2701 reflections
316 parameters
H-atom positions calculated and refined as riding
$w = 1/[\sigma^2(F) + 0.000025F^2]$

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²)

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	$U_{\rm eq}$
O1	0.6617 (4)	0.5654 (2)	−0.4059 (2)	0.069 (2)
O3	0.4420 (3)	0.6217 (2)	−0.1197 (1)	0.061 (2)
O2	0.1801 (4)	0.6560 (2)	−0.1645 (2)	0.084 (2)
O4	0.2322 (5)	0.6661 (3)	0.4593 (2)	0.107 (3)
C1	0.6926 (7)	0.5277 (3)	−0.5679 (3)	0.081 (3)
C2	0.7137 (6)	0.5706 (3)	−0.5185 (2)	0.073 (3)
C3	0.5764 (6)	0.5802 (3)	−0.4669 (2)	0.065 (3)
C4	0.5701 (5)	0.5796 (2)	−0.3511 (2)	0.051 (2)
C5	0.3932 (5)	0.6019 (3)	−0.3480 (2)	0.059 (2)
C6	0.3194 (5)	0.6176 (3)	−0.2879 (2)	0.057 (3)
C7	0.4153 (5)	0.6109 (2)	−0.2319 (2)	0.051 (2)
C8	0.5919 (5)	0.5864 (2)	−0.2368 (2)	0.056 (2)
C9	0.6688 (5)	0.5716 (3)	−0.2937 (2)	0.058 (3)
C10	0.3305 (6)	0.6314 (3)	−0.1703 (2)	0.059 (3)
C11	0.3863 (5)	0.6495 (2)	−0.0563 (2)	0.052 (2)
C12	0.2536 (5)	0.5943 (2)	−0.0249 (2)	0.057 (2)
C13	0.2179 (5)	0.6156 (2)	0.0446 (2)	0.045 (2)
C14	0.3822 (4)	0.6277 (2)	0.0868 (2)	0.043 (2)
C15	0.5125 (4)	0.6820 (2)	0.0508 (2)	0.050 (2)
C16	0.5555 (5)	0.6568 (2)	−0.0181 (2)	0.058 (2)
C17	0.4725 (5)	0.5482 (2)	0.0981 (2)	0.057 (2)
C18	0.3308 (4)	0.6666 (2)	0.1513 (2)	0.042 (2)
C19	0.1605 (4)	0.6318 (2)	0.1815 (2)	0.042 (2)
C20	0.0053 (4)	0.6397 (2)	0.1339 (2)	0.050 (2)
C21	0.0536 (4)	0.6237 (2)	0.0670 (2)	0.050 (2)
C22	0.4847 (4)	0.6705 (3)	0.1999 (2)	0.056 (2)
C23	0.4353 (5)	0.7085 (2)	0.2637 (2)	0.056 (2)
C24	0.2760 (5)	0.6683 (2)	0.2946 (2)	0.044 (2)
C25	0.1222 (4)	0.6704 (2)	0.2453 (2)	0.043 (2)
C26	−0.0423 (5)	0.6470 (2)	0.2848 (2)	0.060 (3)
C27	−0.0042 (5)	0.6782 (3)	0.3527 (2)	0.065 (3)
C28	0.1847 (5)	0.7113 (2)	0.3519 (2)	0.052 (2)
C29	0.3234 (6)	0.5847 (3)	0.3149 (2)	0.066 (3)
C30	0.2811 (6)	0.7060 (3)	0.4148 (2)	0.068 (3)
C31	0.4454 (7)	0.7559 (3)	0.4227 (3)	0.094 (4)

Table 2. *Selected torsion angles* (°)

C3—O1—C4—C5	5.5 (6)	C22—C18—C19—C20	172.3 (3)
C11—O3—C10—O2	−6.6 (6)	C22—C23—C24—C29	66.5 (4)
C6—C7—C10—O2	−1.9 (7)	C23—C24—C25—C26	−167.1 (3)
C12—C13—C14—C17	−70.2 (4)	C27—C28—C30—O4	14.0 (7)
C21—C13—C14—C15	−131.2 (4)	C27—C28—C30—C31	−163.9 (4)

