International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
LARSON, A. C. (1967). *Acta Cryst.* **23**, 664–665.
MIAO, F. M. & PROUT, K. (1981). Private communication.
PILOTY, O. & STEINBOCK, H. (1902). *Chem. Ber.* **35**, 3101–3117.
PROUT, K. & MIAO, F. M. (1982*a*). *Acta Cryst.* B**38**, 685–687.
PROUT, K. & MIAO, F. M. (1982*b*). *Acta Cryst.* B**38**, 687–689.
ROLLETT, J. S. (1969). *Crystallographic Computing*, edited by F. R. AHMED, pp. 169–172. Copenhagen: Munksgaard.
TALBERG, H. J. (1978). *Acta Chem. Scand. Ser. A*, **32**, 401–405.
TURNER, P. H. & COX, A. P. (1976). *Chem. Phys. Lett.* **39**, 585–587.
TURNER, P. H. & COX, A. P. (1978). *J. Chem. Soc. Faraday Trans.* 2, **74**, 533–559.
WASER, J. (1963). *Acta Cryst.* **16**, 1091–1095.

Acta Cryst. (1982). B**38**, 3155–3157

(*E*)-1-(2-Bromo-4,5-dimethoxyphenyl)-2-(3,4-dimethoxyphenyl)ethylene

BY J. M. ARRIETA,* E. LETE AND E. DOMÍNGUEZ

Departamento de Química, Facultad de Ciencias, Universidad del País Vasco, Apartado 644, *Bilbao, Spain*

G. GERMAIN AND J. P. DECLERCQ

Laboratoire de Chimie-Physique et de Cristallographie, Université de Louvain, 1 *Place Louis Pasteur, B*1348, *Louvain-la-Neuve, Belgium*

AND J. M. AMIGÓ†

Departamento de Cristalografía y Mineralogía, Facultad de Ciencias, Universidad del País Vasco, Apartado 644, *Bilbao, Spain*

(*Received* 22 *January* 1982; *accepted* 29 *June* 1982)

Abstract. $C_{18}H_{19}BrO_4$, M_r = 379·26, monoclinic, $C2/c$, a = 28·65 (3), b = 9·340 (5), c = 12·795 (9) Å, β = 95·05 (7)°, V = 3410 (4) Å^3, Z = 8, D_x = 1·48 Mg m^{-3}, $F(000)$ = 1552, Mo $K\alpha$ radiation (λ = 0·71069 Å), μ(Mo $K\alpha$) = 2·58 mm^{-1}. Final R = 0·047 for 1384 observed reflections. The molecule is in the *trans* form and the bromobenzene ring shows a slight distortion.

Introduction. In the course of studies directed towards the synthesis of 3-arylisoquinolines by the Bischler–Napieralski reaction (Fodor & Nagubandi, 1980), we have obtained several symmetrical and asymmetrical stilbenes (Lete, 1981). The crystal structure determination of the title compound was undertaken to find out the correct substitution pattern, which could not be unambiguously inferred from chemical or spectroscopic evidence.

Colourless plate-like crystals were grown from methanol. Cell parameters for use in the intensity data collection were calculated by a least-squares analysis of the angular settings of 15 reflections. Intensities of 2525 independent reflections with $2\theta < 47°$ were collected from a crystal with dimensions 0·4 × 0·2 × 0·04 mm on a Syntex $P2_1$ diffractometer by an ω scan with graphite-monochromatized Mo $K\alpha$ radiation. The intensity of one standard reflection measured every 50 reflections showed no significant variation. 1384 reflections with $I > 2{\cdot}5\sigma(I)$ were considered as observed and included in the refinement. Corrections were made for the Lorentz and polarization effects, but not for absorption.

The structure was solved by Patterson and Fourier methods. Full-matrix least-squares refinement with *SHELX* (Sheldrick, 1976) was completed with anisotropic thermal parameters for non-hydrogen atoms. The H atoms were included at the final stage of the refinement in calculated positions with an overall isotropic temperature factor. The weighting scheme was $w = K/[\sigma^2(F) + 0{\cdot}0007(F)^2]$. The final R was 0·047. Table 1‡ gives the final atomic coordinates with

---

* Correspondence should be addressed to this author.

