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ORIGINAL PAPER

Tetraallyl Ethers of Thiacalix[4]arenes in the 1,3-Alternate Conformation

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Abstract The *O*-alkylation of thiacalix[4]arene and of two derivatives substituted in *p*-position by NO_2 and *p*-N=N–C_6H_4–NO_2 with allylbromide leads to tetraallyl ethers in the 1,3-alternate conformation (**1**–**3**) as proved by X-ray crystallography. Compound **1** crystallized in the trigonal space group $P3_221$ with unit cell parameters $a = 10.9608(4)$ Å, $b = 10.9608(4)$ Å, $c = 24.6730(12)$ Å; $\alpha = 90°$, $\beta = 90°$, $\gamma = 120°$ and $Z = 3$. Compound **2** crystallized in the orthorhombic space group *Pbca* with unit cell parameters $a = 12.8608(4)$ Å, $b = 17.5209(5)$ Å, $c = 33.6527(9)$ Å; $\alpha = 90°$, $\beta = 90°$, $\gamma = 90°$ and $Z = 8$. Compound **3** crystallized in the monoclinic space group $P2_1/n$ with unit cell parameters $a = 18.7825(19)$ Å, $b = 17.6662(13)$ Å, $c = 19.7828(18)$ Å; $\alpha = 90°$, $\beta = 114.152(7)°$, $\gamma = 90°$ and $Z = 4$. Subtle differences in the molecular shape of the calix[4]arene core were found. The unsubstituted compound **1** forms three alternating layers with parallel tubes of different orientation, while for **3** all molecules are arranged in tubes parallel to the *a*-axis. Layers of molecules parallel to the *a*, *b*-plane are found for compound **2**.

Keywords Thiacalix[4]arenes · 1,3-Alternate · Allyl ethers · X-ray analysis

Introduction

Calix[4]arenes fixed in the cone-conformation by sufficiently sized ether residues (propyl or larger) and substituted in *p*-position by amino groups (e.g. [1]) are suitable educts for the introduction of various further functional groups, which may be attached e.g. via amide links. Tetra-CMPO (CMPO = carbamoylmethylphosphine oxide used here for the diphenyl substituted residue exclusively) derivatives [2], as an example, are excellent extractants for actinides or lanthanides. Tetra-picolylamides have been proposed for similar purposes [3]. Tetra-urea derivatives, on the other hand, are interesting due to their ability to form dimeric capsules [4, 5], which may be used also to construct larger self-assembled structures [6–8]. Thiacalix[4]arenes, in which phenolic units are connected via sulfur bridges, are somewhat larger and slightly different in shape [9]. This difference might be useful to change or to fine-tune selectivities for certain derivatives in comparison to their calix[4]arene analogues.

As starting material for the desired amino thiacalix[4]arenes we envisaged their *p*-nitro- or *p*-azo-analogues, from which the amino compounds finally should be available by reduction. To prepare their tetraethers, the more reactive allylbromide (compared to alkyl bromides) was chosen, since allyl ethers can be easily hydrogenated to propylethers. However, in both cases we obtained the tetrallyl ether in the 1,3-alternate conformation in

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reasonable to good yield. This result, not entirely unexpected [10], may be explained by the observation that the 1,3-alternate conformation is already found for the 1,3-diallylether (confirmed by a crystal structure [11]), in strong contrast to 1,3-diethers of "normal" calix[4]arenes, which prefer the cone conformation [12]. An explanation could be the compensation of dipole-moments of the aromatic units (nitrophenol and nitrophenylether) which might be stronger than the stabilization via intramolecular hydrogen bonds which usually determines the conformation of calix[4]arene (derivatives). The tetraallyether in the 1,3-alternate conformation was also obtained for the unsubstituted thiacalix[4]arene. (The 1,3-alternate conformation is not stable in $CDCl_3$ where it is slowly converted to the partial cone conformation already at room temperature. A complete transformation was achieved at 50 °C, where the partial cone isomer seems to be stable.)