All non-H atoms were refined anisotropically by full-matrix least-squares methods.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *Xtal*3.2 *DIFDAT ADDREF ABSORB SORTRF* (Hall, Flack & Stewart, 1992). Program(s) used to solve structure: *Xtal*3.2 *GENTAN*. Program(s) used to refine structure: *Xtal*3.2 *CRYLSQ*. Molecular graphics: *Xtal*3.2 *ORTEP*. Software used to prepare material for publication: *Xtal*3.2 *BONDLA CIFIO*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1161). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bunning, T. J., Klei, H. E., Samulski, E. T., Adams, W. W. & Crane, R. L. (1993). *Mol. Cryst. Liq. Cryst.* **231**, 163–174.

Bunning, T. J., Klei, H. E., Samulski, E. T., Crane, R. L. & Linville, R. J. (1991). *Liq. Cryst.* **10**, 445–446.

Bunning, T. J. & Kreuzer, F. H. (1995). *TRIP*, **3**, 318–323.

Bunning, T. J., Socci, E. P., Farmer, B. L., Campbell, A. L. & Adams, W. W. (1996). *Mol. Cryst. Liq. Cryst.* In the press.

Chabinyc, M. L., Fratini, A. V., Socci, E. P., Farmer, B. L., Bunning, T. J. & Adams, W. W. (1995). *Acta Cryst*, **C51**, 1444–1447.

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5. Enraf–Nonius, Delft, The Netherlands.

Guerina, N. G. & Craven, B. M. (1979). *J. Chem. Soc. Perkin Trans.* 2, pp. 1414–1419

Hall, S. R., Flack, H. D. & Stewart, J. M. (1992). Editors. *Xtal*3.2 *Reference Manual*. Universities of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.

McHugh, C. M. & Bunning, T. J. (1995). *Am. Chem. Soc. Polym. Prep.* **36**, 379–380.

Shibaev, V. P. & Freidzon, Ya. S. (1989). *Side-Chain Liquid Crystal Polymers*, edited by C. B. McArdle, pp. 260–286. Glasgow: Blackie.

Socci, E. P., Farmer, B. L., Chabinyc, M. L., Fratini, A. V., Bunning, T. J. & Adams, W. W. (1995). *Acta Cryst.* **C51**, 888–891.

Sawzik, P. & Craven, B. M. (1980). *Acta Cryst.* **B36**, 215–218.

Acta Cryst. (1996). C**52**, 3183–3185

A Bicyclo[2.2.1]heptenone System

Kraig A. Wheeler and Christopher J. Carrow

Department of Chemistry, Delaware State University, Dover, DE 19901, USA. E-mail: kwheeler@desu.edu

(*Received 4 December 1995; accepted 22 July 1996*)

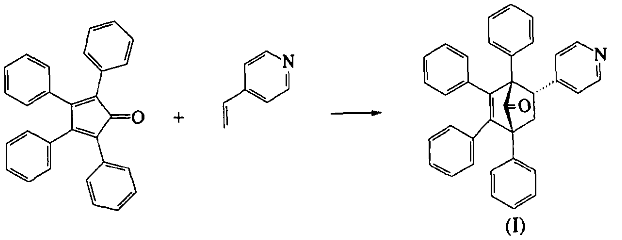
Abstract

The crystal and molecular structure of the title compound, 1,2,3,4-tetraphenyl-5-(4-pyridyl)bicyclo[2.2.1]hept-2-en-7-one, $C_{36}H_{27}NO$, has been determined to confirm the stereochemistry and investigate its potential inclusion behavior.

Comment

Interest in the systematic design and application of organic clathrates has increased markedly in recent years. Studies have shown that sterically encumbered compounds may exhibit inclusion behavior without the cohesive force of hydrogen bonding (Atwood, Davies & MacNicol, 1984, 1991). These inclusion complexes, stabilized by weak van der Waals interactions, are controlled primarily by the topological complementarity of the guest and host molecules.

