

1-Undecyl-4,7-diaza-1-azoniatricyclo[5.2.1.0^{4,10}]-
decane iodideAndrew R. Battle and Lisandra L.
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The title compound, C₁₈H₃₆IN₃, crystallizes in a monoclinic setting. Packing for this compound results in a head-to-head configuration of the cation with the C₁₁ tails orienting themselves in an overlapping multilayer structure. This novel surfactant can be readily hydrolysed to provide a triaza-macrocylic ligand.

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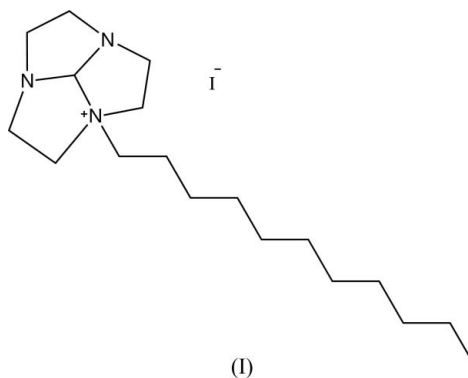
Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ (C—C) = 0.003 Å
R factor = 0.025
wR factor = 0.051
Data-to-parameter ratio = 24.1

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

Comment

Triazatricyclo[5.2.1.0^{4,10}]decane, tacn-orthoamide (tacnoa) has proved a very useful precursor for the synthesis of *N*-substituted derivatives of 1,4,7-triazacyclononane (tacn) (Atkins, 1980; Erhardt, *et al.*, 1980; Weisman *et al.*, 1980, 1987). These compounds readily form amidinium salts in aprotic solvents which are readily hydrolysed in water to form formyl derivatives. The functional group chemistry of tacn macrocycles and their derivatives has been extensively studied (Chaudhuri & Wiegardt, 1987). However, the excellent metal coordination characteristics of tacn have led to renewed interest, especially as components for protein immobilization for protein arrays (Johnson & Martin, 2005), drug delivery in medicine (Shi *et al.*, 2006) and DNA engineering (Hirohama *et al.*, 2004; Tamil *et al.*, 2005; Warden, *et al.*, 2005). We report a surfactant-like tacnoa derivative with a long C₁₁ chain on the tacn amidinium cation and an iodide anion as the counter-ion.



The compound crystallizes in a monoclinic setting ($P2_1/c$), with the asymmetric unit containing the amidinium cation and iodide anion. The molecular structure (Fig. 1) shows the three N atoms oriented facially to form a bridge with C7. The C—N and C—C bond lengths and angles (Table 1) are not atypical with the exception of the C7—N bond lengths, in particular the long N1—C7 bond length [1.682 (3) Å]. This results in both the N2—C7 and N3—C7 bonds being slightly shorter than average Csp^3 —N distances [1.406 (3) Å and 1.411 (3) Å, respectively]. This observation has also been noted in struc-

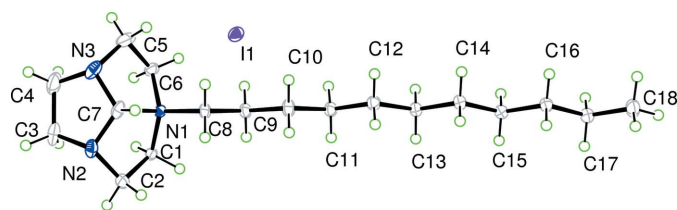


Figure 1
View of (I) (50% probability displacement ellipsoids).

