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Structure of Perhydrotriphenylene

By Richard L. Harlow and Gautam R. Desiraju*

Central Research and Development Department, E. I. Du Pont de Nemours and Company, Inc., Experimental Station, Wilmington, Delaware 19880-356, *USA*

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Abstract. $C_{18}H_{30}$, $M_r = 246\cdot44$, monoclinic, $P2_1/n$, $Z = 4$, $a = 18\cdot315\ (2)$, $b = 15\cdot319\ (3)$, $c = 5\cdot298\ (1)$ Å, $\beta = 95\cdot53\ (1)°$, $V = 1479\cdot5\ (1)$ Å^3, $F(000) = 552$, $D_x = 1\cdot106$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0\cdot057$ mm^{-1}, $T = 173$ K, $R = 0\cdot041$, $wR = 0\cdot037$ for 932 independent reflections. The present report is an accurate redetermination of the crystal structure using low-temperature data. The crystal structure is close-packed and therefore distinct from those several clathrates formed by the title compound. All chemically equivalent bonds are in agreement.

Introduction. Perhydrotriphenylene, PHTP, $C_{18}H_{30}$ forms a large number of inclusion compounds with small organic guest molecules. The crystal structure of the stable Form I of uncomplexed PHTP has been reported previously (Allegra *et al.*, 1967) but is of very limited accuracy (two-dimensional data, $R = 0\cdot20$). The present study was undertaken since good crystals of the substance were obtained upon slow cooling of its saturated solutions in 1-methylnaphthalene, the solvent molecules being presumably too large to be included in a host:guest structure.

Experimental. Intensity data were collected on a colorless flat needle 0·24 × 0·14 × 0·55 mm on a Syntex $P3$ diffractometer. Unit-cell parameters were obtained from 31 reflections in the range $16 \leq 2\theta \leq 26°$. The unit cell was redefined in order to obtain a monoclinic angle closer to 90°. The ω-scan method was used with a scan width of 1·80°ω (the crystals were very mosaic, a typical FWHM value for an ω scan was 0·80°) and the scan speed was between 2·90 and 14·60° min^{-1}. A total of 2690 reflections (2174 unique) was collected with $4\cdot5 < 2\theta < 47\cdot0°$ (beyond 40°, the number of observed intensities fell off rapidly) and in the range $-20 \leq h \leq 20$; $0 \leq k \leq 17$; $-5 \leq l \leq 0$. Of these, 932 unique reflections were found to be non-zero with $I \geq 3\cdot0\sigma(I)$. Three standard reflections ($02\bar{2}$, $\bar{7}4\bar{1}$ and $\bar{8}50$) were measured every 97 reflections and showed less than 2% variation. No absorption corrections were applied. The structure was solved by direct methods using the program *MULTAN* (Germain, Main & Woolfson, 1971) and full-matrix least-squares refinement was performed on F_o using a package of local programs (Calabrese, 1989). The C atoms were refined anisotropically, the six H atoms on the central ring isotropically, and the twelve other H atoms were placed in calculated positions. Refinement converged at $R = 0\cdot041$, $wR = 0\cdot037$, error-of-fit = 1·22, $\Delta/\sigma_{\max} = 0\cdot11$.† The maximum peak in the final Fourier was 0·12 e Å^{-3}. The scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Discussion. Atomic positions and equivalent isotropic temperature factors are listed in Table 1. The bond lengths and angles are given in Table 2. The atomic numbering scheme is given in Fig. 1. Each ring adopts a chair conformation and the configuration of every fused-ring junction is *trans*. The C—C bond lengths cluster into three groups: in the central ring, they vary over a narrow range from 1·529 to 1·540 Å (2 e.s.d.'s); bonds emanating from the central ring vary from 1·520 to 1·532 Å; the remaining exterior bonds are shorter still, 1·508 to 1·522 Å. The crystal packing is shown in Fig. 2 and is characterized by normal or van der Waals H···H contacts. The closest intermolecular contacts are: H(14)—H(15), 2·13; H(7)—H(18), 2·14; H(10)—H(11), 2·16 Å. The crystal structure is, not surprisingly, quite different from those of the PHTP clathrates (Allegra *et al.*, 1967). A considerable amount of recent work describes the use of PHTP as a complexing agent in host:guest compounds (Tsoucaris, 1987; Weber, 1987). It is normally difficult to obtain guest-free crystals of the PHTP host, uncommon solvents such as α-pinene and hexachlorobutadiene having been used for this purpose in the past (Allegra *et al.*, 1967). Although naphthalene molecules are easily

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* Permanent address: School of Chemistry, University of Hyderabad, PO Central University, Hyderabad 500 134, India.

† Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52590 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional coordinates and isotropic thermal parameters*

	x	y	z	B_{iso}/B_{eq}
C(1)	0·5211 (2)	0·1765 (2)	0·6832 (7)	1·8 (1)*
C(2)	0·4953 (2)	0·2439 (2)	0·4801 (7)	1·8 (1)*
C(3)	0·5531 (2)	0·3156 (2)	0·4636 (7)	1·9 (1)*
C(4)	0·6289 (2)	0·2782 (2)	0·4198 (7)	1·9 (1)*
C(5)	0·6537 (2)	0·2062 (2)	0·6113 (6)	1·7 (1)*
C(6)	0·5947 (2)	0·1365 (2)	0·6295 (7)	1·7 (1)*
C(7)	0·4620 (2)	0·1081 (2)	0·7123 (7)	2·6 (1)*
C(8)	0·3883 (2)	0·1470 (2)	0·7566 (7)	2·7 (1)*
C(9)	0·3630 (2)	0·2096 (2)	0·5449 (7)	2·7 (1)*
C(10)	0·4193 (2)	0·2802 (2)	0·5196 (7)	2·7 (1)*
C(11)	0·5306 (2)	0·3865 (2)	0·2675 (7)	2·7 (1)*
C(12)	0·5875 (2)	0·4580 (2)	0·2584 (7)	3·3 (1)*
C(13)	0·6621 (2)	0·4204 (2)	0·2141 (7)	3·0 (1)*
C(14)	0·6847 (2)	0·3512 (2)	0·4105 (6)	2·6 (1)*
C(15)	0·7271 (2)	0·1653 (2)	0·5596 (6)	2·4 (1)*
C(16)	0·7522 (2)	0·0942 (2)	0·7483 (8)	2·7 (1)*
C(17)	0·6938 (2)	0·0254 (2)	0·7611 (7)	2·7 (1)*
C(18)	0·6214 (2)	0·0655 (2)	0·8185 (6)	2·4 (1)*
H(1)	0·5295 (16)	0·2104 (22)	0·8474 (63)	3·4 (8)
H(2)	0·4923 (14)	0·2115 (18)	0·3163 (55)	1·7 (7)
H(3)	0·5601 (15)	0·3450 (20)	0·6229 (54)	1·9 (7)
H(4)	0·6233 (13)	0·2520 (16)	0·2462 (51)	0·6 (6)
H(5)	0·6598 (15)	0·2344 (18)	0·7841 (54)	1·5 (6)
H(6)	0·5889 (17)	0·1086 (20)	0·4684 (62)	2·9 (8)

* Refined with anisotropic thermal parameters.

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)—C(2)	1·533 (5)	C(9)—C(10)	1·510 (5)
C(1)—C(6)	1·531 (5)	C(11)—C(12)	1·515 (5)
C(1)—C(7)	1·526 (5)	C(12)—C(13)	1·522 (5)
C(2)—C(3)	1·533 (5)	C(13)—C(14)	1·515 (5)
C(2)—C(10)	1·532 (5)	C(15)—C(16)	1·519 (5)
C(3)—C(4)	1·540 (4)	C(16)—C(17)	1·508 (5)
C(3)—C(11)	1·531 (5)	C(17)—C(18)	1·517 (5)
C(4)—C(5)	1·537 (4)	C(1)—H(1)	1·01 (3)
C(4)—C(14)	1·520 (5)	C(2)—H(2)	1·00 (3)
C(5)—C(6)	1·529 (5)	C(3)—H(3)	0·95 (3)
C(5)—C(15)	1·532 (4)	C(4)—H(4)	1·00 (3)
C(6)—C(18)	1·528 (4)	C(5)—H(5)	1·01 (3)
C(7)—C(8)	1·514 (5)	C(6)—H(6)	0·95 (3)
C(8)—C(9)	1·514 (5)		

