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1-(2-Hydroxyethyl)-4-{4,5,6,7-tetrahydro-1-[1-(2-hydroxyethyl)-pyridin-4(1*H*)-ylidene]-1*H*-inden-3-yl}pyridinium iodide

Graeme J. Gainsford,* Andrew J. Kay and A. D. Woolhouse

Industrial Research Limited, PO Box 31-310, Lower Hutt, New Zealand

Correspondence e-mail: g.gainsford@irl.cri.nz

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Key indicators

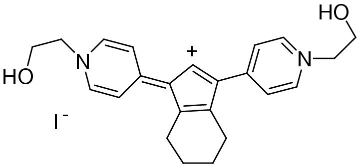
Single-crystal synchrotron study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.025
wR factor = 0.071
Data-to-parameter ratio = 8.8

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

The structure of the title compound, $C_{23}H_{27}N_2O_2^+\cdot I^-$, was determined from a 0.14 × 0.10 × 0.003 mm crystal using synchrotron X-radiation. The cation charge is delocalized and there is strong O—H···I⁻ intermolecular hydrogen bonding.

Comment

The title compound, (I), was prepared as part of a study into the use of zwitterionic merocyanines as potential non-linear optical (NLO) chromophores (Kay *et al.*, 2001). Initial single-crystal and powder diffraction studies using conventional X-ray sources showed that the larger crystals suffered from both twinning and different hydration states. Access to the X9B beamline at the National Synchrotron Light Source gave sufficient data with a crystallite 0.14 × 0.10 × 0.003 mm, leading to the ordered structure reported here. The crystal contains independent $C_{23}H_{27}N_2O_2^+$ cations (Fig. 1) linked in pairs about inversion centres by hydrogen bonds ($O—H\cdots I^-$, Table 2). Similar hydroxyethyl $O—H\cdots I^-$ interactions have been found in the Cambridge Structural Database (Allen, 2002) by use of *Conquest* (Bruno *et al.*, 2002), in entries QIGHET, a zwitterionic cyanine dye (Lacroix *et al.*, 2001), NUYDOA (Grobosch *et al.*, 1998) and ABILIG (Wang *et al.*, 2001). The pairs form a 'herring-bone' pattern along the *b* axis, with the normals to the cation molecular planes approximately in the [101] and [$\overline{1}$01] directions.



(I)

The bond distances illustrate both self-consistency (*e.g.* C8—C16 and C10—C21 in Table 1) and the molecule delocalization, spreading the overall positive charge, *e.g.* C8—C9 = 1.412 (5) Å and C9—C13 = 1.403 (5) Å. The main molecular fragment excluding the hydroxyethyl and atoms C1/C2/C17/C18/C19 is approximately planar, with an r.m.s. displacement of 0.023 (3) Å. The two pyridine rings are subtly different, with their mean planes inclined at 5.6 (2) and 1.5 (2)° (for the C3 and C13 rings, respectively) to the planar central five-membered ring (atoms C8/C9/C10/C21/C16). Likewise, there is a small difference in the dihedral angles within the hydroxyethyl groups, with N—C—C—O(H) values of 70.7 (4) and −59.1 (4)°. Such deviations are consistent with the observed intermoleculer hydrogen bonding.

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Experimental

The compound was prepared as previously described [compound 29*a* in Kay *et al.* (2001)].

Crystal data

$C_{23}H_{27}N_2O_2^{+}\cdot I^{-}$
$M_r = 490.37$
Monoclinic, $P2_1/c$
$a = 9.0520\ (18)$ Å
$b = 24.134\ (5)$ Å
$c = 9.5330\ (19)$ Å
$\beta = 96.22\ (3)^\circ$
$V = 2070.3\ (7)$ Å^3
$Z = 4$
$D_x = 1.573$ Mg m^{-3}
Synchrotron radiation, $\lambda = 0.9204$ Å
Cell parameters from 340 reflections
$\theta = 3.8$–22.5°
$\mu = 3.03$ mm^{-1}
$T = 100\ (2)$ K
Plate, black
$0.14 \times 0.10 \times 0.003$ mm

Data collection

Quantum4 CCD detector diffractometer
φ scans
Absorption correction: none
2300 measured reflections
2300 independent reflections
2191 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 29.0^\circ$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 25$
$l = -10 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.071$
$S = 1.06$
2300 reflections
262 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.025P)^2 + 2.8457P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.51$ e Å^{-3}
$\Delta\rho_{\min} = -0.58$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.412 (5)	C8—C9	1.412 (5)
N1—C1	1.342 (4)	C8—C16	1.464 (4)
N1—C6	1.484 (4)	C9—C10	1.403 (5)
C1—C2	1.363 (5)	C10—C21	1.470 (4)
C2—C3	1.420 (4)	C16—C21	1.364 (5)
C3—C8	1.420 (4)	C17—C18	1.532 (5)
C10—C9—C8	109.7 (3)	C10—C21—C20	128.3 (3)
C4—C3—C8—C9	−6.4 (5)	C3—C8—C16—C17	−9.2 (5)
C21—C10—C13—C14	0.9 (5)	C17—C16—C21—C20	6.5 (5)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2O···I1^{i}	0.82 (4)	2.69 (5)	3.459 (3)	158 (2)
O1—H1O···I1^{ii}	0.76 (4)	2.76 (5)	3.517 (3)	173 (5)

Symmetry codes: (i) $1-x, \frac{1}{2}+y, \frac{3}{2}-z$; (ii) $x-1, \frac{3}{2}-y, \frac{1}{2}+z$.

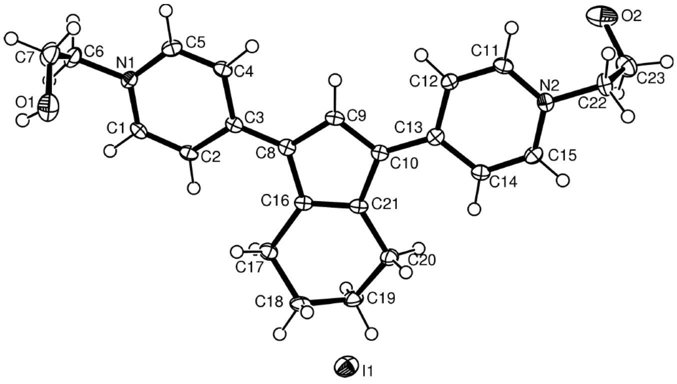

Figure 1
The asymmetric unit of (I) (Farrugia, 1997). Displacement ellipsoids are shown at the 50% probability level and H atoms are of arbitrary radii.

It was not possible to carry out an analytical absorption correction, as the crystal was not uniform or accurately measurable, and no empirical correction method was available for this experimental arrangement. H atoms on O1, O2 and C9 were refined freely, while others were constrained to geometrically calculated positions, riding on their parent atoms. For all H atoms, $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$ of the parent atom.

Data collection: *DENZO* (Otwinowski & Minor, 1997); cell refinement: *DENZO*; data reduction: *DENZO*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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