A long thin molecule, 2,6-dimethyl-6-tetradecyl-2-tetradecyloxy-1,3,6-dioxazocan-6-ium iodide

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An unusually long thin molecule is found in the crystal structure of 2,6-dimethyl-6-tetradecyl-2-tetradecyloxy-1,3,6-dioxazocan-6-ium iodide, $C_{35}H_{72}NO_3^+ \cdot I^-$. The length of the molecule results from two extended tetradecyl alkyl chains bonded to a central dioxazocine group. From H tip-to-tip it measures 40.336 (5) Å. At its waist, the maximum dimension is 6.770 (3) Å.
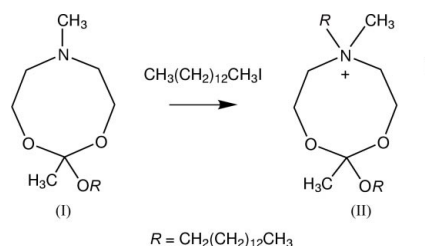
Comment

Compound (II) was synthesized from a dioxazocine ring and tetradecyl iodide, as shown in the Scheme below.

Since both *cis*- and *trans*-addition occur, the crystal structure of the major diastereomer (86%) was determined. It was found to be the *trans* form. Fig. 1 shows the somewhat remarkable dimensions of the molecule. All of the alkyl chains have torsion angles between 176.0 (2) and 180.0 (2)°. The distance from H22*B* to H36*B* is the longest, at 40.336 (5) Å. In the central portion of the molecule, the longest distance is 6.770 (3) Å from H1*C* to H6*C*, resulting in a molecule that is
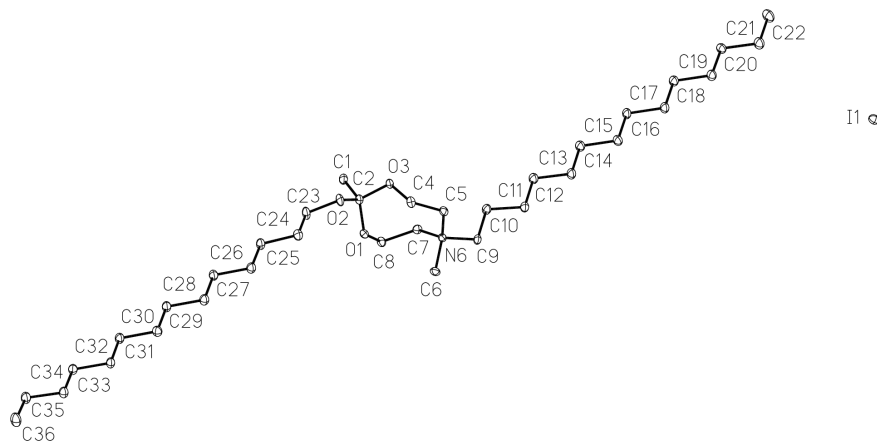

Figure 1
The molecular structure and atom-numbering scheme for (II), with displacement ellipsoids at the 50% probability level.
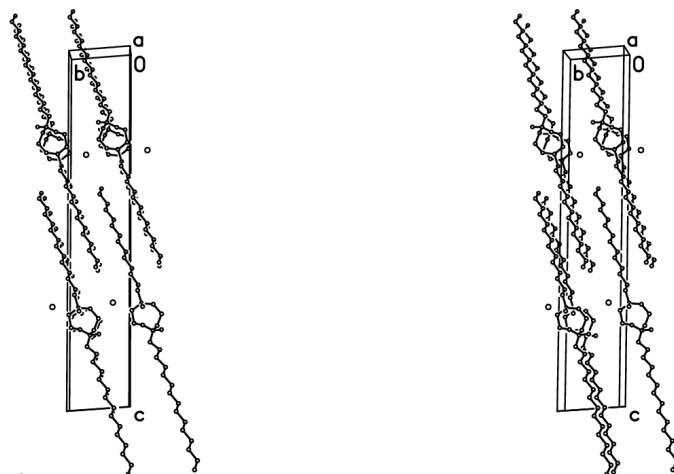
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Figure 2
A stereo-projection of the molecular packing, viewed down *a*.

six times longer than it is wide. The dominant feature in the packing, as shown in Fig. 2, is the overlay of parallel *n*-alkyl groups. The iodide is located in the region of the positively charged ring, forming the closest contact to H8*B* at 2.980 (2) Å. Fig. 3 shows how the zigzag array leads to a number of pairwise C···C contacts in the range 3.944 (3)–4.322 (3) Å. At the temperature of data collection, 91 (2) K, the only increase in thermal motion with position along the chain occurs at the two terminal C atoms. The average equivalent isotropic displacement parameter for the first 12 C atoms is 0.016 (1) Å^2; for the terminal two, it is 0.023 (2) Å^2.