1,2,3,4-Tetraphenyl-5-(4-pyridyl)bicyclo[2.2.1]hept-2-en-7-one, (I), was prepared by a Diels–Alder reaction (Wasserman, 1965; Huisgen, Grashey & Sauer,



(I)

1964) between tetraphenylcyclopentadienone and 4-vinylpyridine. Investigation of the molecular structure (Fig. 1) of (I) indicates the expected *endo* stereochemistry of the pyridyl group. Fig. 1 also reveals the sterically crowded conformation of (I) resulting from the rigidity of the bicyclo[2.2.1]heptenone moiety and close phenyl–phenyl intramolecular contacts. These anticipated conformational features of (I) are evident by the out-of-plane position of the phenyl rings with respect to the C2 and C3 sp^2 atoms [C3—C2—C19—C24 28.4 (5)°, C2—C3—C25—C30 82.1 (4)°].

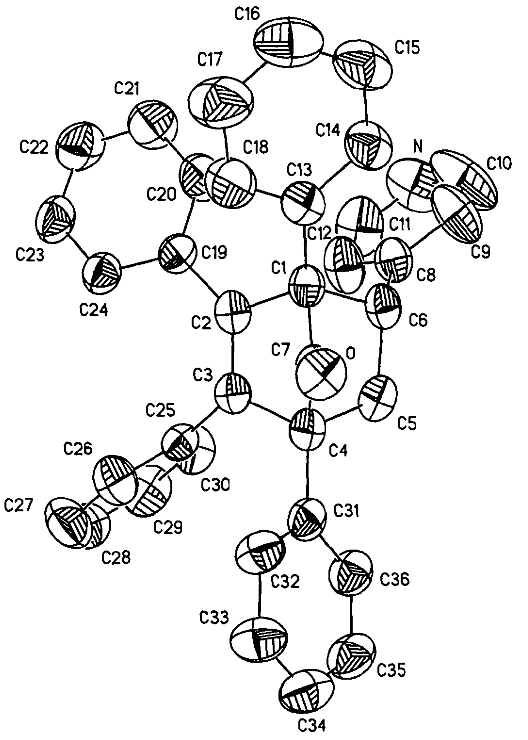


Fig 1. The molecular structure of (I) showing 50% probability displacement ellipsoids. The H atoms are omitted for clarity.

Analysis of the packing pattern (Fig. 2) of (I) reveals a modest 17 Å^3 cavity between centrosymmetrically related molecules [H26···H26′ 4.20 Å; H32···H32′ 4.40 Å; O···O′ 6.282 (3) Å (symmetry code: $-x$, $-y$, $-z$)]. This cavity results from the inability of molecules of (I) to form close-packing arrangements attributable to the crowded conformation of (I). Even though the volume of this lattice cavity is relatively small for direct inclusion of a guest molecule, such cavities suggest (I) as a potential host molecule. Expansion of host crystal lattices often occurs with guest inclusion, presumably the result of a more efficient packing motif (Atwood, Davies & MacNicol, 1984, 1991). Investigation of the inclusion behavior of (I) with varying guest molecules is in progress.

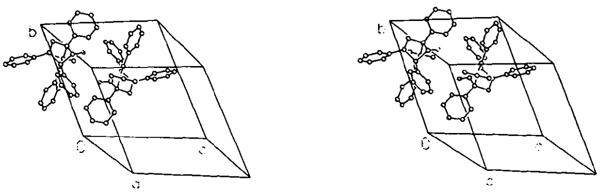


Fig 2. Stereographic representation showing the molecular packing of (I).

Experimental

The title compound was prepared by a Diels–Alder reaction between tetraphenylcyclopentadienone and 4-vinylpyridine (molar ratio 1:1) at room temperature. The resulting *endo* product was recrystallized from acetone by slow evaporation at room temperature.

Crystal data

$C_{36}H_{27}NO$
$M_r = 489.59$
Triclinic
$P\overline{1}$
$a = 10.689\ (1)$ Å
$b = 11.577\ (1)$ Å
$c = 12.355\ (1)$ Å
$\alpha = 117.86\ (1)°$
$\beta = 94.35\ (1)°$
$\gamma = 100.75\ (1)°$
$V = 1304.4\ (2)$ Å^3
$Z = 2$
$D_x = 1.247$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 16.82$–$19.89°$
$\mu = 0.091$ mm^{-1}
$T = 293\ (2)$ K
Transparent plate
0.38 × 0.15 × 0.09 mm
Light yellow

