C2′ and C3′ in (*6a*) and (*6b*) strongly reduced the biological activity. So it seems that this X-ray analysis provides a structural description of the taxol-like active molecule.

References

BEGLEY, M. J., FRECKNALL, E. A. & PATTENDEN, G. (1984). *Acta Cryst.* **C40**, 1745–1747.

CHAUVIÈRE, G., GUÉNARD, D., PASCARD, C., PICOT, F., POTIER, P. & PRANGÉ, T. (1982). *Chem. Soc. Chem. Commun.* pp. 495–496.

COLIN, M., GUÉNARD, D., GUERITTE-VOEGELEIN, F. & POTIER, P. (1986). Rhone-Poulenc Santé Eur. Pat. Appl. EP 253,738 (Cl.C07D305/14), 20 January 1988; Fr. Appl. 86/10,400,17 July 1986. *Chem. Abstr.* (1988). **109**, No. 22762 *W*.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.

DELLA CASA DE MARCANO, D. P., HALSALL, T. G., CASTELLANO, E. & HODDER, J. R. (1970). *Chem. Commun.* pp. 1382–1383.

GRAF, E., KIRFEL, A., WOLF, G. J. & BREITMAIER, E. (1982). *Justus Liebigs Ann. Chem.* 376–381.

HO, T. I., LEE, G. H., PENG, S. M., YEH, M. K. & CHEN, F. C. (1987). *Acta Cryst.* **C43**, 1378–1380.

HO, T. I., LIN, Y. C., LEE, G. M., PENG, S. M., YEH, M. K. & CHEN, F. C. (1987). *Acta Cryst.* **C43**, 1380–1382.

RICHE, C. (1982). 7th European Crystallographic Meeting, Jerusalem, Abstr. 25.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination, Univ. of Cambridge, England.

SHIRO, M. & KOYAMA, M. (1971). *J. Chem. Soc. B*, pp. 1342–1346.

WANI, M. C., TAYLOR, H. L., WALL, M. E., COGGON, P. & MCPHAIL, A. T. (1971). *J. Am. Chem. Soc.* **93**, 2325–2327.

Acta Cryst. (1990). **C46**, 784–788

The Structure of Hexaphenylbenzene Anisole Clathrate, $2C_6(C_6H_5)_6.C_6H_5OCH_3$

BY E. M. LARSON,* R. B. VON DREELE,†‡ P. HANSON AND J. DEVENS GUST

Department of Chemistry, Arizona State University, Tempe, AZ 85287, USA

(*Received* 28 *November* 1988; *accepted* 20 *June* 1989)

Abstract. Hexaphenylbenzene–anisole (2/1), $2C_{42}H_{30}.C_7H_8O$, $M_r \simeq 1177.44$, monoclinic, Aa, a = 11·648 (6), b = 45·99 (2), c = 12·428 (4) Å, β = 99·82 (4)°, V = 6560·1 Å^3, Z = 4, D_m = 1·182, D_x = 1·192 g cm^{-3}, $\lambda(\text{Cu }K\alpha_1)$ = 1·54051 Å, μ = 4·87 cm^{-1}, $F(000)$ = 2488, T = 298 K, R = 9·2 and wR = 3·8% for 3616 unique observed reflections having $I_o > \sigma(I_o)$. The asymmetric unit consists of two hexaphenylbenzene molecules and one anisole molecule. The hexaphenylbenzene molecules adopt a propeller conformation with the phenyl rings twisted by an average of 75° from the molecular plane. The hexaphenylbenzene forms an unusual array, where the molecules are arranged in layers which alternate between body-centered and hexagonal packing. The anisole occupies one set of the pseudo-octahedral holes in this array.

Introduction. The study of clathrated and intercalated molecules forms a broad discipline which has been reviewed extensively (Davies, 1981; MacNicol, McKendrick & Wilson, 1978). The available structural data obtained from X-ray diffraction have also been reviewed (Andreetti, 1981). One common feature of these complexes is the formation of six-membered rings *via* hydrogen bonding between adjacent molecules. The six-membered rings serve as the ceiling and floor of the cavity, which encloses the guest molecule. This feature is important because the geometry and overall dimensions of the aforementioned rings closely resemble those of a substituted benzene. Therefore, hexasubstituted benzenes were proposed as host compounds in an effort to understand better the structures of clathrates (MacNicol *et al.*, 1978). Several of these complexes have been synthesized and their structures reported (Burns, Gilmore, Mallinson, MacNicol & Swanson, 1981; Freer, Gall & MacNicol, 1982; Freer, Gilmore, MacNicol & Swanson, 1980; Gilmore, MacNicol, Murphy & Russell, 1983). We now report the structure of one of these non hydrogen bonded clathrates, hexaphenyl–anisole, recently synthesized in our laboratory.

