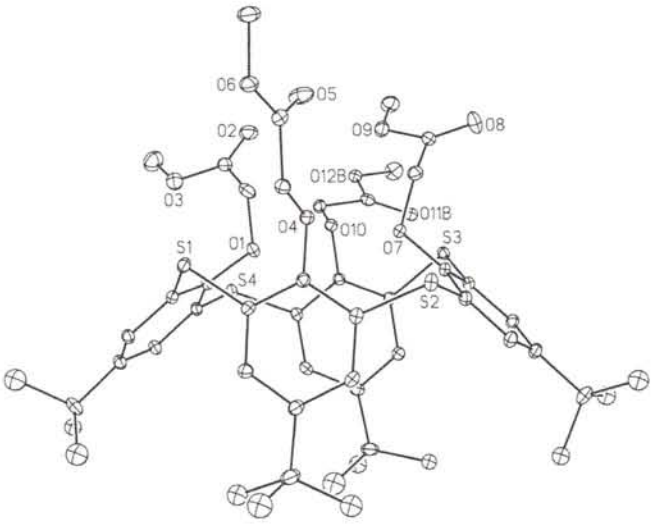
Crystal structure of tetra[(methoxycarbonyl)methoxy]-*p*-*tert*-butyl-thiacalix[4]arene, $C_{52}H_{64}O_{12}S_4$

P. Thuéry*[I], M. Nierlich[I], A. K. Hall[II], J. M. Harrowfield[II], Z. Asfari[III] and J. Vicens[III]

[I] CEA/Saclay, SCM (CNRS URA 331), Bât. 125, F-91191 Gif-sur-Yvette, France
[II] University of Western Australia, Department of Chemistry, and Special Research Centre for Advanced Minerals and Materials Processing, Nedlands, WA 6907, Australia
[III] ECPM, Laboratoire de Chimie des Interactions Moléculaires Spécifiques, (CNRS UMR 7512), 25 rue Becquerel, F-67087 Strasbourg, France

Received January 27, 2000, CCDC-No. 1267/390



Abstract

$C_{52}H_{64}O_{12}S_4$, triclinic, $P\bar{1}$ (No. 2), a = 11.9539(4) Å, b = 14.0875(3) Å, c = 18.2336(7) Å, α = 77.005(2)°, β = 85.833(1)°, γ = 65.372(2)°, V = 2719.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.072, $wR_{gt}(F^2)$ = 0.188, T = 273 K.

Source of material

The title compound has been prepared by reacting 5,11,17,23-tetra-*tert*-butyl-2,8,14,20-tetrathiacalix[4]arene-25,26,27,28-tetrol with bromomethyl acetate in refluxing acetonitrile for 15 h in the presence of potassium carbonate and precipitation with methanol from the crude residue (62% yield; mp 398 K – 399 K). All analytical data were in agreement with a partial cone conformation for the calixarene derivative, as previously reported by Akdas et al. in the case of the tetraethyl ester derivative [1]. Recrystallization from CH_2Cl_2:CH_3CN (1:1) gave single crystals of the calixarene in the cone conformation.

Experimental details

The four *tert*-butyl and one carboxy groups are disordered and modelled with two or four positions. All non-hydrogen atoms refined anisotropically, unless the disordered ones. Hydrogen atoms introduced at calculted positions as riding atoms, unless in the disordered parts, with a displacement factor equal to 1.2 (CH, CH_2) or 1.5 (CH_3) times that of the attached carbon atom.

* Correspondence author (e-mail: thuery@drecam.cea.fr)

Discussion

During the last two decades, calixarenes have drawn much attention as useful starting materials for the design of supramolecular systems or for molecular recognition [2, 3]. In order to introduce new coordinating functions into the macroring, Sone et al. first reported the replacement of the methylene bridges by sulfur atoms, by way of an acid-catalyzed cyclisation of an acyclic tetramer [4]. Recently, Kumagai et al. reported the one-step synthesis of 5,11,17,23-tetra-*tert*-butyl-2,8,14,20-tetrathiacalix[4]arene-25,26,27,28-tetrol by heating *p*-*tert*-butylphenol with elemental sulfur in presence of a base [5]. Subsequently, studies have been made of thiacalix[4]arenes and their derivatives as metal sequestrants, involving both chelation by S and O in the parent compounds [6, 7] and more complicated multidentate binding in the O-derivatives [8].

