Bastiansen, Fritsch & Hedberg, 1964), Allen states that substituent influence on net ring bonding is absent in carbonyl- and cyano-substituted cyclopropanes. However, there appears to be a tendency to increase the ring bond length at high degrees of substitution, as seen in the present study, where we find 1·529 Å. The two 1,2,2-tricyanocyclopropanecarboxylic esters mentioned above display average ring bond lengths of 1·529 and 1·531 Å, and 1,1,2,2-cyclopropanetetracarbonitrile 1·529 Å. The most plausible interpretation for such an increase is bond-length relaxation of steric strain between the substituents. This has been observed in hexachloro- and in hexabromocyclopropane (1·529 and 1·544 Å; Schrumpf & Jones, 1987*b,c*).

The various steric interactions in the title molecule can be demonstrated by the angle deformations. The bond angle between the substituents in the 1-cyano-acid (117·2°; Jones & Schrumpf, 1987*b*) is a little smaller than in the 1,1-diacid (118·3°; Meester, Schenk & MacGillavry, 1971); we consider these as 'normal' bond angles between geminal CN and carboxyl groups without steric interference. The bond angle between the geminal substituents in the tricyanotriester studied here at C(2) is 115·7° and at C(3) 115·5°, *i.e.* somewhat smaller than in the cyano-acid. The bond angle at C(1), however, is drastically reduced (109·9°), obviously on account of substantial steric strain. The origin of the repulsion can be traced by analysing the angles between the individual exocyclic CC bonds and the adjacent ring CC bonds. In the cyano-acid model, these angles are practically the same for the cyano (117·2°) and the ester C atoms (117·5°). In the title molecule, the angles to the cyano C atoms have a narrower range (117·4–119·8°) than those to the ester C atoms (115·5–121·5°). Within each group, however, the angles between the two substituents at C(1) and the C(1)—C(3) bond are substantially larger than the average of each group with the ester C atom responding by an increase of 4°, the cyano carbon by 2° to the steric pressure of the substituents at C(3).

Since there are so few X-ray studies of highly cyano- and carboxyl-substituted cyclopropanes, several such compounds are currently being studied in our laboratories.

This work was supported by the Fonds der Chemischen Industrie, Frankfurt.

References

ALLEN, F. H. (1980). *Acta Cryst.* B**36**, 81–96.

BASTIANSEN, O., FRITSCH, J. E. & HEDBERG, K. (1964). *Acta Cryst.* **17**, 538–543.

CLEGG, W. (1981). *Acta Cryst.* A**37**, 22–28.

GRIFFIN, G. W. & PETERSON, L. I. (1963). *J. Org. Chem.* **28**, 3219–3220.

JONES, P. G. & SCHRUMPF, G. (1987*a*). *Acta Cryst.* C**43**, 1179–1182.

JONES, P. G. & SCHRUMPF, G. (1987*b*). *Acta Cryst.* C**43**, 1576–1579.

LEMLEY, J. T., SKARSTAD, P. M. & HUGHES, R. E. (1976). *Acta Cryst.* B**32**, 35–40.

MEESTER, M. A. M., SCHENK, H. & MACGILLAVRY, C. H. (1971). *Acta Cryst.* B**27**, 630–634.

RAMASUBBU, N., RAJARAM, J. & VENKATESAN, K. (1982). *Acta Cryst.* B**38**, 196–199.

RAMASUBBU, N. & VENKATESAN, K. (1982). *Acta Cryst.* B**38**, 1372–1375.

SCHRUMPF, G. & JONES, P. G. (1987*a*). *Acta Cryst.* C**43**, 1182–1185.

SCHRUMPF, G. & JONES, P. G. (1987*b*). *Acta Cryst.* C**43**, 1185–1187.

SCHRUMPF, G. & JONES, P. G. (1987*c*). *Acta Cryst.* C**43**, 1188–1192.

SHELDRICK, G. M. (1978). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen, Federal Republic of Germany.

