

Refinement

Refinement on F	$\Delta\rho_{\max} = 0.14 \text{ e } \text{\AA}^{-3}$
R = 0.045	$\Delta\rho_{\min} = -0.13 \text{ e } \text{\AA}^{-3}$
wR = 0.054	Extinction correction:
S = 2.566	isotropic (Zachariasen, 1963)
1712 reflections	Extinction coefficient:
194 parameters	$0.232(2) \times 10^{-4}$
H atoms refined isotropically	Atomic scattering factors
$w = 4F_o^2/[\sigma^2(F_o^2) + 0.0004F_o^4]$	from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV)
$(\Delta/\sigma)_{\max} = 0.021$	

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

U_{iso} for H atoms; $U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$ for all others.

	x	y	z	$U_{\text{eq}}/U_{\text{iso}}$
O1	0.55026 (9)	0.6516 (2)	0.64473 (7)	0.0624 (3)
C1	0.5015 (1)	0.4492 (3)	0.65840 (9)	0.0428 (4)
C2	0.5938 (1)	0.2828 (3)	0.68246 (9)	0.0501 (4)
C3	0.6678 (1)	0.1537 (4)	0.7050 (1)	0.0717 (6)
C4	0.4016 (1)	0.3851 (3)	0.57852 (8)	0.0443 (4)
C5	0.3183 (1)	0.5401 (3)	0.5415 (1)	0.0674 (6)
C6	0.2248 (2)	0.4888 (4)	0.4706 (1)	0.0858 (7)
C7	0.2133 (2)	0.2834 (4)	0.4365 (1)	0.0798 (6)
C8	0.2951 (2)	0.1297 (3)	0.4724 (1)	0.0695 (5)
C9	0.3902 (1)	0.1803 (3)	0.54372 (9)	0.0547 (5)
C10	0.4565 (1)	0.4806 (3)	0.73422 (8)	0.0471 (4)
C11	0.4766 (1)	0.6685 (3)	0.7816 (1)	0.0715 (6)
C12	0.4373 (2)	0.6863 (4)	0.8516 (1)	0.0987 (7)
C13	0.3783 (2)	0.5217 (5)	0.8736 (1)	0.1005 (8)
C14	0.3572 (2)	0.3347 (5)	0.8266 (1)	0.0903 (7)
C15	0.3965 (1)	0.3132 (3)	0.7571 (1)	0.0662 (5)
H10H	0.582 (2)	0.641 (3)	0.608 (1)	0.121 (8)
H3	0.722 (2)	0.039 (4)	0.722 (1)	0.126 (8)

Table 2. Selected geometric parameters (\AA , $^\circ$)

O1—C1	1.430 (2)	C1—C4	1.530 (2)
O1—H10H	0.83 (2)	C1—C10	1.530 (2)
C1—C2	1.476 (2)	C2—C3	1.169 (2)
C1—O1—H10H	111 (1)	C2—C1—C10	107.9 (1)
O1—C1—C2	109.5 (1)	C4—C1—C10	110.2 (1)
O1—C1—C4	110.1 (1)	C1—C2—C3	177.2 (2)
O1—C1—C10	107.0 (1)	C2—C3—H3	174 (1)
C2—C1—C4	112.1 (1)		

The 12 phenyl C—C distances have a mean value of 1.377 \AA ; individual e.s.d.'s are 0.002–0.004 \AA , and the e.s.d. of their distribution is 0.010 \AA . Isotropic displacement parameters for the H atoms are in the range 5.9 (4)–11.2 (7) \AA^2 , and C—H distances are 0.90 (2)–1.04 (2) \AA .

