

N(3)	0.7726 (5)	-0.0308 (4)	0.4638 (4)	0.0616
N(4)	0.8378 (5)	0.2834 (4)	0.4044 (4)	0.0685
N(5)	0.7331 (6)	0.1763 (4)	0.0245 (4)	0.0678
N(6)	0.4645 (5)	0.0590 (4)	0.2900 (4)	0.0681
O(1)	0.7095 (6)	0.3597 (5)	0.6440 (6)	0.1292
O(2)	0.5962 (7)	0.2744 (5)	0.7269 (8)	0.1392
O(3)	1.0231 (5)	0.0914 (4)	0.9055 (4)	0.0969
O(4)	1.0818 (5)	-0.0307 (4)	0.8041 (5)	0.0917
O(5)	0.8627 (5)	-0.0785 (4)	0.4381 (4)	0.0896
O(6)	0.6632 (4)	-0.0419 (3)	0.4215 (3)	0.0721
O(7)	0.9231 (6)	0.2386 (5)	0.4553 (5)	0.1115
O(8)	0.8257 (7)	0.3721 (6)	0.4130 (8)	0.1401
O(9)	0.8153 (6)	0.2349 (4)	0.0047 (4)	0.0945
O(10)	0.6763 (5)	0.1166 (5)	-0.0391 (4)	0.0937
O(11)	0.4480 (5)	-0.0251 (4)	0.2460 (4)	0.0949
O(12)	0.3906 (4)	0.1001 (4)	0.3454 (4)	0.0865

Table 2. Geometric parameters (Å, °)

C(1)—C(2)	1.390 (7)	C(1)—C(6)	1.384 (7)
C(1)—C(13)	1.515 (7)	C(2)—C(3)	1.370 (7)
C(2)—N(1)	1.473 (7)	C(3)—C(4)	1.374 (7)
C(4)—C(5)	1.371 (7)	C(4)—N(2)	1.479 (7)
C(5)—C(6)	1.391 (7)	C(6)—N(3)	1.480 (7)
C(7)—C(8)	1.397 (7)	C(7)—C(12)	1.402 (7)
C(7)—C(13)	1.529 (7)	C(8)—C(9)	1.377 (7)
C(8)—N(4)	1.467 (7)	C(9)—C(10)	1.364 (8)
C(10)—C(11)	1.370 (7)	C(10)—N(5)	1.493 (6)
C(11)—C(12)	1.368 (7)	C(12)—N(6)	1.478 (7)
N(1)—O(1)	1.187 (7)	N(1)—O(2)	1.116 (7)
N(2)—O(3)	1.211 (6)	N(2)—O(4)	1.205 (6)
N(3)—O(5)	1.217 (6)	N(3)—O(6)	1.220 (6)
N(4)—O(7)	1.182 (7)	N(4)—O(8)	1.153 (8)
N(5)—O(9)	1.205 (7)	N(5)—O(10)	1.200 (7)
N(6)—O(11)	1.211 (6)	N(6)—O(12)	1.229 (6)
C(6)—C(1)—C(2)	114.1 (5)	C(13)—C(1)—C(2)	120.5 (5)
C(13)—C(1)—C(6)	125.4 (5)	C(3)—C(2)—C(1)	124.8 (5)
N(1)—C(2)—C(1)	120.2 (5)	N(1)—C(2)—C(3)	115.0 (5)
C(4)—C(3)—C(2)	117.7 (5)	C(5)—C(4)—C(3)	121.6 (5)
N(2)—C(4)—C(3)	118.8 (5)	N(2)—C(4)—C(5)	119.6 (5)
C(6)—C(5)—C(4)	117.8 (5)	C(5)—C(6)—C(1)	123.9 (5)
N(3)—C(6)—C(1)	121.2 (5)	N(3)—C(6)—C(5)	114.9 (5)
C(12)—C(7)—C(8)	112.9 (5)	C(13)—C(7)—C(8)	123.0 (5)
C(13)—C(7)—C(12)	124.1 (5)	C(9)—C(8)—C(7)	124.8 (5)
N(4)—C(8)—C(7)	120.0 (5)	N(4)—C(8)—C(9)	115.2 (5)
C(10)—C(9)—C(8)	117.1 (5)	C(11)—C(10)—C(9)	123.0 (5)
N(5)—C(10)—C(9)	118.5 (5)	N(5)—C(10)—C(11)	118.6 (5)
C(12)—C(11)—C(10)	117.0 (5)	C(11)—C(12)—C(7)	125.1 (5)
N(6)—C(12)—C(11)	119.5 (5)	N(6)—C(12)—C(11)	115.3 (5)
C(7)—C(13)—C(1)	117.9 (5)	O(1)—N(1)—C(2)	118.5 (6)
O(2)—N(1)—C(2)	120.1 (6)	O(2)—N(1)—O(1)	121.4 (7)
O(3)—N(2)—C(4)	117.2 (5)	O(4)—N(2)—C(4)	117.2 (6)
O(4)—N(2)—O(3)	125.6 (6)	O(5)—N(3)—C(6)	117.2 (5)
O(6)—N(3)—C(6)	117.7 (5)	O(6)—N(3)—O(5)	125.0 (5)
O(7)—N(4)—C(8)	118.5 (6)	O(8)—N(4)—C(8)	120.0 (6)
O(8)—N(4)—O(7)	121.3 (7)	O(9)—N(5)—C(10)	116.8 (6)
O(10)—N(5)—C(10)	117.4 (6)	O(10)—N(5)—O(9)	125.7 (5)
O(11)—N(6)—C(12)	117.1 (5)	O(12)—N(6)—C(12)	118.3 (5)
O(12)—N(6)—O(11)	124.4 (6)		