Acta Cryst. (1987). C**43**, 1582–1584

Structure of 1,2,3,4-Tetraphenylbenzene

BY HERMANN IRNGARTINGER AND DIETMAR KALLFASS

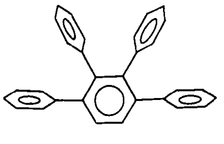
Organisch-Chemisches Institut der Universität, Im Neuenheimer Feld 270, *D*-6900 *Heidelberg, Federal Republic of Germany*

(*Received* 1 *December* 1986; *accepted* 23 *March* 1987)

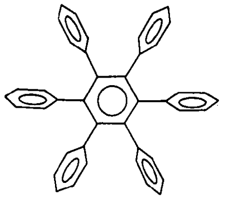
Abstract. $C_{30}H_{22}$, $M_r = 382\cdot51$, orthorhombic, *Pbcn*, $a = 23\cdot577$ (4), $b = 9\cdot013$ (2), $c = 9\cdot950$ (2) Å, $V = 2114\cdot4$ (1·3) Å^3, $Z = 4$, $D_x = 1\cdot20$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0\cdot632$ cm^{-1}, $F(000) = 808$, $T = 213$ (1) K, $R = 0\cdot043$ for 1726 unique observed reflections [$I > 3\sigma(I)$]. The title compound adopts a propeller configuration. The peripheral rings are not perpendicular to the central ring but twisted about 40·2 and 22·2° from this position. The observed interplanar angles indicate absence of electronic interactions between the phenyl rings. The bond distances in the benzene rings range from 1·373 (3) to 1·415 (2) Å.

0108-2701/87/081582-03$01.50

Introduction. In hexaphenylbenzene (2) (Bart, 1968) the tendency of the individual aromatic rings to adopt a coplanar configuration is counteracted by steric requirements of the molecule. We were interested to see whether similar effects would be observed in the title compound (1). In the course of our investigations on the synthesis and structure determination of prismanes (Maier, Bauer, Huber-Patz, Jahn, Kallfass, Rodewald & Irngartinger, 1986; Irngartinger, Kallfass, Litterst & Gleiter, 1987), we obtained (1) as a by-product.



(1)



(2)

Experimental. Colourless crystals 0·3 × 0·4 × 0·35 mm, crystallized from dichloromethane solution. Data measured on a CAD-4 diffractometer (Enraf–Nonius) at 213 K. ω–2θ scan method, cell dimensions from 2θ angles of 25 high-order reflections ($20 < 2\theta < 40°$), 2548 unique reflections up to $(\sin\theta)/\lambda = 0{\cdot}70$ Å^{-1}, $0 < h < 31$, $0 < k < 11$, $0 < l < 13$, three standard reflections: $3,\overline{11},\overline{3}$, 3,11,3, 283 (2% intensity variation), 1726 intensities with $I > 3\sigma(I)$, $R_{\rm int} = 0{\cdot}024$. Absorption ignored.

The structure was solved with *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) by using only reflections with $(\sin\theta)/\lambda < 0{\cdot}63$ Å^{-1} and refined by full-matrix least squares on F^2 with anisotropic thermal parameters for C atoms; H atoms located from difference Fourier syntheses and refined isotropically. 180 parameters, $R = 0{\cdot}043$ for 1726 observed reflections, $wR = 0{\cdot}058$, $w = 4F^2/\sigma^2(F^2) + (0{\cdot}03F^2)^2$, $S = 2{\cdot}498$, $(\Delta/\sigma)_{\rm max} < 0{\cdot}01$, largest peak 0·23 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974); *SDP* programs (Frenz, 1982) used on a PDP 11/44.

Discussion. The final atomic parameters for (1) are given in Table 1.* Bond lengths and bond angles are listed in Table 2. The numbering scheme is shown in Fig. 1.

The molecule lies on a crystallographic twofold axis. The four phenyl rings substituted on the central benzene ring have a propeller arrangement. The individual molecules are thus chiral. The twist angles of the phenyl rings *A* and *B* to the central benzene ring are 49·8 and 67·8° respectively. The observed interplanar angles

