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The Crystal and Molecular Structure of 1,4-Diethoxybenzene

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The crystal structure of 1,4-diethoxybenzene has been determined from visually estimated Cu $K\alpha$ X-ray data. The crystals are monoclinic, space group $P2_1$, with $a = 9.518$ (6), $b = 7.61$ (1), $c = 7.137$ (6) Å, $\beta = 110.40$ (6)° and $Z = 2$. The structure was solved by the Patterson method and refined by block-diagonal least-squares calculations to $R = 0.069$ for 672 non-zero reflexions. The molecule is nearly centrosymmetric and planar. The ethoxy groups take *trans* conformations. The benzene ring seems to adopt a Kekulé structure. The C(aromatic)—O bond lengths, 1.378 (7) and 1.377 (7) Å, are longer than the C(sp^2)—O length in aromatic esters, while the C(aliphatic)—O lengths, 1.416 (8) and 1.426 (8) Å, are shorter than the corresponding length in the esters. The C—O—C angles, 119.4 (5) and 118.9 (5)° are close to the sp^2 angle. The molecules related by a twofold screw axis are stacked with a dihedral angle of 75.5° to form a chevron in a similar manner to 1,4-dimethoxybenzene and terphenyl. The molecular sheets parallel to (100) are stacked through H \cdots O and loose CH₃ \cdots CH₃ contacts.

Introduction

The dialkyl ethers of *p*-hydroquinone have a residual dipole moment; in benzene solution the moments of the dimethyl and diethyl ethers are 1.70 and 1.80 D respectively (Mizushima, Morino & Okazaki, 1938). Goodwin, Przybylska & Robertson (1950) have established, however, that the molecule of 1,4-dimethoxybenzene is centrosymmetric in the crystalline state. This apparent discrepancy may be ascribed to the rotation of the alkyl group about the ring C—O bond in phenyl alkyl ethers. This sort of rotation is considered to play an important role in the β -blocking adrenergic action (Fukui, Nagata & Imamura, 1970; Petrongolo & Tomasi, 1975) or in the nematic aggregate in liquid crystals (Bryan, 1967).

As an extension of the structural study of bis(2-hydroxyethyl) terephthalate (Kashino & Haisa, 1975), this paper reports the conformation of ethoxy groups attached to the benzene ring and the stacking mode of the molecules in the crystal, and compares the geometry of the ethereal linkage in ethers and esters.

Experimental

The crystals were grown from an acetone solution by slow evaporation as colourless plates with developed (100).

Crystal data

1,4-Diethoxybenzene, C₁₀H₁₄O₂, $M_r = 166.2$, m.p. 70–71°C. Monoclinic, space group $P2_1$, $a = 9.518$ (6), $b = 7.61$ (1), $c = 7.137$ (6) Å, $\beta = 110.40$ (6)°, $V = 484.5$ (8) Å³, $D_m = 1.14$ (by flotation in aqueous solution of KI), $D_x = 1.139$ g cm⁻³ for $Z = 2$, $\mu = 6.4$ cm⁻¹ for Cu $K\alpha$ ($\lambda = 1.5418$ Å), $F(000) = 180$. Systematic absences $0k0$ with k odd. The higher symmetry was ruled out by the determination of the structure.

By using specimens with dimensions 0.18 × 0.29 × 0.70 and 0.15 × 0.60 × 0.38 mm in glass capillaries, intensity data were collected on Weissenberg photographs for the layers $hk0$ to $hk5$ and $h0l$ to $h5l$ respectively. Visually estimated intensities were corrected for

Table 1. *The final positional and thermal parameters ($\times 10^4$) with standard deviations in parentheses*

