C22	0.6154 (2)	0.1452 (3)	0.0140 (2)	0.047 (2)
C23	0.7774 (2)	−0.1795 (3)	0.0887 (2)	0.048 (2)
C24	0.8326 (2)	−0.2724 (3)	0.0766 (2)	0.066 (3)
C25	0.8221 (3)	−0.3780 (3)	0.1168 (2)	0.078 (3)
C26	0.7597 (3)	−0.3881 (3)	0.1666 (2)	0.075 (3)
C27	0.7052 (2)	−0.2932 (3)	0.1788 (2)	0.061 (2)
C28	0.7149 (2)	−0.1882 (3)	0.1398 (2)	0.047 (2)
B	0.6980 (2)	−0.0025 (4)	0.0850 (2)	0.050 (2)
H1	0.767 (2)	−0.164 (4)	−0.124 (2)	0.10 (1)
H2	0.821 (2)	−0.053 (3)	−0.123 (2)	0.07 (1)
H3	0.724 (2)	−0.040 (3)	−0.131 (2)	0.06 (1)
H4	0.774 (2)	−0.070 (3)	−0.057 (2)	0.06 (1)

Table 2. *Geometric parameters* (Å, °)

O1—C1	1.419 (5)	C2—C3	1.474 (6)
O1—C2	1.415 (5)	C4—C5	1.478 (7)
O2—C3	1.421 (5)	C6—C7	1.468 (6)
O2—C4	1.418 (5)	C8—C9	1.487 (6)
O3—C5	1.429 (5)	C10—C11	1.492 (6)
O3—C6	1.393 (5)	C11—C12	1.385 (6)
O4—C7	1.425 (5)	C11—C16	1.381 (5)
O4—C8	1.396 (5)	C12—C13	1.371 (7)
O5—C9	1.415 (5)	C13—C14	1.361 (6)
O5—C10	1.425 (4)	C14—C15	1.370 (5)
O6—C17	1.362 (4)	C15—C16	1.384 (5)
O6—B	1.467 (4)	C17—C18	1.364 (5)
O7—C22	1.361 (4)	C17—C22	1.396 (4)
O7—B	1.472 (4)	C18—C19	1.385 (5)
O8—C23	1.369 (4)	C19—C20	1.377 (6)
O8—B	1.502 (4)	C20—C21	1.403 (5)
O9—C28	1.349 (4)	C21—C22	1.359 (5)
O9—B	1.472 (4)	C23—C24	1.366 (5)
N—H1	1.02 (4)	C23—C28	1.391 (4)
N—H2	0.88 (3)	C24—C25	1.392 (5)
N—H3	0.83 (3)	C25—C26	1.376 (6)
N—H4	0.89 (4)	C26—C27	1.375 (5)
C1—C15	1.488 (5)	C27—C28	1.374 (5)
C2—O1—C1	113.7 (3)	C16—C15—C14	118.6 (3)
C4—O2—C3	113.5 (3)	C16—C15—C1	120.1 (3)
C6—O3—C5	113.9 (3)	C14—C15—C1	121.2 (3)
C8—O4—C7	113.8 (3)	C15—C16—C11	121.1 (3)
C10—O5—C9	112.3 (3)	C22—C17—C18	121.1 (3)
B—O6—C17	106.5 (2)	C22—C17—O6	111.2 (3)
B—O7—C22	107.6 (2)	C18—C17—O6	127.7 (3)
B—O8—C23	106.7 (2)	C19—C18—C17	117.4 (3)
B—O9—C28	107.7 (2)	C20—C19—C18	121.9 (3)
H4—N—H3	109 (3)	C21—C20—C19	120.5 (3)
H4—N—H2	110 (3)	C22—C21—C20	117.2 (3)
H4—N—H1	110 (3)	C21—C22—C17	121.9 (3)
H3—N—H2	114 (3)	C21—C22—O7	128.8 (3)
H3—N—H1	111 (3)	C17—C22—O7	109.3 (3)
H2—N—H1	103 (3)	C28—C23—C24	122.1 (3)
C15—C1—O1	106.8 (3)	C28—C23—O8	109.9 (3)
C3—C2—O1	109.1 (3)	C24—C23—O8	128.1 (3)
C2—C3—O2	108.5 (3)	C25—C24—C23	116.7 (3)
C5—C4—O2	108.5 (3)	C26—C25—C24	121.7 (4)
C4—C5—O3	109.9 (3)	C27—C26—C25	120.9 (3)
C7—C6—O3	110.3 (3)	C28—C27—C26	118.1 (3)
C6—C7—O4	109.4 (3)	C27—C28—C23	120.5 (3)
C9—C8—O4	109.7 (3)	C27—C28—O9	128.3 (3)
C8—C9—O5	110.4 (3)	C23—C28—O9	111.2 (3)
C11—C10—O5	109.2 (3)	O9—B—O8	104.1 (3)
C16—C11—C12	118.5 (4)	O9—B—O7	111.8 (3)
C16—C11—C10	119.3 (3)	O9—B—O6	113.6 (3)
C12—C11—C10	122.2 (3)	O8—B—O7	111.0 (3)
C13—C12—C11	120.5 (4)	O8—B—O6	111.2 (3)
C14—C13—C12	120.0 (4)	O7—B—O6	105.3 (3)
C15—C14—C13	121.2 (4)		