† Present address: Facultad de Quimicas, Universidad de Valencia, Burjasot, Valencia, Spain.

‡ Lists of structure factors, anisotropic thermal parameters and H atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38029 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0567-7408/82/123155-03$01.00 © 1982 International Union of Crystallography

Table 1. *Atomic coordinates* ($\times 10^4$; *for* Br $\times 10^5$) *and* B_{eq} (Å^2)

	x	y	z	B_{eq}
Br(1)	77204 (5)	22250 (10)	23740 (10)	4·74
C(2)	7880 (2)	3923 (8)	562 (6)	2·96
C(3)	8079 (2)	2984 (8)	1307 (5)	2·99
C(4)	8543 (2)	2515 (8)	1346 (5)	2·98
C(5)	8826 (2)	3017 (8)	611 (5)	2·91
C(6)	8643 (2)	3929 (8)	−172 (6)	3·00
C(7)	8185 (3)	4354 (8)	−189 (6)	3·35
C(8)	7406 (2)	4419 (8)	558 (6)	3·28
C(9)	7231 (2)	5469 (8)	−37 (6)	3·36
C(10)	6732 (3)	6016 (8)	−106 (6)	3·43
C(11)	6412 (2)	5448 (8)	540 (6)	3·22
C(12)	5960 (3)	5966 (9)	495 (6)	3·42
C(13)	5823 (2)	7083 (9)	−212 (6)	3·59
C(14)	6141 (2)	7640 (9)	−853 (6)	3·70
C(15)	6591 (2)	7107 (9)	−785 (6)	4·07
O(16)	9287 (2)	2688 (6)	582 (4)	4·01
C(17)	9499 (3)	1706 (9)	1352 (6)	4·72
O(18)	8951 (2)	4336 (6)	−870 (4)	4·37
C(19)	8783 (3)	5268 (9)	−1697 (6)	4·58
O(20)	5616 (2)	5494 (6)	1095 (4)	4·88
C(21)	5702 (3)	4230 (10)	1685 (7)	5·35
O(22)	5364 (2)	7518 (6)	−201 (4)	4·68
C(23)	5212 (3)	8637 (9)	−905 (7)	4·98

Table 2. *Torsion angles* (°) ($\sigma < 1·4°$)

C(2)–C(8)–C(9)–C(10)	180·0	C(7)–C(6)–O(18)–C(19)	0·0
Br(1)–C(3)–C(4)–C(5)	180·0	C(4)–C(5)–O(16)–C(17)	2·1
C(8)–C(2)–C(3)–Br(1)	2·0	C(6)–C(5)–O(16)–C(17)	−178·0
C(7)–C(2)–C(3)–Br(1)	−178·2	C(14)–C(13)–O(22)–C(23)	0·0
C(7)–C(2)–C(8)–C(9)	−10·6	C(12)–C(13)–O(22)–C(23)	180·0
C(8)–C(9)–C(10)–C(15)	−178·5	C(11)–C(12)–O(20)–C(21)	−10·9
C(3)–C(2)–C(8)–C(9)	169·2	C(13)–C(12)–O(20)–C(21)	169·5
C(8)–C(9)–C(10)–C(11)	3·6	C(9)–C(10)–C(15)–C(14)	−179·0
C(5)–C(6)–O(18)–C(19)	180·0	C(8)–C(2)–C(3)–C(4)	178·2

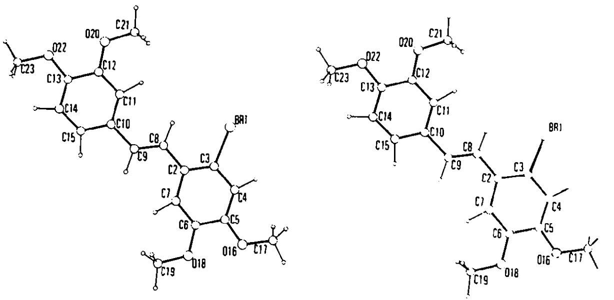

Fig. 1. Stereoscopic view of the molecule with the atom numbering (Motherwell & Clegg, 1978).

their B_{eq} values (Willis & Pryor, 1975). Selected torsion angles are given in Table 2. Fig. 1 shows a stereoscopic view of the molecule and the atom numbering used.