Experimental. Hexaphenylbenzene was prepared using a modification of the published procedure (Fieser & Williams, 1975). It was sublimed under vacuum as a last purification step, and a saturated solution was prepared by dissolving 3 g in 100 ml of boiling anisole which had been distilled three times. After cooling to room temperature, the solution was filtered into Petri dishes containing 5–10 drops of anisole. After four weeks of slow evaporation, small colorless crystals formed. A crystal having dimensions 0·1 × 0·5 × 0·1 mm, after being cut, was chosen

---

* Present address: L-356, Lawrence-Livermore National Laboratory, Mail Box 808, Livermore, CA 94551, USA.

† Present address: Los Alamos National Laboratory, LANSCE, MS H805, Los Alamos, NM 87545, USA.

‡ Author to whom inquiries should be addressed.

0108-2701/90/050784-05$03.00

Table 1. *Atomic coordinates and calculated isotropic thermal parameters*

$U_{\rm iso} = (U_1U_2U_3)^{1/3}$ where U_1, U_2 and U_3 are the principal axes of the thermal ellipsoid.

	x	y	z	$U_{\rm iso}$(Å^2)
C(*A*1)	0·3564 (5)	0·1901 (1)	0·7612 (5)	0·040 (2)
C(*A*2)	0·3298 (5)	0·1611 (1)	0·7339 (5)	0·038 (2)
C(*A*3)	0·3188 (5)	0·1406 (1)	0·8131 (5)	0·032 (2)
C(*A*4)	0·3328 (5)	0·1491 (1)	0·9230 (5)	0·037 (2)
C(*A*5)	0·3541 (5)	0·1782 (1)	0·9502 (5)	0·035 (2)
C(*A*6)	0·3667 (5)	0·1987 (1)	0·8714 (5)	0·034 (2)
C(*B*1)	0·3314 (5)	0·1265 (1)	1·0109 (5)	0·033
C(*B*2)	0·2327 (6)	0·1120 (1)	1·0264 (5)	0·048
C(*B*3)	0·2381 (6)	0·0903 (1)	1·1035 (6)	0·051
C(*B*4)	0·3423 (8)	0·0825 (1)	1·1641 (6)	0·061
C(*B*5)	0·4411 (6)	0·0965 (1)	1·1489 (5)	0·060
C(*B*6)	0·4379 (6)	0·1183 (1)	1·0733 (5)	0·054
C(*C*1)	0·3618 (7)	0·1875 (1)	1·0676 (6)	0·050
C(*C*2)	0·4682 (6)	0·1909 (2)	1·1344 (6)	0·072
C(*C*3)	0·4701 (8)	0·1995 (2)	1·2441 (7)	0·085
C(*C*4)	0·3668 (10)	0·2027 (2)	1·2804 (6)	0·084
C(*C*5)	0·2649 (8)	0·2002 (2)	1·2180 (7)	0·083
C(*C*6)	0·2621 (7)	0·1922 (1)	1·1095 (6)	0·068
C(*D*1)	0·3888 (6)	0·2300 (1)	0·9001 (5)	0·039
C(*D*2)	0·3015 (6)	0·2488 (2)	0·9178 (5)	0·052
C(*D*3)	0·3215 (6)	0·2777 (1)	0·9376 (6)	0·061
C(*D*4)	0·4317 (7)	0·2886 (1)	0·9423 (5)	0·056
C(*D*5)	0·5236 (6)	0·2707 (1)	0·9268 (5)	0·054
C(*D*6)	0·4988 (6)	0·2419 (1)	0·9044 (5)	0·055
C(*E*1)	0·3852 (6)	0·2114 (1)	0·6782 (5)	0·036
C(*E*2)	0·3096 (5)	0·2330 (1)	0·6360 (5)	0·050
C(*E*3)	0·3414 (6)	0·2524 (1)	0·5631 (5)	0·057
C(*E*4)	0·4465 (7)	0·2513 (2)	0·5307 (5)	0·056
C(*E*5)	0·5247 (6)	0·2303 (2)	0·5721 (6)	0·064
C(*E*6)	0·4921 (6)	0·2097 (1)	0·6446 (5)	0·059
C(*F*1)	0·3098 (6)	0·1522 (1)	0·6163 (5)	0·036
C(*F*2)	0·2153 (6)	0·1623 (1)	0·5474 (5)	0·046
C(*F*3)	0·1879 (6)	0·1531 (1)	0·4376 (5)	0·061
C(*F*4)	0·2618 (7)	0·1340 (1)	0·4003 (5)	0·061
C(*F*5)	0·3585 (6)	0·1238 (1)	0·4685 (6)	0·056
