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Benzophenone iminium tetrafluoroborate

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Key indicators

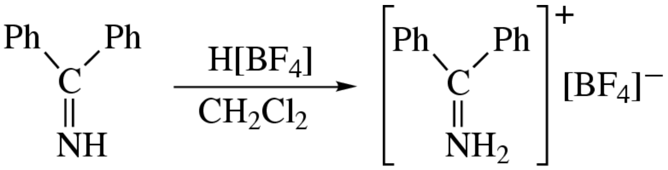
Single-crystal X-ray study
T = 200 K
Mean σ(C–C) = 0.004 Å
R factor = 0.060
wR factor = 0.196
Data-to-parameter ratio = 10.5

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

The reaction of benzophenone imine with an excess of tetrafluoroboric acid in dichloromethane yielded the title compound, $C_{13}H_{12}N^{+}\cdot BF_4^{-}$. The cation is non-planar and the crystal structure is stabilized by N—H···F and C—H···F short contacts.

Comment

Interest in the reactivity of late-transition-metal–amido complexes has grown considerably in the recent years, as a consequence of the relative scarcity of such compounds and of their potential use in C—N bond-forming reactions (Cabeza *et al.*, 1998). In this field, we have recently reported the first insertion of a non-activated alkyne into a metal–nitrogen bond, achieved with a triruthenium cluster derived from benzophenone imine (Cabeza *et al.*, 1997). In an extension of the interesting reactivity observed for these ruthenium complexes, we have studied the reactivity of benzophenone imine–ruthenium complexes with alkyne ligands and protic acids. In this context, the reaction of the binuclear ruthenium complex $[Ru_2(N{=}CPh_2)(CPh{=}CHPh)(CO)_6]$ (Cabeza *et al.*, 1997) with an excess of tetrafluoroboric acid under a CO atmosphere gave the title compound as a by-product. This can also be prepared in high yield by treating benzophenone imine with an excess of tetrafluoroboric acid in dichloromethane.



(I)

The cation is non-planar; dihedral angle between the two phenyl rings is 57.8 (2)°. The crystal structure is stabilized by N—H···F and C—H···F short contacts (Table 2).

Experimental

An excess of tetrafluoroboric acid (54% wt in diethyl ether, *ca* 0.1 ml) was added to a solution of benzophenone imine (103 µl, 0.614 mmol) in dichloromethane (20 ml). The mixture was stirred at room temperature for 5 min and the solvent removed under reduced pressure. The white residue was washed with diethyl ether (2 × 10 ml) and dried *in vacuo* to afford 155 mg (94%) of the title compound. Crystallization from CH_2Cl_2/hexane at room temperature was achieved by slow liquid–liquid diffusion.

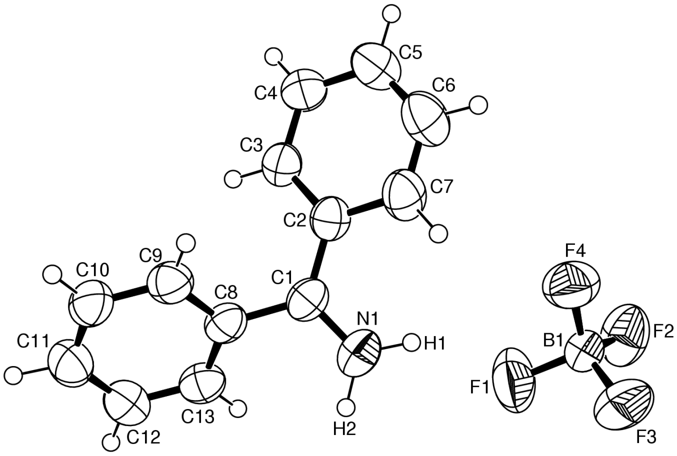


Figure 1
A view of the title compound. Displacement ellipsoids are shown at the 50% probability level.

Crystal data

$C_{13}H_{12}N^{+}\cdot BF_4^{-}$
$M_r = 269.05$
Monoclinic, $P2_1/n$
$a = 5.9727 (4)$ Å
$b = 15.082 (1)$ Å
$c = 14.336 (1)$ Å
$\beta = 91.895 (4)^\circ$
$V = 1290.72 (15)$ Å^3
$Z = 4$

$D_x = 1.385$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 2348 reflections
$\theta = 2$–70°
$\mu = 1.05$ mm^{-1}
$T = 200 (2)$ K
Block, white
0.25 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
φ scans
Absorption correction: none
11038 measured reflections
2306 independent reflections
1624 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.065$
$\theta_{\rm max} = 68.4^\circ$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 18$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.196$
$S = 1.10$
2306 reflections
220 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.1069P)^2 + 0.3137P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.32$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.28$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.300 (3)	C1—C8	1.474 (4)
C1—C2	1.460 (4)		
C8—C1—C2—C3	−28.8 (4)	C2—C1—C8—C9	−38.0 (4)
N1—C1—C2—C7	−26.8 (4)	N1—C1—C8—C13	−36.7 (4)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···F1	0.93 (4)	1.96 (4)	2.786 (4)	147 (4)
N1—H2···F3$^{\rm i}$	0.97 (5)	1.83 (4)	2.804 (3)	175 (4)
C7—H7···F4	0.95 (3)	2.43 (3)	3.230 (4)	142 (2)
C11—H11···F4$^{\rm ii}$	1.02 (4)	2.31 (4)	3.183 (4)	143 (3)

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

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985); 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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