An orange crystal of the title compound was obtained according to the procedure of Shipp, Kaplan & Sitzmann (1972). The structure was solved using direct methods (SHELXS86; Sheldrick, 1986) and successive Fourier maps. H-atom positions were calculated. All calculations were performed using CRYSTALS (Watkin, Carruthers & Betteridge, 1985) on a VAX 6310 computer. Final atomic coordinates are given in Table 1, intramolecular bond distances and angles in Table 2. Fig. 2 shows the molecule with the numbering system and Fig. 3 is a stereoscopic view of the packing; both were drawn using ORTEP (Johnson, 1965).

Financial support from 'Institut Curie' (Contrat coopératif, décision No. 88R/03) is gratefully acknowledged.

0108-2701/93/030612-04\$06.00

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry, together with least-squares-planes data, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55250 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1007]

References

- Boonstra, E. G. (1963). *Acta Cryst.* **16**, 816–823.
 Bryden, J. H. (1972). *Acta Cryst.* **B28**, 1395–1398.
 Carper, W. R., Davis, L. P. & Exline, M. W. (1982). *J. Phys. Chem.* **86**, 459–462.
 Clifford, G. & Dudis, D. S. (1990). *Acta Cryst.* **C46**, 650–652.
 Gérard, F. & Hardy, A. (1988). *Acta Cryst.* **C44**, 1283–1287.
 Grammacioli, C. M., Destro, R. & Simonetta, M. (1968). *Acta Cryst.* **B24**, 129–136.
 Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
 Larson, A. C. (1969). In *Crystallographic Computing*, edited by F. R. Ahmed, pp. 291–294. Copenhagen: Munksgaard.
 Nyburg, S. C., Faerman, C. H., Prasad, L., Palleros, D. & Nudelman, N. (1987). *Acta Cryst.* **C43**, 686–689.
 Sheldrick, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.
 Shipp, K. G., Kaplan, L. A. & Sitzmann, M. E. (1972). *J. Org. Chem.* **37**, 1966–1970.
 Simonnin, M. P., Xié, H. Q., Terrier, F., Lelièvre, J. & Farrell, P. G. (1989). *J. Chem. Soc. Perkin Trans. 2*, pp. 1553–1558.
 Trotter, J. (1959). *Acta Cryst.* **12**, 605–607.
 Watkin, D. J., Carruthers, J. R. & Betteridge, P. W. (1985). *Crystals User Guide*. Chemical Crystallography Laboratory, Univ. of Oxford, England.

