C(1)	0.26630 (21)	0.83009 (17)	0.97825 (16)	0.0216 (7)
C(2)	0.33520 (21)	0.93646 (19)	0.87369 (16)	0.0222 (7)
C(3)	0.31755 (22)	1.08584 (19)	0.88933 (16)	0.0248 (8)
C(4)	0.23836 (23)	1.12924 (19)	1.00489 (18)	0.0262 (8)
C(5)	0.17739 (22)	1.01860 (19)	1.10861 (16)	0.0243 (7)
C(6)	0.18936 (21)	0.86878 (19)	1.09728 (16)	0.0220 (7)
C(7)	0.43206 (22)	0.89243 (20)	0.74979 (16)	0.0252 (8)
C(8)	0.2195 (3)	1.29339 (23)	1.01610 (21)	0.0374 (10)
C(9)	0.11680 (22)	0.75239 (19)	1.21043 (16)	0.0243 (7)
HO(2)	0.166 (4)	1.080 (3)	0.527 (3)	0.065 (9)
HO(3)	0.407 (3)	0.788 (3)	0.456 (3)	0.039 (6)
HO(4)	0.371 (4)	0.553 (3)	1.125 (3)	0.051 (8)
HO(5)	0.226 (5)	0.479 (4)	1.515 (4)	0.090 (12)
HO(7)	0.251 (4)	0.689 (3)	0.891 (3)	0.046 (7)

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

P(1)—O(1)	1.5015 (14)	C(1)—C(2)	1.396 (2)
P(1)—O(2)	1.5450 (15)	C(1)—C(6)	1.397 (2)
P(1)—O(3)	1.5496 (14)	C(2)—C(3)	1.396 (2)
P(1)—C(7)	1.7857 (17)	C(2)—C(7)	1.505 (2)
P(2)—O(4)	1.5688 (13)	C(3)—C(4)	1.387 (2)
P(2)—O(5)	1.5395 (14)	C(4)—C(5)	1.389 (3)
P(2)—O(6)	1.4981 (13)	C(4)—C(8)	1.514 (3)
P(2)—C(9)	1.7893 (17)	C(5)—C(6)	1.392 (2)
O(7)—C(1)	1.387 (2)	C(6)—C(9)	1.511 (2)
O(1)—P(1)—O(2)	108.48 (8)	C(3)—C(4)—C(5)	118.24 (15)
O(1)—P(1)—O(3)	114.68 (8)	C(4)—C(5)—C(6)	121.71 (15)
O(1)—P(1)—C(7)	110.34 (8)	C(1)—C(6)—C(5)	118.46 (15)
O(2)—P(1)—O(3)	109.81 (9)	C(1)—C(6)—C(9)	121.11 (15)
O(2)—P(1)—C(7)	109.45 (9)	C(5)—C(6)—C(9)	120.42 (15)
O(3)—P(1)—C(7)	103.96 (8)	P(1)—C(7)—C(2)	114.48 (12)
O(4)—P(2)—O(5)	105.73 (8)	P(2)—C(9)—C(6)	114.21 (11)
O(4)—P(2)—O(6)	109.86 (8)	P(1)—O(1)—HO(7)	104.0 (8)
O(4)—P(2)—C(9)	107.22 (8)	P(1)—O(2)—HO(2)	121.6 (19)
O(5)—P(2)—O(6)	112.57 (7)	P(1)—O(3)—HO(3)	128.2 (17)
O(5)—P(2)—C(9)	108.10 (8)	P(2)—O(4)—HO(4)	112.4 (19)
O(6)—P(2)—C(9)	112.98 (8)	P(2)—O(5)—HO(5)	116.2 (18)
O(7)—C(1)—C(2)	121.04 (14)	P(2)—O(6)—HO(3)^{ii}	140.6 (9)
O(7)—C(1)—C(6)	117.43 (14)	C(1)—O(7)—HO(7)	104.1 (18)
C(2)—C(1)—C(6)	121.49 (14)	C(1)—O(7)—HO(4)	106.8 (9)
C(1)—C(2)—C(3)	117.75 (15)	HO(4)—O(7)—HO(7)	148.5 (20)
C(2)—C(3)—C(4)	122.28 (15)		
O(1)—P(1)—C(7)—C(2)	−62.1 (1)	O(4)—P(2)—C(9)—C(6)	−65.1 (1)
O(3)—P(1)—C(7)—C(2)	174.4 (2)	O(5)—P(2)—C(9)—C(6)	−178.7 (2)
P(1)—O(1)—H(1*W*2)^{i}	107.3 (10)		
P(1)—O(1)—H(2*W*1)	129.9 (8)		
HO(7)—O(1)—H(1*W*2)^{i}	128.1 (12)		
HO(7)—O(1)—H(2*W*1)	104.7 (11)		
H(1*W*2)^{i}—O(1)—H(2*W*1)	85.4 (13)		
P(2)—O(5)—H(2*W*2)	126.2 (9)		
HO(5)—O(5)—H(2*W*2)	107.5 (20)		
P(2)—O(6)—H(1*W*1)^{iii}	122.0 (10)		
HO(3)^{ii}—O(6)—H(1*W*1)^{iii}	95.3 (13)		
HO(5)^{iv}—O*W*(1)—H(1*W*1)	99.7 (25)		
HO(5)^{iv}—O*W*(1)—H(2*W*1)	118.8 (21)		
H(1*W*1)—O*W*(1)—H(2*W*1)	112.7 (25)		
HO(2)^{v}—O*W*(2)—H(1*W*2)	110.5 (22)		
HO(2)^{v}—O*W*(2)—H(2*W*2)	114.5 (21)		
H(1*W*2)—O*W*(2)—H(2*W*2)	97 (3)		