Discussion. Figs. 2 and 3 show bond lengths and angles. The introduction of a Br atom on the benzene ring causes an appreciable distortion of this ring. The ethylenic double-bond length C(8)–C(9) [1·313 (9) Å] is basically the same as the longest values found earlier in stilbene molecules, 1·318 Å (Finder, Newton & Allinger, 1974) and 1·313 Å (Bernstein, 1975). However, one exocyclic single-bond length, C(2)–C(8) [1·436 (9) Å], is shorter, and the other, C(9)–C(10) [1·516 (9) Å], longer than the distances reported in symmetrical *trans*-stilbenes, 1·471 and 1·484 Å (Bernstein, 1975). This is accompanied by a decrease of the angle C(9)–C(10)–C(11), which is 2·9 (1·4)° less than the corresponding angle C(8)–C(2)–C(7). Fig. 3 shows the irregularity of the bromobenzene ring angles, the angle C(3)–C(2)–C(7) being only 114·1 (6)°.

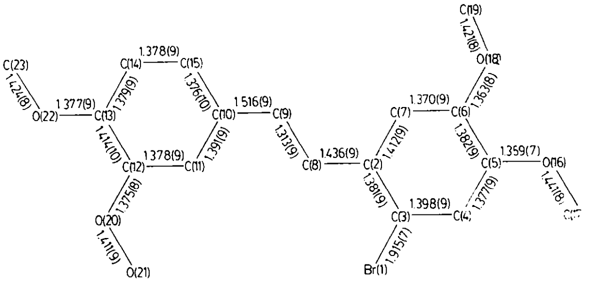

Fig. 2. Bond lengths (Å).

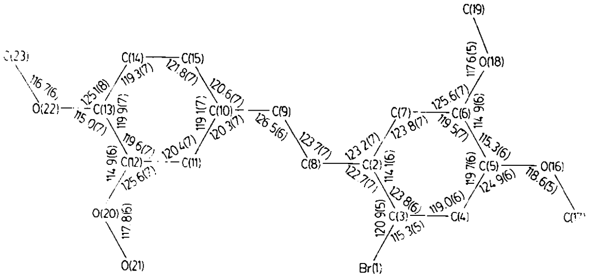

Fig. 3. Bond angles (°).

Both benzene rings are planar within experimental error [largest deviations, atom to mean plane, are: bromobenzene ring: C(5) 0·015 (11) Å; other ring: C(15) −0·05 (3) Å]. The entire molecule is approximately planar with a dihedral angle of 9·1 (1·5)° between the two benzene planes. This can also be seen from the torsion angles in Table 2.

One of us (JMA) is grateful for the financial assistance provided by MUI (Ministerio de Universidades e Investigación).

References

Bernstein, J. (1975). *Acta Cryst.* B**31**, 1268–1271.

Finder, C. J., Newton, M. G. & Allinger, N. L. (1974). *Acta Cryst.* B**30**, 411–415.

Fodor, G. & Nagubandi, S. (1980). *Tetrahedron Lett.* **36**, 1279.

Lete, E. (1981). Doctoral Thesis. Univ. del País Vasco, Bilbao, Spain.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO*. A program for plotting molecular and crystal structures. Univ. of Cambridge, England.

SHELDRICK, G. M. (1976). *SHELX* 76. A computer program for crystal structure determination. Univ. of Cambridge, England.

WILLIS, B. T. M. & PRYOR, A. W. (1975). *Thermal Vibrations in Crystallography*. pp. 101–102. Cambridge Univ. Press.

