obtenu n'est pas une 1,5-benzothiazépine mais un dérivé non cyclisé, la 2-(2-aminophénylthio)-2-(2-thiényl)éthyl 2-thiényl cétone (I). Dans ce cas, les

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