The asymmetric unit in the title compound comprises one calixarene molecule in a distorted cone conformation. The four sulfur bridges define a mean plane with deviations of ±0.33 Å, i.e. more irregular than usual with methylene-bridged calix[4]arenes. The dihedral angles between the four aromatic rings and this mean plane are 40.25(7)°, 88.94(7)°, 45.87(8)° and 88.90(7)°: such a pseudo-C_2 geometry is rather frequent in calix[4]arenes bearing bulky substituents and prevents solvent inclusion in the calixarene cavity. The mean C—S distance and C–S–C angle are 1.785(4) Å and 100(1)°, respectively. The size of the cavity is enlarged by the replacement of carbon by sulfur atoms, with a mean distance between adjacent sulfur atoms of 5.53(2) Å, compared to the typical distance of about 5.1 Å between methylene carbon atoms in calix[4]arenes. These features are close to those observed in the related compound tetra[(ethoxycarbonyl)-methoxy]-*p*-*tert*-butylthiacalix[4]arene in the cone conformation [1]. The terminal atoms of four *tert*-butyl and one carboxylic groups are highly disordered and have been modelled by two or three positions (noted A, B, C, not represented on the drawing for clarity). The packing consists in alternate columns along the *a*-axis, defining intra- and intermolecular infinite channels, as previously observed in this family of compounds [9].

Table 1. Data collection and handling.

Crystal:	colourless, parallelepipedic, size 0.25 × 0.25 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.32 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa-CCD, φ
$2\theta_{max}$:	57.86°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	22318, 10491
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 7916
$N(param)_{refined}$:	661
Program:	SHELXTL [10]