Acta Cryst. (1982). **B38**, 3157–3159

A 1,4,7,10,13,16-Hexaoxacyclooctadecane(18-crown-6)–4,4′-Biphenyldiol Dihydrate Ternary Complex

BY DAVID A. GROSSIE AND WILLIAM H. WATSON

FASTBIOS Laboratory, Department of Chemistry, Texas Christian University, Fort Worth, Texas 76129, *USA*

AND F. VÖGTLE AND W. M. MÜLLER

Institut für Organische Chemie und Biochemie der Universität Bonn, Gerhard-Domagk-Strasse 1, *D*-5000 *Bonn, Federal Republic of Germany*

(*Received* 8 *March* 1982; *accepted* 7 *July* 1982)

Abstract. $C_{12}H_{24}O_6.C_{12}H_{10}O_2.2H_2O$, $M_r = 486{\cdot}6$, monoclinic, $P2_1/c$, $a = 9{\cdot}264\,(1)$, $b = 11{\cdot}007\,(2)$, $c = 14{\cdot}165\,(2)$ Å, $\beta = 109{\cdot}10\,(1)°$, $U = 1364{\cdot}8\,(4)$ Å^3, $Z = 2$, $D_c = 1{\cdot}18$ Mg m^{-3}, μ(Cu $K\alpha$) = 0·844 mm^{-1}. Full-matrix least-squares refinement (calculated and observed H-atom positions were included in the structure-factor calculations but the parameters were not refined) based on 1138 observed reflections led to a final R of 0·073 and R_w of 0·035. The 18-crown-6 ether sits on a center of symmetry. The six O atoms are alternately 0·23 (2) Å above and below the mean plane and form an approximate hexagon with averaged sides of 2·83 (1) Å. The two water molecules are each hydrogen bonded to two O atoms of the crown ether. The planar 4,4′-biphenyldiol molecules sit on centers of symmetry and are hydrogen-bonded to the water molecules. The 4,4′-biphenyldiol molecules bridge two crown ethers *via* hydrogen bonds to water molecules.

Introduction. The weak interactions between crown ethers and neutral organic molecules are of chemical and biological interest. The mediation of these interactions by water molecules is of particular interest because of the importance of water in living organisms. The study of such interactions may lead to a better understanding of secondary interactions that occur at biological receptor sites. The change in the hydrophobic or hydrophilic nature of a site and the imposed stereospecificity are areas of interest. The specific role of water as an integral component of molecular recognition has not been emphasized. Although a number of structures of 18-crown-6 ethers with charged species have been described, only the neutral complexes with dimethyl acetylenedicarboxylate (1:1) (Goldberg, 1975), dimethyl sulfone (1:1) (Weber & Vögtle, 1980), urea (1:5) (Harkema, Van Hummel, Daasvatn & Reinhoudt, 1981), benzenesulfonamide (1:2) (Knochel, Kopf, Oehler & Rudolph, 1978) and phenyl carbamate (1:2) (Galloy, Watson, Vögtle & Müller, 1982) have been reported.

Recently the structures of two ternary complexes composed of a cryptand, a salt and a small neutral molecule have been reported (de Boer, Reinhoudt, Uiterwijk & Harkema, 1982; Hilgenfeld & Saenger, 1981). The present structure and a ternary complex of a cryptand, hydroquinone and water (Vögtle, Puff, Friedricks & Müller, 1982) are the first comprehensive reports on neutral ternary complexes involving cryptands.

A crystal of dimensions 0·33 × 0·17 × 0·17 mm was selected for all X-ray measurements. Intensity data were collected on a Syntex $P2_1$ diffractometer system by the θ:2θ scanning technique using a variable scan speed, Cu $K\alpha$ radiation ($\lambda = 1{\cdot}54178$ Å) and a graphite monochromator. Room-temperature lattice parameters were refined by a least-squares procedure utilizing 13 reflections whose angles were measured by a centering routine associated with the diffractometer. A periodically monitored reflection showed no significant change in intensity. Of the 2113 independent reflections measured ($2\theta < 120°$) 1239 had intensities greater than $3\sigma(I)$. Lorentz and polarization corrections were applied, but no absorption corrections were made.

An E map phased by *MULTAN* 78 (Main, Lessinger, Woolfson, Germain & Declercq, 1978) revealed the positions of all non-H atoms. After several cycles of refinement H-atom positions were calculated and compared with those obtained from a difference

0567-7408/82/123157-03$01.00 © 1982 International Union of Crystallography