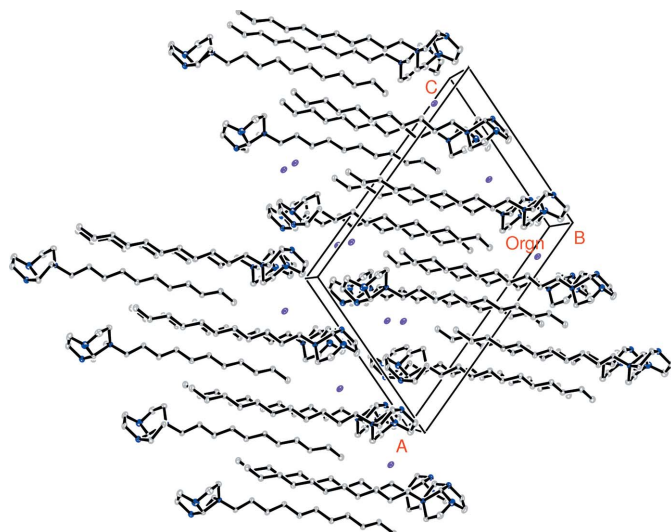


Figure 2
Crystal packing in (I).

tures of similar compounds (Battle & Spiccia, 2005; Farrugia *et al.*, 1993). The large charged polar head group and highly hydrophobic C_{11} tail results in a multilayered structure in which the tails are overlapping in two dimensions and the polar amindinium cations are oriented in a head-to-head fashion. The iodide ion occupies holes between the head groups (Fig. 2). The low-temperature data gave excellent displacement parameters, in particular for the C_{11} carbon chain.

Non-classical hydrogen bonding is also a feature. Bonds are formed between I1 and several neighboring C–H atoms. Four interactions with distances under 3.2 \AA occur, the closest being $3.01(1) \text{ \AA}$ [$C1-H1 \cdots I1(x, y+1, z)$].

Experimental

The title compound was synthesized using a general published procedure (Zhang *et al.*, 1995). Triazatricyclo[5.2.1.0^{4,10}]decane (0.41 g, 2.9 mmol) was dissolved in MeCN (10 ml) and the solution was stirred. To this solution was added iodoundecane (0.97 g, 3.45 mmol) in MeCN (5 ml) and the solution was stirred at room temperature for 18 h. Diethyl ether (20 ml) was introduced, causing a white precipitate to form. The solid was collected by vacuum filtration, washed with diethyl ether, and then air dried to yield (I). (Yield 1.13 g, 0.27 mmol; 93%). Single crystals suitable for diffraction were grown by vapour diffusion from a mixture of acetonitrile and diethyl ether. An approximately square plate ($0.12 \times 0.05 \times 0.01 \text{ mm}$) was selected for X-ray analysis.

Crystal data

$C_{18}H_{36}N_3^+ \cdot I^-$
 $M_r = 421.4$
Monoclinic, $P2_1/c$
 $a = 20.336(4) \text{ \AA}$
 $b = 7.0062(14) \text{ \AA}$
 $c = 14.748(3) \text{ \AA}$
 $\beta = 109.17(3)^\circ$
 $V = 1984.8(7) \text{ \AA}^3$

$Z = 4$
 $D_x = 1.41 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
 $\mu = 1.62 \text{ mm}^{-1}$
 $T = 123(2) \text{ K}$
Plate, colorless
 $0.12 \times 0.05 \times 0.01 \text{ mm}$

Data collection

Nonius KappaCCD diffractometer
 φ and ω thick slice scans
Absorption correction: multi-scan
(SADABS; Sheldrick, 2000)
 $T_{\min} = 0.830$, $T_{\max} = 0.984$

26808 measured reflections
4830 independent reflections
3967 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$
 $\theta_{\max} = 28.2^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.051$
 $S = 1.03$
4830 reflections
200 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0173P)^2 + 1.4994P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.62 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.42 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

N1–C8	1.504 (2)	N2–C7	1.406 (3)
N1–C7	1.682 (3)	N3–C7	1.411 (3)
C1–N1–C7	102.08 (14)	N2–C7–N3	110.85 (18)
N1–C8–C9	116.08 (16)	N2–C7–N1	106.00 (16)
N1–C1–C2	103.86 (16)		

H atoms were placed in calculated positions, the C–H bond distances ranging from 0.96 – 0.98 \AA , and were included in the refinement in the riding-model approximation with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

Data collection: COLLECT (Hooft, 2000); cell refinement: SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO (Otwinowski & Minor, 1997) and SCALEPACK; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

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