Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **perbromobishomocubane (1)**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	1578(7)	2865(12)	4677(7)	44(2)
Br(1)	308(1)	3513(2)	4056(1)	62(1)
C(2)	2053(9)	2827(16)	5730(9)	63(3)
Br(2)	1306(1)	2584(2)	6405(1)	63(1)
C(3)	2750(9)	4079(18)	6023(8)	67(4)
Br(3)	2043(1)	6021(1)	5842(1)	72(1)
Br(6)	3674(1)	3860(2)	7276(1)	64(1)
C(4)	3173(7)	3840(12)	5321(8)	48(3)
Br(4)	4072(1)	5304(1)	5387(1)	60(1)
C(5)	2308(7)	3562(10)	4364(7)	40(2)
Br(5)	1953(1)	5093(1)	3449(1)	61(1)

Table 3. Anisotropic displacement parameters ($Å^2$x 10^3) for **perbromobishomocubane (1)**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	36(6)	49(5)	45(6)	2(5)	15(5)	-8(4)
Br(1)	40(1)	69(1)	67(1)	-12(1)	13(1)	10(1)
C(2)	60(8)	75(8)	62(7)	1(6)	34(7)	-7(6)
Br(2)	71(1)	79(1)	59(1)	-13(1)	45(1)	-23(1)
C(3)	51(7)	102(10)	49(7)	-1(7)	20(6)	-17(7)
Br(3)	99(1)	40(1)	93(1)	-5(1)	54(1)	15(1)
Br(6)	54(1)	89(1)	42(1)	-9(1)	11(1)	4(1)
C(4)	31(5)	55(6)	59(7)	10(5)	19(5)	-7(4)
Br(4)	58(1)	53(1)	81(1)	-13(1)	41(1)	-18(1)
C(5)	55(6)	33(5)	31(5)	1(4)	17(5)	0(4)
Br(5)	85(1)	46(1)	52(1)	20(1)	28(1)	11(1)

Table 4. Bond lengths [Å] and angles [°] for **perbromobishomocubane (1)**.

C(1)-C(2)	1.521(17)	C(3)-Br(6)	1.927(13)
C(1)-C(5)	1.548(13)	C(3)-Br(3)	2.001(16)
C(1)-C(4)#1	1.557(15)	C(4)-C(1)#1	1.557(15)
C(1)-Br(1)	1.914(10)	C(4)-C(5)	1.585(15)
C(2)-C(3)	1.490(19)	C(4)-Br(4)	1.885(9)
C(2)-C(5)#1	1.629(16)	C(5)-C(2)#1	1.629(16)
C(2)-Br(2)	1.891(11)	C(5)-Br(5)	1.892(9)
C(3)-C(4)	1.521(16)		
C(2)-C(1)-C(5)	105.2(8)	C(2)-C(3)-Br(3)	107.5(9)
C(2)-C(1)-C(4)#1	88.1(9)	C(4)-C(3)-Br(3)	113.3(9)
C(5)-C(1)-C(4)#1	99.7(7)	Br(6)-C(3)-Br(3)	111.9(7)
C(2)-C(1)-Br(1)	120.7(8)	C(3)-C(4)-C(1)#1	108.4(9)
C(5)-C(1)-Br(1)	117.9(7)	C(3)-C(4)-C(5)	105.1(8)
C(4)#1-C(1)-Br(1)	119.9(7)	C(1)#1-C(4)-C(5)	88.1(8)
C(3)-C(2)-C(1)	107.0(10)	C(3)-C(4)-Br(4)	113.8(9)
C(3)-C(2)-C(5)#1	100.9(10)	C(1)#1-C(4)-Br(4)	119.0(7)
C(1)-C(2)-C(5)#1	87.8(8)	C(5)-C(4)-Br(4)	119.2(7)
C(3)-C(2)-Br(2)	117.0(9)	C(1)-C(5)-C(4)	102.3(7)
C(1)-C(2)-Br(2)	118.7(8)	C(1)-C(5)-C(2)#1	106.9(8)
C(5)#1-C(2)-Br(2)	120.7(8)	C(4)-C(5)-C(2)#1	83.5(8)
C(2)-C(3)-C(4)	98.8(10)	C(1)-C(5)-Br(5)	120.3(7)
C(2)-C(3)-Br(6)	113.2(10)	C(4)-C(5)-Br(5)	119.3(7)
C(4)-C(3)-Br(6)	111.5(8)	C(2)#1-C(5)-Br(5)	118.0(7)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1/2,-y+1/2,-z+1

Table 5. Crystal data and structure refinement for **dimethoxyperbromobishomocubane (4)**.