Data collection: CAD-4 (Enraf–Nonius, 1977). Cell refinement: CAD-4. Data reduction: PROCESS in MolEN (Fair, 1990). Program(s) used to solve structure: MULTAN (Main *et al.*, 1980) (direct methods). Program(s) used to refine structure: LSFM in MolEN (Fair, 1990). Molecular graphics: ORTEPII (Johnson, 1976). Software used to prepare material for publication: CIF in MolEN (Fair, 1990).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1146). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 2676–2680

Absolute Configuration of Mutilin, 5-Acetylmutilin and 5-Bromoacetylmutilin

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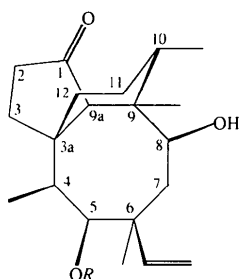
Abstract

The structures of the tricyclic diterpenoids mutilin, C₂₀H₃₂O₃, (1), 5-acetylmutilin, C₂₂H₃₄O₄, (2), and 5-bromoacetylmutilin, C₂₂H₃₃BrO₄, (3), are reported (mu-

tilin is 6-ethenyldecahydro-5,8-dihydroxy-4,6,9,10-tetramethyl-1-oxo-3a,9-propano-3a*H*-cyclopentacyclooctene). All these compounds present similar conformations, confirming the rigidity of their common tricyclic skeleton. The absolute configuration of these natural products is confirmed by the X-ray diffraction analysis of the bromo derivative (3).

Comment

Mutilin (1), isolated from several species of basidiomycetes (Kavanagh, Hervey & Robbins, 1951, 1952), is representative of the now well known family of important diterpenoid metabolites. Of the structurally related derivatives of (1) isolated to date, many exhibit significant *in vitro* antibiotic activity against gram-positive bacteria, and low animal toxicity (Hogenauer, 1979; Berner, Vypel & Schulz, 1987, and references cited therein). The chemical structure of (1), established by Arigoni (1968) and by Birch, Holzappel & Rickards (1966), contains a rigid tricyclic carbon network with eight stereocentres, seven of which are consecutively installed around the eight-membered ring. The only crystallographic study reported for a congener of mutilin (Dobler & Dürr, 1975) is on the 5-bromoacetyl derivative (3). The data reported for this structure are of quite poor quality, giving very unusual distances and a high *R* factor, so we decided to collect new data for (3); the stereostructure assigned to (3) on the basis of ORD spectral comparison with model compounds, has been unequivocally confirmed by our investigation. In fact, in spite of the use of Mo *K* α radiation, which is not the most appropriate for the determination of the absolute configuration, the Flack (1983) parameter of 0.017 (11) indicates that our data are reliable.



- (1) $R = H$
 (2) $R = -CO-Me$
 (3) $R = -CO-CH_2Br$

The conformations of the five-, six- and eight-membered rings are envelope, chair and chair-boat, respectively. A comparison of the geometric parameters of the three molecules does not reveal any unusual features. Table 4 reports selected bond distances, bond angles and torsion angles of the common part of the compounds;