Experimental

General Experimental

Melting points are uncorrected. ^1H and ^{13}C NMR spectra were recorded on a Bruker Avance DRX 400 spectrometer at 400 and 100.6 MHz respectively. Chemical shifts are reported in δ units (ppm) with reference to the residual solvent peaks. Mass spectra were recorded on a Waters/Micromass QTof Ultima 3 mass spectrometer. All solvents were HPLC grade and used without further purification.

As previously verified, [13, 14] data for elemental analyses of organic calixarenes are often misleading, due to inclusion of solvent molecules. Especially in the case of isomers, they cannot be considered appropriate criteria of purity. However, the identities of the reported compounds were unambiguously established by their spectroscopic data and their purity was controlled by TLC.

Syntheses

*25,26,27,28-Tetraallyloxy-2,8,14,20-thiacalix[4]arene (**1**)*

Allylbromide (2.86 g, 2.1 mL, 23.70 mmol) was added to the suspension of 25,26,27,28-tetrahydroxy-2,8,14,20-thiacalix[4]arene [15] (0.40 g, 0.81 mmol) and Na_2CO_3 (2.51 g, 23.70 mmol) in dry acetonitrile (16 mL) and the reaction mixture was refluxed for 7 days with stirring. 1 N HCl was added ($pH < 7$) and the aqueous layer was extracted with chloroform (3 × 20 mL). The combined organic fractions were dried ($MgSO_4$) and evaporated. Diethyl ether (25 mL) was added to the residue and the formed solid was filtered, washed with diethyl ether (3 × 5 mL) and dried for 1 h under vacuum (10 mmHg) at 100 °C. Compound **1** (0.24 g, 44%) was obtained as a colorless powder. Mp = 210–214 °C. ^1H NMR (400 MHz, $CDCl_3$, 25 °C): δ 4.56 (m, 8H, OCH_2), 4.73–4.96 (m, 8H, $CH{=}CH_2$), 5.64–5.77 (m, 4H, $CH{=}CH_2$), 6.74 (t, 4H, $^3J_{HH} = 7.7$ Hz, H_{Ar}), 7.34 (d, 8H, $^3J_{HH} = 7.7$ Hz, H_{Ar}). ^{13}C–{^1H} NMR (100.6 MHz, $CDCl_3$, 25 °C): δ 69.6 (s, OCH_2), 115.5 (s, $CH{=}CH_2$), 123.1 (s, C_{Ar}), 129.0 (s, C_{Ar}), 133.2 (s, $CH{=}CH_2$), 134.2 (s, C_{Ar}), 159.4 (s, C_{Ar}). MS (FD): m/z (%): 656.4 (100) $[M]^+$, calcd for $C_{36}H_{32}O_4S_4$ 656.91.

*5,11,17,23-Tetranitro-25,26,27,28-tetraallyloxy-2,8,14,20-thiacalix[4]arene (**2**)*

Allylbromide (2.86 g, 2.1 mL, 23.70 mmol) was added to the suspension of 5,11,17,23-tetranitro-25,26,27,28-tetrahydroxy-2,8,14,20-thiacalix[4]arene [16] (0.40 g, 0.59 mmol) and Na_2CO_3 (2.51 g, 23.70 mmol) in dry acetonitrile (16 mL) and the reaction mixture was refluxed for 5 days with stirring. 1 N HCl was added ($pH < 7$) and the aqueous layer was washed with chloroform (4 × 30 mL). The combined organic fractions were dried ($MgSO_4$) and evaporated. Diethyl ether (80 mL) was added and the crystalline residue was filtered, washed with diethyl ether (3 × 5 mL) and dried for 1 h under vacuum (10 mmHg) at r.t. The compound thus obtained (0.21 g, 47%) as a brown powder was identified as 5,11,17,23-tetranitro-25,27-diallyloxy-2,8,14,20-thiacalix[4]arene [11]. Mp = 185–189 °C. ^1H NMR (300 MHz, $CDCl_3$, 25 °C): δ 4.90 (d, 4H, $^3J_{HH} = 6.1$ Hz, OCH_2), 5.38–5.52 (m, 4H, $CH{=}CH_2$), 6.10–6.27 (m, 2H, $CH{=}CH_2$), 7.81 (s, 2H, OH), 8.05 (s, 4H, H_{Ar}), 8.65 (s, 4H, H_{Ar}). ^{13}C–{^1H} NMR (100.6 MHz, $CDCl_3$, 25 °C): δ 78.3 (s, OCH_2), 121.1 (s, $CH{=}CH_2$), 121.7 (s, C_{Ar}), 129.6 (s, C_{Ar}), 131.0 (s, $CH{=}CH_2$), 131.4 (s, C_{Ar}), 132.6 (s, C_{Ar}), 140.3 (s, C_{Ar}), 144.1 (s, C_{Ar}), 162.6 (s, C_{Ar}), 163.6 (s, C_{Ar}). MS (FD): m/z (%): 756.2 (100) $[M]^+$, calcd for $C_{30}H_{20}N_4O_{12}S_4$ 756.77.