O—H···O	O—H	H···O	O···O	O—H···O
O(2)—HO(2)···O*W*(2)^{vi}	0.89 (3)	1.65 (3)	2.522 (2)	164 (3)
O(3)—HO(3)···O(6)^{iv}	0.88 (3)	1.66 (3)	2.529 (2)	170 (2)
O(4)—HO(4)···O(7)	0.80 (3)	1.88 (3)	2.640 (2)	164 (3)
O(5)—HO(5)···O*W*(1)^{ii}	1.14 (4)	1.32 (4)	2.458 (2)	173 (3)
O(7)—HO(7)···O(1)	0.85 (3)	1.92 (3)	2.753 (2)	173 (3)
O*W*(1)—H(1*W*1)···O(6)^{iii}	0.92 (3)	1.77 (3)	2.693 (2)	172 (3)
O*W*(1)—H(2*W*1)···O(1)	0.91 (3)	1.79 (3)	2.688 (2)	168 (2)
O*W*(2)—H(1*W*2)···O(1)^{i}	0.93 (4)	1.94 (4)	2.866 (2)	173 (3)
O*W*(2)—H(2*W*2)···O(5)	0.95 (3)	1.87 (3)	2.801 (2)	167 (3)

Symmetry code: (i) $-x, 1-y, 2-z$; (ii) $x, y, 1+z$; (iii) $1-x, 1-y, 2-z$; (iv) $x, y, z-1$; (v) $x, y-1, 1+z$; (vi) $x, 1+y, z-1$.

The data were collected using a θ-scan width of $(0.8 + 0.35\tan\theta)^\circ$ and a θ-scan rate of 5.49° $\min^{-1}$ with background counts for 5 s on each side of every scan. The absorption correction used nine strong low-angle (χ values close to 90°) reflections. All H atoms were visible in difference maps; the hydroxyl H atoms were refined isotropically while those attached to the C atoms were positioned geometrically (C—H 0.95 Å) and included as riding atoms in the structure-factor calculations.

GF thanks NSERC Canada for Grants in Aid of Research.

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 55824 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1012]

References

Ageeva, A. B. & Ivanov, B. E. (1967). *Izv. Akad. Nauk SSSR Ser. Khim.* **7**, 1494-1498.