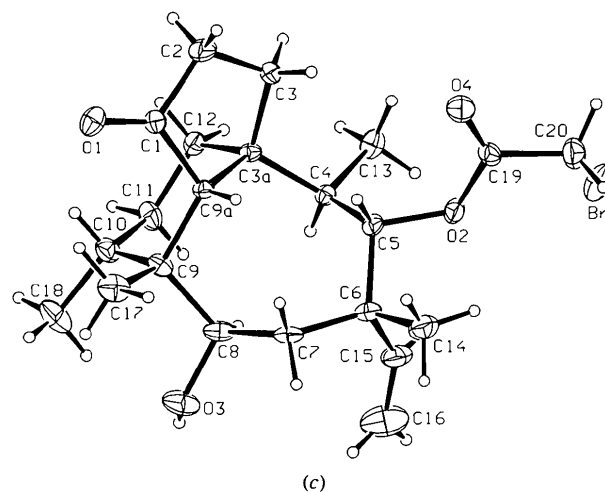
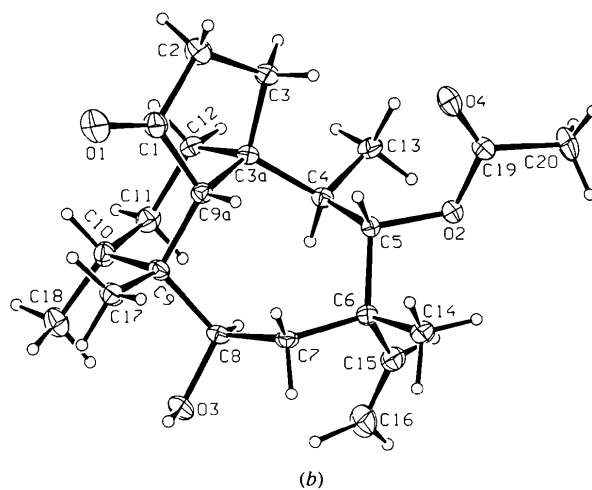
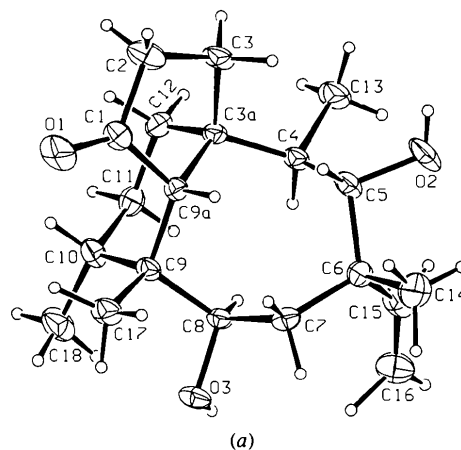


Fig. 1. ORTEP (Johnson, 1976) plots of (a) mutilin, (b) 5-acetylmutilin and (c) 5-bromoacetylmutilin. Displacement ellipsoids are plotted at the 20% probability level.

the greatest differences in chemically equivalent bonds are found for the terminal ethylene group C15=C16, affected by high thermal motion, and for the O2—C5 bond, in a different chemical environment in (1) with respect to (2) and (3). The differences in bond angles and torsion angles are in some cases, but not surprisingly, more pronounced. In particular, the widest torsion angle along the tricyclic skeleton is C(4)—C(5)—C(6)—C(7), with values ranging from -67.5 (3) to -63.3 (3) $^\circ$. These differences are partially due to the different packing of the three compounds. Packing in (1) is characterized by a hydrogen bond between the hydroxy group O2—HO2 and O3ⁱ, with O2 \cdots O3ⁱ 2.798 (4) Å and O2—HO2 \cdots O3ⁱ 171 (3) $^\circ$, and a weaker one between the hydroxy group O3—HO3 and O1ⁱⁱ, with O3 \cdots O1ⁱⁱ 2.872 (4) Å and O3—HO3 \cdots O4ⁱⁱ 167 (3) $^\circ$ [symmetry codes: (i) $\frac{1}{2} - x, -y, -\frac{1}{2} + z$; (ii) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$]. Packing of (2) and of (3) is looser, showing only a weak hydrogen bond between the hydroxy group and an acetyl O atom: in (2) the distance O3 \cdots O4ⁱⁱⁱ is 3.024 (2) Å and the angle O3—HO3 \cdots O4ⁱⁱⁱ is 154 (3) $^\circ$, while in (3), O3 \cdots O4^{iv} is 2.950 Å and O3—HO3 \cdots O4^{iv} is 162 (3) $^\circ$ [symmetry codes: (iii) $-x, \frac{1}{2} + y, \frac{1}{2} - z$; (iv) $1 + x, y, z$].

Experimental

(1) and (2) were produced by basidiomycetes *Pleurotus mutilus* (Fr.) Sacc. and *Pleurotus passeckerianus* Pilat, purified by silica gel chromatography and crystallized from chloroform. (3) was synthesized by esterification of (1) with bromoacetic acid in dichloromethane at room temperature in the presence of dicyclohexylcarbodiimide, and crystallized from *n*-hexane—diisopropyl ether.