Identification code	eat84b	
Empirical formula	C12 H6 Br10 O2	
Formula weight	981.27	
Temperature	294(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 12.5823(3) Å	α= 90°.
	b = 11.7851(5) Å	β= 90°.
	c = 13.6700(8) Å	γ = 90°.
Volume	2027.04(15) $Å^3$	
Z	4	
Density (calculated)	3.215 Mg/m^3	
Absorption coefficient	23.619 mm^{-1}	
F(000)	1776	
Crystal size	.4 x .4 x .4 mm^3	
Theta range for data collection	6.08 to 57.99°.	
Index ranges	0<=h<=13, -12<=k<=9, -2<=l<=15	
Reflections collected	2647	
Independent reflections	1407 [R(int) = 0.0201]	
Completeness to theta = 57.99°	100.0 %	
Absorption correction	Integration	
Max. and min. transmission	0.1690 and 0.0774	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1407 / 1 / 115	
Goodness-of-fit on F^2	1.079	
Final R indices [I>2sigma(I)]	R1 = 0.0242, wR2 = 0.0575	
R indices (all data)	R1 = 0.0280, wR2 = 0.0593	
Extinction coefficient	0.00063(3)	
Largest diff. peak and hole	0.438 and -0.432 $e.Å^{-3}$	

Table 6. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **dimethoxyperbromobishomocubane (4)**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(2)	10337(4)	1074(3)	4981(4)	20(1)
Br(2)	10551(7)	2631(5)	4738(7)	27(1)
O(2)	10500(4)	2223(4)	5065(4)	27(1)
C(2A)	10660(6)	2905(6)	4206(5)	37(2)
C(3)	9186(3)	600(4)	4655(4)	20(1)
Br(3)	8192(1)	1678(1)	4205(1)	33(1)
C(4)	8899(4)	-209(4)	5538(4)	21(1)
Br(4)	7447(1)	-635(1)	5689(1)	34(1)
C(5)	9442(4)	271(4)	6443(4)	24(1)
Br(5A)	8816(1)	1705(1)	6843(1)	35(1)
Br(5B)	9476(1)	-745(1)	7546(1)	40(1)
C(6)	10521(4)	460(4)	5974(4)	22(1)
Br(6)	11597(1)	1045(1)	6804(1)	33(1)

Table 6a. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for a **dimethoxyperbromobishomocubane (4)**.

	x	y	z	U(eq)
H(2C)	11264	2625	3850	56
H(2A)	10784	3678	4394	56
H(2B)	10040	2865	3798	56

Table 7. Bond lengths [Å] and angles [°] for a **dimethoxyperbromobishomocubane (4)**.