Acta Cryst. (1993). **C49**, 612–615

4-[2-Benzoyl-1-(benzoylmethyl)ethyl][2.2]-paracyclophane

VLADIMIR G. KHARITONOV, DMITRII YU. ANTONOV,
 VALERIA I. ROZENBERG, VALERII A. NIKANOROV,
 ALEXANDER I. YANOVSKY AND YURI T. STRUCHKOV

Institute of Organoelement Compounds, 28 Vavilov Street, Moscow 117813, Russia

(Received 5 August 1992; accepted 9 October 1992)

Abstract

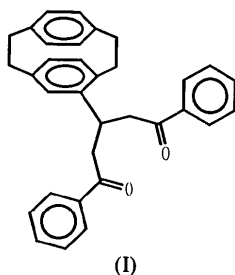
The unsymmetrically substituted [2.2]paracyclophane moiety of the title compound, 1,5-diphenyl-3-(tricyclo[8.2.2.2^{4,7}]hexadeca-4,6,10,12,13,15-hexaen-5-yl)-1,5-pentanedione, exhibits geometrical features typical of known paracyclophane derivatives; the benzene rings are non-planar and have a shallow boat conformation with folding angles of 11.8–14.0°. The dimethylene-bridge

© 1993 International Union of Crystallography

planes are very nearly normal to the mean planes through the paracyclophane benzene nuclei. Both benzoylmethyl groups have very similar bond lengths and angles, although their orientations relative to the paracyclophane moiety differ significantly.

Comment

In the course of our studies into the synthesis and reactivity of alkenyl-substituted [2.2]paracyclophanes we have undertaken the synthesis of 4-[2-benzoyl-1-(benzoylmethyl)ethyl][2.2]paracyclophane (I), an important precursor of paracyclophane-substituted pyrilium salts. As it was impossible to make unambiguous assignment of the structure of (I) on the basis of spectroscopic data, we have carried out an X-ray diffraction study.



Compound (I) belongs to the rather poorly investigated class of unsymmetrically substituted [2.2]paracyclophane derivatives. To our knowledge, only one full structural study of the monosubstituted paracyclophane, *viz* camphenoyloxymethyl[2.2]paracyclophane (II) (Tochtermann, Olsson, Vogt, Peters, Peters & von Schnering, 1987), has been reported as yet. Naturally, the most important geometrical characteristics of the paracyclophane moiety are not affected by substitution. Thus (I) exhibits the same distortions of the aromatic rings previously observed in [2.2]paracyclophane structures, *e.g.* tetraacetoxy[2.2]paracyclophane (Knobler, Maverick, Parker, Trueblood, Weiss, Cram & Helgesen, 1986), 4,16-dichloro[2.2]paracyclophane (Lindeman, Struchkov & Guryshv, 1986). Indeed, both the paracyclophane benzene rings in (I) are distinctly non-planar and have a shallow-boat conformation [the folding angles along the C4...C8, C5...C7, C12...C16 and C13...C15 lines being 14.0(1), 13.0(2), 11.8(2) and 11.7(3) $^{\circ}$] and the endocyclic angles at the four *ipso* atoms within the aromatic nuclei (C3, C6, C11 and C14) are substantially smaller than 120 $^{\circ}$ (116.4–117.0 $^{\circ}$). The dihedral angle formed by the planes of the paracyclophane benzene ring bottoms C4, C5, C7, C8 and C12, C13, C15, C16 is equal to 1.13(8) $^{\circ}$.

The additional substitution in one of the benzene rings causes some geometrical peculiarities. Along with the small differences in folding angles (see above) the paracyclophane benzene rings are characterized by somewhat

different distributions of endocyclic bond lengths. In particular, the C3—C8, C7—C8 and C6—C7 bonds are consistently shorter than the corresponding C3—C4, C4—C5 and C5—C6 bonds on the 'substituted side' of the ring; there are no noticeable bond-length differences in the other paracyclophane benzene ring. The rather significant difference between the exocyclic bond angles at the C3 *ipso* atom [C2—C3—C4 123.3(2), C2—C3—C8 118.5(2) $^{\circ}$] is obviously the result of repulsion of the substituents at the C3 and C4 atoms.