Table 2. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
O(11A)	2*i*	0.08(2)	1.011(4)	0.090(4)	0.047(3)	0.04(1)
O(12A)	2*i*	0.08	1.132(4)	0.145(7)	0.022(4)	0.07(2)
O(11B)	2*i*	0.52(2)	1.000(1)	0.0719(7)	0.0648(7)	0.049(3)
O(12B)	2*i*	0.52	1.095(1)	0.176(1)	0.0010(8)	0.046(3)
O(11C)	2*i*	0.40(2)	1.030(2)	0.0695(7)	0.0764(9)	0.049(3)
O(12C)	2*i*	0.40	1.070(2)	0.194(1)	–0.0129(9)	0.055(3)
H(3)	2*i*		0.4332(3)	0.6173(3)	0.0800(2)	0.055
H(5)	2*i*		0.3704(3)	0.6490(3)	0.2929(2)	0.052
H(7A)	2*i*		0.8672(3)	0.2980(4)	0.3001(2)	0.078
H(7B)	2*i*		0.7901(3)	0.4105(4)	0.3188(2)	0.078
H(9A)	2*i*		0.821(3)	0.6500(7)	0.124(2)	0.165
H(9B)	2*i*		0.872(5)	0.549(3)	0.089(1)	0.165
H(9C)	2*i*		0.951(2)	0.559(4)	0.149(1)	0.165
C(11A)	2*i*	0.59(1)	0.1574(8)	0.745(1)	0.2112(7)	0.106(4)
C(12A)	2*i*	0.59	0.2296(6)	0.7735(6)	0.0751(3)	0.058(2)
C(13A)	2*i*	0.59	0.2705(9)	0.8468(6)	0.1740(6)	0.091(3)
C(11B)	2*i*	0.41(1)	0.1533(9)	0.709(1)	0.184(1)	0.101(5)
C(12B)	2*i*	0.41	0.264(1)	0.807(1)	0.0788(6)	0.095(5)
C(13B)	2*i*	0.41	0.219(1)	0.822(1)	0.2106(9)	0.101(5)
H(17)	2*i*		0.4302(3)	0.1412(3)	0.4215(2)	0.056
H(19)	2*i*		0.3457(3)	0.4537(3)	0.3592(2)	0.059
H(20A)	2*i*		0.7109(3)	0.2286(3)	0.5643(2)	0.056
H(20B)	2*i*		0.6651(3)	0.3489(3)	0.5214(2)	0.056
H(22A)	2*i*		0.979(1)	0.322(3)	0.652(1)	0.127
H(22B)	2*i*		1.007(2)	0.342(3)	0.566(1)	0.127
H(22C)	2*i*		1.0379(9)	0.2250(7)	0.612(2)	0.127
C(24A)	2*i*	0.42(2)	0.174(1)	0.426(1)	0.3047(8)	0.088(4)
C(25A)	2*i*	0.42	0.225(1)	0.2247(9)	0.350(1)	0.085(4)
C(26A)	2*i*	0.42	0.149(1)	0.374(2)	0.4269(9)	0.105(4)
C(24B)	2*i*	0.33(1)	0.206(2)	0.398(1)	0.2825(7)	0.101(5)
C(25B)	2*i*	0.33	0.207(1)	0.2260(8)	0.380(1)	0.047(3)
C(26B)	2*i*	0.33	0.139(1)	0.409(1)	0.398(1)	0.104(5)
C(24C)	2*i*	0.25(1)	0.235(2)	0.353(2)	0.2729(5)	0.084(5)
C(25C)	2*i*	0.25	0.194(2)	0.240(2)	0.407(1)	0.087(5)
C(26C)	2*i*	0.25	0.142(1)	0.432(1)	0.366(1)	0.095(5)
H(30)	2*i*		0.7670(3)	–0.1331(3)	0.2357(2)	0.052
H(32)	2*i*		0.6106(3)	–0.1054(3)	0.4353(2)	0.057
H(33A)	2*i*		0.9260(3)	–0.0081(3)	0.4354(2)	0.060
H(33B)	2*i*		0.9196(3)	0.1082(3)	0.4222(2)	0.060
H(35A)	2*i*		1.147(2)	0.1536(6)	0.229(2)	0.110
H(35B)	2*i*		1.123(2)	0.058(3)	0.215(1)	0.110
H(35C)	2*i*		1.2181(7)	0.038(2)	0.2776(5)	0.110
C(37A)	2*i*	0.47(1)	0.670(1)	–0.263(1)	0.2606(6)	0.084(3)
C(38A)	2*i*	0.47	0.4848(6)	–0.1564(8)	0.3343(6)	0.066(3)
C(39A)	2*i*	0.47	0.674(1)	–0.3075(7)	0.3986(6)	0.086(3)
C(37B)	2*i*	0.21(1)	0.627(2)	–0.225(1)	0.2453(5)	0.031(4)
C(38B)	2*i*	0.21	0.5058(9)	–0.193(1)	0.3620(9)	0.058(5)
C(39B)	2*i*	0.21	0.714(2)	–0.3257(7)	0.3741(9)	0.106(7)
C(37C)	2*i*	0.32(1)	0.578(2)	–0.192(1)	0.2472(6)	0.070(4)
C(38C)	2*i*	0.32	0.542(1)	–0.229(1)	0.3867(7)	0.078(4)
C(39C)	2*i*	0.32	0.742(1)	–0.3283(7)	0.3417(9)	0.088(4)
H(43)	2*i*		0.4850(3)	0.3706(3)	0.0657(2)	0.056
H(45)	2*i*		0.6379(3)	0.0571(3)	0.1528(2)	0.056
H(46A)	2*i*		0.9935(3)	0.2832(3)	0.0967(2)	0.057
