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Bis(pyridine)iodonium(I) tetrafluoroborate

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The title compound, $C_{10}H_{10}IN_2^+\cdot BF_4^-$, or IPy_2BF_4, is found to crystallize in the monoclinic crystal system. The asymmetric unit contains two tetrafluoroborate anions, one independent IPy_2 cation and two half-cations; for the latter the I atoms lie on centres of symmetry. A slight deviation from planarity is observed in the crystallographically independent IPy_2 cation.

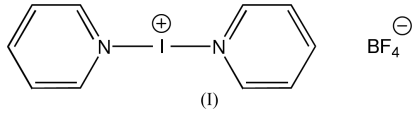
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.007 Å
R factor = 0.036
wR factor = 0.141
Data-to-parameter ratio = 14.7

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

Comment

The title compound, (I), is a well known iodination reagent in organic synthesis (Barluenga *et al.*, 1985; Barluenga, 1999).



(I)

The crystal structures of some related compounds have already been published (Hassel & Hope, 1961; Brayer & James, 1982). However, the crystal structure of IPy_2BF_4 has not been reported to date.

This compound is found to crystallize in the monoclinic space group $P2_1/c$. It is of note that the I atoms in the crystal cell exhibit a 'frustrated' $C2/m$ symmetry, which would result in a new cell given by the transformation $a' = a$, $b' = b/2$ and $c' = c/2$, with only one I atom per asymmetric unit. In the actual unit cell ($P2_1/c$ symmetry), the I atom corresponding to the independent IPy_2 cation (I1) occupies a position with coordinates [0.48947 (3), 0.74618 (2), 0.99242 (2)], quite close to the special position ($\frac{1}{2}$, $\frac{3}{4}$, 1), which could have generated a centred cell. All attempts to solve the crystal structure using the transformed unit cell failed. Furthermore, a careful visual inspection of the X-ray diffraction pattern, by superposition of the predicted reflections on the experimental ones, revealed that several strong reflections were not being indexed when the centred cell was imposed. The choice of the primitive cell

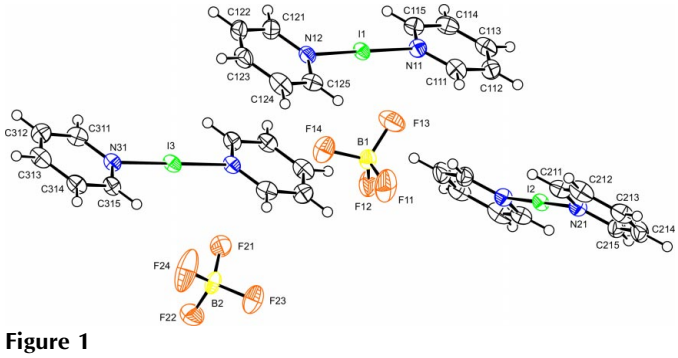


Figure 1
View of the independent ions in the asymmetric unit. The non-labelled atoms were generated by application of the crystal symmetry. Ellipsoids are drawn at the 50% probability level.

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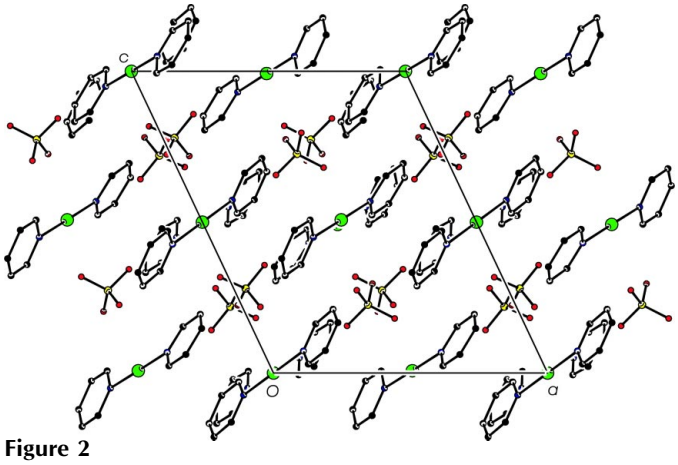

Figure 2
Packing diagram of the crystal structure, viewed down the monoclinic *b* axis.