The mother liquor was evaporated and the residue was triturated with acetone (10 mL). The insoluble part was filtered off and acetone was evaporated. Finally diethyl ether (10 mL) was added and the crystalline residue was filtered, washed with diethyl ether (3 × 2 mL) and dried for 2 h under vacuum (10 mmHg) at r.t. The tetraether **2** (0.05 g, 10%) was obtained as a colorless powder. Mp = 161–165 °C. ^1H NMR (400 MHz, $CDCl_3$, 25 °C): δ 4.57–4.98 (m, 16H, OCH_2 and $CH{=}CH_2$), 5.64–5.76 (m, 4H, $CH{=}CH_2$), 8.27 (s, 8H, H_{Ar}); ^{13}C–{^1H} NMR (100.6 MHz, $CDCl_3$, 25 °C): δ 70.3 (s, OCH_2), 117.1 (s, $CH{=}CH_2$), 129.2 (s, C_{Ar}), 129.4 (s, C_{Ar}), 131.2 (s, $CH{=}CH_2$), 142.9 (s, C_{Ar}), 163.9 (s, C_{Ar}). MS (ESI): m/z (%): 859.1 (100) $[M + Na]^+$, calcd for $C_{36}H_{28}N_4O_{12}S_4$ 836.90.

*5,11,17,23-Tetrakis(4-nitrophenylazo)-25,26,27,28-tetraallyloxy-2,8,14,20-thiacalix[4]arene (**3**)*

Allylbromide (2.20 g, 1.6 mL, 18.30 mmol) was added to the suspension of 5,11,17,23-tetrakis(4-nitrophenylazo)-25,26,27,28-tetrahydroxy-2,8,14,20-thiacalix[4]arene [17] (0.50 g, 0.46 mmol) and Na_2CO_3 (1.94 g, 18.30 mmol) in dry acetonitrile (10 mL) and DMF (3 mL) and the reaction mixture was refluxed for 3 days with stirring. 1 N HCl was added (pH < 7) and the aqueous layer was washed with chloroform (3 × 100 ml). The combined organic fractions were dried ($MgSO_4$) and evaporated. Diethyl ether (50 mL) was added to the residue and the formed precipitate was filtered, washed with diethyl ether (3 × 3 ml) and dried for 1 h under vacuum (10 mmHg) at r.t. Compound **3** (0.41 g, 72%) was obtained as a brownish powder. Mp = 180–183 °C. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ 4.60–4.80 (m, 16H, OCH_2 and $CH{=}CH_2$), 5.58–5.74 (m, 4H, $CH{=}CH_2$), 7.92 (d, 8H, $^3J_{HH} = 8.8$ Hz, H_{Ar}), 8.04 (s, 8H, H_{Ar}), 8.33 (d, 8H, $^3J_{HH} = 8.8$ Hz, H_{Ar}). $^{13}C\text{–}\{^1H\}$ NMR (100.6 MHz, $CDCl_3$, 25 °C): δ 70.0 (s, OCH_2), 116.4 (s, $CH{=}CH_2$), 123.3 (s, C_{Ar}), 124.8 (s, C_{Ar}), 128.8 (s, C_{Ar}), 129.7 (s, C_{Ar}), 131.9 (s, $CH{=}CH_2$), 147.5 (s, C_{Ar}), 148.8 (s, C_{Ar}), 155.5 (s, C_{Ar}), 162.2 (s, C_{Ar}). MS (FD): m/z (%): 1252.8 (100) $[M]^+$, calcd for $C_{60}H_{44}N_{12}O_{12}S_4$ 1253.35.