C(*F*6)	0·3817 (5)	0·1328 (1)	0·5762 (5)	0·041
C(*G*1)	0·2842 (6)	0·1100 (1)	0·7778 (5)	0·034
C(*G*2)	0·3684 (6)	0·0883 (1)	0·7930 (5)	0·052
C(*G*3)	0·3378 (8)	0·0607 (2)	0·7498 (6)	0·070
C(*G*4)	0·2307 (9)	0·0557 (2)	0·6919 (6)	0·073
C(*G*5)	0·1481 (7)	0·0763 (2)	0·6813 (6)	0·062
C(*G*6)	0·1749 (6)	0·1039 (1)	0·7239 (5)	0·047
C(*H*1)	0·2851 (5)	0·4107 (1)	0·7276 (5)	0·037 (2)
C(*H*2)	0·2612 (5)	0·4314 (1)	0·6426 (5)	0·034 (2)
C(*H*3)	0·2204 (5)	0·4587 (1)	0·6655 (5)	0·034 (2)
C(*H*4)	0·2067 (5)	0·4663 (1)	0·7714 (5)	0·034 (2)
C(*H*5)	0·2310 (5)	0·4458 (1)	0·8556 (5)	0·035 (2)
C(*H*6)	0·2664 (5)	0·4178 (1)	0·8319 (5)	0·033 (2)
C(*I*1)	0·1768 (6)	0·4967 (1)	0·8006 (5)	0·036
C(*I*2)	0·2610 (6)	0·5142 (1)	0·8590 (5)	0·043
C(*I*3)	0·2354 (6)	0·5422 (1)	0·8900 (5)	0·050
C(*I*4)	0·1238 (7)	0·5532 (1)	0·8607 (5)	0·051
C(*I*5)	0·0410 (6)	0·5359 (1)	0·8043 (5)	0·052
C(*I*6)	0·0656 (6)	0·5081 (1)	0·7725 (5)	0·050
C(*J*1)	0·2155 (7)	0·4539 (1)	0·9674 (6)	0·050
C(*J*2)	0·1099 (7)	0·4579 (2)	0·9972 (7)	0·082
C(*J*3)	0·0966 (10)	0·4666 (2)	1·1041 (10)	0·114
C(*J*4)	0·1954 (13)	0·4720 (2)	1·1711 (7)	0·111
C(*J*5)	0·3006 (10)	0·4686 (2)	1·1485 (7)	0·096
C(*J*6)	0·3123 (7)	0·4596 (1)	1·0445 (7)	0·071
C(*K*1)	0·2799 (6)	0·3951 (1)	0·9194 (5)	0·040
C(*K*2)	0·3876 (6)	0·3858 (1)	0·9712 (5)	0·051
C(*K*3)	0·3961 (7)	0·3646 (1)	1·0512 (5)	0·064
C(*K*4)	0·2989 (8)	0·3517 (1)	1·0756 (5)	0·054
C(*K*5)	0·1918 (7)	0·3602 (2)	1·0254 (6)	0·076
C(*K*6)	0·1836 (6)	0·3823 (2)	0·9480 (6)	0·058
C(*L*1)	0·3311 (5)	0·3812 (1)	0·7072 (4)	0·034
C(*L*2)	0·2649 (5)	0·3564 (1)	0·7082 (5)	0·047
C(*L*3)	0·3108 (7)	0·3289 (1)	0·6934 (5)	0·053
C(*L*4)	0·4241 (7)	0·3267 (1)	0·6759 (5)	0·062
C(*L*5)	0·4903 (5)	0·3507 (2)	0·6772 (5)	0·050
C(*L*6)	0·4464 (6)	0·3780 (1)	0·6909 (5)	0·051
C(*M*1)	0·2822 (6)	0·4241 (1)	0·5312 (5)	0·037
C(*M*2)	0·1950 (6)	0·4146 (1)	0·4529 (6)	0·056
C(*M*3)	0·2164 (7)	0·4076 (2)	0·3498 (6)	0·070
C(*M*4)	0·3245 (8)	0·4105 (1)	0·3234 (6)	0·064
C(*M*5)	0·4128 (6)	0·4210 (1)	0·4006 (6)	0·062
C(*M*6)	0·3906 (6)	0·4280 (1)	0·5030 (5)	0·057
C(*N*1)	0·1867 (6)	0·4806 (1)	0·5748 (5)	0·039
C(*N*2)	0·0751 (6)	0·4796 (1)	0·5156 (5)	0·051
C(*N*3)	0·0430 (6)	0·4992 (2)	0·4304 (6)	0·064
C(*N*4)	0·1183 (7)	0·5199 (1)	0·4086 (6)	0·058
C(*N*5)	0·2311 (7)	0·5207 (1)	0·4675 (6)	0·060
C(*N*6)	0·2634 (6)	0·5002 (2)	0·5499 (5)	0·058
O(1)*	0·2086	0·3089 (1)	0·3608	0·075
C(1)	0·1708 (7)	0·3345 (2)	0·3992 (7)	0·099
C(2)	0·3168 (8)	0·3081 (2)	0·3349 (7)	0·056
C(3)	0·4035 (10)	0·3280 (2)	0·3699 (6)	0·072
C(4)	0·5104 (9)	0·3232 (3)	0·3361 (9)	0·068
C(5)	0·5262 (13)	0·3006 (3)	0·2727 (11)	0·088
C(6)	0·4370 (14)	0·2813 (2)	0·2402 (9)	0·092
C(7)	0·3345 (8)	0·2855 (2)	0·2717 (8)	0·067