The anisotropic thermal parameters have the form $\exp(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - \beta_{12}hk - \beta_{13}hl - \beta_{23}kl)$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
O(1)	7126 (3)	2533 (5)	1480 (4)	170 (5)	270 (8)	345 (9)	-53 (11)	189 (10)	-134 (16)
O(2)	2145 (3)	2428 (6)	3839 (4)	170 (5)	280 (7)	327 (8)	-45 (12)	171 (10)	-99 (16)
C(1)	5865 (5)	2500 (7)	2008 (6)	164 (6)	183 (8)	235 (10)	10 (14)	120 (12)	-13 (17)
C(2)	4579 (5)	1575 (6)	981 (6)	182 (7)	193 (9)	223 (9)	14 (14)	124 (13)	-51 (17)
C(3)	3363 (5)	1596 (7)	1650 (6)	161 (6)	201 (9)	262 (11)	-38 (14)	64 (13)	-38 (18)
C(4)	3393 (5)	2500 (7)	3286 (6)	161 (6)	170 (8)	255 (10)	10 (13)	81 (12)	18 (17)
C(5)	4682 (5)	3519 (7)	4315 (6)	168 (7)	196 (8)	257 (11)	-31 (14)	97 (13)	-45 (18)
C(6)	5895 (5)	3480 (7)	3678 (7)	163 (7)	202 (9)	311 (12)	-37 (15)	71 (14)	-44 (20)
C(7)	7147 (6)	1550 (8)	-196 (7)	199 (7)	284 (12)	308 (12)	35 (17)	231 (15)	-40 (22)
C(8)	8656 (7)	1814 (10)	-445 (10)	272 (11)	339 (17)	541 (20)	69 (23)	437 (26)	-36 (33)
C(9)	2137 (5)	3411 (9)	5536 (8)	182 (7)	293 (12)	341 (14)	-44 (18)	146 (16)	-82 (23)
C(10)	709 (7)	2944 (11)	5900 (9)	255 (11)	452 (23)	476 (19)	-114 (26)	400 (24)	-175 (37)

Lorentz-polarization factors and for spot shape. Intensities of 672 independent non-zero reflexions (57% of those accessible in the Cu sphere) were placed on an approximately absolute scale by the Wilson plot ($B = 6.4 \text{ \AA}^2$).

Structure determination and refinement

The structure was solved by the Patterson method and refined by block-diagonal least-squares calculations. When R was 0.11 all the H atoms were located by a difference Fourier synthesis. With anisotropic thermal parameters for the non-hydrogen atoms and the weighting scheme $w = 1.0$ for $0 < |F_o| \leq 4.0$ and $w = (4.0/|F_o|)^2$ for $|F_o| > 4.0$, the refinement reduced the R value to 0.069 for 672 non-zero reflexions.*

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32068 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

The final atomic parameters are listed in Tables 1 and 2. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962). The computation was carried out on an NEAC 2200-500 computer at the Okayama University Computer Center. The programs used were *HBL5-5* and *DAPH* (Ashida, 1973).

Table 2. *The final parameters of the hydrogen atoms*

	x	y	z	$B (\text{\AA}^2)$
H(1)	0.450 (4)	0.099 (6)	-0.013 (6)	1.8 (0.9)
H(2)	0.244 (6)	0.100 (11)	0.099 (9)	7.6 (1.7)
H(3)	0.479 (5)	0.418 (6)	0.555 (6)	2.7 (1.0)
H(4)	0.677 (6)	0.426 (9)	0.445 (8)	5.9 (1.4)
H(5)	0.626 (5)	0.190 (8)	-0.131 (7)	4.2 (1.2)
H(6)	0.692 (6)	0.030 (8)	0.002 (7)	5.1 (1.4)
H(7)	0.868 (8)	0.326 (14)	-0.100 (11)	10.4 (2.1)
H(8)	0.881 (7)	0.061 (11)	-0.105 (10)	8.0 (1.8)
H(9)	0.939 (7)	0.144 (12)	0.093 (10)	9.3 (2.1)
H(10)	0.304 (5)	0.303 (9)	0.681 (7)	5.4 (1.3)
H(11)	0.210 (8)	0.478 (13)	0.527 (10)	9.7 (2.1)
H(12)	0.075 (6)	0.161 (10)	0.628 (8)	7.1 (1.6)
H(13)	0.072 (5)	0.344 (9)	0.726 (7)	5.4 (1.3)
H(14)	-0.000 (9)	0.336 (13)	0.469 (12)	9.9 (2.0)

