S = 0.741
3415 reflections
236 parameters
H atoms not refined, except H101 on N10
Weighting by Chebychev polynomial (Carruthers & Watkin, 1979)
Extinction correction: Larson (1970)
Extinction coefficient: 174 (14)
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

C1—C23	1.517 (2)	C23—C24	1.529 (2)
C1—C23—C24	114.5 (1)		

All H atoms were located in a difference map, although those connected to C were replaced at ideal positions. A local implementation of the *DIFABS* algorithm (Walker & Stuart, 1983), which evaluates a contribution to F_c (rather than F_o), was applied to the unmerged data and the structure refined to convergence with a Chebychev weighting scheme (Carruthers & Watkin, 1979). At convergence, the reciprocal absorption correction was applied to F_o, equivalent reflections merged and the final difference synthesis computed.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *RC*93 (Watkin, Prout & Lilley, 1994). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *CRYSTALS* (Watkin, Prout, Carruthers & Betteridge, 1996). Molecular graphics: *CAMERON* (Watkin, Prout & Pearce, 1996). Software used to prepare material for publication: *CRYSTALS*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1165). Services for accessing these data are described at the back of the journal.

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A *trans*-1,2-Diarylhexafluorocyclobutane

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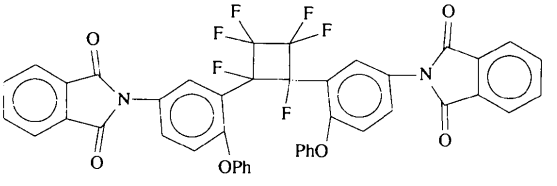
Abstract

Crystal structure analysis establishes the configuration of one of two isomers of the thermal dimerization of *N*-[3-(1,2,2-trifluoroethenyl)-4-phenoxyphenyl]phthalimide to be *trans*-5,5′-[1,2,3,3,4,4-hexafluorocyclobutane-1,2-diylbis(2-phenoxyphenyl)]-*N*,*N*′-bis(phthalimide), $C_{44}H_{24}F_6N_2O_6$. The cyclobutane ring puckers and has a dihedral angle of 23.5 (4)°. The crystal also contains a diethyl ether molecule of solvation, $C_4H_{10}O$.

Comment

N-[3-(1,2,2-Trifluoroethenyl)-4-phenoxyphenyl]phthalimide undergoes [2+2] cycloaddition to give a major and a minor product (64 and 5% yield, respectively) that are *cis*/*trans* isomers of the octa-substituted cyclobutane. Recrystallization of the major product from ether gave crystals suitable for X-ray diffraction analysis. On consideration of steric interactions between adjacent aryl substituents, one would predict that the *trans* isomer would be the major product. This prediction was confirmed as the crystal structure determination established the major isomer to be *trans*-5,5′-[1,2,3,3,4,4-hexafluorocyclobutane-1,2-diylbis(2-phenoxyphenyl)]-*N*,*N*′-bis(phthalimide), (I). A diethyl ether molecule of solvation is also included in the crystal.



(I)

The cyclobutane ring is puckered. The C1—C2—C3 plane forms a dihedral angle of 23.5 (4)° with the C3—C4—C1 plane. A survey of the geometry of cyclobutanes (Allen, 1984) reports the average pucker in acyclic-substituted cyclobutane rings to be 24.3°. The aryl substituents have three planar regions. For the aryl group bonded to the C1 cyclobutane atom, the plane of the phenoxyphenyl group forms a dihedral angle of 67.3 (1)° with the plane of the phenyl ring bonded to the

cyclobutane ring; the plane of the phthalimide group forms a dihedral angle of 47.3 (1)° with the plane of the phenyl ring bonded to the cyclobutane ring. The corresponding dihedral angles for the other aryl group are 99.6 (1)° for the phenoxyphenyl group and 48.8 (1)° for the phthalimide group.

The molecules pack in such a way that allows the phenoxyphenyl groups to form parallel stacking interactions with phenoxyphenyl groups of adjacent molecules. Similarly, the phthalimide groups interact with neighboring phthalimide groups to form parallel stacks. The diethyl ether molecules occupy voids and exhibit high thermal motion.