Data were corrected for Lorentz and polarization effects. $\sigma(F)$ was calculated from $[\sigma(I)^2 + (Ik)^2]^{1/2}/2F$, where $k = 0.02$. The H atoms attached to the N atom were located on a difference Fourier map and refined with isotropic temperature factors. The remaining H atoms were fixed at calculated positions [d(C—H) = 0.95 Å; $U_H = 0.05$ Å^2].

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 55685 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1034]

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Acta Cryst. (1993). C**49**, 404–406

Structure of 1:1 Clathrate between Tris(2,3-naphthalenedioxy)cyclotriphosphazene and *p*-Xylene

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(*Received* 28 *April* 1992; *accepted* 24 *August* 1992)

Abstract

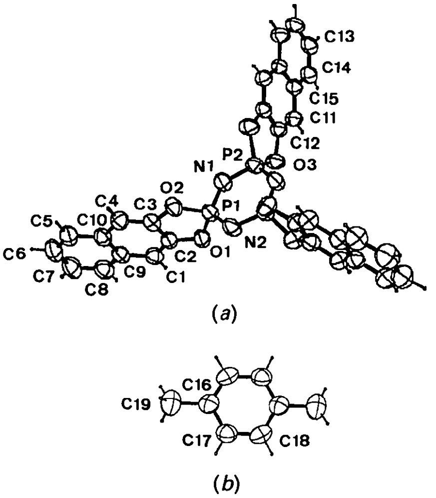
The host molecule lies on the crystallographic twofold axis. The centroid of the guest, *p*-xylene, is located at a center of symmetry. The host forms a cage-type 1:1 clathrate with *p*-xylene. The *p*-xylene is

0108-2701/93/020404-03$06.00

accommodated in the cage-like cavity produced by the naphthalenedioxy groups of the host molecules.

Comment

The host compound, tris(2,3-naphthalenedioxy)-cyclotriphosphazene (TNDOCTP), is known to form channel-type clathrates of isomorphous structures with benzene, chloroform, *etc.* (Allcock & Stein, 1974; Allcock, 1984). Tris(1,8-naphthalenedioxy)-cyclotriphosphazene (1,8-isomer of TNDOCTP) also forms a channel-type clathrate with *p*-xylene (Allcock, Stein & Bissell, 1974). Allcock (1984) confirmed that TNDOCTP forms a clathrate with *p*-xylene. The host was synthesized from hexachlorocyclotriphosphazene and 2,3-dihydroxynaphthalene as described by Allcock & Kugel (1966). The *p*-xylene clathrate, TNDOCTP/*p*-xylene, obtained by recrystallization from *p*-xylene solution, was relatively unstable in air. The cell dimensions are completely different from those observed in the channel-type clathrates and it is of great interest how the hosts include *p*-xylene molecules.



(*a*)

(*b*)

Fig. 1. Atomic numbering for (*a*) host and (*b*) guest with thermal ellipsoids drawn at 50% probability.

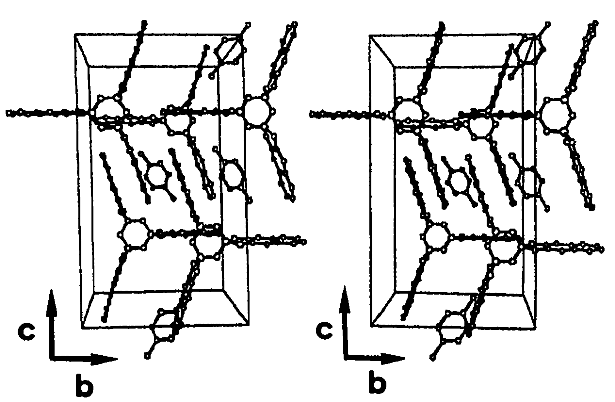


Fig. 2. Stereoview of the unit-cell packing projected along the *a* axis.