C(2)-O(2)	1.374(6)	C(4)-C(5)	1.522(7)
C(2)-C(6)	1.555(7)	C(4)-C(2)#1	1.570(6)
C(2)-C(4)#1	1.570(6)	C(4)-Br(4)	1.907(5)
C(2)-C(3)	1.616(6)	C(5)-C(6)	1.518(7)
C(2)-Br(2)	1.884(4)	C(5)-Br(5B)	1.926(5)
O(2)-C(2A)	1.438(8)	C(5)-Br(5A)	1.943(5)
C(3)-C(6)#1	1.562(7)	C(6)-C(3)#1	1.562(7)
C(3)-C(4)	1.580(7)	C(6)-Br(6)	1.896(5)
C(3)-Br(3)	1.885(4)		
O(2)-C(2)-C(6)	111.3(4)	C(5)-C(4)-C(3)	107.1(4)
O(2)-C(2)-C(4)#1	125.9(4)	C(2)#1-C(4)-C(3)	84.7(3)
C(6)-C(2)-C(4)#1	90.1(4)	C(5)-C(4)-Br(4)	116.0(3)
O(2)-C(2)-C(3)	119.8(4)	C(2)#1-C(4)-Br(4)	117.7(3)
C(6)-C(2)-C(3)	102.3(3)	C(3)-C(4)-Br(4)	117.4(3)
C(4)#1-C(2)-C(3)	101.5(3)	C(6)-C(5)-C(4)	96.4(4)
O(2)-C(2)-Br(2)	15.0(4)	C(6)-C(5)-Br(5B)	113.7(3)
C(6)-C(2)-Br(2)	125.9(4)	C(4)-C(5)-Br(5B)	114.5(3)
C(4)#1-C(2)-Br(2)	117.7(4)	C(6)-C(5)-Br(5A)	110.7(3)
C(3)-C(2)-Br(2)	114.6(4)	C(4)-C(5)-Br(5A)	111.7(3)
C(2)-O(2)-C(2A)	120.2(5)	Br(5B)-C(5)-Br(5A)	109.2(2)
C(6)#1-C(3)-C(4)	89.5(3)	C(5)-C(6)-C(2)	107.7(4)
C(6)#1-C(3)-C(2)	102.5(3)	C(5)-C(6)-C(3)#1	109.0(4)
C(4)-C(3)-C(2)	101.7(3)	C(2)-C(6)-C(3)#1	85.8(3)
C(6)#1-C(3)-Br(3)	121.1(3)	C(5)-C(6)-Br(6)	116.1(3)
C(4)-C(3)-Br(3)	120.3(3)	C(2)-C(6)-Br(6)	117.4(3)
C(2)-C(3)-Br(3)	116.9(3)	C(3)#1-C(6)-Br(6)	116.8(3)
C(5)-C(4)-C(2)#1	109.5(4)		

Symmetry transformations used to generate equivalent atoms:

#1 -x+2,-y,-z+1

Table 8. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for a **dimethoxyperbromobishomocubane (4)**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(2)	20(2)	14(3)	26(3)	0(2)	3(2)	-3(2)
Br(2)	40(2)	18(3)	25(3)	7(2)	-2(2)	-5(3)
O(2)	40(2)	18(3)	25(3)	7(2)	-2(2)	-5(3)
C(2A)	53(4)	22(4)	37(5)	18(3)	-5(3)	-7(3)
C(3)	15(2)	20(3)	24(3)	3(2)	-5(2)	5(2)
Br(3)	32(1)	30(1)	38(1)	5(1)	-4(1)	9(1)
C(4)	23(2)	20(3)	19(3)	2(2)	7(2)	-2(2)
Br(4)	23(1)	37(1)	42(1)	-1(1)	6(1)	-6(1)
C(5)	30(3)	23(3)	19(3)	1(2)	-1(2)	0(2)
Br(5A)	38(1)	31(1)	36(1)	-11(1)	10(1)	1(1)
Br(5B)	54(1)	43(1)	25(1)	10(1)	3(1)	-4(1)
C(6)	25(2)	23(3)	18(2)	-1(2)	-2(2)	-4(2)
Br(6)	33(1)	39(1)	28(1)	-7(1)	-8(1)	-6(1)

