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1,5-Di-*tert*-butyl-3-hydroxy-7-methylene-1-azonia-5-azacyclooctane iodide

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.007 Å
R factor = 0.044
wR factor = 0.117
Data-to-parameter ratio = 21.5

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

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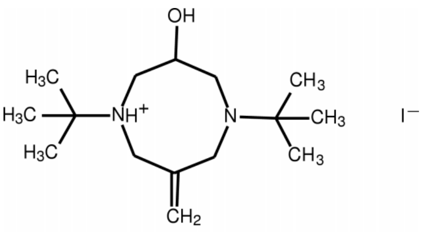
This paper reports the solid-state structure of the title compound, $C_{15}H_{31}IN_2O$. The molecule has a non-crystallographic mirror plane that accommodates all atoms except the hydroxyl-H and the H atom on the positively charged N atom.

Comment

Incorporating both the difluoramino and nitramino functional groups in the diazocine ring system is predicted to enhance the energy density of this system. The synthesis of such materials has been the focus of much effort in recent years. One approach that has been developed is to construct a precursor eight-membered ring with functional groups that can be readily transformed to the desired end product. In this case, it involved the cyclocondensation of 3-iodo-2-(iodomethyl)-1-propene with 1,5-di(*tert*-butylamino)-2-propanol to produce the title compound, (I), as an iodide salt.



(I)

The conformation of the eight-membered heterocyclic ring can be described as consisting of two fused envelopes having N1 and N5 in common. The flap of one envelope (C2, C3 and C4) is folded *cis* to the two *tert*-butyl groups, while the other flap (C6, C7 and C8) is folded *trans* to the *tert*-butyl groups. The molecule, excluding the H atoms on O3 and N5, could have a non-crystallographic mirror plane passing through C3, O3, C7 and C7a. Because of this near-symmetry it would not have been surprising to find the positive charge disordered across the two N atoms. However, the H atom on N5 was clearly visible in a difference map and there was no indication of a comparable peak near N1. Both available H atoms participate in hydrogen bonds. There is an intramolecular N5···N1 bond and an intermolecular O3···I1 bond (see Table 1).

Experimental

The title compound was produced as an iodide salt by the cyclocondensation of 3-iodo-2-(iodomethyl)-1-propene with 1,5-di(*tert*-butylamino)-2-propanol.

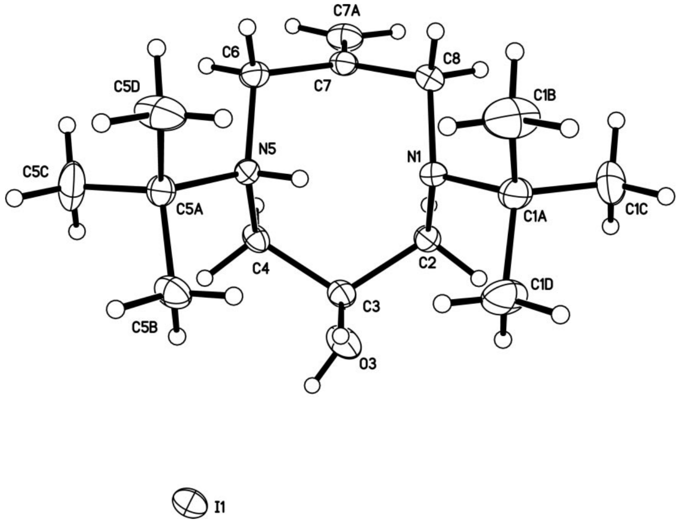


Figure 1
View of 1,5-di-*tert*-butyl-3-hydroxy-7-*exo*-methylene-1-azonia-5-azacyclooctane iodide with 20% probability ellipsoids.

Crystal data

$C_{15}H_{31}IN_2O$
$M_r = 382.33$
Monoclinic, $C2/c$
$a = 26.8737$ (10) Å
$b = 14.6191$ (6) Å
$c = 10.1557$ (4) Å
$\beta = 110.323$ (1)°
$V = 3741.5$ (3) Å^3
$Z = 8$

$D_x = 1.357$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8072 reflections
$\theta = 2.5$–$28.2°$
$\mu = 1.71$ mm^{-1}
$T = 294$ (2) K
Thick rod, colorless
0.48 × 0.40 × 0.16 mm

Data collection

Bruker SMART 1000 diffractometer
φ and ω scans
Absorption correction: by integration (*XPREP*; Bruker, 2001)
$T_{\min} = 0.757$, $T_{\max} = 0.815$
12311 measured reflections

3821 independent reflections
3074 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$
$\theta_{\max} = 26.4°$
$h = -33 \rightarrow 32$
$k = -18 \rightarrow 18$
$l = -12 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.117$
$S = 1.02$
3821 reflections
178 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0613P)^2 + 9.4177P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.010$
$\Delta\rho_{\max} = 1.01$ e Å^{-3}
$\Delta\rho_{\min} = -0.70$ e Å^{-3}

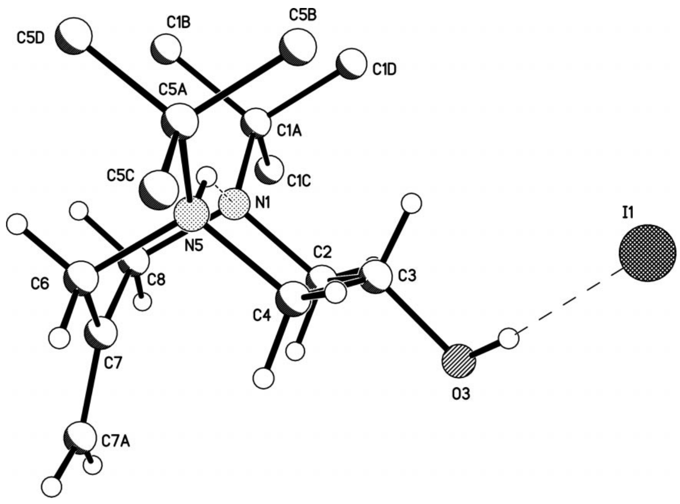


Figure 2
A second view of the molecule showing the double-envelope conformation of the eight-membered ring and the hydrogen bonding.

Table 1
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N5—H5$\cdots$N1	0.89 (5)	1.92 (5)	2.668 (5)	141 (4)
O3—H3$\cdots$I1	0.87 (7)	2.61 (7)	3.462 (4)	167 (6)

The coordinates of the H atoms (on N5 and O3) involved in hydrogen bonding were refined. All others were included in the refinement using a riding model.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*.

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References

Bruker (2001). *SMART* (Version 5.624), *SAINT* (Version 6.04) and *XPREP* for Windows NT. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.10. Bruker AXS, Inc., Madison, Wisconsin, USA.

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