Experimental

Crystal data

$C_{30}H_{18}N_3O_6P_3.C_8H_{10}$
$M_r = 715.58$
Orthorhombic
Pbcn
$a = 10.609\ (4)$ Å
$b = 13.705\ (7)$ Å
$c = 23.535\ (6)$ Å
$V = 3422\ (2)$ Å^3
$Z = 4$
$D_x = 1.389$ Mg m^{-3}
$D_m = 1.38$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 19 reflections
$\theta = 17.1$–$19.5°$
$\mu = 2.034$ mm^{-1}
$T = 293$ K
Prism
0.3 × 0.1 × 0.1 mm
Colorless

Data collection

Rigaku AFC-5*RU* diffractometer
$\omega/2\theta$ scans
5256 measured reflections
2768 independent reflections
2231 observed reflections $[I>3\sigma(I)]$
$R_{\rm int} = 0.0194$

$\theta_{\rm max} = 60°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 15$
$l = -26 \rightarrow 26$
3 standard reflections monitored every 56 reflections intensity variation: 5%

Refinement

Refinement on F
Final $R = 0.0410$
$wR = 0.0484$
$S = 2.24$
2200 reflections
284 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o)+(0.023F_o)^2]$
$(\Delta/\sigma)_{\rm max} = 0.4$
$\Delta\rho_{\rm max} = 0.78$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.42$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$B_{\rm eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$B_{\rm eq}$
P1	0.01199 (6)	0.73229 (5)	0.69157 (3)	4.00 (2)
P2	0.0	0.55760 (6)	0.75	4.10 (2)
N1	0.0109 (2)	0.6175 (1)	0.69349 (8)	4.55 (5)
N2	0.0	0.7869 (2)	0.75	4.7 (1)
O1	−0.0920 (2)	0.7710 (1)	0.64772 (7)	4.65 (4)
O2	0.1333 (2)	0.7704 (1)	0.65776 (7)	4.61 (4)
O3	−0.1128 (2)	0.4801 (1)	0.74550 (8)	4.71 (4)
C1	−0.0900 (2)	0.8315 (2)	0.5495 (1)	4.5 (1)
C2	−0.0326 (2)	0.8036 (2)	0.5980 (1)	4.04 (9)
C3	0.0980 (2)	0.8044 (2)	0.6039 (1)	4.07 (9)
C4	0.1755 (2)	0.8326 (2)	0.5619 (1)	4.5 (1)
C5	0.1957 (3)	0.8910 (2)	0.4630 (1)	5.4 (1)
C6	0.1416 (3)	0.9168 (2)	0.4130 (1)	6.4 (1)
C7	0.0113 (3)	0.9163 (2)	0.4066 (1)	6.6 (1)
C8	−0.0639 (3)	0.8898 (2)	0.4506 (1)	5.6 (1)
C9	−0.0123 (3)	0.8615 (2)	0.5036 (1)	4.48 (9)
C10	0.1207 (3)	0.8621 (2)	0.5097 (1)	4.4 (1)
C11	−0.1333 (2)	0.3025 (2)	0.7449 (1)	4.08 (8)
C12	−0.0654 (2)	0.3852 (2)	0.7476 (1)	3.73 (4)
C13	−0.0654 (3)	0.0365 (2)	0.7478 (1)	5.08 (9)
C14	−0.1292 (3)	0.1222 (2)	0.7455 (1)	4.70 (9)

C15	−0.0668 (2)	0.2126 (2)	0.7474 (1)	3.88 (4)
C16	0.0191 (3)	0.5595 (2)	0.4521 (1)	5.0 (1)
C17	0.0614 (3)	0.5868 (2)	0.5050 (1)	5.3 (1)
C18	0.0430 (3)	0.5289 (2)	0.5519 (1)	5.5 (1)
C19	0.0424 (3)	0.6218 (3)	0.4007 (1)	7.6 (1)