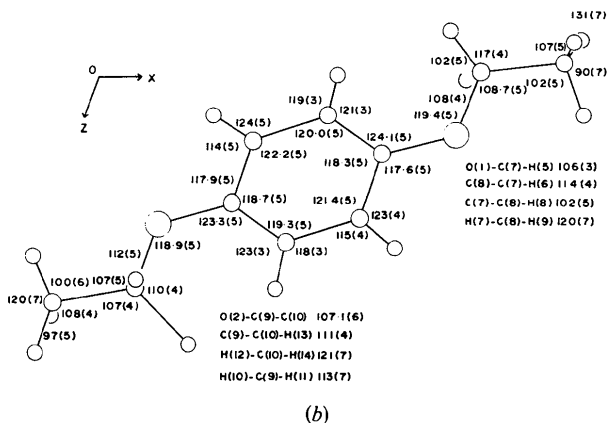
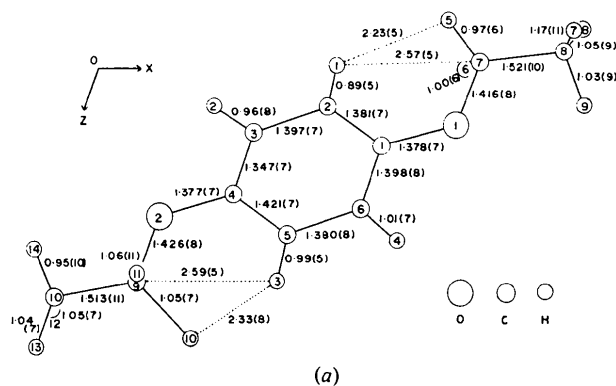


Fig. 1. The molecular structure: (a) bond lengths (\AA), intra-molecular contacts (\AA) and numbering of atoms, (b) bond angles ($^\circ$). The e.s.d.'s are shown in parentheses.

Results and discussion

Molecular structure

Bond lengths and angles are shown in Fig. 1. The least-squares plane of the benzene ring and deviations of atoms from the plane are listed in Table 3. The molecule is nearly centrosymmetric and planar, even though it lies at a general position. The conformations of the two ethoxy groups show only a slight difference on close examination. However, simple molecules of symmetric 1,4-disubstituted benzenes generally have an inversion centre in the crystals, as shown in Table 4. This occurs even for molecules with different substituents, by means of disorder, e.g. *p*-chlorobromobenzene ($P2_1/a$, $Z = 2$, Hendricks, 1932; Klug, 1947),

Table 3. Least-squares plane of the benzene ring and deviations (Å) of atoms from the plane

Atoms with an asterisk were used for the calculation of the plane.

$$X = ax + cz \cos \beta, Y = by, Z = cz \sin \beta.$$

$$-0.1866X + 0.7902Y - 0.5838Z + 0.2153 = 0$$

C(1)*	-0.014	C(8)	-0.078	H(7)	0.97
C(2)*	0.012	C(9)	-0.018	H(8)	-0.62
C(3)*	0.010	C(10)	-0.171	H(9)	-0.91
C(4)*	-0.015	H(1)	0.06	H(10)	-0.85
C(5)*	0.015	H(2)	0.04	H(11)	0.90
C(6)*	-0.005	H(3)	-0.03	H(12)	-1.11
O(1)	-0.036	H(4)	0.04	H(13)	-0.35
O(2)	-0.027	H(5)	0.70	H(14)	0.62
C(7)	-0.055	H(6)	-0.84		

p-chloronitrobenzene ($P2_1/c$, $Z = 2$; Mak & Trotter, 1962), and the triclinic form of 4-formylbenzoic acid ($P\bar{1}$, $Z = 1$; Haisa, Kashino, Ikejiri, Ohno & Teranishi, 1976).