Ageeva, A. B. & Ivanov, B. E. (1968). *Chem. Abstr.* **68**, 3870, Nr. 39738*a*.

Allen, F. H., Kennard, O. & Taylor, R. (1983). *Acc. Chem. Res.* **16**, 146-153.

Gabe, E. J., Le Page, Y., Charland, J. P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384-389.

Gebert, E., Reis, A. H. Jr, Druyan, M. E., Peterson, S. W., Mason, G. W. & Peppard, D. F. (1977). *J. Phys. Chem.* **81**, 471-475.

Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kaiser, J., Schied, B., Trautmann, N. & Vogt, W. (1993). *Makromol. Chem.* In the press.

Peterson, S. W., Gebert, E., Reis, A. H. Jr, Druyan, M. E., Mason, G. W. & Peppard, D. F. (1977). *J. Phys. Chem.* **81**, 466-471.

Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

Vogt, W. (1978). *Phosphorus Sulfur*, **5**, 123-125.

Acta Cryst. (1993). C**49**, 1026–1028

Structure of 4,6-Bis(trifluoromethyl)-2,2-bis[2,4,6-tris(trifluoromethyl)phenylthio]-1-thiabenzocyclobutene

EHMKE POHL, REGINE HERBST-IRMER AND
GEORGE M. SHELDRICK

*Institut für Anorganische Chemie, Universität
Göttingen, Tammannstrasse 4, 3400 Göttingen,
Germany*

(*Received* 5 *October* 1992; *accepted* 26 *November* 1992)

Abstract

The structure of the title compound, 3,5-bis(trifluoromethyl)-8,8-bis[2,4,6-tris(trifluoromethyl)phenylthio]-7-thiabicyclo[4.2.0]octa-1,3,5-triene, is reported.

Comment

Over the last few years the substituent 2,4,6-tris(trifluoromethyl)phenyl (R_f) has turned out to be a highly versatile ligand in main-group chemistry. It combines high steric demand with a strong electron-withdrawing effect (Edelmann, 1992). The reaction of Ga(Me)$_3$ with HSR_f resulted in the unexpected formation of a condensation product. The product arises from coupling of three R_fS units under elimination of three *ortho* F atoms. A similiar reaction of three R_fO units has been reported by Whitmire, Roesky, Brooker & Sheldrick (1991).

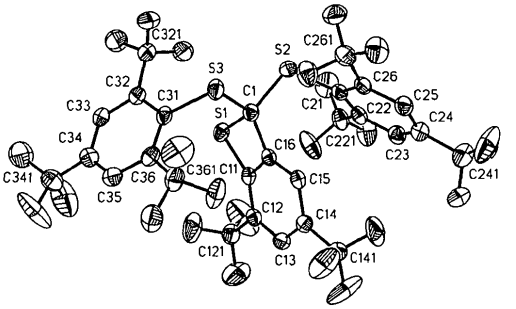


Fig. 1. Structure of the title compound showing 50% probability displacement ellipsoids. The H atoms are omitted for clarity.

Experimental

Crystal data

$C_{27}H_6F_{24}S_3$
$M_r = 882.50$
Monoclinic
$P2_1/c$
$a = 17.721$ (4) Å
$b = 10.062$ (2) Å
$c = 17.284$ (3) Å
$\beta = 93.19$ (2)°
$V = 3077.1$ (11) Å^3
$Z = 4$
$D_x = 1.905$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 48 reflections
$\theta = 10$-12.5°
$\mu = 0.409$ mm^{-1}
$T = 153.0$ (10) K
Block
0.6 × 0.4 × 0.4 mm
Colourless

Data collection

Stoe-Siemens AED four-circle diffractometer
Profile data from $2\theta/\omega$ scans
Absorption correction: empirical
$T_{\min} = 0.738$, $T_{\max} = 0.786$
6164 measured reflections
5372 independent reflections