Compound (1)

Crystal data

C ₂₀ H ₃₂ O ₃	Mo K α radiation
$M_r = 320.46$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 45 reflections
$P2_12_12_1$	$\theta = 5.56$ – 14.44 $^\circ$
$a = 9.816$ (1) Å	$\mu = 0.075$ mm ⁻¹
$b = 12.422$ (1) Å	$T = 293$ (2) K
$c = 15.234$ (1) Å	Prism
$V = 1857.5$ (3) Å ³	$0.36 \times 0.30 \times 0.24$ mm
$Z = 4$	Colourless
$D_x = 1.146$ Mg m ⁻³	

Data collection

Siemens P4 diffractometer	$\theta_{\max} = 24.99$ $^\circ$
$\theta/2\theta$ scans	$h = 0 \rightarrow 11$
Absorption correction: none	$k = 0 \rightarrow 14$
	$l = -18 \rightarrow 18$
3618 measured reflections	3 standard reflections
3263 independent reflections	monitored every 50 reflections
2430 observed reflections	
$[I > 2\sigma(I)]$	intensity decay: 2.59%
$R_{\text{int}} = 0.0195$	

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.0463$
 $wR(F^2) = 0.1166$
 $S = 1.032$
 3263 reflections
 323 parameters
 All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.0662P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.020$

$\Delta\rho_{\max} = 0.131$ e Å⁻³
 $\Delta\rho_{\min} = -0.124$ e Å⁻³
 Extinction correction: SHELXL93 (Sheldrick, 1993)
 Extinction coefficient: 0.023 (2)
 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) for (1)

	$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$			
	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
O1	0.6151 (3)	-0.2474 (2)	0.10621 (15)	0.0894 (8)
O2	0.1160 (3)	0.0789 (2)	-0.01141 (15)	0.0879 (9)
O3	0.3843 (2)	0.0350 (2)	0.33007 (11)	0.0604 (6)
C1	0.5535 (3)	-0.1773 (2)	0.0686 (2)	0.0576 (8)
C2	0.5508 (5)	-0.1688 (3)	-0.0299 (2)	0.0831 (12)
C3	0.4735 (4)	-0.0660 (3)	-0.0497 (2)	0.0633 (8)
C3a	0.4752 (2)	0.0005 (2)	0.03633 (15)	0.0425 (6)
C4	0.3569 (3)	0.0846 (2)	0.0369 (2)	0.0468 (6)
C5	0.2150 (3)	0.0326 (3)	0.0468 (2)	0.0556 (7)
C6	0.1504 (3)	0.0439 (2)	0.1399 (2)	0.0528 (7)
C7	0.2311 (3)	-0.0130 (2)	0.2128 (2)	0.0502 (7)
C8	0.3760 (3)	0.0238 (2)	0.23558 (14)	0.0426 (6)
C9a	0.4660 (3)	-0.0881 (2)	0.1083 (2)	0.0411 (6)
C9	0.4923 (3)	-0.0536 (2)	0.20497 (15)	0.0438 (6)
C10	0.6345 (3)	0.0008 (2)	0.2070 (2)	0.0554 (7)
C11	0.6442 (3)	0.0916 (3)	0.1392 (2)	0.0567 (7)
C12	0.6149 (3)	0.0543 (3)	0.0466 (2)	0.0565 (7)
C13	0.3629 (5)	0.1584 (3)	-0.0446 (2)	0.0833 (11)
C14	0.0090 (4)	-0.0097 (4)	0.1383 (3)	0.0905 (12)
C15	0.1322 (4)	0.1632 (3)	0.1599 (2)	0.0677 (9)
C16	0.1280 (5)	0.2121 (3)	0.2323 (2)	0.0847 (11)
C17	0.4889 (5)	-0.1546 (3)	0.2637 (2)	0.0693 (9)
C18	0.6829 (4)	0.0402 (4)	0.2969 (2)	0.0851 (12)