The C—C bond lengths in the benzene ring are alternately longer and shorter than the average 1.387 Å, the ring seeming to adopt the Kekulé form. The inner angles at the 1 and 4 positions are slightly smaller than the sp^2 angle. The exocyclic C—O bonds deviate on the same side of the benzene ring. The molecule displays a slight but significant bowing.

The C(ar)—O bond lengths are longer than those found in other aromatic ethers, while the C(al)—O lengths are shorter, as shown in Table 5. From Fig. 2, a plot of the C(sp^2)—O lengths vs C(al)—O lengths in both ethers and esters, it may be concluded that the C(sp^2)—O bonds in esters are shorter than those in ethers and that the longer C(sp^2)—O bonds are accompanied by the shorter C(al)—O bonds. The former may be ascribed to the higher π -electron density around the C atom owing to participation in the carbonyl group. The latter seems to result from an efflux of π -electrons from the C(sp^2)—O bond into the C(al)—O bond.

The bond angles of the ethereal O atoms are significantly different from the 110 or 125° expected for the sp^3 hybridization in all the ethers in Table 5. This is consistent with the suggestion by Petrongolo & Tomasi (1975) that aromatic ethers are better described by sp^2 hybridization. The torsion angles about the C(ar)—O and O—C(al) bonds are close to 180° in the ethers listed in Table 5.

The residual dipole moment of 1.80 D cannot be explained by the present structure. This moment is con-

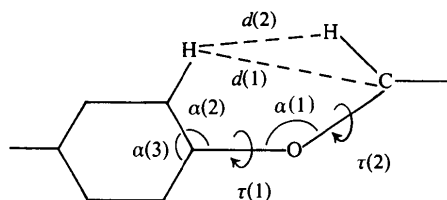
Table 4. Crystal structures of symmetric 1,4-disubstituted benzenes

		Space group	Z	Symmetry of molecules in crystals	Type of molecular overlapping
(I)	1,4-Dimethoxybenzene	<i>Pbca</i>	4	$\bar{1}$	Oblique
(II)	Ammonium hydrogen terephthalate	<i>C2/c</i>	4	$\bar{1}$	Parallel
(III)	Hydroquinone (γ -form)	$P2_1/c$	4*	$\bar{1}$	Parallel
(IV)	Bis(2-hydroxyethyl) terephthalate	$P2_1/a$	4*	$\bar{1}$	Parallel
(V)	Diethyl terephthalate	$P2_1/n$	2	$\bar{1}$	Parallel
(VI)	Piperazinium terephthalate	$P2_1/c$	2	$\bar{1}$	Oblique
(VII)	Terphenyl	$P2_1/c$	2	$\bar{1}$	Oblique
(VIII)	<i>p</i> -Dichlorobenzene (monoclinic form)	$P2_1/a$	2	$\bar{1}$	Parallel
(IX)	<i>p</i> -Dinitrobenzene	$P2_1/n$	2	$\bar{1}$	Parallel
(X)	1,4-Diethoxybenzene	$P2_1$	2	1	Oblique
(XI)	Terephthalic acid (form I)	$P\bar{1}$	1	$\bar{1}$	Parallel
(XII)	Terephthalamide	$P\bar{1}$	1	$\bar{1}$	Parallel
(XIII)	<i>p</i> -Dichlorobenzene (triclinic form)	$P\bar{1}$	1	$\bar{1}$	Parallel
(XIV)	Terephthalic acid (form II)	<i>P1</i>	1	1	Parallel

References: (I) Goodwin, Przybylska & Robertson (1950). (II) Cobbleddick & Small (1972a). (III) Maartmann-Moe (1966). (IV) Kashino & Haisa (1975). (V) Bailey (1949). (VI) Kashino, Sasaki & Haisa (1973). (VII) Hertel & Römer (1933). (VIII) Panattoni, Frasson & Bezzi (1963). (IX) Trotter (1961). (X) This work. (XI) Bailey & Brown (1967). (XII) Cobbleddick & Small (1972b). (XIII) Housty & Clastre (1957). (XIV) Bailey & Brown (1967).