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

P1—N1	1.574 (2)	P1—O2	1.601 (2)
P1—O1	1.601 (2)	P2—O3	1.604 (2)
P2—N1	1.567 (2)	O1—C2	1.402 (3)
N2—P1	1.571 (2)	O3—C12	1.395 (3)
O2—C3	1.401 (3)	C1—C9	1.420 (4)
C1—C2	1.349 (3)	C3—C4	1.343 (3)
C2—C3	1.393 (3)	C5—C6	1.356 (4)
C4—C10	1.418 (3)	C6—C7	1.391 (5)
C5—C10	1.413 (4)	C8—C9	1.416 (4)
C7—C8	1.357 (4)	C11—C12	1.344 (4)
C9—C10	1.418 (5)	C12—C12^{ii}	1.392 (3)
C11—C15	1.421 (4)	C13—C13^{ii}	1.392 (5)
C13—C14	1.357 (4)	C15—C15^{ii}	1.423 (3)
C14—C15	1.405 (4)	C16—C19	1.501 (4)
C16—C18^{i}	1.382 (4)	C17—C18	1.373 (4)
N1—P1—N2	116.8 (1)	N1—P1—O1	110.1 (1)
N1—P1—O2	110.2 (1)	N2—P1—O1	110.52 (9)
N2—P1—O2	110.17 (9)	O1—P1—O2	97.2 (1)
N1—P2—O3	110.2 (1)	N1—P2—N1^{ii}	116.8 (1)
N1—P2—O3^{ii}	110.3 (1)	O3—P2—N1	110.4 (1)
O3—P2—O3^{ii}	97.0 (1)	P1—N1—P2	123.3 (1)
P1—N2—P1^{ii}	123.1 (2)	P1—O1—C2	109.5 (2)
P1—O2—C3	110.1 (2)	P2—O3—C12	110.2 (2)
C2—C1—C9	117.6 (2)	O1—C2—C1	126.4 (2)
O1—C2—C3	111.5 (2)	C1—C2—C3	122.1 (2)
O2—C3—C2	110.7 (2)	O2—C3—C4	126.7 (2)
C2—C3—C4	122.6 (2)	C3—C4—C10	118.0 (2)
C6—C5—C10	120.6 (3)	C5—C6—C7	120.9 (3)
C6—C7—C8	120.2 (2)	C7—C8—C9	121.2 (3)
C1—C9—C8	121.7 (3)	C1—C9—C10	120.2 (2)
C8—C9—C10	118.2 (2)	C4—C10—C5	121.5 (3)
C4—C10—C9	119.6 (2)	C5—C10—C9	118.9 (2)
C12—C11—C15	117.6 (2)	O3—C12—C11	126.2 (2)
O3—C12—C12^{ii}	111.2 (2)	C11—C12—C12^{ii}	122.5 (2)
C14—C13—C13^{ii}	120.0 (3)	C13—C14—C15	121.8 (3)
C11—C15—C14	121.9 (2)	C11—C15—C15^{ii}	119.9 (2)
C14—C15—C15^{ii}	118.2 (2)	C17—C16—C18^{i}	117.1 (2)
C17—C16—C19	121.4 (3)	C16—C17—C18	121.6 (3)
C19—C16—C18^{i}	121.5 (2)	C17—C18—C16^{i}	121.3 (2)

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $-x, y, \frac{3}{2}-z$.

D_m was determined by flotation in an *n*-heptane/carbon tetrachloride mixture. The space group *Pbcn* was determined from systematic absences (0*kl*, *k* odd; *h*0*l*, *l* odd; *hk*0, *h* + *k* odd; *h*00, *h* odd; 0*k*0, *k* odd; 00*l*, *l* odd). A Lorentz–polarization correction was applied but no absorption or extinction corrections. The structure was solved by direct methods with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined by a block-diagonal least-squares method. The C, N, O and P atoms were refined with anisotropic temperature factors. H atoms were determined from difference Fourier syntheses and refined with isotropic temperature factors. N2 and P2 were fixed on the twofold axes. All computations were performed on a FACOM M382 computer at the Data Processing Center of Kyoto University using *KPPXRAY* (Taga, Masuda, Higashi & Iizuka, 1991).

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55579 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1012]

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Acta Cryst. (1993). C**49**, 406–408

Structure of 2-Phenylacetophenone

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(*Received* 12 *May* 1992; *accepted* 7 *September* 1992)

Abstract

The crystal structure of 2-phenylacetophenone (C_6H_5—CH_2—CO—C_6H_5) (CAS registry number 451-40-1) has been determined from 945 measured X-ray reflections with $I \geq \sigma(I)$. The dihedral angle between the two benzene ring planes is 64.2°.

0108-2701/93/020406-03$06.00