Crystal Structures Determination

Crystal and data collection parameters are given in Table 1. Data were collected on a STOE-IPDS-II two-circle diffractometer employing graphite-monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) with ω-scans. Data reduction was performed with the X-Area software [18]. An empirical absorption correction was performed using the MULABS [19] option in PLATON [20]. The structures were solved by direct methods with SHELXS-90 [21] and refined by

Table 1 Crystallographic data and refinement details for compounds **1**–**3**

Compound	1	2	3
CCDC deposition no.	766609	766610	766611
Empirical formula	$C_{36}H_{32}O_4S_4$	$C_{36}H_{28}N_4O_{12}S_4$	$C_{60}H_{44}N_{12}O_{12}S_4$
Formula weight (g/mol)	656.86	836.86	1253.31
Temperature (K)	173(2)	173(2)	173(2)
Radiation type/λ (Å)	Mo K_α/0.71073	Mo K_α/0.71073	Mo K_α/0.71073
Crystal system	Trigonal	Orthorhombic	Monoclinic
Space group	$P3_221$	$Pbca$	$P2_1/n$
Z	3	8	4
a (Å)	10.9608(4)	12.8608(4)	18.7825(19)
b (Å)	10.9608(4)	17.5209(5)	17.6662(13)
c (Å)	24.6730(12)	33.6527(9)	19.7828(18)
α (°)	90	90	90
β (°)	90	90	114.152(7)
γ (°)	120	90	90
Volume (Å^3)	2567.07(18)	7583.1(4)	5989.6(9)
Calculated density (Mg/m^3)	1.275	1.466	1.390
Absorption coeff. (mm^{-1})	0.315	0.319	0.232
F(000)	1032	3456	2592
Crystal size (mm)	0.46 × 0.44 × 0.42	0.48 × 0.45 × 0.44	0.42 × 0.12 × 0.07
$2\theta_{max}$ (°)	49.8	50.16	51.48
Reflections measured	26,618	80,191	43,335
Independent reflections	2953	6698	11,253
R_{int}	0.0406	0.0757	0.1765
Reflections with $I > 2\sigma(I)$	2875	5897	4176
Data/restraints/parameter	2953/0/200	6698/0/516	11,253/56/803
Weighting scheme[a] x/y	0.0469/0.2946	0.0467/4.3387	0.1573/0.0
Goodness-of-fit on F^2	1.069	1.027	0.986
R_1 [$I > 2\sigma(I)$]	0.0243	0.0379	0.1188
wR_2 [$I > 2\sigma(I)$]	0.0668	0.0962	0.2501
wR_2 [all data]	0.0678	0.1002	0.3422
$\Delta\rho_{max}$ (e/Å^3)	0.293	0.324	1.420

[a] $w = 1/[\sigma^2(F_o^2) + (x \times P)^2 + y \times P]$; $P = (F_o^2 + 2F_c^2)/3$

full-matrix least-squares techniques with SHELXL-97 [21]. All non-H atoms were refined with anisotropic displacement parameters. Hydrogens bonded to C were included at calculated positions and allowed to ride on their parent atoms. In **2** and **3**, one terminal vinyl group is disordered over two positions. For **3** restraints were used to keep the geometric parameters of some moieties in a reasonable range.

Results and Discussion

X-ray crystal structures could be obtained for the three thiacalix[4]arene tetraallyl ethers (**1**–**3**, Fig. 1) in the 1,3-alternate conformation. Single crystals of **1** and **2** (*p*-H, *p*-NO$_2$) were obtained by slow evaporation of a solution in acetone and of a chloroform solution in the case of the tetraazo compound **3**. No solvent was included in the crystal in all cases.