4392 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0360$
$\theta_{\max} = 24.98°$
$h = -17 \rightarrow 21$
$k = -11 \rightarrow 9$
$l = -20 \rightarrow 20$
3 standard reflections
frequency: 90 min
intensity variation: none

Refinement

Refinement on F^2
$R1 = 0.0465$ for $F > 4\sigma(F)$
$wR2 = 0.1254$ for all data

$(\Delta/\sigma)_{\max} = 0.000$
$\Delta\rho_{\max} = 0.663$ e Å^{-3}
$\Delta\rho_{\min} = -0.409$ e Å^{-3}

$S = 1.07$
5355 reflections
517 parameters
Calculated weights
$w = 1/[\sigma^2(F_o^2)+(0.0424P)^2 + 5.6388P]$
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)

Refinement on F^2 for all reflections except those flagged for possible systematic errors; the observed threshold $I > 2\sigma(I)$ is used only for calculating $R1$ *etc.*, given here for comparison with refinements on F. Three *para* CF$_3$ groups (F141-F143, F241-F243, F341-F343) are rotationally disordered. Two positions were refined for each CF$_3$ group with restraints on the C—F and F···F distances. The anisotropic displacement parameters of opposite F atoms were constrained to be equal. The site occupancies were 0.837 (4), 0.860 (7) and 0.776 (5), respectively, for the major components of these three CF$_3$ groups. A riding model was employed for the H atoms, with $U({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: Stoe *DIF*4. Cell refinement: Stoe *DIF*4. Data reduction: Stoe *REDU*4. Program(s) used to solve structure: *SHELXS*-92 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*-92 (Sheldrick, 1992). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXL*-92.

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

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

	x	y	z	$U_{\rm eq}$
C1	0.2510 (2)	0.5766 (4)	0.5447 (2)	0.028 (2)
S1	0.35736 (5)	0.56424 (9)	0.53927 (6)	0.0316 (4)
C11	0.3202 (2)	0.4250 (3)	0.4888 (2)	0.026 (2)
C12	0.3452 (2)	0.3169 (4)	0.4485 (2)	0.030 (2)
C121	0.4269 (2)	0.2991 (4)	0.4360 (3)	0.043 (2)
F121	0.4668 (2)	0.2761 (5)	0.5010 (2)	0.1057 (15)
F122	0.4403 (2)	0.1992 (3)	0.3891 (2)	0.087 (2)
F123	0.4570 (2)	0.4054 (3)	0.4064 (2)	0.081 (2)
C13	0.2896 (2)	0.2278 (4)	0.4212 (2)	0.032 (2)
C14	0.2137 (2)	0.2488 (4)	0.4331 (2)	0.029 (2)
C141	0.1555 (2)	0.1510 (4)	0.4035 (2)	0.037 (2)
F141 †	0.1092 (3)	0.1185 (7)	0.4557 (2)	0.116 (4)
F142 †	0.1849 (2)	0.0418 (3)	0.3764 (4)	0.085 (2)
F143 †	0.1135 (2)	0.1987 (3)	0.3446 (3)	0.071 (2)
F144	0.1626 (13)	0.1136 (25)	0.3320 (7)	0.116 (4)
F145	0.0857 (4)	0.1928 (15)	0.4068 (17)	0.085 (2)
F146	0.1573 (12)	0.0392 (13)	0.4439 (12)	0.071 (2)
C15	0.1890 (2)	0.3613 (4)	0.4728 (2)	0.026 (2)
C16	0.2445 (2)	0.4475 (3)	0.4999 (2)	0.025 (2)
S2	0.21823 (5)	0.57716 (10)	0.64345 (5)	0.0330 (5)
C21	0.1685 (2)	0.4234 (4)	0.6509 (2)	0.028 (2)
C22	0.2049 (2)	0.3036 (4)	0.6726 (2)	0.032 (2)
C221	0.2882 (2)	0.2964 (4)	0.6978 (2)	0.040 (2)
F221	0.30930 (15)	0.3911 (3)	0.74770 (15)	0.060 (2)
F222	0.33214 (13)	0.3044 (3)	0.63867 (14)	0.0591 (13)
F223	0.30476 (14)	0.1825 (3)	0.7335 (2)	0.0578 (14)
C23	0.1647 (2)	0.1855 (4)	0.6723 (2)	0.035 (2)
C24	0.0876 (2)	0.1842 (4)	0.6538 (2)	0.036 (2)
C241	0.0461 (2)	0.0544 (4)	0.6515 (2)	0.051 (3)
F241 †	−0.0274 (2)	0.0691 (3)	0.6445 (4)	0.085 (2)
F242 †	0.0685 (3)	−0.0254 (4)	0.5990 (3)	0.095 (4)
F243 †	0.0572 (2)	−0.0113 (4)	0.7191 (2)	0.068 (3)
F244	0.0902 (8)	−0.0451 (10)	0.6344 (17)	0.085 (2)
F245	0.0120 (15)	0.0249 (18)	0.7119 (9)	0.095 (4)
F246	−0.0058 (11)	0.0567 (16)	0.5918 (11)	0.068 (3)
C25	0.0501 (2)	0.3023 (4)	0.6376 (2)	0.035 (2)
C26	0.0895 (2)	0.4212 (4)	0.6365 (2)	0.030 (2)
C261	0.0449 (2)	0.5467 (4)	0.6191 (2)	0.038 (2)

