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1,1-Bis(*p*-methoxyphenyl)-2,2-dimethylpropane

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Abstract. $C_{19}H_{24}O_2$, $M_r = 284\cdot4$, orthorhombic, $P2_12_12_1$, $a = 17\cdot675$ (2), $b = 12\cdot294$ (2), $c = 7\cdot756$ (1) Å. $Z = 4$, $D_x = 1\cdot12$ Mg m^{-3}, μ(Cu $K\alpha$) = 0·562 mm^{-1}, $F(000) = 616$, $R = 0\cdot088$ for 1268 observed reflections. The molecules have pseudo-mirror symmetry similar to that found for the analogous compound 1,1-bis(*p*-ethoxyphenyl)-2,2-dimethylpropane (GH44). Whereas in GH44 the ethoxy groups have *trans* conformations with respect to one another, the methoxy groups in the title compound are *cis* related. Most atoms have large thermal motion, in particular the methyl groups attached to the central isobutyl group and the *p*-methoxy groups.

Introduction. Holan (1969) defined some stereochemical requirements for insecticidal activity among a series of DDT compounds. Since then, the crystal structures of a number of these compounds have been determined. The *p,p'*-dimethoxy analogue of DDT (methoxychlor) (Smith, Kennard & White, 1977) and the *p,p'*-diethyl analogue of DDD (Perthane) (Hovmöller, Smith & Kennard, 1978) are two commercial insecticides having no halogen substituents in the phenyl ring. Of greater interest are those compounds which are insecticidally active yet possess no Cl substituents. An example, synthesized by Holan, is 1,1-bis(*p*-ethoxyphenyl)-2,2-dimethylpropane (GH44) (DeLacy & Kennard, 1972). The title compound, 1,1-bis(*p*-methoxyphenyl)-2,2-dimethylpropane, is analogous to GH44 and was earlier reported by Rogers, Brown, Rasmussen & Heal (1953). Activities (CSIRO, 1969) indicate that DDT, methoxychlor, GH44 and this compound are comparable. Rogers, Brown, Rasmussen & Heal (1953) also found that the compound had a decreased effectiveness against a strain of DDT-resistant flies. This structural study was undertaken as part of a programme to determine precise stereochemical parameters for DDT-type insecticides.

A sample provided by Dr Rogers was recrystallized from a 2-propanol–water mixture. Three-dimensional X-ray data were obtained from a single crystal, measuring 0·18 × 0·25 × 0·40 mm, on a Philips PW 1100 four-circle diffractometer using monochromatic Cu $K\alpha$ radiation and a 2θ–θ scanning mode with a fixed scan width of 1·6°. 1268 reflections with $I > 2\cdot5\sigma(I)$ were considered observed out of 1762 collected up to $2\theta = 134°$. The structure was solved by direct methods using the *TANG* non-centrosymmetric program incorporated in *SHELX* (Sheldrick, 1976). Full-matrix least-squares refinement with anisotropic thermal parameters for all non-hydrogen atoms reduced R $(= \sum||F_o - F_c||/\sum|F_o|)$ to 0·088. Unit weights were used. The H atoms were located in a difference Fourier synthesis and included in the refinement at fixed positions with each isotropic U set invariant at 0·05 Å^2. Neutral-element scattering factors were used for the non-hydrogens (Doyle & Turner, 1968) while those for H were from Stewart, Davidson & Simpson (1965). No corrections were made for absorption [μ(Cu $K\alpha$) = 0·562 mm^{-1}]. Three intense reflections (310, 120, 302) were considered to be affected by extinction and were removed before the last cycle of refinement. A final difference Fourier map revealed no features larger than 0·11 e Å^{-3}. Although complete convergence had not been reached, reduction of the residual R was very small with each successive cycle. High thermal motion for most atoms in the molecule, particularly the C atoms of the tertiary butyl group and the peripheral methoxy groups, *e.g.* U_{11}, U_{22} and U_{33} for C(15), C(16), C(17), C(18) and C(19) [0·122, 0·135, 0·101; 0·081, 0·121, 0·101; 0·118, 0·194, 0·125; 0·065, 0·070, 0·119; and 0·069, 0·094, 0·140 Å^2, respectively] has probably contributed to the

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relatively large R. Positional parameters are given in Table 1.*

Interatomic distances and angles are listed in Table 2.