As in unsubstituted [2.2]paracyclophane (Hope, Bernstein & Trueblood, 1972) and some of the previously studied less-substituted paracyclophane derivatives (Tochtermann, Olssen, Vogt, Peters, Peters & von Schnering, 1987; Lindeman, Struchkov & Guryshv, 1986), both of the dimethylene bridges in (I) are almost planar [torsion angles C3—C2—C1—C14 and C6—C9—C10—C11 in (I) are 1.7(4) and $-2.0(5)^{\circ}$, respectively], their mean planes being very nearly normal to the mean planes of the benzene rings.

The different orientations of two —CH₂COPh groups relative to the paracyclophane moiety in (I) may be characterized by the torsion angles C4—C17—C18—C19 and C4—C17—C26—C27 [$-69.1(2)$ and 162.7(2) $^{\circ}$, respectively]. This conformational difference gives rise to the different steric environments of the PhCO groups [one of them forms short intramolecular contacts with the atoms of the paracyclophane nucleus: C19...C4 3.236(3), C19...C5 3.219(3) Å] and thus leads to a significant difference between the C4—C17—C18 and C4—C17—C26 bond angles [114.8(1) and 108.3(1) $^{\circ}$, respectively]. The bond lengths and angles in both benzoylmethyl moieties in (I) are quite similar. The carbonyl groups may be considered coplanar with the corresponding Ph groups to some approximation; the dihedral angles (C18, C19, C20, O1) \wedge (C20, C21, C22, C23, C24, C25) and (C26, C27, C28, O2) \wedge (C28, C29, C30, C31, C32, C33) are 23.6(1) and 11.3(1) $^{\circ}$, respectively.

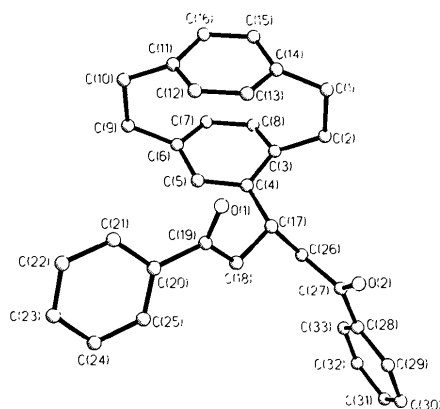


Fig. 1. View of the title compound showing the labelling of the non-H atoms.

Experimental

Crystal data

C₃₃H₃₀O₂M_r = 458.57

Monoclinic

P2₁/c

a = 13.032 (3) Å

b = 21.366 (4) Å

c = 9.041 (2) Å

β = 100.77 (2)°

V = 2473.0 (9) Å³

Z = 4

D_x = 1.232 Mg m⁻³

Mo Kα radiation

λ = 0.71073 Å

Cell parameters from 24 reflections

θ = 13–14°

μ = 0.075 mm⁻¹

T = 293.0 (10) K

Cubes

0.4 × 0.3 × 0.3 mm

Colourless

Data collection

Siemens P3/PC diffractometer

θ/2θ scans

Absorption correction: none

5007 measured reflections

4797 independent reflections

2872 observed reflections

[I > 2σ(I)]

R_{int} = 0.0111θ_{max} = 26.05°

h = 0 → 15

k = 0 → 26

l = -11 → 10

2 standard reflections

monitored every 98

reflections

intensity variation: 1%

Refinement

Refinement on F²

Final R (on F) = 0.0458 for observed reflections

wR (on F²) = 0.0984 for all reflections

S = 1.084

Calculated weights

$$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.1277P]$$

$$P = (F_o^2 + 2F_c^2)/3$$

4766 reflections

Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1987). Program(s) used to refine structure: *SHELXL92* (Sheldrick, 1992).