H(46B)	2*i*		0.8816(3)	0.3089(3)	0.0442(2)	0.057
C(50A)	2*i*	0.26(2)	0.416(2)	0.219(2)	–0.0031(6)	0.093(5)
C(51A)	2*i*	0.26	0.432(2)	0.088(1)	0.112(1)	0.065(4)
C(52A)	2*i*	0.26	0.3121(9)	0.293(1)	0.104(1)	0.085(5)
C(50B)	2*i*	0.51(1)	0.454(1)	0.173(2)	0.0009(6)	0.082(3)
C(51B)	2*i*	0.51	0.404(1)	0.111(1)	0.1356(8)	0.062(3)
C(52B)	2*i*	0.51	0.3138(7)	0.3040(6)	0.070(1)	0.063(3)
C(50C)	2*i*	0.23(2)	0.466(2)	0.130(2)	0.022(1)	0.086(6)
C(51C)	2*i*	0.23	0.384(2)	0.144(2)	0.1551(8)	0.067(5)
C(52C)	2*i*	0.23	0.333(1)	0.303(1)	0.039(1)	0.084(6)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	2*i*	0.56295(8)	0.47716(7)	0.38046(5)	0.0538(5)	0.0366(5)	0.0455(5)	–0.0221(3)	–0.0071(3)	–0.0073(3)
S(2)	2*i*	0.66996(8)	0.04630(7)	0.48296(5)	0.0625(5)	0.0356(5)	0.0422(4)	–0.0203(4)	0.0055(4)	–0.0086(3)
S(3)	2*i*	0.87753(7)	0.01364(6)	0.20424(4)	0.0358(4)	0.0347(4)	0.0463(4)	–0.0104(3)	0.0030(3)	–0.0084(3)
S(4)	2*i*	0.66370(8)	0.44589(6)	0.08218(5)	0.0505(5)	0.0330(5)	0.0497(5)	–0.0099(3)	0.0079(3)	–0.0085(3)
O(1)	2*i*	0.7042(2)	0.3731(2)	0.2492(1)	0.044(1)	0.034(1)	0.059(1)	–0.0060(9)	–0.007(1)	–0.007(1)
O(2)	2*i*	0.9835(3)	0.3709(3)	0.2015(2)	0.048(2)	0.092(3)	0.119(3)	–0.028(2)	0.007(2)	–0.047(2)
O(3)	2*i*	0.8138(3)	0.5219(3)	0.1930(2)	0.072(2)	0.071(2)	0.092(2)	–0.028(2)	0.003(2)	–0.015(2)
O(4)	2*i*	0.7193(2)	0.2472(2)	0.4537(1)	0.041(1)	0.042(1)	0.052(1)	–0.0154(9)	0.0005(9)	–0.017(1)
O(5)	2*i*	0.9373(3)	0.2352(3)	0.4897(2)	0.064(2)	0.135(3)	0.089(2)	–0.056(2)	0.024(2)	–0.061(2)
O(6)	2*i*	0.8601(2)	0.3100(2)	0.5882(2)	0.053(2)	0.065(2)	0.067(2)	–0.021(1)	–0.008(1)	–0.027(1)
O(7)	2*i*	0.8153(2)	0.0927(2)	0.3493(1)	0.044(1)	0.035(1)	0.051(1)	–0.0196(9)	–0.0027(9)	–0.0095(9)
O(8)	2*i*	1.1091(3)	–0.0684(2)	0.3514(2)	0.077(2)	0.049(2)	0.088(2)	–0.001(2)	0.007(2)	–0.006(2)
O(9)	2*i*	1.0451(2)	0.1070(2)	0.3062(2)	0.049(1)	0.050(2)	0.080(2)	–0.023(1)	0.013(1)	–0.021(1)
O(10)	2*i*	0.8732(2)	0.2301(2)	0.1466(1)	0.038(1)	0.042(1)	0.048(1)	–0.0163(9)	0.0019(9)	–0.0131(9)
C(1)	2*i*	0.6028(3)	0.4679(2)	0.2293(2)	0.037(2)	0.030(2)	0.050(2)	–0.010(1)	–0.003(1)	–0.006(1)
C(2)	2*i*	0.5672(3)	0.5053(3)	0.1533(2)	0.041(2)	0.031(2)	0.047(2)	–0.010(1)	0.000(1)	–0.009(1)
C(3)	2*i*	0.4568(3)	0.5946(3)	0.1307(2)	0.044(2)	0.040(2)	0.044(2)	–0.008(1)	–0.003(1)	–0.009(1)
C(4)	2*i*	0.3819(3)	0.6499(3)	0.1823(2)	0.043(2)	0.042(2)	0.048(2)	–0.009(1)	–0.002(1)	–0.013(1)
C(5)	2*i*	0.4190(3)	0.6125(3)	0.2575(2)	0.042(2)	0.037(2)	0.047(2)	–0.010(1)	0.001(1)	–0.014(1)
C(6)	2*i*	0.5269(3)	0.5219(2)	0.2817(2)	0.041(2)	0.033(2)	0.046(2)	–0.016(1)	–0.004(1)	–0.007(1)
C(7)	2*i*	0.8127(3)	0.3714(4)	0.2786(2)	0.042(2)	0.066(3)	0.067(2)	–0.003(2)	–0.013(2)	–0.012(2)
C(8)	2*i*	0.8802(4)	0.4190(4)	0.2205(3)	0.050(2)	0.060(3)	0.079(3)	–0.017(2)	–0.010(2)	–0.026(2)
C(9)	2*i*	0.8690(7)	0.5741(6)	0.1337(4)	0.119(5)	0.109(5)	0.114(5)	–0.067(4)	0.008(4)	–0.009(4)

Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(10)	2*i*	0.2594(3)	0.7467(3)	0.1586(2)	0.049(2)	0.060(2)	0.053(2)	0.008(2)	–0.008(2)	–0.017(2)
C(14)	2*i*	0.5193(3)	0.3678(3)	0.3994(2)	0.045(2)	0.041(2)	0.042(2)	–0.021(1)	0.001(1)	–0.011(1)
C(15)	2*i*	0.6022(3)	0.2649(3)	0.4333(2)	0.041(2)	0.040(2)	0.041(2)	–0.018(1)	0.003(1)	–0.014(1)
C(16)	2*i*	0.5678(3)	0.1801(3)	0.4411(2)	0.051(2)	0.037(2)	0.041(2)	–0.020(1)	0.005(1)	–0.010(1)
C(17)	2*i*	0.4515(3)	0.1987(3)	0.4170(2)	0.056(2)	0.047(2)	0.048(2)	–0.031(2)	0.004(2)	–0.012(1)
C(18)	2*i*	0.3661(3)	0.3011(3)	0.3864(2)	0.054(2)	0.055(2)	0.053(2)	–0.031(2)	–0.005(2)	–0.007(2)
C(19)	2*i*	0.4020(3)	0.3843(3)	0.3788(2)	0.047(2)	0.047(2)	0.052(2)	–0.020(2)	–0.008(1)	–0.006(1)
C(20)	2*i*	0.7262(3)	0.2770(3)	0.5220(2)	0.044(2)	0.050(2)	0.045(2)	–0.018(1)	–0.002(1)	–0.011(1)
C(21)	2*i*	0.8538(3)	0.2714(3)	0.5289(2)	0.052(2)	0.051(2)	0.054(2)	–0.024(2)	–0.002(2)	–0.014(2)
C(22)	2*i*	0.9812(4)	0.2986(5)	0.6059(3)	0.060(3)	0.105(4)	0.103(4)	–0.035(3)	–0.015(2)	–0.042(3)
C(23)	2*i*	0.2359(4)	0.3240(4)	0.3614(3)	0.074(3)	0.080(3)	0.102(4)	–0.050(3)	–0.033(3)	0.012(3)
C(27)	2*i*	0.7020(3)	–0.0120(3)	0.4019(2)	0.045(2)	0.035(2)	0.046(2)	–0.016(1)	0.003(1)	–0.009(1)
C(28)	2*i*	0.7730(3)	0.0177(2)	0.3444(2)	0.039(2)	0.027(2)	0.047(2)	–0.013(1)	–0.001(1)	–0.007(1)
C(29)	2*i*	0.7942(3)	–0.0260(2)	0.2809(2)	0.037(2)	0.034(2)	0.044(2)	–0.013(1)	–0.000(1)	–0.008(1)
C(30)	2*i*	0.7496(3)	–0.1023(3)	0.2775(2)	0.048(2)	0.038(2)	0.049(2)	–0.019(1)	0.002(1)	–0.015(1)
C(31)	2*i*	0.6806(3)	–0.1338(3)	0.3339(2)	0.052(2)	0.041(2)	0.055(2)	–0.025(2)	0.003(2)	–0.012(1)
C(32)	2*i*	0.6573(3)	–0.0864(3)	0.3965(2)	0.056(2)	0.042(2)	0.051(2)	–0.026(2)	0.009(2)	–0.011(1)
C(33)	2*i*	0.9230(3)	0.0532(3)	0.3971(2)	0.048(2)	0.052(2)	0.053(2)	–0.022(2)	–0.004(2)	–0.017(2)
C(34)	2*i*	1.0361(3)	0.0215(3)	0.3503(2)	0.044(2)	0.045(2)	0.057(2)	–0.014(1)	–0.007(2)	–0.013(2)
C(35)	2*i*	1.1413(4)	0.0876(4)	0.2525(3)	0.057(2)	0.083(3)	0.086(3)	–0.034(2)	0.020(2)	–0.025(2)
C(36)	2*i*	0.6302(4)	–0.2159(3)	0.3280(2)	0.084(3)	0.063(3)	0.067(2)	–0.051(2)	0.008(2)	–0.020(2)
C(40)	2*i*	0.7530(3)	0.1284(3)	0.1549(2)	0.037(2)	0.037(2)	0.042(2)	–0.012(1)	0.004(1)	–0.013(1)
C(41)	2*i*	0.7637(3)	0.2256(2)	0.1323(2)	0.033(1)	0.037(2)	0.040(2)	–0.011(1)	0.003(1)	–0.013(1)
C(42)	2*i*	0.6605(3)	0.3170(3)	0.1017(2)	0.041(2)	0.038(2)	0.042(2)	–0.011(1)	0.003(1)	–0.010(1)
C(43)	2*i*	0.5523(3)	0.3091(3)	0.0874(2)	0.034(2)	0.047(2)	0.049(2)	–0.008(1)	0.000(1)	–0.006(1)
C(44)	2*i*	0.5420(3)	0.2119(3)	0.1047(2)	0.041(2)	0.054(2)	0.051(2)	–0.020(2)	0.001(1)	–0.006(2)
C(45)	2*i*	0.6433(3)	0.1228(3)	0.1394(2)	0.045(2)	0.043(2)	0.052(2)	–0.021(1)	0.000(1)	–0.006(1)
C(46)	2*i*	0.9392(3)	0.2545(3)	0.0827(2)	0.043(2)	0.043(2)	0.058(2)	–0.020(1)	0.009(1)	–0.013(1)
C(47)	2*i*	1.0145(3)	0.1570(3)	0.0507(2)	0.038(2)	0.058(2)	0.047(2)	–0.018(2)	0.004(1)	–0.015(2)
C(48)	2*i*	1.1613(5)	0.0976(5)	–0.0417(3)	0.083(3)	0.106(4)	0.087(3)	–0.041(3)	0.040(3)	–0.057(3)
C(49)	2*i*	0.4275(4)	0.1988(3)	0.0819(3)	0.047(2)	0.080(3)	0.092(3)	–0.032(2)	–0.015(2)	0.014(2)