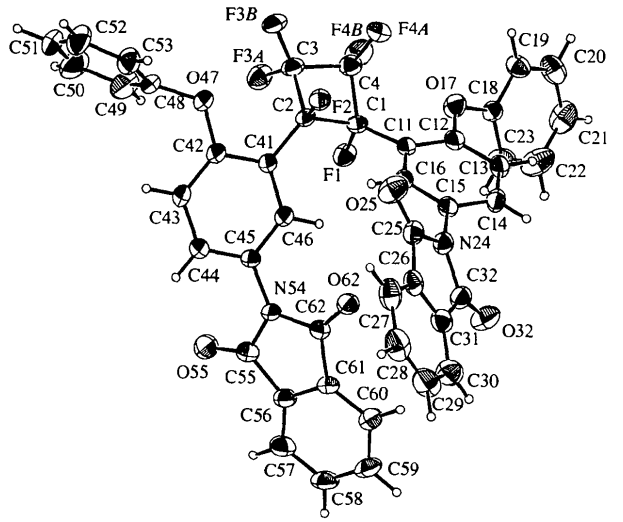


Fig. 1. *ORTEP*II (Johnson, 1976) view of the title molecule with displacement ellipsoids drawn at the 35% probability level.

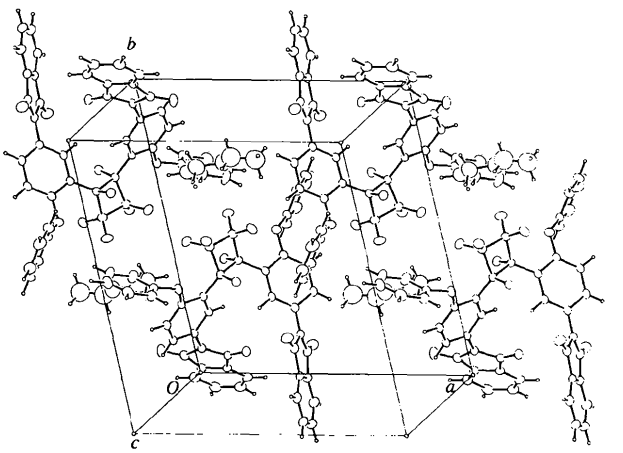


Fig. 2. View of the molecular packing in the unit cell.

Experimental

Details of the preparation and crystallization of the title compound are given in Yamamoto, Swenson & Burton (1994).

Crystal data

$C_{44}H_{24}F_6N_2O_6.C_4H_{10}O$
M_r = 864.81
Triclinic
$P\bar{1}$
a = 13.891 (2) Å
b = 15.426 (3) Å
c = 11.014 (2) Å
α = 102.81 (2)°
β = 104.94 (2)°
γ = 102.71 (2)°
V = 2125 (2) Å^3
Z = 2
D_x = 1.35 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 11–12°
μ = 0.102 mm^{-1}
T = 291 K
Thick plate
0.55 × 0.35 × 0.20 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
8643 measured reflections
7401 independent reflections
4348 reflections with $I > 2\sigma(I)$

R_{int} = 0.022
θ_{max} = 25.0°
h = −16 → 16
k = −17 → 18
l = −13 → 3
4 standard reflections
frequency: 240 min
intensity decay: 17.7%

Refinement

Refinement on F
R = 0.054
wR = 0.078
S = 1.469
4348 reflections
558 parameters
H atoms refined using a riding model, U = 1.3U of the bonded atom

Weighting scheme based on measured e.s.d.'s (Killean & Lawrence, 1969)
$(\Delta/\sigma)_{max}$ = 0.024
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.07 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

F1—C1	1.410 (3)	C1—C2	1.573 (4)
F2—C2	1.395 (3)	C1—C4	1.560 (4)
F3*B*—C3	1.341 (3)	C1—C11	1.491 (5)
F3*A*—C3	1.350 (4)	C2—C3	1.552 (5)
F4*A*—C4	1.348 (3)	C2—C41	1.496 (4)
F4*B*—C4	1.328 (4)	C3—C4	1.541 (5)
F1—C1—C2	107.5 (2)	F3*B*—C3—F3*A*	108.1 (3)
F1—C1—C4	104.7 (3)	F3*B*—C3—C2	118.2 (3)
F1—C1—C11	109.1 (2)	F3*B*—C3—C4	116.5 (2)
C2—C1—C4	88.2 (2)	F3*A*—C3—C2	112.1 (2)
C2—C1—C11	120.8 (3)	F3*A*—C3—C4	111.4 (3)
C4—C1—C11	124.0 (2)	C2—C3—C4	89.7 (3)
F2—C2—C1	107.2 (2)	F4*A*—C4—F4*B*	108.8 (3)
F2—C2—C3	105.5 (2)	F4*A*—C4—C1	111.6 (3)
F2—C2—C41	109.8 (2)	F4*A*—C4—C3	110.8 (2)
C1—C2—C3	88.2 (2)	F4*B*—C4—C1	118.4 (2)
C1—C2—C41	120.6 (2)	F4*B*—C4—C3	117.0 (3)
C3—C2—C41	122.9 (3)	C1—C4—C3	89.0 (2)