Compound (2)

Crystal data

C ₂₂ H ₃₄ O ₄	Mo K α radiation
$M_r = 362.49$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 25 reflections
$P2_12_12_1$	$\theta = 5.2$ – 14.65 $^\circ$
$a = 7.063$ (1) Å	$\mu = 0.080$ mm ⁻¹
$b = 15.863$ (2) Å	$T = 293$ (2) K
$c = 18.027$ (3) Å	Prism
$V = 2019.8$ (5) Å ³	$0.34 \times 0.34 \times 0.16$ mm
$Z = 4$	Colourless
$D_x = 1.192$ Mg m ⁻³	

Data collection

Siemens P4 diffractometer	$\theta_{\max} = 25.00$ $^\circ$
$\theta/2\theta$ scans	$h = 0 \rightarrow 8$
Absorption correction: none	$k = 0 \rightarrow 18$
	$l = -21 \rightarrow 21$
3976 measured reflections	3 standard reflections
3544 independent reflections	monitored every 197 reflections
2694 observed reflections	
$[I > 2\sigma(I)]$	intensity decay: 5.73%
$R_{\text{int}} = 0.0232$	

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.0448$
 $wR(F^2) = 0.0994$
 $S = 1.067$
 3544 reflections
 346 parameters
 H atoms bonded to C20
 calculated, all others
 refined isotropically
 $w = 1/[\sigma^2(F_o^2) + (0.0323P)^2 + 0.1808P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = -0.002$

$\Delta\rho_{\max} = 0.131 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.124 \text{ e } \text{\AA}^{-3}$
 Extinction correction:
SHELXL93 (Sheldrick,
 1993)
 Extinction coefficient:
 0.0065 (7)
 Atomic scattering factors
 from *International Tables*
 for *Crystallography* (1992,
 Vol. C, Tables 4.2.6.8 and
 6.1.1.4)

Absorption correction:
 ψ scan (*SHELXL/PC*;
 Sheldrick, 1990)
 $T_{\min} = 0.397$, $T_{\max} =$
 0.458
 3909 measured reflections
 3677 independent reflections
 2407 observed reflections
 $[I > 2\sigma(I)]$

$h = 0 \rightarrow 11$
 $k = -13 \rightarrow 13$
 $l = -12 \rightarrow 11$
 3 standard reflections
 monitored every 197
 reflections
 intensity decay: 5.50%

Table 2. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
O1	0.1416 (5)	0.09879 (14)	0.46734 (12)	0.0886 (9)
O2	0.0078 (3)	0.08047 (10)	0.10276 (9)	0.0396 (4)
O3	0.3968 (3)	0.33879 (11)	0.27704 (11)	0.0483 (5)
O4	-0.1835 (3)	-0.01154 (13)	0.16127 (11)	0.0557 (6)
C1	0.1477 (4)	0.0745 (2)	0.4041 (2)	0.0469 (7)
C2	0.1054 (7)	-0.0158 (2)	0.3826 (2)	0.0602 (9)
C3	0.1453 (5)	-0.0215 (2)	0.3003 (2)	0.0462 (7)
C3a	0.2712 (4)	0.05548 (14)	0.28135 (14)	0.0341 (6)
C4	0.2633 (4)	0.0753 (2)	0.19657 (14)	0.0342 (6)
C5	0.0745 (4)	0.11699 (15)	0.17300 (13)	0.0327 (6)
C6	0.0826 (4)	0.21334 (15)	0.15716 (14)	0.0364 (6)
C7	0.1218 (4)	0.26447 (15)	0.22870 (10)	0.0363 (6)
C8	0.3153 (4)	0.25596 (15)	0.26777 (14)	0.0346 (6)
C9a	0.1938 (4)	0.1251 (2)	0.33413 (14)	0.0334 (6)
C9	0.3113 (4)	0.20759 (15)	0.34293 (13)	0.0335 (6)
C10	0.5134 (4)	0.1807 (2)	0.3681 (2)	0.0403 (7)
C11	0.5971 (4)	0.1146 (2)	0.3159 (2)	0.0456 (7)
C12	0.4758 (4)	0.0369 (2)	0.3066 (2)	0.0451 (7)
C13	0.3098 (5)	-0.0034 (2)	0.1498 (2)	0.0519 (8)
C14	-0.1150 (5)	0.2411 (2)	0.1298 (2)	0.0502 (7)
C15	0.2257 (5)	0.2311 (2)	0.0969 (2)	0.0483 (7)
C16	0.3493 (6)	0.2928 (2)	0.0936 (2)	0.0663 (10)
C17	0.2208 (5)	0.2647 (2)	0.4019 (2)	0.0438 (7)
C18	0.6556 (5)	0.2522 (2)	0.3797 (2)	0.0569 (8)
C19	-0.1207 (4)	0.0187 (2)	0.1051 (2)	0.0431 (7)
C20	-0.1727 (6)	-0.0094 (2)	0.0281 (2)	0.0744 (11)