C(2)—C(1)—C(6)	110·9 (3)	C(12)—C(13)—C(14)	110·5 (3)
C(2)—C(1)—C(7)	111·1 (3)	C(4)—C(14)—C(13)	113·4 (3)
C(6)—C(1)—C(7)	112·9 (3)	C(5)—C(15)—C(16)	113·0 (3)
C(1)—C(2)—C(3)	110·8 (3)	C(15)—C(16)—C(17)	111·0 (3)
C(1)—C(2)—C(10)	111·8 (3)	C(16)—C(17)—C(18)	111·4 (3)
C(3)—C(2)—C(10)	112·9 (3)	C(6)—C(18)—C(17)	112·8 (3)
C(2)—C(3)—C(4)	112·3 (3)	C(2)—C(1)—H(1)	106 (2)
C(2)—C(3)—C(11)	113·9 (3)	C(6)—C(1)—H(1)	108 (2)
C(4)—C(3)—C(11)	110·4 (3)	C(7)—C(1)—H(1)	108 (2)
C(3)—C(4)—C(5)	112·4 (3)	C(1)—C(2)—H(2)	105 (2)
C(3)—C(4)—C(14)	110·5 (3)	C(3)—C(2)—H(2)	107 (2)
C(5)—C(4)—C(14)	113·1 (3)	C(10)—C(2)—H(2)	109 (2)
C(4)—C(5)—C(6)	112·2 (3)	C(2)—C(3)—H(3)	109 (2)
C(4)—C(5)—C(15)	112·6 (3)	C(4)—C(3)—H(3)	105 (2)
C(6)—C(5)—C(15)	111·3 (3)	C(11)—C(3)—H(3)	106 (2)
C(1)—C(6)—C(5)	111·8 (3)	C(3)—C(4)—H(4)	106 (1)
C(1)—C(6)—C(18)	113·3 (3)	C(5)—C(4)—H(4)	109 (1)
C(5)—C(6)—C(18)	110·8 (3)	C(14)—C(4)—H(4)	106 (1)
C(1)—C(7)—C(8)	113·4 (3)	C(4)—C(5)—H(5)	107 (2)
C(7)—C(8)—C(9)	110·4 (3)	C(6)—C(5)—H(5)	105 (2)
C(8)—C(9)—C(10)	110·9 (3)	C(15)—C(5)—H(5)	108 (2)
C(2)—C(10)—C(9)	113·0 (3)	C(1)—C(6)—H(6)	109 (2)
C(3)—C(11)—C(12)	113·2 (3)	C(5)—C(6)—H(6)	106 (2)
C(11)—C(12)—C(13)	111·1 (3)	C(18)—C(6)—H(6)	106 (2)

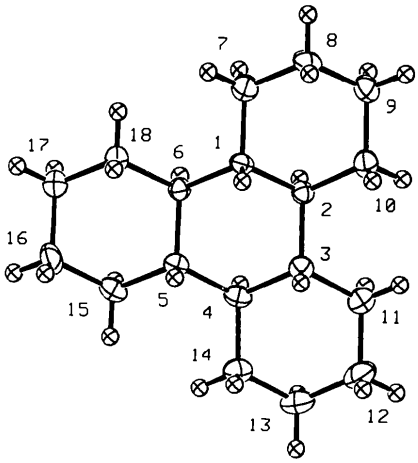

Fig. 1. Atomic numbering scheme for perhydrotriphenylene, PHTP.

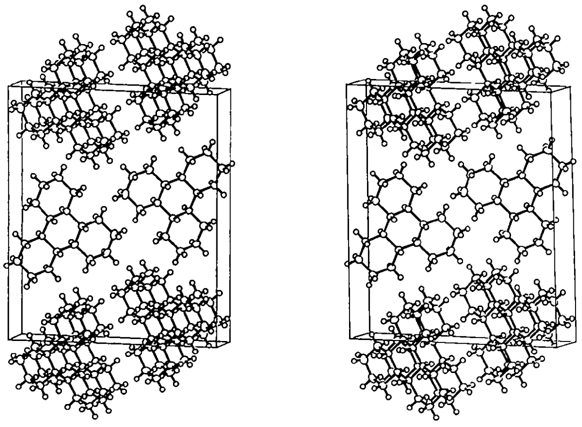

Fig. 2. Stereodrawing of the crystal structure of PHTP, with the *b* axis horizontal and the *a* axis vertical. The molecules stack translationally along the short *c* axis.

accommodated in the host PHTP lattice, the molecular size of 1-methylnaphthalene seems to be critically large so as to preclude complexation.

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References

Allegra, G., Farina, M., Immirzi, A., Colombo, A., Rossi, U., Broggi, R. & Natta, G. (1967). *J. Chem. Soc. B*, pp. 1020–1028.

Calabrese, J. C. (1989). Central Research & Development, E. I. Du Pont de Nemours & Co., Inc., PO Box 80228, Wilmington, DE 19880-0228, USA.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99–102. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Tsoucaris, G. (1987). In *Organic Solid State Chemistry*, edited by G. R. Desiraju, pp. 207–270. Amsterdam: Elsevier.

Weber, E. (1987). Editor. *Molecular Inclusion and Molecular Recognition – Clathrates* 1. Berlin: Springer.