Discussion. The molecules of the title compound are very similar to those of the analogous compound GH44 (DeLacy & Kennard, 1972) with an approximate mirror plane containing the two ethyl carbons (Fig. 1). The methoxy groups have a *cis* relationship as found for the *p*-ethyl groups in Perthane (Hovmöller *et al.*, 1978). In GH44, the *p*-ethoxy groups are *trans* related. Methoxychlor [1,1,1-trichloro-2,2-bis(*p*-methoxyphenyl)ethane] (Smith *et al.*, 1977) is comparable to the present compound but possesses neither the pseudo mirror nor the *cis*-related methoxy groups. In GH44 and methoxychlor, all atoms of the *para* substituent groups of the ring are essentially coplanar with the benzene rings. In the case of this compound, one methoxy group is coplanar with the ring but the second lies slightly out of the plane. Torsion angles down the C(4)–O(4) and C(10)–O(10) vectors [C(3)–C(4)–O(4)–C(18) and C(9)–C(10)–O(10)–C(19)] are +176·8 (8) and +159·8 (8)° respectively. The ethyl groups of Perthane lie considerably

Table 1. *Atomic positional parameters* (×10^4) *with estimated standard deviations in parentheses*

	x	*y*	*z*
O(4)	4312 (3)	1796 (4)	2663 (9)
O(10)	2402 (3)	9319 (4)	2144 (9)
C(1)	3700 (4)	4706 (6)	5107 (11)
C(2)	4255 (4)	4652 (7)	3789 (11)
C(3)	4431 (4)	3685 (7)	3010 (11)
C(4)	4079 (4)	2728 (6)	3528 (12)
C(5)	3530 (5)	2745 (7)	4794 (12)
C(6)	3353 (5)	3741 (8)	5555 (11)
C(7)	3208 (5)	6662 (6)	4982 (11)
C(8)	3622 (4)	7170 (6)	3677 (11)
C(9)	3341 (5)	8025 (6)	2781 (12)
C(10)	2630 (5)	8448 (6)	3145 (11)
C(11)	2197 (4)	7948 (7)	4388 (13)
C(12)	2484 (5)	7081 (7)	5292 (12)
C(13)	3495 (6)	5736 (8)	6134 (13)
C(14)	4102 (6)	6100 (8)	7454 (13)
C(15)	3790 (6)	6954 (8)	8565 (14)
C(16)	4842 (6)	6543 (8)	6691 (14)
C(17)	4284 (6)	5050 (8)	8608 (15)
C(18)	3942 (6)	811 (8)	3048 (16)
C(19)	1810 (5)	9982 (9)	2849 (16)
H(2)	4335	5436	3009
H(3)	4840	3638	2117
H(5)	3268	2009	4985
H(6)	2972	3775	6575
H(8)	4159	6928	3228
H(9)	3666	8207	1554
H(11)	1612	8314	4660
H(12)	2183	6869	6492
H(13)	3023	5397	6897
H(151)	4233	7351	9190
H(152)	3847	7639	8039
H(153)	3220	6726	9242
H(161)	4988	6016	6280
H(162)	5213	6245	7917
H(163)	4666	6522	5268
H(171)	4844	5145	8893
H(172)	4572	4771	7941
H(173)	3833	5057	8955
H(181)	4076	782	4422
H(182)	4187	0	3337
H(183)	3513	930	2563
H(191)	1424	9602	4023
H(192)	1852	10340	1941
H(193)	1394	9812	2649

Table 2. *Bond distances* (Å) *and angles* (°) *with estimated standard deviations in parentheses*

C(1)–C(2)	1·418 (11)	C(10)–C(11)	1·376 (11)
C(2)–C(3)	1·370 (10)	C(11)–C(12)	1·373 (11)
C(3)–C(4)	1·390 (11)	C(12)–C(7)	1·400 (10)
C(4)–C(5)	1·381 (11)	C(10)–O(10)	1·383 (10)
C(5)–C(6)	1·395 (10)	O(10)–C(19)	1·435 (11)
C(6)–C(1)	1·380 (10)	C(1)–C(13)	1·539 (11)
C(4)–O(4)	1·390 (10)	C(7)–C(13)	1·534 (11)
O(4)–C(18)	1·408 (11)	C(13)–C(14)	1·549 (10)
C(7)–C(8)	1·396 (11)	C(14)–C(15)	1·466 (11)
C(8)–C(9)	1·354 (11)	C(14)–C(16)	1·536 (11)
C(9)–C(10)	1·389 (11)	C(14)–C(17)	1·603 (11)