Compound (3)

Crystal data

$\text{C}_{22}\text{H}_{33}\text{BrO}_4$

$M_r = 441.39$

Monoclinic

$P2_1$

$a = 9.320 (1) \text{\AA}$

$b = 11.251 (1) \text{\AA}$

$c = 10.671 (1) \text{\AA}$

$\beta = 109.266 (8)^\circ$

$V = 1056.3 (2) \text{\AA}^3$

$Z = 2$

$D_x = 1.388 \text{ Mg m}^{-3}$

Data collection

Siemens P4 diffractometer

$\theta/2\theta$ scans

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{\AA}$

Cell parameters from 37
 reflections

$\theta = 3.62\text{--}17.15^\circ$

$\mu = 1.970 \text{ mm}^{-1}$

$T = 293 (2) \text{ K}$

Prism

$0.45 \times 0.28 \times 0.25 \text{ mm}$

Colourless

$R_{\text{int}} = 0.0267$

$\theta_{\max} = 25.00^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.0444$

$wR(F^2) = 0.0912$

3677 reflections

299 parameters

H atoms of methyl and ethyl
 groups calculated, all
 others refined isotropically
 with $X\text{—H}$ distance
 restraints

$w = 1/[\sigma^2(F_o^2) + (0.0310P)^2 + 0.1348P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 0.241 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.239 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

Atomic scattering factors
 from *International Tables*
 for *Crystallography* (1992,
 Vol. C, Tables 4.2.6.8 and
 6.1.1.4)

Absolute configuration:

Flack (1983) parameter

= 0.017 (11)