* Unit-cell origin detemined by not refining x and z coordinates of this atom.

for X-ray diffraction and sealed in a capillary to avoid sublimation of the material. Weissenberg layer photographs gave initial lattice constants and showed the Laue group to be monoclinic, and A centered. The systematic extinctions were consistent with the space groups Aa and $A2/a$; subsequent solution and refinement of the structure confirmed the final choice as Aa. The least-squares refined cell dimensions based on 15 diffractometer-centered reflections ($5 < 2\theta < 23°$) are given in the *Abstract*. Intensities for 5045 reflections were collected by θ–2θ scans (Cu $K\alpha$, graphite monochromator, $2\theta \le 110 \pm 1°$ about $K\alpha_1$–$K\alpha_2$ doublet at $1°\ \text{min}^{-1}$, background counts for 1/4 of scan time at each end of scan) on a Syntex $P\bar{1}$ diffractometer. Preliminary data reduction and calculation of weights as previously described (Post, Von Dreele & Buseck, 1982) and sort and merge of these data yielded 3616 intensities with $I_o > \sigma(I_o)$. Variations in intensity of two monitor reflections were less than 1%. No absorption or extinction corrections were performed. Range of hkl: $-12 \le h \le 12$, $0 \le k \le 50$, $0 \le l \le 13$. 4125 reflections unique, $R_{\rm int} = 5{\cdot}6\%$.

Initial attempts to solve the structure using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) failed. Since the b axis of the monoclinic cell is approximately twice that of the original hexaphenylbenzene orthorhombic cell (Bart, 1968) while the a and c axes are similar to that cell, the published y positions of one molecule were divided by two and input with x and z unchanged to *RANTAN* (Yao Jia-Xing, 1981) as a positioned group. This yielded one solution; $R = 18{\cdot}33\%$; ABSFOM = 1·2274; PSIZERO = 1·4, which gave atom positions for one molecule and two-thirds of the second molecule.

The 68 atom positions were input to *CRYSTALS* (Watkin & Prout, 1985) and successive refinements and difference Fourier maps provided locations of all remaining atoms ($R \sim 25\%$). Atomic scattering factors were used for C, O and H (*International Tables for X-ray Crystallography*, 1974). The atom positions and isotropic temperature factors were refined by full-matrix least-squares minimization of

$\sum w(F_o - F_c)^2$ to give $R = 18{\cdot}2$ and $wR = 9{\cdot}3\%$. Introduction of anisotropic temperature factors for the anisole and the outer rings of the hexaphenylbenzene molecules necessitated the use of large-block least-

Table 2. *Bond distances* (Å) *and angles* (°) *within the rings*

Ring	1—2	2—3	3—4	4—5	5—6	6—1	Mean
A	1·399 (7)	1·385 (7)	1·403 (7)	1·395 (7)	1·386 (7)	1·411 (6)	1·396 (10)
B	1·370 (7)	1·379 (7)	1·362 (8)	1·359 (8)	1·369 (8)	1·397 (7)	1·373 (14)
C	1·368 (8)	1·392 (8)	1·308 (10)	1·362 (10)	1·417 (9)	1·378 (8)	1·371 (36)
D	1·381 (7)	1·366 (7)	1·369 (8)	1·389 (8)	1·376 (7)	1·386 (7)	1·378 (9)
E	1·371 (7)	1·368 (7)	1·352 (8)	1·368 (8)	1·404 (8)	1·381 (7)	1·374 (17)
F	1·356 (7)	1·411 (8)	1·366 (8)	1·370 (7)	1·383 (7)	1·373 (7)	1·376 (19)
G	1·363 (7)	1·389 (7)	1·342 (9)	1·350 (10)	1·401 (8)	1·388 (7)	1·372 (24)
H	1·389 (7)	1·414 (7)	1·389 (7)	1·396 (7)	1·403 (7)	1·399 (7)	1·398 (9)
I	1·376 (7)	1·389 (7)	1·385 (7)	1·350 (8)	1·382 (7)	1·385 (7)	1·378 (14)
J	1·357 (8)	1·422 (10)	1·324 (14)	1·313 (13)	1·386 (9)	1·374 (8)	1·363 (40)
K	1·366 (7)	1·391 (8)	1·353 (8)	1·357 (9)	1·383 (7)	1·377 (7)	1·371 (15)
L	1·377 (7)	1·400 (8)	1·377 (8)	1·347 (8)	1·376 (7)	1·400 (7)	1·380 (20)
M	1·354 (8)	1·384 (8)	1·361 (9)	1·369 (8)	1·378 (8)	1·379 (7)	1·371 (12)
N	1·380 (7)	1·391 (7)	1·353 (8)	1·391 (8)	1·396 (8)	1·341 (7)	1·375 (23)