436 parameters

(Δ/σ)_{max} = -0.038Δρ_{max} = 0.114 e Å⁻³Δρ_{min} = -0.131 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 thermal parameters (Å²)
$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U _{eq}
O1	0.71976 (12)	0.08376 (8)	0.2614 (2)	0.0734 (10)
O2	0.44027 (12)	0.20677 (8)	0.2657 (2)	0.0794 (9)
C1	0.7883 (2)	0.23729 (13)	0.6642 (5)	0.090 (2)
C2	0.6776 (2)	0.21628 (11)	0.6841 (3)	0.070 (2)
C3	0.67028 (14)	0.14678 (9)	0.7152 (2)	0.0515 (10)
C4	0.64383 (13)	0.10138 (9)	0.6004 (2)	0.0441 (9)
C5	0.68464 (14)	0.04148 (9)	0.6283 (2)	0.0460 (10)
C6	0.74747 (14)	0.02443 (9)	0.7655 (2)	0.0502 (11)
C7	0.7480 (2)	0.06489 (10)	0.8845 (2)	0.0562 (12)
C8	0.7107 (2)	0.12497 (11)	0.8586 (2)	0.0576 (13)
C9	0.8263 (2)	-0.02765 (11)	0.7763 (3)	0.068 (2)
C10	0.9367 (2)	-0.00632 (14)	0.7530 (6)	0.099 (2)
C11	0.9418 (2)	0.06235 (11)	0.7187 (3)	0.0713 (11)
C12	0.9020 (2)	0.08447 (13)	0.5758 (3)	0.0688 (12)
C13	0.8646 (2)	0.14484 (12)	0.5537 (3)	0.0631 (12)

C14	0.8666 (2)	0.18516 (10)	0.6747 (3)	0.0639 (13)
C15	0.9268 (2)	0.16727 (13)	0.8110 (3)	0.0720 (15)
C16	0.9637 (2)	0.10696 (13)	0.8327 (3)	0.0773 (14)
C17	0.57489 (14)	0.12030 (10)	0.4513 (2)	0.0493 (10)
C18	0.5548 (2)	0.06838 (12)	0.3335 (2)	0.0558 (11)
C19	0.6474 (2)	0.04798 (11)	0.2665 (2)	0.0546 (11)
C20	0.64774 (15)	-0.01695 (10)	0.2051 (2)	0.0530 (12)
C21	0.7428 (2)	-0.04473 (13)	0.1921 (3)	0.0650 (14)
C22	0.7449 (2)	-0.10425 (13)	0.1337 (3)	0.077 (2)
C23	0.6538 (2)	-0.13644 (14)	0.0852 (3)	0.078 (2)
C24	0.5597 (2)	-0.10971 (12)	0.0961 (3)	0.075 (2)
C25	0.5562 (2)	-0.05063 (11)	0.1578 (2)	0.0612 (13)
C26	0.4688 (2)	0.14215 (12)	0.4835 (2)	0.0543 (11)
C27	0.40255 (15)	0.17988 (9)	0.3614 (2)	0.0514 (11)
C28	0.28769 (14)	0.18372 (8)	0.3601 (2)	0.0468 (10)
C29	0.2219 (2)	0.20791 (10)	0.2345 (3)	0.0608 (13)
C30	0.1155 (2)	0.20840 (12)	0.2274 (3)	0.0745 (14)
C31	0.0735 (2)	0.18544 (11)	0.3449 (3)	0.0705 (13)
C32	0.1367 (2)	0.16267 (11)	0.4706 (3)	0.0640 (13)
C33	0.2437 (2)	0.16120 (10)	0.4790 (2)	0.0551 (12)

Table 2. Bond lengths (Å) and angles (°)