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
5046 measured reflections
4289 independent reflections
2275 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.032$

$\theta_{\rm max} = 24.99°$
$h = -1 \rightarrow 12$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 14$
3 standard reflections monitored every 97 reflections intensity decay: 5.7%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0589$
$wR(F^2) = 0.142$
$S = 1.03$

$(\Delta/\sigma)_{\rm max} = -0.02$
$\Delta\rho_{\rm max} = 0.17$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.18$ e Å^{-3}
Extinction correction: none

4289 reflections
343 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0356P)^2 + 0.0168P]$
where $P = (F_o^2 + 2F_c^2)/3$

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
N	−0.6564 (4)	−0.2164 (4)	−0.5840 (3)	0.0965 (12)
O	−0.2757 (2)	0.0336 (2)	0.0596 (2)	0.0551 (6)
C1	−0.3493 (3)	−0.1034 (3)	−0.1701 (3)	0.0404 (7)
C2	−0.2333 (3)	−0.1031 (3)	−0.2363 (3)	0.0418 (8)
C3	−0.1621 (3)	0.0259 (3)	−0.1871 (3)	0.0404 (7)
C4	−0.2278 (3)	0.1201 (3)	−0.0874 (3)	0.0411 (8)
C5	−0.3577 (3)	0.1129 (3)	−0.1594 (3)	0.0510 (9)
C6	−0.4442 (3)	−0.0313 (3)	−0.2059 (3)	0.0468 (8)
C7	−0.2812 (3)	0.0220 (3)	−0.0422 (3)	0.0431 (8)
C8	−0.5167 (3)	−0.0992 (3)	−0.3384 (3)	0.0501 (8)
C9	−0.6493 (4)	−0.1385 (4)	−0.3654 (4)	0.0792 (12)
C10	−0.7126 (4)	−0.1936 (5)	−0.4867 (5)	0.112 (2)
C11	−0.5295 (5)	−0.1783 (4)	−0.5568 (4)	0.0852 (13)
C12	−0.4558 (4)	−0.1201 (4)	−0.4386 (3)	0.0707 (11)
C13	−0.4055 (3)	−0.2268 (3)	−0.1606 (3)	0.0458 (8)
C14	−0.5369 (3)	−0.2780 (3)	−0.1747 (3)	0.0574 (9)
C15	−0.5824 (4)	−0.3914 (4)	−0.1638 (3)	0.0724 (12)
C16	−0.4971 (5)	−0.4543 (4)	−0.1379 (4)	0.0806 (13)
C17	−0.3667 (5)	−0.4031 (4)	−0.1208 (4)	0.0854 (13)
C18	−0.3218 (4)	−0.2903 (4)	−0.1321 (3)	0.0667 (10)
C19	−0.2134 (3)	−0.2269 (3)	−0.3407 (3)	0.0421 (8)
C20	−0.3161 (4)	−0.3369 (3)	−0.4150 (3)	0.0648 (10)
C21	−0.3005 (4)	−0.4525 (4)	−0.5138 (3)	0.0798 (12)
C22	−0.1794 (4)	−0.4611 (4)	−0.5408 (3)	0.0730 (11)
C23	−0.0757 (4)	−0.3544 (4)	−0.4685 (3)	0.0611 (10)
C24	−0.0914 (3)	−0.2386 (3)	−0.3692 (3)	0.0507 (9)
C25	−0.0476 (3)	0.0787 (3)	−0.2262 (3)	0.0421 (8)
C26	0.0761 (4)	0.1030 (4)	−0.1667 (3)	0.0651 (10)
C27	0.1831 (4)	0.1502 (4)	−0.2035 (4)	0.0834 (13)
C28	0.1680 (4)	0.1738 (4)	−0.3015 (4)	0.0803 (13)
C29	0.0466 (5)	0.1517 (4)	−0.3609 (4)	0.0767 (12)
C30	−0.0617 (4)	0.1043 (3)	−0.3241 (3)	0.0614 (10)
C31	−0.1472 (3)	0.2577 (3)	0.0081 (3)	0.0453 (8)
C32	−0.0549 (3)	0.2734 (3)	0.1014 (3)	0.0563 (9)
C33	0.0202 (4)	0.3996 (4)	0.1914 (3)	0.0661 (10)
C34	0.0026 (4)	0.5124 (4)	0.1888 (3)	0.0662 (11)
C35	−0.0883 (4)	0.4992 (3)	0.0962 (4)	0.0643 (10)
C36	−0.1624 (3)	0.3727 (3)	0.0059 (3)	0.0562 (9)