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$U_{\rm eq}$ is one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U_{\rm eq}(\times 10^4$ Å$^2)$
C(1)	0·52865 (7)	0·44630 (18)	0·73143 (16)	320 (7)
C(2)	0·55839 (7)	0·58172 (19)	0·71668 (16)	344 (8)
C(3)	0·52854 (7)	0·71400 (19)	0·73483 (17)	378 (8)
C(4)	0·61967 (7)	0·59321 (18)	0·67935 (17)	352 (8)
C(5)	0·65439 (8)	0·69041 (20)	0·75098 (20)	434 (9)
C(6)	0·71069 (8)	0·71308 (23)	0·71258 (21)	521 (11)
C(7)	0·73220 (8)	0·64027 (25)	0·60187 (23)	532 (11)
C(8)	0·69898 (8)	0·54285 (23)	0·53074 (21)	503 (10)
C(9)	0·64226 (7)	0·51924 (21)	0·56913 (18)	428 (9)
C(10)	0·55755 (7)	0·30180 (18)	0·70120 (16)	329 (8)
C(11)	0·60178 (7)	0·24715 (21)	0·77920 (18)	405 (9)
C(12)	0·62938 (8)	0·11637 (21)	0·74230 (22)	488 (10)
C(13)	0·61320 (9)	0·04073 (19)	0·62706 (22)	487 (10)
C(14)	0·56860 (8)	0·09349 (21)	0·55026 (20)	452 (10)
C(15)	0·54093 (8)	0·22225 (19)	0·58751 (18)	389 (9)

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

C(1)–C(1′)	1·401 (2)	C(6)–C(7)	1·379 (3)
C(1)–C(2)	1·415 (2)	C(7)–C(8)	1·373 (3)
C(1)–C(10)	1·500 (2)	C(8)–C(9)	1·407 (3)
C(2)–C(3)	1·396 (2)	C(10)–C(11)	1·390 (2)
C(2)–C(4)	1·495 (2)	C(10)–C(15)	1·396 (2)
C(3)–C(3′)	1·379 (2)	C(11)–C(12)	1·396 (3)
C(4)–C(5)	1·395 (2)	C(12)–C(13)	1·387 (3)
C(4)–C(9)	1·390 (2)	C(13)–C(14)	1·384 (3)
C(5)–C(6)	1·396 (3)	C(14)–C(15)	1·382 (3)

C(1′)–C(1)–C(2)	120·3 (1)	C(6)–C(7)–C(8)	120·4 (2)
C(1′)–C(1)–C(10)	119·4 (1)	C(7)–C(8)–C(9)	119·9 (2)
C(2)–C(1)–C(10)	120·2 (1)	C(4)–C(9)–C(8)	120·4 (2)
C(1)–C(2)–C(3)	118·3 (2)	C(1)–C(10)–C(11)	122·4 (1)
C(1)–C(2)–C(4)	124·3 (1)	C(1)–C(10)–C(15)	118·7 (1)
C(3)–C(2)–C(4)	117·4 (1)	C(11)–C(10)–C(15)	118·8 (2)
C(2)–C(3)–C(3′)	121·3 (2)	C(10)–C(11)–C(12)	120·1 (2)
C(2)–C(4)–C(5)	118·9 (2)	C(11)–C(12)–C(13)	120·3 (2)
C(2)–C(4)–C(9)	122·2 (2)	C(12)–C(13)–C(14)	119·8 (2)
C(5)–C(4)–C(9)	118·7 (2)	C(13)–C(14)–C(15)	119·9 (2)
C(4)–C(5)–C(6)	120·7 (2)	C(10)–C(15)–C(14)	121·1 (2)
C(5)–C(6)–C(7)	119·9 (2)		

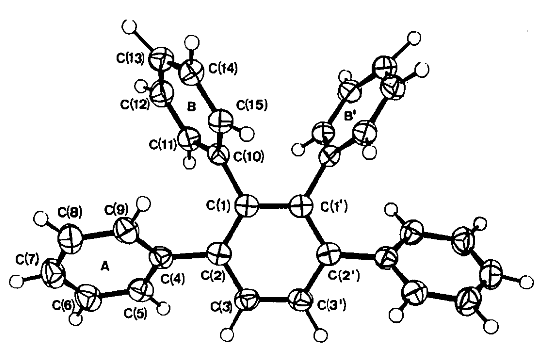


Fig. 1. *ORTEP* (Johnson, 1976) plot of compound (1) with 50% probability thermal ellipsoids showing the numbering scheme.