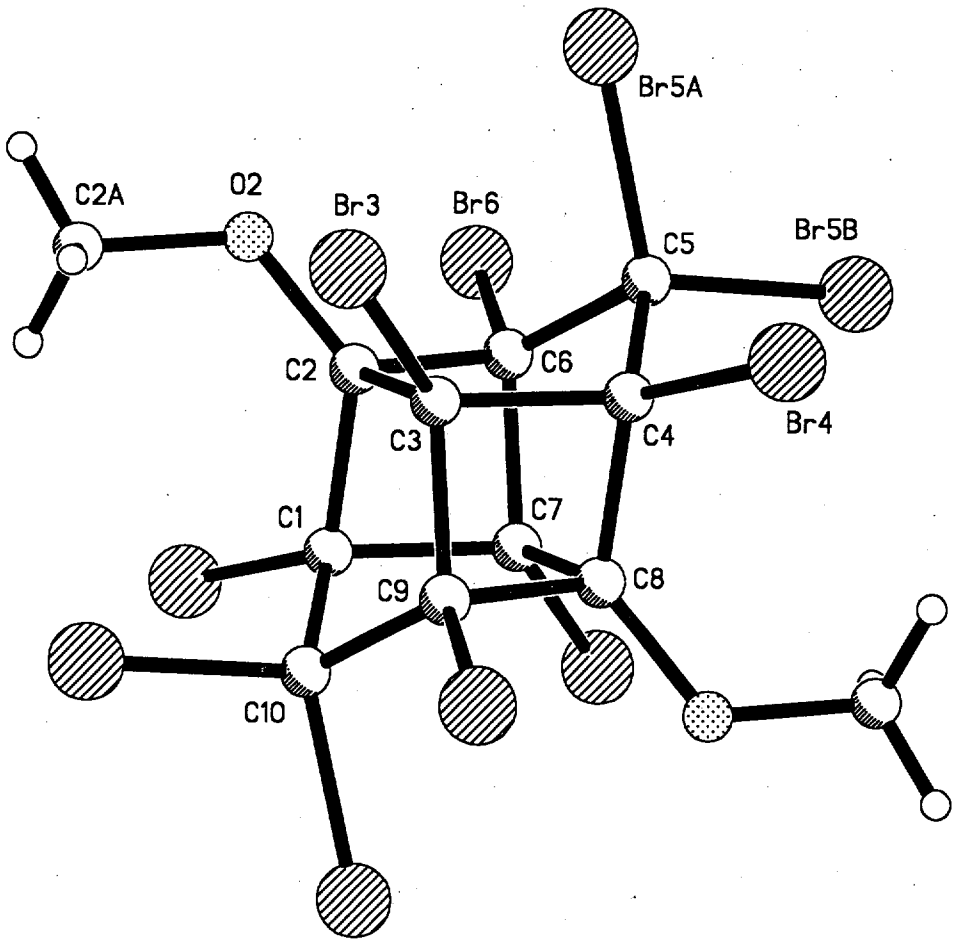

Figure S1. A view of the structure of **4** as it occurs in the crystal. Although the X-ray intensity data could be explained quite well (R = 0.028) with only this structure, there were slight residual density peaks and distortions of bond distances in the vicinity of the –OCH3 groups. Inclusion of a small (10%) admixture of a bromo-substituent impurity at these sites led to a slight improvement in the agreement (to R = 0.024) and more reasonable bond distances. There was no hint of disorder elsewhere in the structure.