* The unit cell contains two kinds of molecules.

Table 5. Geometrical features of related ethers



See Fig. 2 for compounds and references.

Compound	C(ar)—O	C(al)—O	$\alpha(1)$	$\alpha(2)$	$\alpha(3)$	$\tau(1)$	$\tau(2)$	$d(1)$	$d(2)$
(1)	1.378 Å	1.416 Å	119.4°	124.1°	118.3°	179.6°	180.0°	2.57 Å	2.23 Å
(1)	1.377	1.426	118.9	123.3	118.7	181.5	185.4	2.59	2.33
(2)	1.377	1.429	117.2	125.1	120.0	188.4	176.3	2.34	—
(3)	1.377	1.418	119.4	124.3	120.2	181.1	179.9	2.57	2.34
(3)	1.373	1.444	117.5	124.6	120.1	178.4	184.9	2.49	2.26
(4)	1.365	1.452	116.8	124.8	120.9	178.1	183.9	2.56	—
(5)	1.358	1.450	118.0	124.0	120.4	181.0	178.4	2.50	2.29
(4)	1.357	1.455	117.8	123.8	120.0	180.7	175.5	2.55	—

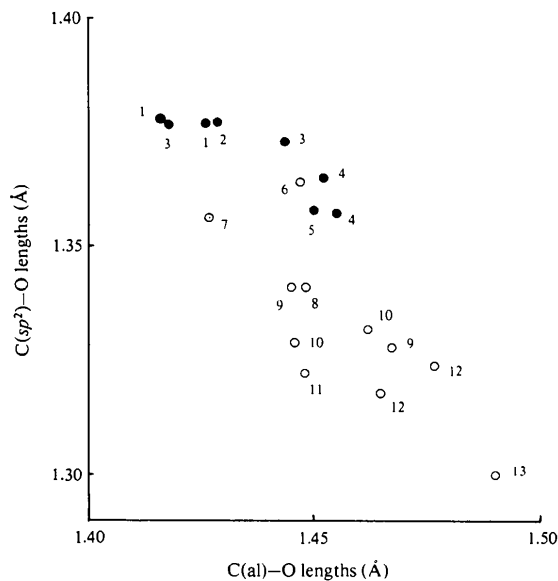


Fig. 2. Plot of the $C(sp^2)$ —O lengths vs $C(al)$ —O lengths in ethers (●) and esters (○). (1) 1,4-Diethoxybenzene (this work). (2) 4-Ethoxyisonitrosoacetanilide (Briansó, Miravittles, Plana & Font-Altaba, 1973). (3) *p-n*-Butoxybenzoic acid (Bryan & Fallon, 1975). (4) 4,4'-Azodiphenetole (Galigné, 1970). (5) *p*-Ethoxybenzoic acid (Bryan & Jenkins, 1975). (6) Procaine-bis-*p*-nitrophenyl phosphate complex (Sax, Pletcher & Gustaffson, 1970). (7) Procaine.HCl (Dexter, 1972). (8) Parpanit (Griffith & Robertson, 1972). (9) 3-Ethoxycarbonyl-4-oxo-6-methylhomopyrimidazole (Sasvári, Horvai & Simon, 1972). (10) Bis-(2-hydroxyethyl) terephthalate (Kashino & Haisa, 1975). (11) 4-Ethoxycarbonylanilinium bis-*p*-nitrophenylphosphate (Pletcher, Sax & Yoo, 1972). (12) Ethyl *p*-azoxybenzoate (Krigbaum & Barber, 1971). (13) Ethyl tyrosinate (Pieret, Durant, Griffé, Germain & Debaerdemaeker, 1970).

sidered to result from the contribution of the *cis* conformation of C(7) and C(9) with respect to the $O(1) \cdots O(2)$ axis (Mizushima, Morino & Okazaki, 1938), to the same degree as the *trans* conformation.