---

* Lists of H-atom bond distances, angles, and atomic positions, anisotropic thermal parameters and a list of structure amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43906 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Short intramolecular distances* (Å)

C(1)···C(9)	3·196 (2)	C(9)···C(10)	3·091 (2)
C(2)···C(11)	3·244 (3)	C(9)···C(11)	3·361 (3)
C(3)···C(5)	2·979 (2)	C(10)···C(10′)	2·882 (2)
C(4)···C(10)	3·015 (2)	C(10)···C(15′)	3·213 (2)
C(4)···C(11)	3·300 (2)		

indicate absence of electronic interactions between the phenyl rings. In compound (2) these angles range from 62·0 to 70·7°. The largest deviations from the best planes in (1) are 0·008 and 0·009 Å in rings *A* and *B*, respectively, whereas in the central ring this value is 0·02 Å [torsion angles C(10)—C(1)—C(1′)—C(10′) 9·7 (2)° and C(4)—C(2)—C(1)—C(10) −3·7 (2)°].

The bond distances in the benzene rings range from 1·373 (3) to 1·415 (2) Å. Despite the repulsion of the phenyl rings (short intramolecular distances are given in Table 3), the bond distances are not lengthened significantly. The bond angles C(1)—C(2)—C(4) [124·3 (1)°] and C(3)—C(2)—C(4) [117·4 (1)°], however, are influenced by this repulsion effect.

Financial support was given by the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie.

References

Bart, J. C. J. (1968). *Acta Cryst.* B**24**, 1277–1287.

Frenz, B. A. (1982). *Enraf–Nonius Structure Determination Package*. College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Irngartinger, H., Kallfass, D., Litterst, E. & Gleiter, R. (1987). *Acta Cryst.* C**43**, 266–269.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Maier, G., Bauer, I., Huber-Patz, U., Jahn, R., Kallfass, D., Rodewald, H. & Irngartinger, H. (1986). *Chem. Ber.* **119**, 1111–1116.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Acta Cryst. (1987). C**43**, 1584–1587

Structures of 5-Bromobicyclo[6.3.0]undecane-2,6-dione (I) and 2-Methyl-4,5,6,6a,8,9,9a,10-octahydro-7*H*-cyclopentacycloocteno[5,6-*b*]furan-6-one (II)

By S. Okumoto, S. Ohba and Y. Saito

Department of Chemistry, Faculty of Science and Technology, Keio University, Hiyoshi 3, *Kohoku-ku, Yokohama* 223, *Japan*

T. Ishii

Chiba Institute of Technology, Tsudanuma 2, *Narashino, Chiba* 275, *Japan*

M. Umehara

Department of Chemistry, Keio University, Hiyoshi 4–1–1, *Kohoku-ku, Yokohama* 223, *Japan*

and S. Hishida

Naka Works, Hitachi Ltd, Ichige, Katsuta, Ibaraki 312, *Japan*

(*Received* 28 *January* 1987; *accepted* 3 *March* 1987)

Abstract. $C_{11}H_{15}BrO_2$ (I): $M_r = 259\cdot1$, monoclinic, $P2_1/c$, $a = 6\cdot401$ (1), $b = 16\cdot493$ (3), $c = 10\cdot928$ (2) Å, $\beta = 111\cdot02$ (2)°, $V = 1076\cdot8$ (4) Å^3, $Z = 4$, $D_m = 1\cdot58$ (3), $D_x = 1\cdot60$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71073$ Å, $\mu = 3\cdot75$ mm^{-1}, $F(000) = 528$, $T = 295$ (1) K, $R = 0\cdot046$ for 1568 observed unique reflections. $C_{14}H_{18}O_2$ (II): $M_r = 218\cdot3$, monoclinic, $P2_1/c$, $a = 4\cdot939$ (1), $b = 12\cdot900$ (2), $c = 19\cdot024$ (2) Å, $\beta = 93\cdot17$ (2)°, $V = 1210\cdot2$ (4) Å^3, $Z = 4$, $D_m = 1\cdot20$ (3), $D_x = 1\cdot19$ Mg m^{-3}, Mo $K\alpha$, $\mu = 0\cdot07$ mm^{-1}, $F(000) = 472$, $T = 300$ (1) K, $R = 0\cdot041$ for 1188 observed unique reflections. The ring fusion of the five- and eight-membered rings in both (I) and (II) is *trans*. The boat–chair conformation of the eight-membered rings is somewhat distorted because of steric repulsion between C=O and C—Br bonds in (I) and the double bond in the eight-membered ring of (II).

Introduction. Compounds (I) and (II) were synthesized by one of the authors (MU) in the course of preparation

0108-2701/87/081584-04$01.50