References

1. Akdas, H.; Mislin, G.; Graf, E.; Hosseini, M. W.; de Cian, A.; Fischer, J.: Synthesis and solid state structural analysis of conformers of tetrakis-((ethoxycarbonyl)methoxy)tetrathiacalix[4]arene. Tetrahedron Lett. **40** (1999) 2113-2116.
2. Gutsche, C. D.: Calixarenes revisited. Monographs in Supramolecular Chemistry (Ed. J. F. Stoddart). Royal Society of Chemistry, London 1998.
3. Vicens, J.; Böhmer, V. (Eds.): Calixarenes: a Versatile Class of Macrocyclic Compounds. Kluwer Academic Publishers, Dordrecht, The Netherlands 1991.
4. Sone, T.; Ohba, Y.; Moriya, K.; Kumada, H.; Ito, K.: Synthesis and properties of sulfur-bridged analogs of *p-tert*-butylcalix[4]arene. Tetrahedron **53** (1997) 10689-10698.
5. Kumagai, H.; Hasegawa, M.; Miyanari, S.; Sugawa, Y.; Sato, Y.; Hori, T.; Ueda, S.; Kamiyama, H.; Miyano, S.: Facile synthesis of *p-tert*-butylthiacalix[4]arene by the reaction of *p-tert*-butylphenol with elemental sulfur in the presence of a base. Tetrahedron Lett. **38** (1997) 3971-3972.
6. Mislin, G.; Graf, E.; Hosseini, M. W.; Bilyk, A.; Hall, A. K.; Harrowfield, J. M.; Skelton, B. W.; White, A. H.: Thiacalixarenes as cluster keepers: synthesis and structural analysis of a magnetically coupled tetracopper(II) square. Chem. Commun. (1999) 373-374.
7. Iki, N.; Morohashi, N.; Kabuto, C.; Miyano, S.: Coordination of epithio groups of *p-tert*-butylthiacalix[4]arene in a Zn^{2+} complex studied by X-ray crystallography. Chem. Lett. (1999) 219-220.
8. Iki, N.; Narumi, F.; Fujimoto, T.; Morohashi, N.; Miyano, S.: Selective synthesis of three conformational isomers of tetrakis(ethoxycarbonyl)-thiacalix[4]arene and their complexation properties towards alkali metal ions. J. Chem. Soc., Perkin Trans. **2** (1998) 2745-2750.
9. Lhoták, P.; Himl, M.; Pakhomova, S.; Stibor, I.: Tetraalkylated 2,8,14,20-tetrathiacalix[4]arenes: novel infinite channels in the solid state. Tetrahedron Lett. **39** (1998) 8915-8918.
10. Sheldrick, G. M.: SHELXTL, Version 5.1, Bruker-AXS, Madison, Wisconsin, USA 1999.