The molecular shape of the thiacalix[4]arene part is similar for all three compounds (and similar to other tetraethers of thiacalixarenes in the 1,3-alternate conformation) [22, 23]. The aromatic rings are bent slightly outwards, including angles of 80° (**1**), 72–82° (**2**) and 81–88° (**3**) with the molecular main plane, defined by the four sulfur atoms. For all compounds bond lengths and bond angles are in the usual range. Characteristic distances and angles are collected in Table 2. The shape of the three molecules is compared in Fig. 2.

In an ideal 1,3-alternate conformation, the four sulfur atoms should be placed on the corners of a regular square. This is (practically) the case for all compounds, since the distance between adjacent S-atoms differs by 0.024 Å for **1**, by max 0.05 Å for **2** and by max 0.017 Å for **3**. The length of the diagonals differs by 0.165 Å for **1** (indicating a rhombic shape) and by 0.133 Å for **3**, while practically no difference exists for **2**.

For comparison the diagonal distance of the phenolic oxygenes (O12–O32 and O22–O42) is distinctly larger for **2** ($\Delta = 0.403$ Å) than for **1** ($\Delta = 0.065$ Å) and **3** ($\Delta = 0.11$ Å), an order which is not reflected for carbon atoms in *p*-position of the phenolic units (opposite to the phenolic oxygen), which differ by $\Delta = 0.021$ Å for **1** and $\Delta = 0.092$ Å for **2**, while the larger difference of $\Delta = 0.565$ Å for **3** cannot be only due to the lower quality of this crystal structure. As can be seen in Fig. 2, this lower quality of **3** is mainly due to disorder in the azophenyl residues while the accuracy of the 1,3-alternate core is similar to **1** and **2**.

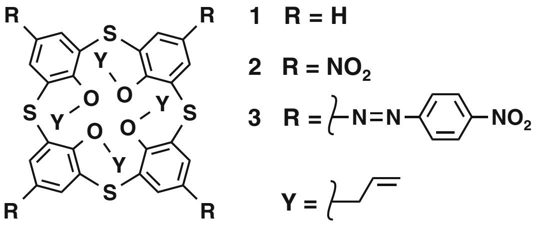


Fig. 1 Molecular structures of compounds **1**–**3**

Table 2 Selected distances (Å) and angles (°) for compounds **1**–**3**

	1	**2**	**3**
Distances			
O12–O32[a]	4.965	4.744	5.169
O22–O42[a]	4.900	4.341	5.062
C15–C35[a]	6.379	6.566	6.157
C25–C45[a]	6.358	6.474	5.590
N15–N35	–	7.079	6.480
N25–N45	–	6.979	5.587
S1–S2	5.555	5.538	5.546
S2–S3[a]	5.579	5.578	5.540
S3[a]–S4[a]	5.555	5.588	5.548
S4–S1	5.579	5.541	5.559
S1–S3[a]	7.780	7.864	7.777
S2–S4[a]	7.945	7.862	7.913
Bond angles			
C11–S1–C43	104.4	102.6	102.6
C21–S2–C13	104.0	104.7	101.5
C31–S3–C23	104.4	106.0	103.3
C41–S4–C33	104.0	104.2	102.8
S1–S2–S3	88.7	90.1	89.1
S2–S3–S4	91.1	89.5	91.1
S3–S4–S1	88.7	89.9	88.9
S4–S1–S2	91.1	90.4	90.9
Angles between planes			
m.p./C11–C16	80.0	81.6	85.0
m.p./C31–C36	80.0	73.8	81.2
m.p./C21–C26	80.2	72.0	88.0
m.p./C41–C46	80.2	81.5	87.2
C11–C16/C31–C36	19.93	24.6	13.8
C21–C26/C41–C46	19.60	26.5	4.9

m.p. main plane, defined by the four S-atoms

[a] For **1**: O32, O42, C35, C45, S3, S4 are the symmetry equivalents of O12, O22, C15, C25, S1, S2, respectively

The slight difference in the 1,3-alternate conformation of the three compounds is evidenced best by the angle between opposite aromatic rings of the calixarene skeleton. It is slightly higher (24.6°/26.5°) for **2** than for **1** (19.9°/19.6°) and lowest for **3** (14°/5°), where both values also show the strongest deviation.