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (3)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
Br	0.07855 (8)	0.22061 (6)	1.24072 (5)	0.0808 (2)
O1	0.0777 (4)	-0.0897 (3)	0.4198 (3)	0.0529 (10)
O2	0.1302 (4)	0.0546 (3)	1.0422 (3)	0.0430 (8)
O3	0.5959 (4)	-0.1214 (4)	0.8338 (4)	0.0714 (12)
O4	-0.1130 (4)	0.0074 (3)	0.9374 (4)	0.0611 (10)
C1	0.0686 (5)	-0.0411 (4)	0.5151 (5)	0.0361 (12)
C2	-0.0693 (6)	0.0313 (6)	0.5167 (6)	0.053 (2)
C3	-0.0246 (5)	0.0978 (5)	0.6462 (5)	0.0382 (12)
C3a	0.1509 (5)	0.0923 (4)	0.7011 (4)	0.0304 (10)
C4	0.2085 (5)	0.1167 (4)	0.8543 (4)	0.0329 (11)
C5	0.1832 (5)	0.0118 (4)	0.9359 (4)	0.0342 (11)
C6	0.3237 (5)	-0.0669 (4)	1.0085 (4)	0.0435 (12)
C7	0.3823 (6)	-0.1336 (5)	0.9106 (5)	0.0472 (13)
C8	0.4536 (6)	-0.0660 (5)	0.8200 (6)	0.0471 (14)
C9	0.3529 (5)	-0.0624 (4)	0.6692 (4)	0.0381 (11)
C9a	0.1865 (5)	-0.0309 (4)	0.6553 (4)	0.0311 (10)
C10	0.4067 (6)	0.0353 (5)	0.5941 (5)	0.0494 (13)
C11	0.3809 (7)	0.1610 (5)	0.6373 (5)	0.0510 (13)
C12	0.2167 (6)	0.1834 (4)	0.6270 (4)	0.0389 (13)
C13	0.1394 (5)	0.2328 (5)	0.8846 (4)	0.0512 (13)
C14	0.2706 (7)	-0.1618 (5)	1.0882 (5)	0.067 (2)
C15	0.4430 (7)	0.0110 (6)	1.1064 (5)	0.063 (2)
C16	0.5792 (9)	-0.0020 (8)	1.1555 (9)	0.114 (3)
C17	0.3593 (6)	-0.1857 (5)	0.6096 (5)	0.0598 (15)
C18	0.5696 (7)	0.0229 (6)	0.5886 (7)	0.083 (2)
C19	-0.0142 (7)	0.0456 (4)	1.0316 (5)	0.0434 (12)
C20	-0.0429 (7)	0.0865 (6)	1.1555 (5)	0.062 (2)

Table 4. Selected values of bond distances (\AA), bond angles ($^\circ$) and torsion angles ($^\circ$)

	Mutilin	5-Acetylmutilin	5-Bromo-acetylmutilin
O1—C1	1.205 (3)	1.203 (3)	1.182 (5)
O2—C5	1.435 (3)	1.470 (3)	1.460 (5)
O3—C8	1.448 (3)	1.444 (3)	1.428 (6)
C1—C2	1.504 (4)	1.514 (4)	1.527 (7)
C1—C9a	1.527 (4)	1.531 (4)	1.541 (6)
C2—C3	1.516 (5)	1.513 (4)	1.504 (7)
C3—C3a	1.550 (4)	1.548 (4)	1.545 (6)