The intramolecular C(7)—H(1) and C(9)···H(3) contacts are significantly shorter than, while the H(1)···H(5) and H(3)···H(10) are comparable with, the sum of the van der Waals radii. The angles C(2)—C(1)—O(1) and C(5)—C(4)—O(2) are significantly larger than the sp^2 angle, as observed in phenols (Coppens & Schmidt, 1965; Fukuyama, Ohkura, Kashino & Haisa, 1973). The mean value for aromatic ethers in Table 5 is $124.3 \pm 0.5^\circ$.

Crystal structure

The molecular packing arrangement in the crystal is illustrated in Fig. 3. The benzene ring makes angles of 100.8 , 37.8 and 125.7° with the a , b and c^* axes respectively, which are close to those in 1,4-dimethoxybenzene. The molecules related by a twofold screw axis are stacked 'en chevron' with a dihedral angle of 75.5° . The short contacts $C(3) \cdots H(3^{vii})$ 2.83 (5) Å and $O(2) \cdots H(4^{vii})$ 2.74 (7) Å seem to determine the mutual arrangement of the molecules as seen from Fig. 4. None of the other intermolecular contacts is less than the sum of the van der Waals radii. The molecular sheets parallel to (100) are stacked through $H(9) \cdots O(2^y)$ and loose methyl···methyl contacts.

An examination of the molecular overlapping of the related compounds listed in Table 4 indicates that the majority are of the parallel type while the remaining four are oblique. Except for piperazinium terephthalate, a very similar oblique overlapping has been observed in

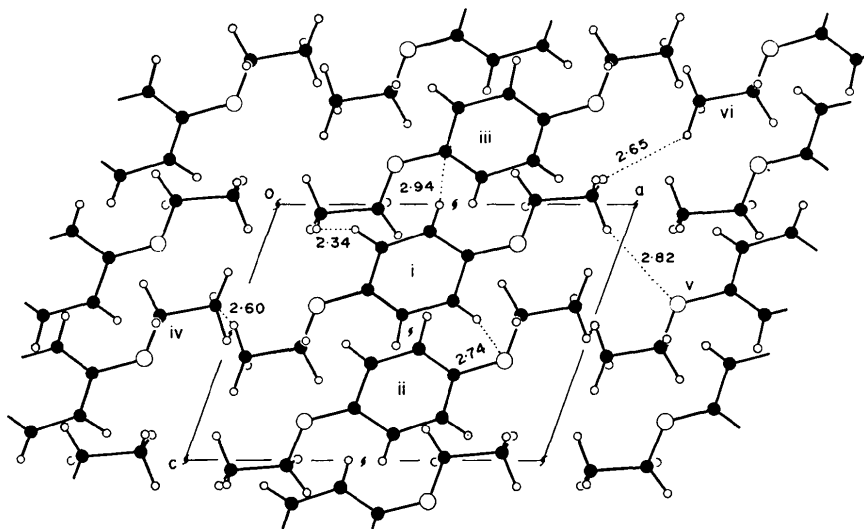


Fig. 3. Projection of the crystal structure along the b axis. Dotted lines show intermolecular contacts. Symmetry code: (i) x, y, z ; (ii) $1 - x, \frac{1}{2} + y, 1 - z$; (iii) $1 - x, -\frac{1}{2} + y, -z$; (iv) $-x, \frac{1}{2} + y, 1 - z$; (v) $1 + x, y, z$; (vi) $1 + x, y, -1 + z$.