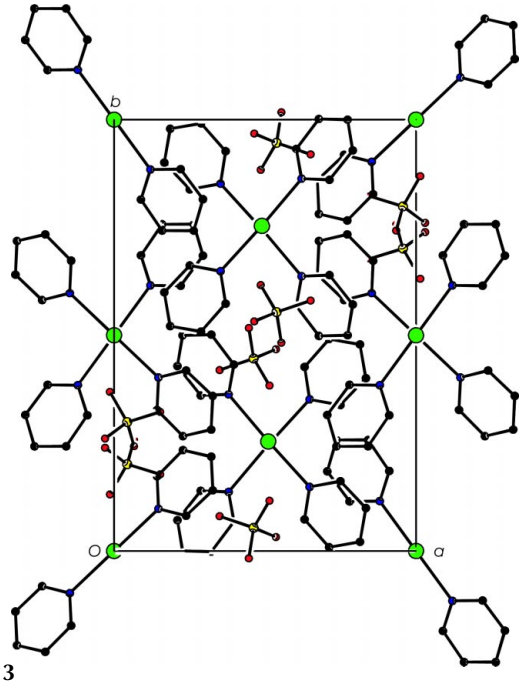

Figure 3
Packing diagram, viewed down the *c* axis, showing the pseudosymmetry defined by the I atoms.

is reinforced by the fact that the spatial arrangement of the remainder of the unit-cell contents, *viz.* the pyridine rings and the counter-anions BF_4, deviates from the pseudosymmetry defined by the I atoms.

The whole IPy_2 cation is basically planar. The torsion angle between the planes of the two pyridines is, of course, exactly 0° in those IPy_2 cations in which the I atom (I2 and I3) lies on a centre of symmetry. A slight deviation from this value [9.7 (2)°] is observed in the other independent IPy_2 cation in the unit cell.

Experimental

Crystals of (I) were grown at ambient temperature by slow diffusion of diethyl ether into a solution of the compound in dichloromethane. These crystals appeared to be light-sensitive and deteriorate after a few days in the presence of visible light. Care was therefore taken during the whole process to reduce to a minimum the exposure of the crystals to direct ambient light.

Crystal data

$C_{10}H_{10}IN_2^+\cdot BF_4^-$	D_x = 1.911 Mg m^{-3}
M_r = 371.91	Cu $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 5040 reflections
a = 12.1840 (4) Å	θ = 2.0–70.0°
b = 15.7830 (4) Å	μ = 19.80 mm^{-1}
c = 14.8550 (3) Å	T = 120 (2) K
β = 115.1760 (16)°	Plate, colourless
V = 2585.25 (12) Å^3	0.10 × 0.05 × 0.02 mm
Z = 8	

Data collection

Nonius KappaCCD diffractometer	3369 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.029
Absorption correction: refined from ΔF (*XABS*2; Parkin *et al.*, 1995)	θ_{max} = 69.8°
T_{min} = 0.416, T_{max} = 0.673	$h = -14 \rightarrow 13$
9437 measured reflections	$k = 0 \rightarrow 19$
4821 independent reflections	$l = 0 \rightarrow 18$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.036	$w = 1/[\sigma^2(F_o^2) + (0.0731P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.141	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.10	$\Delta\rho_{max}$ = 0.97 e Å^{-3}
4821 reflections	$\Delta\rho_{min}$ = −1.35 e Å^{-3}
328 parameters	

Table 1
Selected geometric parameters (Å, °).

I1—N11	2.255 (3)	I2—N21	2.260 (3)
I1—N12	2.261 (3)	I3—N31	2.261 (3)
N11—I1—N12	177.66 (12)	N31—I3—N31^{ii}	180.0
N21^{i}—I2—N21	180.0		
I1—N11—C115—C114	178.7 (4)	I2—N21—C215—C214	179.5 (3)
I1—N12—C125—C124	−177.4 (3)	I3—N31—C315—C314	177.7 (3)

Symmetry codes: (i) $-x, 2-y, -z$; (ii) $2-x, 1-y, 2-z$.

The correct assignment of the space group was performed after a careful analysis of the experimental data. The reflection conditions corresponding to space group $P2_1/c$ were clearly fulfilled. In addition, some other doubtful systematic absences seemed to suggest that a possible centring was present in the crystal structure. However, the intensities of these reflections, although not very strong, could be considered as observed. All possible space groups compatible with these doubtful systematic absences were checked, but the crystal structure could only be solved when all of these absences were discarded and the correct space group, $P2_1/c$, was chosen. All H atoms were positioned geometrically and allowed to ride on their respective parent atoms, with U_{iso}(H) values equal to $1.2U_{eq}$(C). The maximum and minimum electron-density peaks are in the vicinity of atom I1.

Data collection: *COLLECT* (Nonius, 1997–2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2001); software used to prepare material for publication: *SHELXL*97.

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