Experimental

Myristyl iodide (1.63 g, 5.03 mmol) was added to a solution of (I) (600 mg, 1.68 mmol) in DMF (8 ml) at room temperature. After stirring for 18 h, diethyl ether was added slowly to the reaction solution to precipitate the product. The solids were filtered, washed with diethyl ether and then dried under vacuum to yield the ammonium iodide (II) (725 mg, 63%) as a 6:1 mixture of diastereomers. An analytical sample of the mixture was dissolved in CH$_2$Cl$_2$ and precipitated by slow addition of diethyl ether, yielding the major diastereomer. Recrystallization was carried out by slow evaporation from a solution in acetonitrile and CH$_2$Cl$_2$.
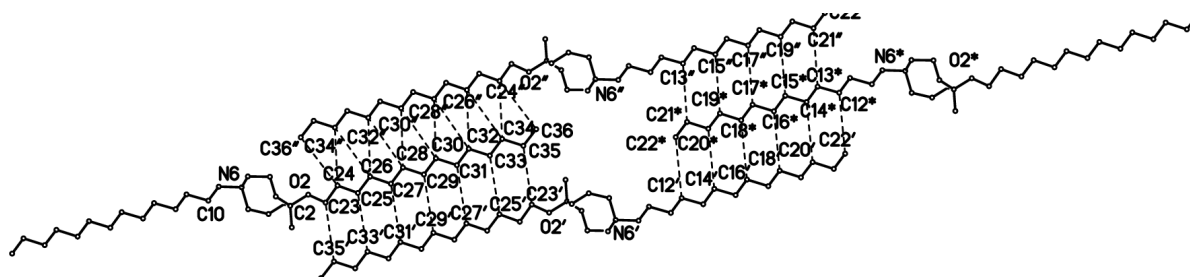
Crystal data

C$_{35}$H$_{72}$NO$_3^+$·I$^-$	Z = 2
M_r = 681.84	D_x = 1.234 Mg m^{-3}
Triclinic, $P\bar{1}$	Mo *K*α radiation
a = 6.0194 (7) Å	Cell parameters from 510
b = 7.2183 (8) Å	reflections
c = 42.384 (4) Å	θ = 2.4–30.7°
α = 85.527 (5)°	μ = 0.90 mm^{-1}
β = 88.810 (4)°	*T* = 91 (2) K
γ = 87.880 (4)°	Plate, colorless
V = 1834.4 (3) Å^3	0.28 × 0.12 × 0.04 mm

Data collection

Bruker SMART 1000 diffractometer	10499 independent reflections
ω scans	9098 reflections with *I* > 2σ(*I*)
Absorption correction: multi-scan	R_{int} = 0.037
(*SADABS*; Sheldrick, 2002)	θ$_{max}$ = 30.0°
T_{min} = 0.786, T_{max} = 0.965	*h* = −8 → 8
25283 measured reflections	*k* = −10 → 10
	l = −59 → 59

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0365P)^2$
R[$F^2 > 2\sigma(F^2)$] = 0.038	$+ 0.6504P]$
$wR(F^2)$ = 0.084	where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.001
10499 reflections	$\Delta\rho_{max}$ = 1.20 e Å^{-3}
365 parameters	$\Delta\rho_{min}$ = −1.31 e Å^{-3}
H-atom parameters constrained	

All H atoms were treated as riding, with C—H distances in the range 0.98–0.99 Å. The highest peak and deepest hole are 0.89 Å from I1 and 1.36 Å from H32*B*, respectively.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1994); software used to prepare material for publication: *SHELXL*97.

References

Bruker (2002). *SMART* (Version 5.054) and *SAINT* (Version 6.22). Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1994). *SHELXTL*. Version 5.1. Siemens Analytical X-ray Instruments, Madison, Wisconsin, USA.
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Figure 3
Close contacts between the alkyl chains in adjacent molecules. Dashed lines represent contacts in the range 3.94–4.10 Å.