Ring	1—2—3	2—3—4	3—4—5	4—5—6	5—6—1	6—1—2
A	121·4 (6)	119·5 (6)	119·4 (6)	121·2 (6)	119·6 (6)	118·8 (6)
B	120·6 (6)	120·4 (6)	119·6 (6)	121·1 (7)	119·7 (6)	118·6 (6)
C	122·0 (8)	117·8 (8)	123·9 (8)	118·6 (7)	118·5 (7)	119·1 (7)
D	122·1 (7)	119·5 (7)	121·1 (6)	117·3 (6)	123·3 (7)	116·6 (6)
E	119·9 (6)	122·3 (7)	119·5 (7)	118·9 (7)	120·8 (7)	118·6 (6)
F	122·2 (6)	117·9 (7)	120·5 (6)	120·1 (6)	120·1 (6)	118·2 (6)
G	120·4 (7)	119·9 (7)	120·8 (7)	118·1 (7)	118·1 (7)	119·8 (6)
H	119·1 (6)	121·2 (6)	119·4 (6)	119·6 (6)	120·8 (6)	119·8 (6)
I	121·4 (6)	120·1 (6)	118·5 (6)	121·9 (6)	120·5 (6)	117·6 (6)
J	122·8 (9)	114·8 (9)	125·8 (10)	118·7 (9)	120·5 (8)	117·3 (7)
K	122·1 (7)	118·6 (7)	120·6 (7)	120·5 (7)	120·2 (7)	117·9 (6)
L	121·3 (6)	119·1 (6)	119·9 (9)	121·8 (7)	119·9 (6)	117·9 (6)
M	120·6 (7)	121·4 (7)	118·7 (7)	119·5 (7)	121·9 (7)	117·8 (6)
N	119·3 (7)	120·6 (7)	120·0 (7)	118·7 (7)	121·1 (7)	120·2 (7)

Table 3. *Bond distances* (Å) *and angles* (°) *between the rings*

		Twist			Twist
C(*A*1)C(*E*1)	1·500 (7)	76·0 (6)	C(*H*1)C(*L*1)	1·496 (7)	71·3 (6)
C(*A*2)C(*F*1)	1·498 (7)	66·3 (8)	C(*H*2)C(*M*1)	1·484 (7)	85·4 (9)
C(*A*3)C(*G*1)	1·506 (7)	74·0 (7)	C(*H*3)C(*N*1)	1·514 (7)	87·4 (7)
C(*A*4)C(*B*1)	1·511 (7)	72·4 (7)	C(*H*4)C(*I*1)	1·500 (7)	72·6 (6)
C(*A*5)C(*C*1)	1·507 (7)	81·5 (6)	C(*H*5)C(*J*1)	1·480 (7)	74·2 (7)
C(*A*6)C(*D*1)	1·492 (7)	77·8 (7)	C(*H*6)C(*K*1)	1·497 (7)	75·8 (7)

C(*A*1)C(*E*1)C(*E*2)	122·1 (6)	C(*H*1)C(*L*1)C(*L*2)	122·0 (6)
C(*A*1)C(*E*1)C(*E*6)	119·3 (6)	C(*H*1)C(*H*1)C(*L*6)	120·0 (6)
C(*A*2)C(*F*1)C(*F*2)	119·8 (6)	C(*H*2)C(*M*1)C(*M*2)	121·4 (6)
C(*A*2)C(*F*1)C(*F*6)	121·9 (6)	C(*H*2)C(*M*1)C(*M*6)	120·8 (6)
C(*A*3)C(G1)C(G6)	121·1 (6)	C(*H*3)C(*N*1)C(*N*6)	121·4 (6)
C(*A*3)C(G1)C(G2)	118·9 (6)	C(*H*3)C(*N*1)C(*N*2)	118·4 (6)
C(*A*4)C(*B*1)C(*B*2)	123·2 (6)	C(*H*4)C(*I*1)C(*I*2)	119·9 (6)
C(*A*4)C(*B*1)C(*B*6)	118·0 (6)	C(*H*4)C(*I*1)C(*I*6)	122·4 (6)
C(*A*6)C(*D*1)C(*D*2)	122·7 (6)	C(*H*5)C(*J*1)C(*J*2)	123·5 (6)
C(*A*6)C(*D*1)C(*D*6)	120·7 (6)	C(*H*5)C(*J*1)C(*J*6)	119·1 (7)
C(*A*5)C(*C*1)C(*C*6)	120·0 (7)	C(*H*6)C(*K*1)C(*K*6)	120·0 (6)
C(*A*5)C(*C*1)C(*C*2)	120·9 (7)	C(*H*6)C(*K*1)C(*K*2)	122·1 (6)
C(*B*1)C(*A*4)C(*A*5)	120·6 (6)	C(*I*1)C(*H*4)C(*H*3)	122·1 (6)
C(*B*1)C(*A*4)C(*A*3)	119·9 (6)	C(*I*1)C(*H*4)C(*H*5)	118·3 (5)
C(*C*1)C(*A*5)C(*A*6)	119·8 (6)	C(*J*1)C(*H*5)C(*H*6)	121·3 (6)
C(*C*1)C(*A*5)C(*A*4)	119·0 (6)	C(*J*1)C(*H*5)C(*H*4)	119·2 (6)
C(*D*1)C(*A*6)C(*A*5)	121·4 (6)	C(*K*1)C(*H*6)C(*H*1)	120·0 (6)
C(*D*1)C(*A*6)C(*A*1)	119·0 (6)	(C*K*1)C(*H*6)C(*H*5)	119·2 (6)
C(*E*1)C(*A*1)C(*A*2)	121·4 (6)	C(*L*1)C(*H*1)C(*H*6)	119·3 (6)
C(*E*1)C(*A*1)C(*A*6)	119·5 (6)	C(*L*1)C(*H*1)C(*H*2)	120·9 (6)
C(*F*1)C(*A*2)C(*A*3)	119·3 (6)	C(*M*1)C(*H*2)C(*H*3)	120·8 (6)
C(*F*1)C(*A*2)C(*A*1)	119·3 (6)	C(*M*1)C(*H*2)C(*H*1)	120·1 (5)
C(*G*1)C(*A*3)C(*A*2)	118·8 (6)	C(*N*1)C(*H*3)C(*H*2)	120·3 (6)
C(*G*1)C(*A*3)C(*A*4)	121·6 (6)	C(*N*1)C(*H*3)C(*H*4)	118·4 (5)