C(1)–C(2)–C(3)	121·1 (7)	C(10)–C(11)–C(12)	119·9 (7)
C(2)–C(3)–C(4)	120·4 (7)	C(10)–O(10)–C(19)	116·0 (8)
C(2)–C(1)–C(13)	125·1 (7)	C(11)–C(12)–C(7)	122·4 (8)
C(3)–C(4)–C(5)	120·5 (8)	C(11)–C(10)–O(10)	125·3 (8)
C(3)–C(4)–O(4)	115·2 (8)	C(12)–C(7)–C(8)	116·0 (8)
C(4)–C(5)–C(6)	118·2 (8)	C(12)–C(7)–C(13)	118·4 (7)
C(4)–O(4)–C(18)	118·0 (8)	C(1)–C(13)–C(14)	114·6 (8)
C(5)–C(6)–C(1)	123·3 (8)	C(7)–C(13)–C(14)	113·6 (8)
C(5)–C(4)–O(4)	124·3 (7)	C(1)–C(13)–C(7)	112·8 (9)
C(6)–C(1)–C(2)	116·7 (8)	C(13)–C(14)–C(15)	109·6 (9)
C(6)–C(1)–C(13)	118·2 (8)	C(13)–C(14)–C(16)	116·0 (9)
C(7)–C(8)–C(9)	121·8 (8)	C(13)–C(14)–C(17)	106·0 (8)
C(8)–C(9)–C(10)	121·2 (8)	C(15)–C(14)–C(16)	107·0 (9)
C(8)–C(7)–C(13)	125·5 (6)	C(15)–C(14)–C(17)	108·9 (9)
C(9)–C(10)–C(11)	118·6 (8)	C(16)–C(14)–C(17)	109·3 (9)
C(9)–C(10)–O(10)	116·1 (8)		

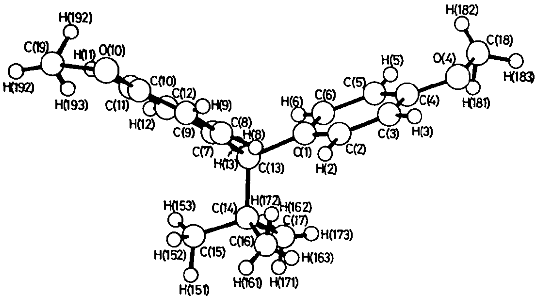

Fig. 1. 1,1-Bis(*p*-methoxyphenyl)-2,2-dimethylpropane viewed perpendicular to the C(1)–C(13)–C(14) plane.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35138 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

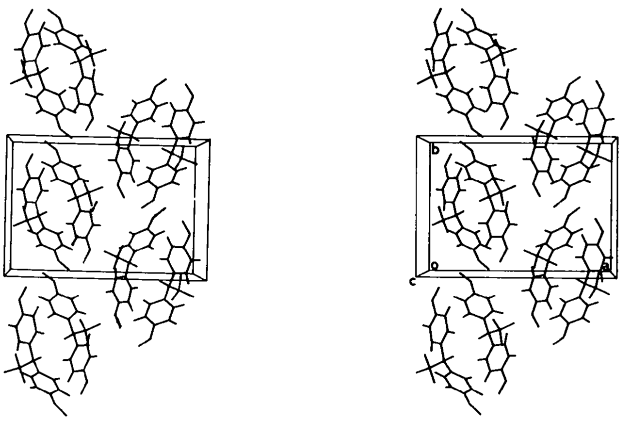

Fig. 2. Stereoscopic view of the packing in the cell viewed perpendicular to the *ab* plane. Methyl hydrogens are omitted for clarity.

out of the ring plane. The methyl groups of the neopentyl residue adopt a normal staggered conformation while the torsion angles down the C(13)–C(1) and C(13)–C(7) vectors between the C(14)–C(13) bond and the plane of the benzene ring are +72·1 and −70·8° respectively. These angles are equivalent to the angles τ_1[C(2)–C(1)–C(13)–C(14)] and τ_2[C(8)–C(7)–C(13)–C(14)] used by Hovmöller, Norrestam & Palm (1977) in their description of the conformational aspects of a number of DDT analogues and compare with the +82 and −66°, respectively, for the equivalent angles in GH44. Intramolecular distances and angles are normal. However, the *exo* C(1), C(7) and C(4), C(10) angles are distorted. Similar effects were found in GH44 and a number of other DDT analogues (Smith, 1978). The mode of packing of the molecules in the cell, van der Waals in nature, is illustrated in Fig. 2 showing discrete molecular units with no significant intermolecular contacts [minimum, 3·36 (1) Å, O(10)···C(18)].

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Structure of *meso*-5,5,7,12,12,14-Hexamethyl-1,4,8,11-tetraazacyclotetradecane Dihydrate*

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Abstract. $C_{16}H_{36}N_4.2H_2O$, $M_r = 320·51$, tetragonal, $I4_1/a$ (origin at $\bar{1}$), $Z = 8$, $a = b = 18·647$ (4), $c = 12·021$ (3) Å, $V = 4179·8$ Å^3, $d_m = 1·026$, $d_c = 1·029$ Mg m^{-3}, μ(Cu $K\alpha$) = 0·465 mm^{-1}. 2288 reflections were measured; of these only 870 independent reflections had $I > 2\sigma_I$. The structure was solved by direct methods and refined by the full-matrix least-squares method to a final $R = 0·091$ ($R_w = 0·051$, weights from counting statistics). The molecule appears to have C_i symmetry and a slightly deformed crown conformation with equatorial methyl

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* For a preliminary communication see Krajewski, Urbańczyk-Lipkowska & Gluziński (1979).

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