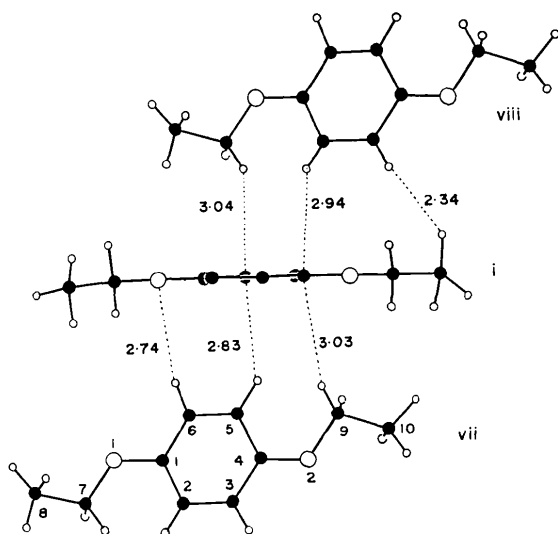


Fig. 4. Arrangement of the molecules, together with intermolecular distances (Å). The H atoms attached to the benzene ring of molecule (i) are omitted. Symmetry code: (i) x, y, z ; (vii) $1 - x, -\frac{1}{2} + y, 1 - z$; (viii) $1 - x, \frac{1}{2} + y, -z$.

Table 6. Molecular stacking and dihedral angles for the nearly planar molecules crystallizing in the space group $P2_1$

	b (Å)	δ (°)	k	In Fig. 5
<i>p</i> -Diethoxybenzene	7.61	75.5	0.733	I
Phenanthrene	6.16	58.6	0.734	IV
1,2-Benzanthracene	6.50	47.6	0.731	V
1,2;5,6-Dibenzanthracene	5.654	44.6	0.760	VI
1,9;5,10-Diperinaphthyleneanthracene	7.83	8.7	0.755	VII

δ : dihedral angle between molecules related by a 2_1 operation.

k : packing coefficient calculated based on the definition of Kitaigorodsky (1973).

1,4-dimethoxybenzene and also terphenyl, as compared in Fig. 5. The present compound included, only a few of the many nearly-planar hydrocarbons crystallize in the space group $P2_1$, as listed in Table 6, and their molecular overlapping, in every case, is of the oblique type (Fig. 5). The long molecular axes are approxi-

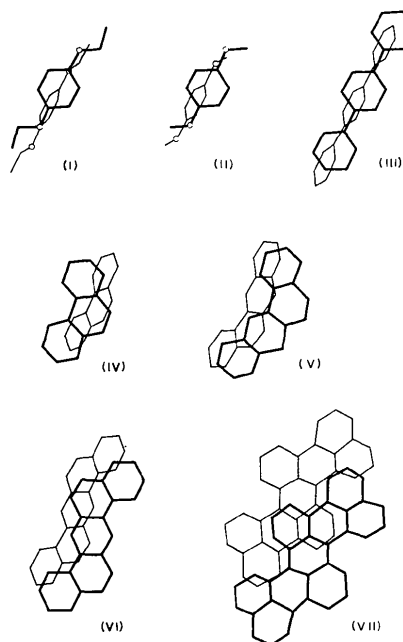


Fig. 5. Molecular overlapping of some related compounds. (I) 1,4-Diethoxybenzene (this work). (II) 1,4-Dimethoxybenzene (Goodwin, Przybylska & Robertson, 1950). (III) Terphenyl (Hertel & Römer, 1933). (IV) Phenanthrene (Trotter, 1963). (V) 1,2-Benzanthracene (Friedlander & Sayre, 1956). (VI) 1,2;5,6-Dibenzanthracene, monoclinic form (Robertson & White, 1956). (VII) 1,9;5,10-Diperinaphthyleneanthracene (Rossmann, 1959).

mately parallel to each other and the dihedral angle between the molecular planes becomes smaller as the molecular width of the aromatic portion increases. The packing coefficients are rather large for these crystals.

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