

Ganna Podoprygorina,^a Volker Böhmer^a and Michael Bolte^{b*}

^aFachbereich Chemie, Pharmazie und Geowissenschaften, Abteilung Lehramt Chemie, Johannes Gutenberg-Universität, Duesbergweg 10-14, 55099 Mainz, Germany, and ^bInstitut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, Germany

Correspondence e-mail: bolte@chemie.uni-frankfurt.de

Key indicators

Single-crystal X-ray study
 $T = 173$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 Disorder in main residue
 R factor = 0.048
 wR factor = 0.121
 Data-to-parameter ratio = 13.3

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Diisopropylethylammonium picrate

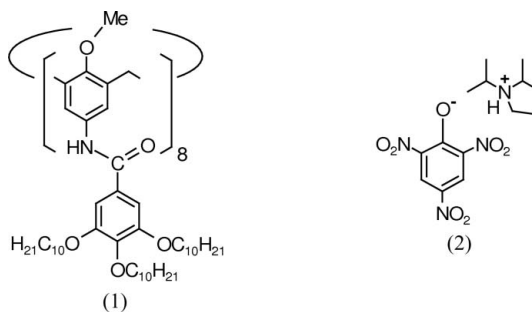
The structure of the title compound, $\text{C}_8\text{H}_{20}\text{N}^+\cdot\text{C}_6\text{H}_2\text{N}_3\text{O}_7^-$, consists of discrete diisopropylethylammonium cations and picrate anions. The protonated N atom of the cation forms a hydrogen bond to the phenol O atom of the picrate anion.

Received 20 November 2006

Accepted 21 November 2006

Comment

Calix[8]arene derivatives functionalized at their *para*-positions with residues allowing intermolecular hydrogen bonding or π - π stacking self-assemble as columnar structures on highly oriented pyrolytic graphite (Podoprygorina *et al.*, 2003; Zhang *et al.*, 2005). As shown by atomic force microscopy the octaamide (1) (prepared by acylation of the octaamino-calix[8]arene octamethylether with 3,4,5-tris-decyloxybenzoyl chloride in the presence of diisopropylethylamine as base) forms on the graphite surface domains of parallel-aligned columns, which are formed by single calixarene molecules stacked like a roll of coins. For a thorough study of the packing of the calix[8]arene molecules in the tubes we tried to obtain single crystals to determine their structure by X-ray crystallography.



The octaamide (1) was dissolved in THF and a few drops of ethanol were added to grow single crystals. Slow evaporation resulted in thin crystalline needles of the calix[8]arene, which were not appropriate for X-ray analysis. Since charge-transfer complexes formed between aromatic donor and acceptor systems are known to form crystals easily, picric acid was added to the solution of calixarene (1) in THF to obtain the respective charge-transfer complex. After dilution with *n*-hexane, single crystals suitable for X-ray analysis were obtained by slow evaporation. However, those crystals consisted of diisopropylethylammonium picrate (2). Obviously, they were formed from picric acid and traces of diisopropylethylamine, which was used in the synthesis of the calix[8]arene octaamide (1).

A perspective view of the title compound is shown in Fig. 1. Bond lengths and angles can be regarded as normal

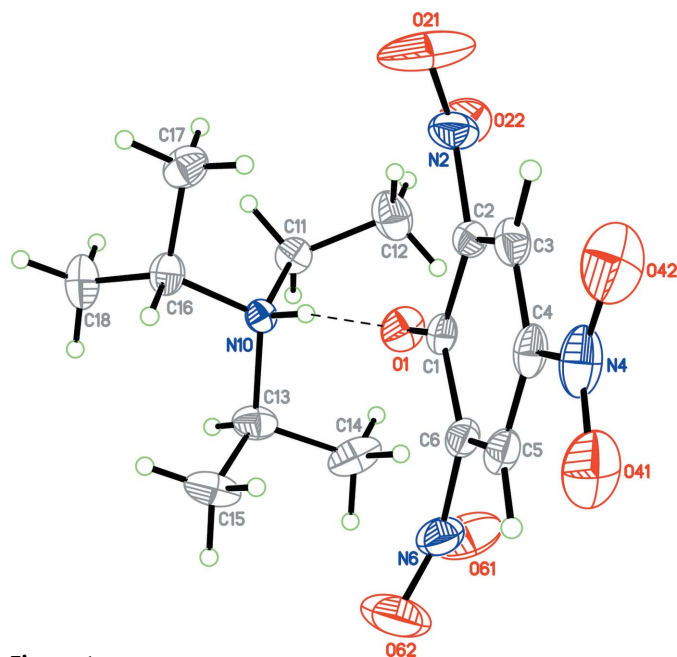


Figure 1

The asymmetric unit of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level. The minor-occupancy O atoms of the disordered nitro group are not shown. The dashed line indicates a hydrogen bond.

(Cambridge Structural Database; Version 5.27, November 2005 updated August 2006; Mogul Version 1.1; Allen, 2002). The structure of the title compound consists of discrete diisopropylethylammonium cations and picrate anions. The N atom of the cation is protonated and forms a hydrogen bond to the phenol O atom of the picrate anion.

Experimental

A sample of the octaamide, (1), obviously containing traces of diisopropylethylamine from its synthesis, was dissolved in THF. An excess of picric acid was added and the solution was diluted by *n*-hexane and slowly evaporated at room temperature. Diffraction quality crystals separated after several days.

Crystal data

$C_8H_{20}N^+ \cdot C_6H_2N_3O_7^-$
 $M_r = 358.36$
 Monoclinic, $P2_1/c$
 $a = 7.3769$ (10) Å
 $b = 11.3828$ (13) Å
 $c = 20.715$ (3) Å
 $\beta = 91.173$ (11)°
 $V = 1739.1$ (4) Å³

$Z = 4$
 $D_x = 1.369$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 173$ (2) K
 Rod, yellow
 $0.21 \times 0.12 \times 0.12$ mm

Data collection

Stoe IPDS-II two-circle diffractometer
 ω scans
 Absorption correction: none
 7320 measured reflections

3182 independent reflections
 2188 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.063$
 $\theta_{max} = 25.6^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.121$
 $S = 0.99$
 3182 reflections
 240 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0655P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.23$ e Å⁻³
 $\Delta\rho_{min} = -0.21$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.013 (2)

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N10—H10···O1	0.84 (2)	2.00 (3)	2.834 (2)	169 (2)

The N-bound H atom was refined freely. All other H atoms were located in a difference electron density map, but refined with fixed individual displacement parameters [$U_{iso}(H) = 1.2U_{eq}(C)$ or $U_{iso}(H) = 1.5U_{eq}(C_{methyl})$] using a riding model with C—H ranging from 0.95 to 1.00 Å. The O atoms of one nitro group are disordered over two positions with site occupation factors of 0.880 (8) and 0.120 (8). The minor occupied site was refined isotropically.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

Financial support by the Deutsche Forschungsgemeinschaft (SFB 625) is gratefully acknowledged.

References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
 Podoprygorina, G., Zhang, J., Brusko, V., Bolte, M., Janshoff, A. & Böhmer, V. (2003). *Org. Lett.* **5**, 5071–5074.
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
 Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
 Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.
 Zhang, J., Podoprygorina, G., Brusko, V., Böhmer, V. & Janshoff, A. (2005). *Chem. Mater.* **17**, 2290–2297.