During refinement, the anisotropic displacement parameters of C atoms C74 and C75 refined to non-positive-definite values. These atoms were included in the final refinement model with isotropic displacement parameters. All H atoms were included using the riding model, C—H = 0.95 Å. Backgrounds were obtained from analysis of the scan profile (Blessing, Coppens & Becker, 1974).

Data collection: *CAD-4 Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD-4 Operations Manual*. Data reduction: *PROCESS* in *MolEN* (Fair, 1990). Program(s) used to solve structure: direct methods (*MULTAN*80; Main *et al.*, 1980). Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *CIF VAX* in *MolEN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1009). Services for accessing these data are described at the back of the journal.

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Tubingensin B, a Cytotoxic Carbazole Alkaloid from the Sclerotia of *Aspergillus tubingensis*

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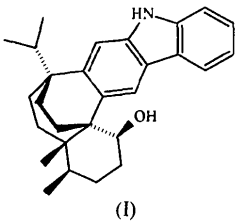
Abstract

The crystal structure of tubingensin B, (1α,4α,4aα,7α,-14bβ)-(−)-2,3,4,4a,5,6,7,9-octahydro-4,4a-dimethyl-7-isopropyl-1*H*-7,14b-ethanobenzo[3,4]cyclohepta[1,2-*b*]carbazol-1-ol, $C_{28}H_{35}NO$, confirms the structure assigned from NMR spectra. The molecule forms hydrogen-bonded sheets parallel to the *bc* plane in the crystal. A disordered molecule of solvent, $CHCl_3$, is included in the crystal.

Comment

Tubingensin B is a cytotoxic carbazole alkaloid with a novel ring system. It was originally isolated from the sclerotia of the fungus *Aspergillus tubingensis*. Tubingensin B is biogenetically related to other metabolites present in the sclerotia of various *Aspergillus* spp.; some of the metabolites may serve as chemical defenses against consumption of the sclerotia by insects (Gloer, 1995). The structure of the title compound, (I), was assigned on the basis of selective INEPT (insensitive nuclei enhanced by polarization transfer), homonuclear decoupling and COSY (correlation spectroscopy) NMR experiments (TePaske, Gloer, Wicklow & Dowd, 1989). The crystal structure analysis confirms the assigned structure.



(I)

The C4–C9 ring is planar (largest deviation from planarity is 0.018 Å for C6). The C2—C3—C10—C11—C12—C27 ring is distorted slightly from planarity to a C11—C12 half-chair conformation [the C10—C11—C12—C27 torsion angle is 6.9 (5)°]. The N1—C2—C3—C4—C9 ring is also slightly distorted from planarity to the N1 envelope conformation (N1 is 0.044 Å from the C2—C3—C4—C9 plane). The C11—C12—C23—C22—C21—C20 ring has a boat conformation. The C15–C20 ring has a chair conformation. The C13—C14—C15—C20—C21—C22—C23 and the C11—C12—C23—C13—C14—C15—C20 seven-membered rings adopt the chair conformation, with the C13, C15, C20 and C23 atoms forming the seat of the chair for both seven-membered rings.

Four symmetry-related molecules are joined by hydrogen bonds (two unique and two related by twofold symmetry). The molecules alternate as donor and acceptor, forming a ring. Each molecule is also involved in a hydrogen bond of a symmetry-related ring of four molecules, thus forming hydrogen-bonded sheets parallel to the *ab* plane. The geometries of the unique hydrogen bonds are given in Table 2.

As the $CHCl_3$ solvent molecule is near a crystallographic twofold axis, the molecule is necessarily disordered. It is further disordered in that it has two orientations with occupancies of 0.30 (1) for orientation *A*