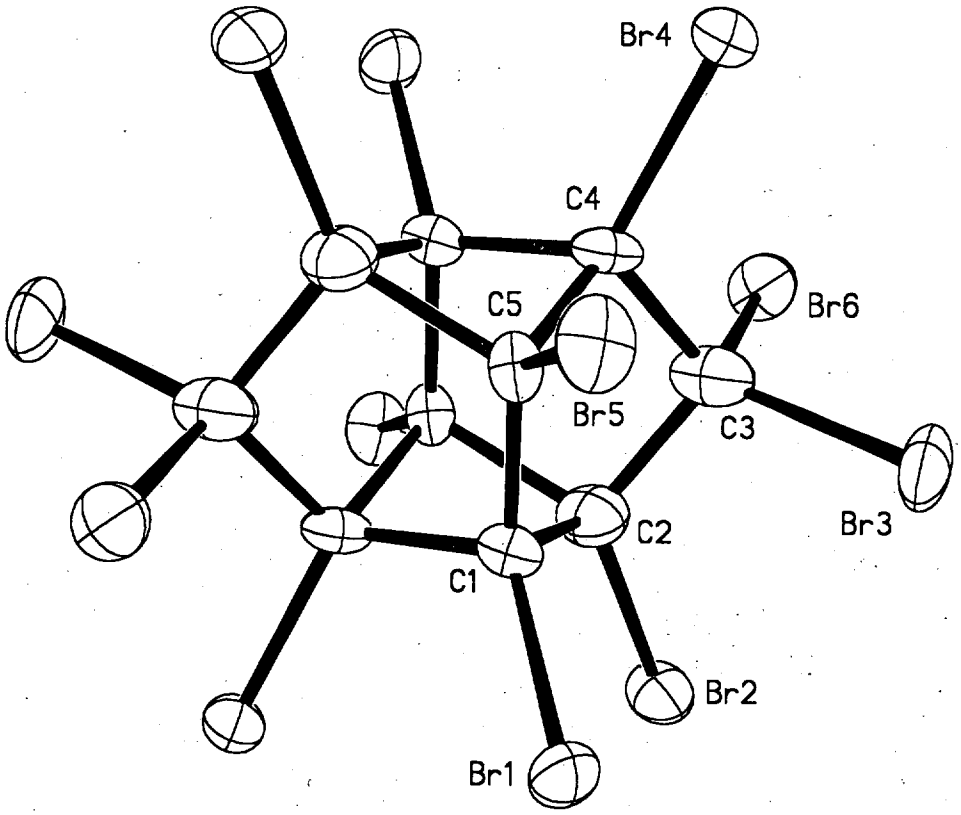

Figure S2. A view of the structure of **1** as it occurs in the crystal, showing the numbering scheme and ellipsoids representing the anisotropic displacement parameters. The molecule sits on an inversion center, so only half the atoms were refined. Although there were numerous residual density peaks, most were in the vicinity of the dense bromine atoms, which is not unusual. No systematic disorder could be seen. The four-membered ring shows unusually small bond angles (83.5 and 88.1); similar values are reported for other D2h-bishomocubanes (cf. ref. 2).

Supplementary Tables and Figures

On the Reaction of Sodium Methoxide with *D2h*-Perbromobishomocubane

A Structural Reassignment

Philip E. Eaton,* Datong Tang and Richard Gilardi

Table 1. Crystal data and structure refinement for 4.

Identification code	eat84b	
Empirical formula	C12 H6 Br10 O2	
Formula weight	981.27	
Temperature	294(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 12.5823(3) Å	α= 90°.
	b = 11.7851(5) Å	β= 90°.
	c = 13.6700(8) Å	γ = 90°.
Volume	2027.04(15) Å^3	
Z	4	
Density (calculated)	3.215 Mg/m^3	
Absorption coefficient	23.619 mm^{-1}	
F(000)	1776	
Crystal size	.4 x .4 x .4 mm^3	
Theta range for data collection	6.08 to 57.99°.	
Index ranges	0<=h<=13, -12<=k<=9, -2<=l<=15	
Reflections collected	2647	
Independent reflections	1407 [R(int) = 0.0201]	
Completeness to theta = 57.99°	100.0 %	
Absorption correction	Integration	
Max. and min. transmission	0.1690 and 0.0774	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1407 / 1 / 115	
Goodness-of-fit on F^2	1.079	
Final R indices [I>2sigma(I)]	R1 = 0.0242, wR2 = 0.0575	
R indices (all data)	R1 = 0.0280, wR2 = 0.0593	
Extinction coefficient	0.00063(3)	
Largest diff. peak and hole	0.438 and -0.432 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 4. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(2)	10337(4)	1074(3)	4981(4)	20(1)
Br(2)	10551(7)	2631(5)	4738(7)	27(1)
O(2)	10500(4)	2223(4)	5065(4)	27(1)
C(2A)	10660(6)	2905(6)	4206(5)	37(2)
C(3)	9186(3)	600(4)	4655(4)	20(1)
Br(3)	8192(1)	1678(1)	4205(1)	33(1)
C(4)	8899(4)	-209(4)	5538(4)	21(1)
Br(4)	7447(1)	-635(1)	5689(1)	34(1)
C(5)	9442(4)	271(4)	6443(4)	24(1)
Br(5A)	8816(1)	1705(1)	6843(1)	35(1)
Br(5B)	9476(1)	-745(1)	7546(1)	40(1)
C(6)	10521(4)	460(4)	5974(4)	22(1)
Br(6)	11597(1)	1045(1)	6804(1)	33(1)

Table 2a. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 4.

	x	y	z	U(eq)
H(2C)	11264	2625	3850	56
H(2A)	10784	3678	4394	56
H(2B)	10040	2865	3798	56

Table 3. Bond lengths [Å] and angles [°] for 4.