Table 4. *Bond distances* (Å) *and angles* (°) *for anisole*

O(1)C(2)	1·353 (8)	C(1)O(1)C(2)	118·1 (7)
O(1)C(1)	1·373 (8)	O(1)C(2)C(7)	113·7 (9)
C(2)C(3)	1·376 (9)	C(3)C(2)C(7)	121·4 (9)
C(3)C(4)	1·397 (11)	C(3)C(2)O(1)	124·9 (10)
C(4)C(5)	1·333 (13)	C(4)C(3)C(2)	116·4 (9)
C(5)C(6)	1·374 (14)	C(3)C(4)C(5)	121·4 (13)
C(6)C(7)	1·334 (12)	C(6)C(5)C(4)	120·2 (15)
C(2)C(7)	1·340 (9)	C(5)C(6)C(7)	119·3 (14)
		C(2)C(7)C(6)	121·3 (10)

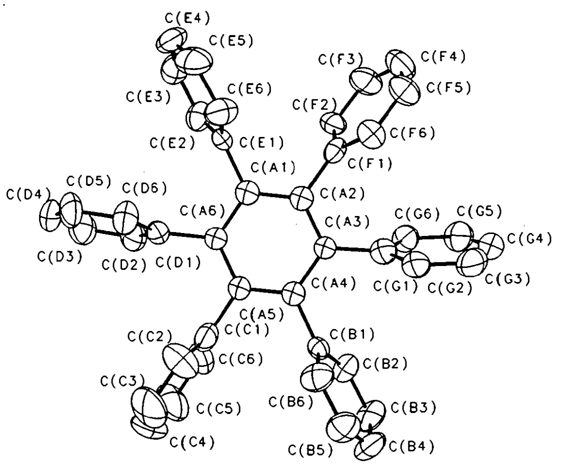

Fig. 1. Perspective representation of hexaphenylbenzene molecule C(*A*1)–C(*G*6). Ellipsoids represent 50% thermal motion surfaces. H atoms are omitted for clarity.

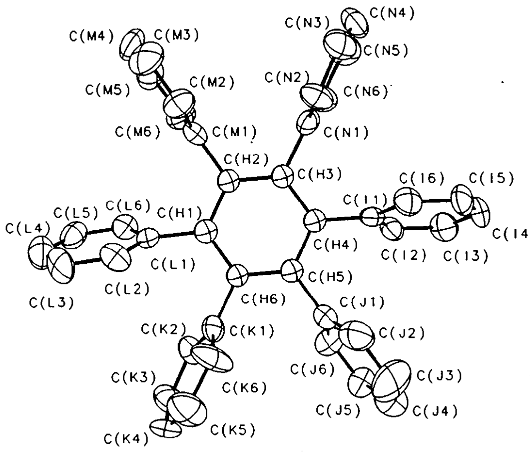

Fig. 2. Perspective representation of hexaphenylbenzene molecule C(*H*1)–C(*N*6). Ellipsoids represent 50% thermal motion surfaces. H atoms are omitted for clarity.

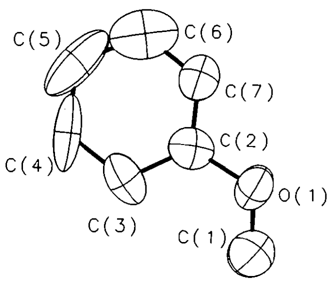

Fig. 3. Perspective representation of anisole molecule. Ellipsoids represent 50% thermal motion surfaces. H atoms are omitted for clarity.