F261	−0.02983 (12)	0.5217 (3)	0.6135 (2)	0.0489 (12)
F262	0.05591 (14)	0.6402 (2)	0.6726 (2)	0.0492 (14)
F263	0.06092 (13)	0.5995 (2)	0.55039 (14)	0.0470 (14)
S3	0.20796 (5)	0.72672 (10)	0.50009 (6)	0.0355 (5)
C31	0.2775 (2)	0.7765 (4)	0.4347 (2)	0.030 (2)
C32	0.3377 (2)	0.8588 (4)	0.4615 (2)	0.031 (2)
C321	0.3409 (2)	0.9212 (4)	0.5411 (2)	0.040 (3)
F321	0.2797 (2)	0.9954 (2)	0.55249 (15)	0.052 (2)
F322	0.4002 (2)	1.0021 (3)	0.55167 (15)	0.059 (2)
F323	0.3463 (2)	0.8321 (2)	0.59911 (13)	0.050 (2)
C33	0.3961 (2)	0.8866 (4)	0.4139 (2)	0.031 (2)
C34	0.3946 (2)	0.8369 (4)	0.3391 (2)	0.034 (2)
C341	0.4593 (2)	0.8614 (4)	0.2889 (2)	0.045 (2)
F341 †	0.4887 (4)	0.9801 (5)	0.2991 (5)	0.103 (4)
F342 †	0.5143 (3)	0.7784 (7)	0.3037 (4)	0.102 (2)
F343 †	0.4404 (2)	0.8516 (10)	0.2154 (2)	0.102 (2)
F344	0.4726 (14)	0.7590 (15)	0.2444 (15)	0.103 (4)
F345	0.4467 (10)	0.9612 (22)	0.2411 (14)	0.102 (2)
F346	0.5225 (6)	0.8874 (34)	0.3272 (6)	0.102 (2)
C35	0.3330 (2)	0.7639 (4)	0.3105 (2)	0.034 (2)
C36	0.2746 (2)	0.7349 (4)	0.3572 (2)	0.032 (2)
C361	0.2070 (2)	0.6631 (4)	0.3196 (2)	0.040 (2)
F361	0.2145 (2)	0.6448 (3)	0.24294 (14)	0.057 (2)
F362	0.1965 (2)	0.5425 (3)	0.34738 (15)	0.059 (2)
F363	0.14385 (15)	0.7312 (3)	0.3245 (2)	0.0683 (15)

† Major components of the disordered CF_3 groups.