O1—C19	1.221 (2)	C15—C16	1.376 (4)
O2—C27	1.216 (2)	C17—C18	1.526 (2)
C1—C2	1.554 (4)	C17—C26	1.538 (2)
C1—C14	1.501 (4)	C18—C19	1.512 (3)
C2—C3	1.518 (3)	C19—C20	1.494 (3)
C3—C4	1.415 (3)	C20—C25	1.391 (3)
C3—C8	1.386 (3)	C20—C21	1.398 (3)
C4—C5	1.391 (3)	C21—C22	1.379 (4)
C4—C17	1.528 (3)	C22—C23	1.371 (4)
C5—C6	1.401 (3)	C23—C24	1.373 (3)
C6—C7	1.379 (3)	C24—C25	1.384 (3)
C6—C9	1.505 (3)	C26—C27	1.503 (3)
C7—C8	1.377 (3)	C27—C28	1.497 (3)
C9—C10	1.560 (4)	C28—C29	1.388 (3)
C10—C11	1.504 (4)	C28—C33	1.395 (3)
C11—C12	1.382 (3)	C29—C30	1.377 (3)
C11—C16	1.394 (4)	C30—C31	1.374 (3)
C12—C13	1.380 (3)	C31—C32	1.363 (3)
C13—C14	1.389 (3)	C32—C33	1.382 (3)
C14—C15	1.385 (3)		
C14—C1—C2	114.4 (2)	C18—C17—C26	108.0 (1)
C3—C2—C1	113.3 (2)	C4—C17—C26	108.3 (1)
C8—C3—C4	117.0 (2)	C19—C18—C17	116.3 (2)
C8—C3—C2	118.5 (2)	O1—C19—C20	120.8 (2)
C4—C3—C2	123.3 (2)	O1—C19—C18	120.9 (2)
C5—C4—C3	117.7 (2)	C20—C19—C18	118.3 (2)
C5—C4—C17	123.0 (2)	C25—C20—C21	118.5 (2)
C3—C4—C17	119.2 (2)	C25—C20—C19	122.2 (2)
C4—C5—C6	122.7 (2)	C21—C20—C19	119.3 (2)
C7—C6—C5	116.4 (2)	C22—C21—C20	120.4 (2)
C7—C6—C9	120.6 (2)	C23—C22—C21	120.4 (3)
C5—C6—C9	121.9 (2)	C22—C23—C24	120.0 (3)
C8—C7—C6	120.1 (2)	C23—C24—C25	120.4 (3)
C7—C8—C3	122.2 (2)	C24—C25—C20	120.3 (2)
C6—C9—C10	114.3 (2)	C27—C26—C17	115.9 (2)
C11—C10—C9	113.0 (2)	O2—C27—C28	120.3 (2)
C12—C11—C10	116.5 (2)	O2—C27—C26	121.5 (2)
C12—C11—C16	120.2 (3)	C28—C27—C26	118.1 (2)
C16—C11—C10	121.7 (3)	C29—C28—C33	118.6 (2)
C13—C12—C11	121.0 (3)	C29—C28—C27	119.4 (2)
C12—C13—C14	120.8 (2)	C33—C28—C27	122.0 (2)
C15—C14—C13	116.9 (2)	C30—C29—C28	120.2 (2)
C15—C14—C1	121.8 (3)	C31—C30—C29	120.3 (2)
C13—C14—C1	119.9 (2)	C32—C31—C30	120.4 (2)
C16—C15—C14	120.7 (3)	C31—C32—C33	120.0 (2)
C15—C16—C11	121.1 (2)	C32—C33—C28	120.4 (2)
C18—C17—C4	114.8 (1)		

Refinement on F² for all reflections except those flagged for possible systematic errors; the observed threshold I > 2σ(I) is used only for calculating R(obs.) etc., given here for comparison with refinements on F.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55725 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1020]

References

- Hope, H., Bernstein, J. & Trueblood, K. N. (1972). *Acta Cryst.* **B28**, 1733–1735.
- Knobler, C. B., Maverick, E. F., Parker, K. M., Trueblood, K. N., Weiss, R. L., Cram, D. J. & Helgesen, R. C. (1986). *Acta Cryst.* **C42**, 1862–1863.
- Lindeman, S. V., Struchkov, Yu. T. & Guryshv, V. N. (1986). *Izv. Akad. Nauk SSSR Ser. Khim.* pp. 2007–2010.
- Sheldrick, G. M. (1987). *SHELXTL-Plus*, PC version. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1992). *SHELXL92*. Beta-test version. Program for the refinement of crystal structures. Univ. of Göttingen, Germany.
- Tochtermann, W., Olsson, G., Vogt, C., Peters, E.-M., Peters, K. & von Schnering, H. G. (1987). *Chem. Ber.* **120**, 1523–1530.