C(2)-O(2)	1.374(6)	C(4)-C(5)	1.522(7)
C(2)-C(6)	1.555(7)	C(4)-C(2)#1	1.570(6)
C(2)-C(4)#1	1.570(6)	C(4)-Br(4)	1.907(5)
C(2)-C(3)	1.616(6)	C(5)-C(6)	1.518(7)
C(2)-Br(2)	1.884(4)	C(5)-Br(5B)	1.926(5)
O(2)-C(2A)	1.438(8)	C(5)-Br(5A)	1.943(5)
C(3)-C(6)#1	1.562(7)	C(6)-C(3)#1	1.562(7)
C(3)-C(4)	1.580(7)	C(6)-Br(6)	1.896(5)
C(3)-Br(3)	1.885(4)		
O(2)-C(2)-C(6)	111.3(4)	C(5)-C(4)-C(3)	107.1(4)
O(2)-C(2)-C(4)#1	125.9(4)	C(2)#1-C(4)-C(3)	84.7(3)
C(6)-C(2)-C(4)#1	90.1(4)	C(5)-C(4)-Br(4)	116.0(3)
O(2)-C(2)-C(3)	119.8(4)	C(2)#1-C(4)-Br(4)	117.7(3)
C(6)-C(2)-C(3)	102.3(3)	C(3)-C(4)-Br(4)	117.4(3)
C(4)#1-C(2)-C(3)	101.5(3)	C(6)-C(5)-C(4)	96.4(4)
O(2)-C(2)-Br(2)	15.0(4)	C(6)-C(5)-Br(5B)	113.7(3)
C(6)-C(2)-Br(2)	125.9(4)	C(4)-C(5)-Br(5B)	114.5(3)
C(4)#1-C(2)-Br(2)	117.7(4)	C(6)-C(5)-Br(5A)	110.7(3)
C(3)-C(2)-Br(2)	114.6(4)	C(4)-C(5)-Br(5A)	111.7(3)
C(2)-O(2)-C(2A)	120.2(5)	Br(5B)-C(5)-Br(5A)	109.2(2)
C(6)#1-C(3)-C(4)	89.5(3)	C(5)-C(6)-C(2)	107.7(4)
C(6)#1-C(3)-C(2)	102.5(3)	C(5)-C(6)-C(3)#1	109.0(4)
C(4)-C(3)-C(2)	101.7(3)	C(2)-C(6)-C(3)#1	85.8(3)
C(6)#1-C(3)-Br(3)	121.1(3)	C(5)-C(6)-Br(6)	116.1(3)
C(4)-C(3)-Br(3)	120.3(3)	C(2)-C(6)-Br(6)	117.4(3)
C(2)-C(3)-Br(3)	116.9(3)	C(3)#1-C(6)-Br(6)	116.8(3)
C(5)-C(4)-C(2)#1	109.5(4)		

Symmetry transformations used to generate equivalent atoms:

#1 -x+2,-y,-z+1

Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for 4. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(2)	20(2)	14(3)	26(3)	0(2)	3(2)	-3(2)
Br(2)	40(2)	18(3)	25(3)	7(2)	-2(2)	-5(3)
O(2)	40(2)	18(3)	25(3)	7(2)	-2(2)	-5(3)
C(2A)	53(4)	22(4)	37(5)	18(3)	-5(3)	-7(3)
C(3)	15(2)	20(3)	24(3)	3(2)	-5(2)	5(2)
Br(3)	32(1)	30(1)	38(1)	5(1)	-4(1)	9(1)
C(4)	23(2)	20(3)	19(3)	2(2)	7(2)	-2(2)
Br(4)	23(1)	37(1)	42(1)	-1(1)	6(1)	-6(1)
C(5)	30(3)	23(3)	19(3)	1(2)	-1(2)	0(2)
Br(5A)	38(1)	31(1)	36(1)	-11(1)	10(1)	1(1)
Br(5B)	54(1)	43(1)	25(1)	10(1)	3(1)	-4(1)
C(6)	25(2)	23(3)	18(2)	-1(2)	-2(2)	-4(2)
Br(6)	33(1)	39(1)	28(1)	-7(1)	-8(1)	-6(1)