C3a—C9a	1.555 (3)	1.557 (3)	1.542 (6)
C3a—C12	1.533 (4)	1.543 (4)	1.540 (6)
C3a—C4	1.563 (3)	1.562 (3)	1.568 (6)
C4—C5	1.542 (4)	1.548 (4)	1.531 (6)
C4—C13	1.545 (4)	1.542 (4)	1.537 (7)
C5—C6	1.560 (4)	1.556 (3)	1.559 (6)
C6—C7	1.537 (4)	1.549 (4)	1.526 (7)
C6—C14	1.540 (5)	1.544 (4)	1.544 (7)
C6—C15	1.523 (4)	1.511 (4)	1.526 (7)
C7—C8	1.534 (4)	1.543 (4)	1.543 (8)
C8—C9	1.564 (4)	1.557 (3)	1.573 (8)
C9—C9a	1.556 (3)	1.558 (3)	1.550 (6)
C9—C10	1.552 (4)	1.557 (4)	1.538 (7)
C9—C17	1.541 (4)	1.536 (4)	1.536 (6)
C10—C11	1.532 (4)	1.529 (4)	1.531 (7)
C10—C18	1.529 (4)	1.528 (4)	1.545 (8)
C11—C12	1.513 (4)	1.512 (4)	1.519 (8)
C15—C16	1.260 (4)	1.313 (5)	1.212 (8)
C2—C1—C9a	109.5 (2)	109.1 (2)	106.4 (4)
C1—C2—C3	105.5 (3)	105.7 (2)	107.2 (4)
C2—C3—C3a	105.9 (2)	106.1 (2)	105.5 (4)
C3—C3a—C9a	102.7 (2)	102.9 (2)	103.1 (3)
C4—C3a—C9a	115.2 (2)	116.3 (2)	116.9 (3)
C9a—C3a—C12	106.8 (2)	106.5 (2)	105.7 (3)
C3a—C4—C5	113.1 (2)	112.7 (2)	113.2 (3)
C4—C5—C6	114.7 (2)	116.0 (2)	117.3 (4)
C5—C6—C7	114.0 (2)	111.6 (2)	111.7 (4)
C6—C7—C8	120.3 (2)	119.5 (2)	120.9 (4)
C7—C8—C9	115.2 (2)	115.1 (2)	117.3 (4)
C9a—C9—C10	106.8 (2)	106.8 (2)	105.9 (4)
C8—C9—C9a	109.3 (2)	109.6 (2)	110.1 (4)
C1—C9a—C3a	101.6 (2)	101.9 (2)	102.4 (3)
C3a—C9a—C9	117.6 (2)	118.1 (2)	119.3 (3)
C9—C10—C11	111.3 (2)	111.3 (2)	113.1 (4)
C10—C11—C12	113.1 (2)	114.1 (2)	112.7 (4)
C3a—C12—C11	113.5 (2)	114.0 (2)	114.5 (4)
C3—C3a—C4—C13	-55.6 (3)	-53.0 (3)	-52.5 (5)
C13—C4—C5—O2	-10.5 (3)	-11.1 (3)	-12.7 (5)
C3a—C4—C5—C6	104.1 (3)	104.5 (2)	102.8 (4)
C4—C5—C6—C7	-63.3 (3)	-67.5 (3)	-65.9 (5)
O2—C5—C6—C14	55.2 (4)	55.3 (3)	55.5 (5)
C5—C6—C7—C8	63.7 (3)	66.8 (3)	66.6 (6)
C6—C7—C8—C9	-107.1 (3)	-108.8 (3)	-108.3 (5)
C3—C3a—C9a—C1	37.4 (3)	36.9 (3)	38.8 (4)
C4—C3a—C9a—C9	-70.9 (3)	-70.9 (3)	-69.8 (5)
C1—C9a—C9—C17	-52.6 (3)	-51.9 (3)	-52.8 (5)
C3a—C9a—C9—C8	66.4 (3)	65.4 (3)	64.6 (5)
O3—C8—C9—C17	48.7 (3)	50.5 (3)	46.4 (5)
C7—C8—C9—C9a	46.2 (3)	46.5 (3)	45.6 (5)
C17—C9—C10—C18	-60.7 (3)	-61.1 (3)	-58.5 (6)
C9a—C9—C10—C11	53.5 (3)	52.9 (3)	52.2 (5)
C8—C9—C10—C11	-66.4 (3)	-66.9 (3)	-67.5 (5)
C9—C10—C11—C12	-57.1 (3)	-55.8 (3)	-54.8 (6)
C10—C11—C12—C3a	56.6 (3)	54.9 (3)	54.2 (6)
C9a—C3a—C12—C11	-51.6 (3)	-49.9 (3)	-50.6 (5)

For all compounds, data collection: *XSCANS* (Fait, 1991); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structures: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *ORTEPII* (Johnson, 1976); 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: NA1195). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-Nitrodibenzofuran and 3-Iododibenzofuran

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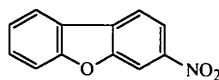
(Received 25 October 1993; accepted 18 July 1995)

Abstract

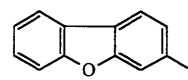
The structures of 3-nitrodibenzofuran, C₁₂H₇NO₃, and 3-iododibenzofuran, C₁₂H₇IO, have been determined. Both molecules are nearly planar and pack in herringbone patterns composed of symmetry related head-to-tail pairs with parallel π systems.

Comment

The title structures, 3-nitrodibenzofuran, (1), and 3-iododibenzofuran, (2), were determined as part of a study of the relationship between NMR spectroscopic parameters and molecular geometry in diphenyl ethers and dibenzofurans.



(1)



(2)