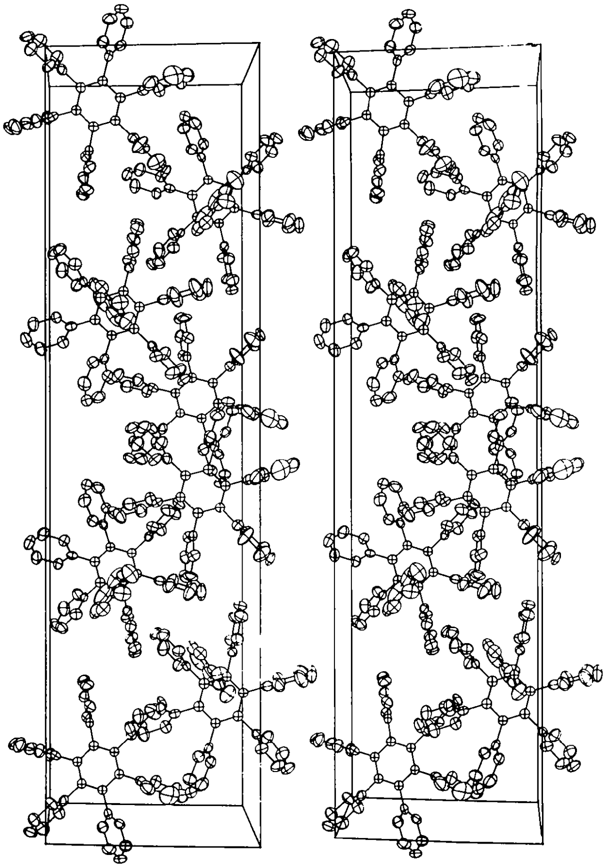

Fig. 4. Stereographic representation of hexaphenylbenzene anisole clathrate. The vertical axis is *b*, the horizontal axis is *c* and the *a* axis is into the paper. Hexaphenylbenzene molecule C(*H*1)–C(*N*6) is in the lower left corner of the cell. H atoms are omitted for clarity.

squares refinement (767 parameters in 6 blocks). H-atom positions were calculated but not refined. The resulting $R = 9{\cdot}2\%$ and $wR = 3{\cdot}8\%$ at convergence; $S = 2{\cdot}38$. Max. $\Delta/\sigma = 0{\cdot}20$; max., min. height in final difference Fourier map 0·2, $-0{\cdot}2$ e Å^{-3}.

Discussion. Atomic coordinates with their standard deviations and 'equivalent' isotropic thermal parameters are listed in Table 1.* Bond distances and angles within the rings are given in Table 2, while the distances and angles between the rings are listed in Table 3. The average aromatic C—C bond length in the central benzene rings is 1·397 (10) Å and the mean C—C distance which connects the rings is 1·499 (10) Å. The outer phenyl ring average C—C distance is 1·373 (21) Å; there is a slight deviation from perfect hexagonal symmetry with average C(1)—C(2), C(2)—C(3) and C(3)—C(4) distances of 1·374 (14), 1·389 (15) and 1·357 (21) Å, respectively. This deviation probably arises from the torsional libration of these rings about the inter-ring bond. The average intra-ring C—C—C angle for all 14 rings is 120·0 (18)°. Torsion angles for the twist of the phenyl rings from the plane of the central benzene ranged from 66 to 87° giving each molecule a propeller conformation. Distances and angles for the anisole molecule are presented in Table 4. Figs. 1–3 show the thermal motion of the atoms in the three

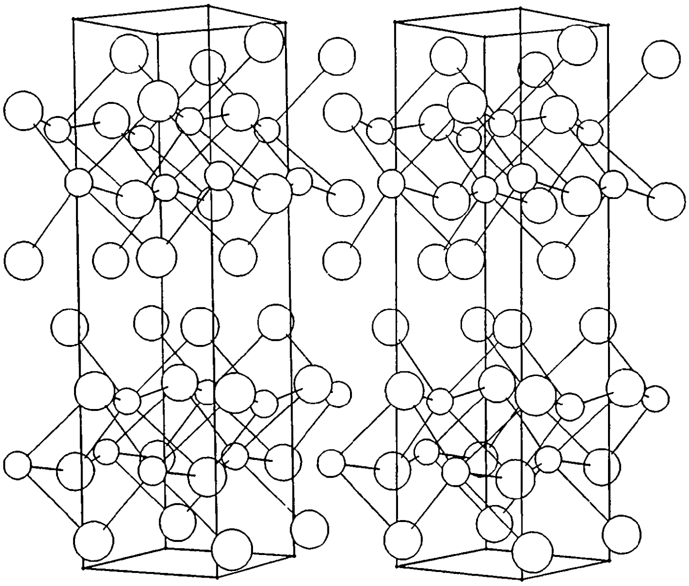

Fig. 5. Stereographic representation of the molecular packing in hexaphenylbenzene anisole clathrate. The centers of the hexaphenylbenzene molecules are the large circles and the centers of the anisole molecules are the small circles. Lines are drawn from the anisole centers to the nearest six hexaphenylbenzene molecules. The *b* axis is vertical, the *c* axis is horizontal and the *a* axis is oblique into the paper.