An interesting packing is found for compound **1**. As shown in Fig. 3 three layers of parallel columns or tubes are found which extend along the *a*-axis, the diagonal in the *a*,*b*-plane (the plane formed by the *a*- and *b*-axis) and the *b*-axis. This means, that these positions may be

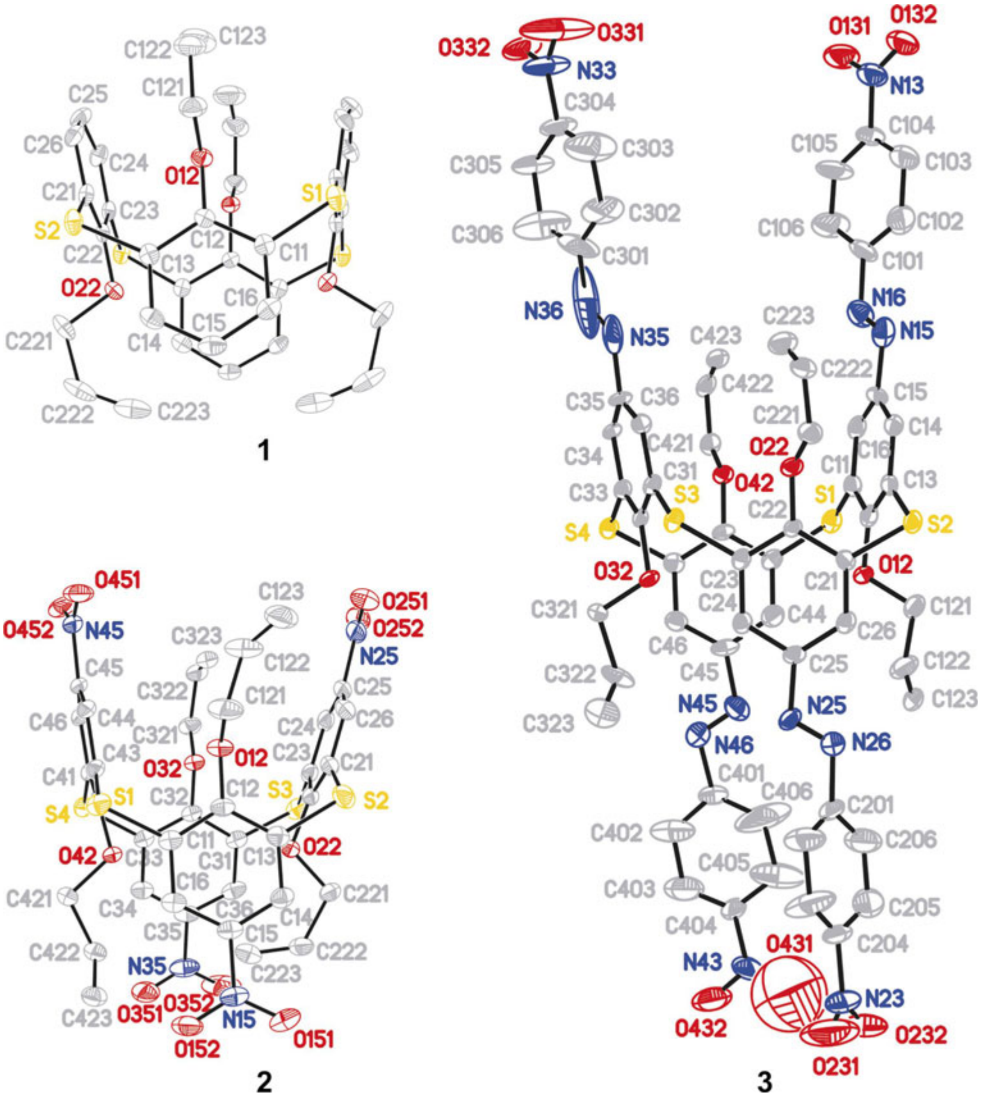
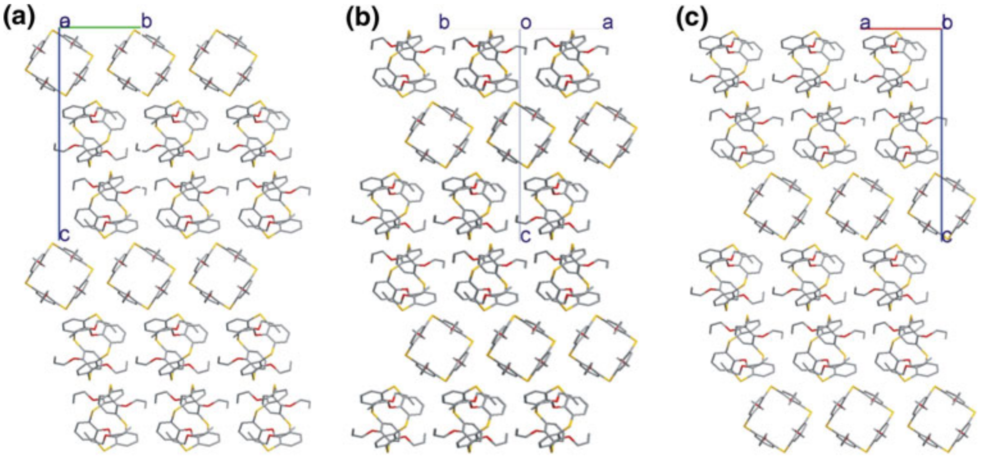