Acta Cryst. (1993). **C49**, 615–616

Structure of *trans*-Decafluoroazobenzene

K. CHINAKALI† AND HOONG-KUN FUN

School of Physics, Universiti Sains Malaysia, Minden,
11800 Penang, Malaysia

OMAR BIN SHAWKATALY*

Chemical Sciences Programme, Center for
Off-Campus Studies, Universiti Sains
Malaysia, Minden, 11800 Penang, Malaysia

SIANG-GUAN TEOH

School of Chemical Sciences, Universiti Sains Malaysia,
Minden, 11800 Penang, Malaysia

(Received 27 May 1992; accepted 7 September 1992)

Abstract

The planar *trans*-decafluoroazobenzene molecules are stacked along the [102] direction with an interlayer distance of 4.22 Å.

Comment

The title compound was prepared (Birchall, Haszeldine & Kemp, 1970) by the coupling of penta-

fluoroaniline in benzene using lead tetraacetate as a catalyst and crystallized from methanol. Individual molecules are situated in the unit cell so that a crystallographic inversion centre bisects the N—N bond. The crystal structure is composed of layers of decafluoroazobenzene molecules which are stacked along the [102] direction with an interlayer spacing of 4.22 Å. The bond lengths and distances are comparable with those reported for the *trans*-decafluoroazobenzene–*trans*-stilbene (1/1) complex (Bruce, Snow & Tiekink, 1987).

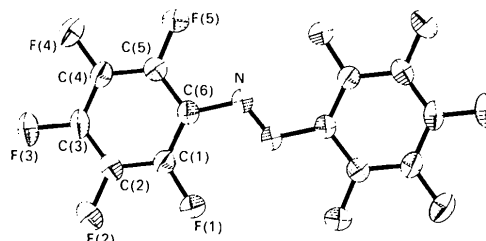


Fig. 1. View of the molecule showing the labelling of the non-H atoms. Thermal ellipsoids are shown at 50% probability levels.

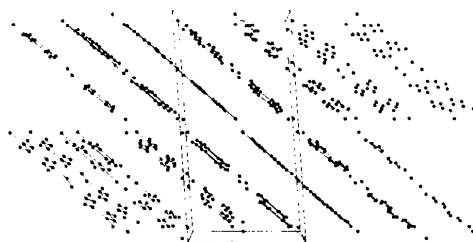


Fig. 2. Packing of the molecules in the unit cell viewed down the *b* axis.

Experimental

Crystal data

$C_{12}F_{10}N_2$
 $M_r = 362.1$
 Monoclinic
 $C2/c$
 $a = 17.371$ (6) Å
 $b = 7.621$ (2) Å
 $c = 8.862$ (3) Å
 $\beta = 94.03$ (2)°
 $V = 1170.3$ (6) Å³
 $Z = 4$
 $D_x = 2.055$ Mg m⁻³
 $D_m = 2.038$ Mg m⁻³

Mo $K\alpha$ radiation
 $\lambda = 0.71069$ Å
 Cell parameters from 50 reflections
 $\theta = 7.5$ – 17.5 °
 $\mu = 0.237$ mm⁻¹
 $T = 298$ K
 Thin plate
 $1.0 \times 0.66 \times 0.06$ mm
 Orange

Data collection

Siemens P4 diffractometer
 $2\theta/\theta$ scans

$R_{int} = 0.044$
 $\theta_{max} = 55.0$ °

† Post Doctoral Research Fellow. Permanent address: Physics Division, Department of Chemical Engineering, AC Tech. Campus, Anna University, Madras 600025, India.