---

* 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 52360 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

molecules. Fig. 4 presents a stereodiagram of the packing arrangement of the molecules in the unit cell and Fig. 5 is a simplification of the packing diagram where only the molecular centers are shown. All hexaphenylbenzene molecules in this structure are positioned with their molecular sixfold axes roughly parallel to the *a* axis. The hexagonal packing near $y = 0$ and $y = 1/2$ corresponds to the packing in the hexaphenylbenzene structure (Bart, 1968). These regions are separated by body-centered packing layers containing anisole molecules in the 'octahedral holes'. Two hexaphenylbenzene molecules from adjacent unit cells along *a* serve to form the top and bottom of a cavity with the anisole perpendicular to the central C_6 rings, and four other hexaphenylbenzene molecules are the sides of the hole. Thus, the anisole is surrounded by six individual host molecules arranged in a pseudo-octahedron (Fig. 5), unlike the other reported structures for hexa-host compounds. In most of the other known clathrate structures, the solvated molecules are enclosed within a cage formed by just two host molecules. While some of the previous studies have shown some disorder in the solvated molecule, none is observed in the case of hexaphenylbenzene anisole. A refinement of the occupancy of anisole produced a value of 0·992 (6), thus this clathrate is stoichiometric. A search of the distances yielded no intermolecular or intramolecular contacts shorter than the sum of the van der Waals radii for the atoms.

References

ANDREETTI, G. D. (1981). *J. Mol. Struct.* **75**, 129–138.

BART, J. C. J. (1968). *Acta Cryst.* **B24**, 1277–1287.

BURNS, K., GILMORE, C. J., MALLINSON, P. R., MACNICOL, D. D. & SWANSON, S. (1981). *J. Chem. Res. Synop.* **2**, 30–31.

DAVIES, J. E. D. (1981). *J. Mol. Struct.* **75**, 1–12.

FIESER, L. F. & WILLIAMS, K. L. (1975). *Organic Experiments*, 3rd ed., p. 294. Lexington, MA: Heath.

FREER, A. A., GALL, J. H. & MACNICOL, D. D. (1982). *J. Chem. Soc. Chem. Commun.* **12**, 674–676.

FREER, A. A., GILMORE, C. J., MACNICOL, D. D. & SWANSON, S. (1980). *Tetrahedron Lett.* **21**, 205–208.

GILMORE, C. J., MACNICOL, D. D., MURPHY, A. & RUSSELL, M. A. (1983). *Tetrahedron Lett.* **24**, 3269–3272.

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

MACNICOL, D. D., MCKENDRICK, J. J. & WILSON, D. R. (1978). *Chem. Soc. Rev.* **7**, 65–87.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

POST, J. E., VON DREELE, R. B. & BUSECK, P. R. (1982). *Acta Cryst.* **B38**, 1056–1065.

WATKIN, D. & PROUT, K. (1985). Private communication.

YAO JIA-XING (1981). *Acta Cryst.* **A37**, 642–644.

Acta Cryst. (1990). C**46**, 788–791

Structure of 2-Amino-6-chloromethyl-3-cyano-5-ethoxycarbonyl-4-(2-furyl)-4*H*-pyran

BY J. LOKAJ

Department of Microanalytical Chemistry, Faculty of Chemical Technology, Slovak Technical University, Radlinskeho 9, *CS*-81237 *Bratislava, Czechoslovakia*

V. KETTMANN AND F. PAVELČÍK

Department of Analytical Chemistry, Faculty of Pharmacy, Komensky University, Odbojárov 10, *CS*-83232 *Bratislava, Czechoslovakia*

AND D. ILAVSKÝ AND Š. MARCHALÍN

Department of Organic Chemistry, Faculty of Chemical Technology, Slovak Technical University, Radlinskeho 9, *CS*-81237 *Bratislava, Czechoslovakia*

(*Received* 2 *June* 1989; *accepted* 14 *August* 1989)

Abstract. Ethyl 6-amino-2-chloromethyl-5-cyano-4-(2-furyl)-4*H*-pyran-3-carboxylate, $C_{14}H_{13}ClN_2O_4$, $M_r = 308·7$, triclinic, $P\bar{1}$, $a = 11·356 (6)$, $b = 9·066 (4)$, $c = 8·390 (3)$ Å, $\alpha = 105·55 (3)$, $\beta = 104·89 (4)$, $\gamma = 110·09 (4)$°, $V = 721·1$ Å^3, $Z = 2$, $D_x = 1·421$, $D_m = 1·42$ Mg m^{-3}. $\lambda(\text{Cu } K\alpha) = 1·54178$ Å, $\mu = 1·18$ mm^{-1}, $F(000) = 320$, $T = 293$ K, final $R = 0·056$ for 1631 unique observed reflections. The molecular structure is analyzed in detail and results are compared with those of three analogues reported earlier with variations in ring substitutions, as well as with results of closely related 1,4-dihydropyridines.

0108-2701/90/050788-04$03.00