Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1052). Services for accessing these data are described at the back of the journal.

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3-(N-Benzyl-N-methylcarbamoyl)-1,2,4-trimethylquinolinium Iodide

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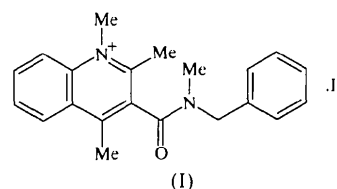
Abstract

The title compound, $C_{21}H_{23}N_2O^+.I^-$, possesses a central planar carbamoyl fragment in which the carbonyl and N-methyl groups are in *anti* positions. Located perpendicular to this central fragment on opposite sides are the N-benzyl group and the quinolinium fragment. This conformation leads to an axial chirality. The compound crystallizes as a racemate in a centrosymmetric space group.

Comment

In previous investigations, it has been shown that the rotation of an aromatic ring system about the connecting bond to a carbamoyl group is sterically hindered if there are two methyl groups in *ortho* positions. The ring is therefore stabilized in a conformation perpendicular to the carbamoyl group (Smeets *et al.*, 1988).

The benzyl group is a popular N-protecting group in organic chemistry. A total of 118 compounds bearing a benzyl-N(R)–C(O)R fragment can be found in the Cambridge Structural Database (October 1997 release; Allen & Kennard, 1993). A statistical analysis shows a nearly Gaussian distribution of the torsion angles of the N—C_{benzyl} bond about the maxima at 90 and −90°. Semi-empirical *MNDO* [Dewar & Thiel (1977); as implemented in *MOPAC*6.0 (Stewart, 1990)] calculations also suggest energy minima at torsion angles of 90 and −90°, with a rotational barrier of 3.5 kcal mol^{-1} (1 kcal mol^{-1} = 4.184 kJ mol^{-1}).

We report here the results of our analysis of the title compound, (I). A view of the molecule with the numbering scheme is shown in Fig. 1. We find a nearly perpendicular situation on both sides of the carbamoyl

(I)

group of the title compound involving the N-benzyl and quinolinium fragments [torsion angles C2—C1—C13—O 79.9 (3) and C13—N2—C15—C16 88.1 (3)°] resulting in an axial chirality. In contrast to similar compounds (van Hooff *et al.*, 1982; Bastiaansen *et al.*, 1986, 1988), the title compound does not undergo spontaneous enantiomeric resolution, but crystallizes as a racemate in the centrosymmetric space group $P2_1/c$. In accordance with these older investigations, the carbonyl and N-methyl groups are in *anti* positions in the carbamoyl group.
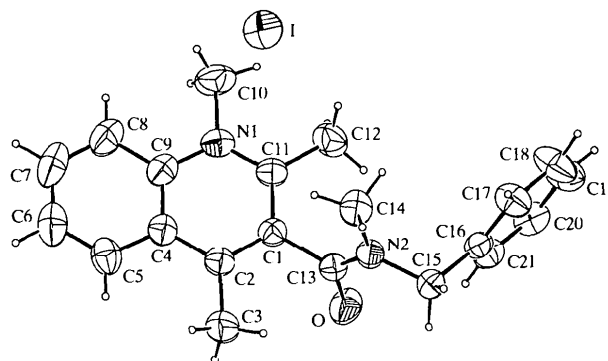

Fig. 1. *PLATON* (Spek, 1990) plot of the title molecule showing 50% probability displacement ellipsoids.

$C_{21}H_{23}N_2O^+.I^-$

The I^- anion is located 3.893 (2) Å from the N1 atom and 3.895 (3) Å from C10. These distances are much too long for bonding interactions and are about 0.2 Å longer than in comparable pyridinium compounds (Kanters *et al.*, 1986).

Experimental

The title compound was prepared according to the procedure described previously for the pyridinium analogue (Bastiaansen *et al.*, 1986).

Crystal data

$C_{21}H_{23}N_2O^+.I^-$	Mo $K\alpha$ radiation
$M_r = 446.31$	$\lambda = 0.71073$ Å
Monoclinic	Cell parameters from 25
$P2_1/c$	reflections
$a = 17.441 (2)$ Å	$\theta = 14.33–16.78°$
$b = 10.9569 (12)$ Å	$\mu = 1.646$ mm^{-1}
$c = 10.756 (3)$ Å	$T = 295 (2)$ K
$\beta = 107.690 (11)°$	Plate
$V = 1958.3 (6)$ Å^3	$0.95 \times 0.75 \times 0.45$ mm
$Z = 4$	Yellow
$D_x = 1.514$ Mg m^{-3}	
D_m not measured	

Data collection

Enraf–Nonius CAD-4 diffractometer	3150 reflections with $I > 2\sigma(I)$
$\omega/2\theta$ scans	$R_{int} = 0.016$
Absorption correction:	$\theta_{max} = 25°$
ψ scans (North *et al.*, 1968)	$h = -20 \rightarrow 19$
	$k = -13 \rightarrow 0$
$T_{min} = 0.33$, $T_{max} = 0.48$	$l = 0 \rightarrow 12$
3649 measured reflections	3 standard reflections
3448 independent reflections	frequency: 60 min
	intensity decay: none

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} = 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.029$	$\Delta\rho_{max} = 0.242$ e Å^{-3}
$wR(F^2) = 0.085$	$\Delta\rho_{min} = -0.668$ e Å^{-3}
$S = 1.136$	Extinction correction: none
3448 reflections	Scattering factors from
226 parameters	*International Tables for*
H atoms constrained	*Crystallography* (Vol. C)
$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2 + 1.0685P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. *Selected geometric parameters (Å, °)*

N1—I	3.893 (2)	C1—C13	1.514 (3)
N2—C13	1.334 (3)	C10—I	3.895 (3)
N2—C14	1.459 (3)	C13—O	1.232 (3)
N2—C15	1.470 (3)	C15—C16	1.512 (3)
C13—N2—C14	124.1 (2)	O—C13—C1	118.3 (2)
C13—N2—C15	118.9 (2)	N2—C13—C1	118.0 (2)
C14—N2—C15	116.8 (2)	N2—C15—C16	111.68 (19)
O—C13—N2	123.7 (2)		

C14—N2—C13—O	179.8 (3)	C2—C1—C13—N2	−100.1 (3)
C15—N2—C13—O	4.2 (4)	C11—C1—C13—N2	84.8 (3)
C14—N2—C13—C1	−0.2 (4)	C13—N2—C15—C16	88.1 (3)
C15—N2—C13—C1	−175.8 (2)	C14—N2—C15—C16	−87.8 (3)
C2—C1—C13—O	79.9 (3)	N2—C15—C16—C17	91.8 (3)
C11—C1—C13—O	−95.2 (3)	N2—C15—C16—C21	−87.8 (3)

The X-ray data were collected on a larger than usual plate crystal using a sufficiently large collimator tube to ensure a homogeneous X-ray beam at the crystal. The adaptation of the collimator to the crystal size is possible here in view of the use of a β-filter as opposed to a graphite monochromator (Alexander & Smith, 1962).

Data collection: locally modified *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *SET4* (de Boer & Duisenberg, 1984). Data reduction: *HELENA* (Spek, 1997). Program(s) used to solve structure: *DIRDIF96* (Beurskens *et al.*, 1996). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *PLATON* (Spek, 1990). Software used to prepare material for publication: *PLATON*.

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