Table 2. *Selected geometric parameters (Å, °)*

N—C11	1.311 (5)	C6—C8	1.509 (4)
N—C10	1.321 (6)	C6—C5	1.555 (4)
O—C7	1.197 (3)	C5—C4	1.557 (4)
C7—C4	1.525 (4)	C4—C31	1.500 (4)
C7—C1	1.556 (4)	C4—C3	1.537 (4)
C1—C13	1.505 (4)	C3—C2	1.355 (4)
C1—C2	1.537 (4)	C3—C25	1.480 (4)
C1—C6	1.585 (4)	C2—C19	1.480 (4)
C13—C1—C2	118.6 (3)	C31—C4—C7	117.7 (2)
C13—C1—C7	113.4 (2)	C31—C4—C3	118.0 (3)
C2—C1—C7	96.5 (2)	C7—C4—C3	96.6 (2)
C13—C1—C6	119.2 (3)	C31—C4—C5	117.1 (3)
C2—C1—C6	108.9 (2)	C7—C4—C5	98.3 (2)
C7—C1—C6	95.4 (2)	C3—C4—C5	105.6 (2)
C3—C2—C19	128.4 (3)	C6—C5—C4	105.5 (2)
C3—C2—C1	108.7 (3)	C8—C6—C5	114.0 (3)
C19—C2—C1	122.9 (3)	C8—C6—C1	118.7 (2)
C2—C3—C25	128.9 (3)	C5—C6—C1	103.4 (2)
C2—C3—C4	108.9 (3)	O—C7—C1	128.6 (3)
C25—C3—C4	121.9 (3)	C4—C7—C1	99.1 (2)
O—C7—C4	132.3 (3)		

The structure was solved by direct methods and refined by anisotropic full-matrix least squares for all non-H atoms. H-atom positions were fixed during the refinement and assigned isotropic displacement parameters 1.2 × B_{eq} of the corresponding C atom.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *SHELXTL* (Sheldrick, 1994). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

KAW gratefully acknowledges support from the National Science Foundation (DMR-9414042).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1217). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Atwood, J. L., Davies, J. E. D. & MacNicol, D. D. (1984). Editors. *Inclusion Compounds*, Vols. 1–3. London: Academic Press.

Atwood, J. L., Davies, J. E. D. & MacNicol, D. D. (1991). Editors. *Inclusion Compounds*, Vol. 4. London: Oxford.

Huisgen, R., Grashey, R. & Sauer, J. (1964). *Chemistry of Alkenes*, pp. 878–928. New York: Interscience.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Sheldrick, G. M. (1994). *SHELXTL Software.* Version 5. Siemens Industrial Autom., Inc., Madison, Wisconsin, USA.

Siemens (1994). *XSCANS Software.* Version 2.0. Siemens Industrial Autom., Inc., Madison, Wisconsin, USA.

Wasserman, A. (1965). In *Diels–Alder Reactions.* New York: Elsevier.

Acta Cryst. (1996). C**52**, 3185–3188

Tetraphenylphosphonium Perchlorate

Jagadese J. Vittal and Philip A. W. Dean

Department of Chemistry, The University of Western Ontario, London, Ontario, Canada N6A 5B7. E-mail: jvittal@julian.uwo.ca

(*Received 1 July 1996; accepted 9 September 1996*)

Abstract

Crystals of the title compound, $C_{24}H_{20}P^+.ClO_4^-$, are composed of Ph_4P^+ cations and ClO_4^- anions separated by normal van der Waals distances. The P and Cl atoms occupy special positions of $\overline{4}$ (S_4) symmetry. In the cation, the P—C distance is 1.833 (4) Å and the C—P—C angles are 110.25 (13) and 107.9 (2)°. The perchlorate anion is disordered.