Fig. 2 Comparison of the molecular shape of compounds **1**–**3**. Hydrogen atoms are omitted for clarity

Fig. 3 Packing of molecules **1**, seen **a** along the *a*-axis, **b** along the "*a*–*b*" diagonal and **c** along the *b*-axis



interconverted by turning around the *c*-axis. Similar columns or tubes are formed by compound **3**, but in this case *all* tubes are parallel to the *a*-axis (Fig. 4b). Such an alignment of the molecular axes (perpendicular to the reference plane defined by the S-atoms) is not found in the crystal lattice of **2**. Here the packing is best described by

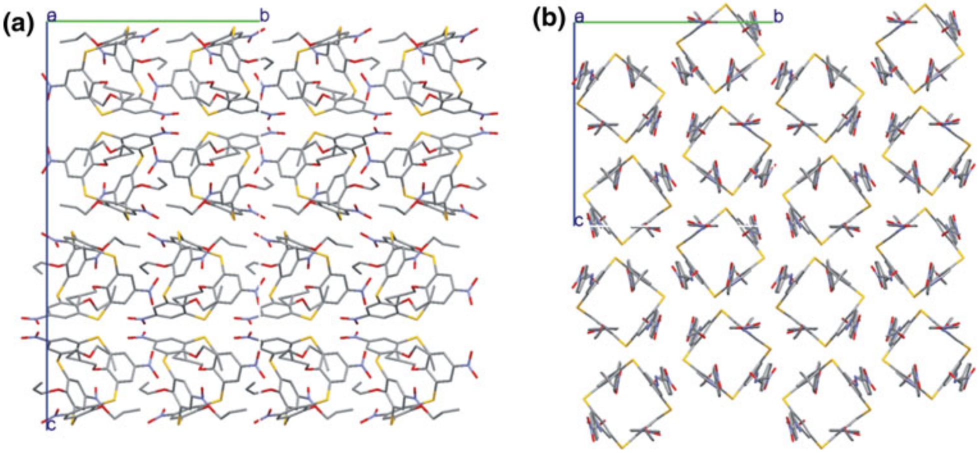


Fig. 4 Packing of molecules **a 2** and **b 3** seen along the *a*-axis

layers of molecules parallel to the *a*, *b*-plane, as shown in Fig. 4a.

Supplementary Material

CCDC-766609, CCDC-766610 and CCDC-766611 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

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