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

C1—C16	1.513 (5)	C12—C13	1.395 (5)
C1—S2	1.834 (4)	C13—C14	1.387 (5)
C1—S3	1.842 (4)	C14—C15	1.407 (5)
C1—S1	1.897 (4)	C15—C16	1.375 (5)
S1—C11	1.759 (4)	S2—C21	1.789 (4)
C11—C12	1.378 (5)	S3—C31	1.790 (4)
C11—C16	1.383 (5)		
C16—C1—S2	117.4 (2)	C11—C12—C13	115.9 (3)
C16—C1—S3	118.2 (2)	C11—C12—C121	120.8 (3)
S2—C1—S3	104.0 (2)	C14—C13—C12	121.6 (3)
C16—C1—S1	88.0 (2)	C13—C14—C15	121.7 (3)
S2—C1—S1	114.4 (2)	C16—C15—C14	115.9 (3)
S3—C1—S1	115.0 (2)	C15—C16—C11	122.1 (3)
C11—S1—C1	74.4 (2)	C15—C16—C1	138.4 (3)
C12—C11—C16	122.7 (3)	C11—C16—C1	99.5 (3)
C12—C11—S1	139.2 (3)	C21—S2—C1	104.3 (2)
C16—C11—S1	98.1 (3)	C31—S3—C1	102.1 (2)

This work was supported by the Deutsche Forschunggemeinschaft and the Fonds der chemischen Industrie. EP is grateful to the Stiftung Stipendienfonds des Verbands der chemischen Industrie for a Fellowship.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55892 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1032]

References

Edelmann, F. T. (1992). *Comments Inorg. Chem.* **12**, 259-284.

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

Sheldrick, G. M. (1991). *SHELXTL-Plus*. Version 4. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1992). *SHELXL*-92. Program for crystal structure refinement. Univ. of Göttingen, Germany.

Whitmire, K. H., Roesky, H. W., Brooker, S. & Sheldrick, G. M. (1991). *J. Organomet. Chem.* **402**, C4-C7.

Acta Cryst. (1993). C**49**, 1028–1031

Structure of a Bromine Derivative of Isostegane

George R. Clark

Department of Chemistry, University of Auckland, Auckland, New Zealand

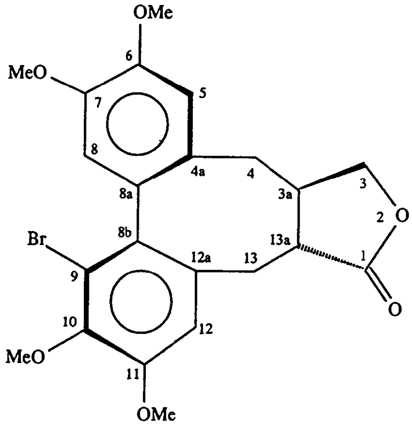
(*Received* 4 *August* 1992; *accepted* 1 *December* 1992)

Abstract

The structure determination establishes that the crystals are (3a*S**,13a*S**;8a*R**,8b*R**)-9-bromo-6,7,-10,11-tetramethoxy-3a,4,13,13a-tetrahydrodibenzo-[4,5;6,7]cycloocta[1,2-*c*]furan-1(3*H*)-one.

Comment

Dibenzocyclooctadiene lignan lactones are of considerable interest because of their pronounced therapeutic activity (Ward, 1982). Co-workers in this Department have been studying the synthesis of such compounds, and have oxidatively coupled dimethylmatairesinol with thallium tristrifluoroacetate and boron trifluoride etherate to give an isostegane whose structure we have established by an X-ray diffraction study (Cambie, Clark, Craw, Rutledge & Woodgate, 1984). The 9-bromo derivative (I) was also prepared (Craw, 1984), and its X-ray structure has now been determined to evaluate the geometric changes consequent upon bromination, and to verify the absolute stereochemistry of the molecule by use of the anomalous dispersion of the Br atom.



(I)

The crystals contain two molecules of compound and one molecule of dichloromethane solvent per asymmetric unit. Fig. 1 depicts the molecular struc-

0108-2701/93/051028-04$06.00