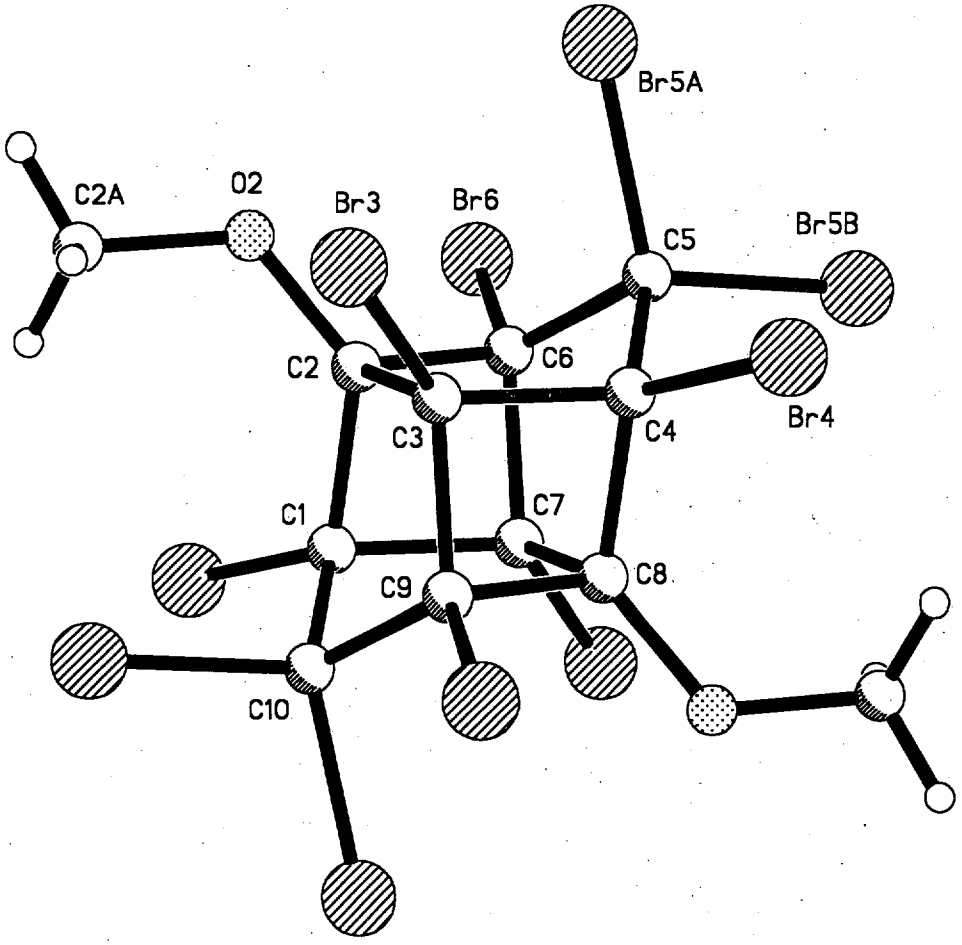

Figure S3. A view of the structure of **4** as it occurs in the crystal. Although the X-ray intensity data could be explained quite well (R = 0.028) with only this structure, there were slight residual density peaks and distortions of bond distances in the vicinity of the $-OCH_3$ groups. Inclusion of a small (10%) admixture of a bromo-substituent impurity at these sites led to a slight improvement in the agreement (to R = 0.024) and reasonable bond distances. There was no hint of disorder elsewhere in the structure