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3-*tert*-Butyl-1-(2-pyridylmethyl)-3*H*-imidazolium bromide hydrate

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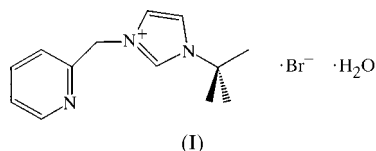
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The structure of the title compound, $C_{13}H_{18}N_3^+ \cdot Br^- \cdot H_2O$, shows that the cation and the bromide anion are linked *via* the water molecule through weak intermolecular hydrogen bonds. The water was not detected by NMR spectroscopy, but the cell content is in agreement with the elemental analysis. The presence of water plays an important role in further reactions.

Comment

It has been reported that mixed-donor carbene complexes of late transition metals, such as Pd, Ru and Rh, are highly active precatalysts for Heck coupling and other important reactions (Tulloch *et al.*, 2000). The title compound, (I), was synthesized in order to prepare *N*-heterocyclic carbene complexes, to act as novel catalysts. Because of a disagreement within the analytical data, obtained from elemental analysis and NMR spectroscopy, the pure compound was recrystallized from acetone. The cell content, obtained from the structure determination, agrees with the results of the elemental analysis, in that one molecule of water is associated with each ion pair. Intermolecular hydrogen bonding is observed between proton H321 from one methyl group of the *tert*-butyl group and the O atom of the water molecule. A proton from each of the other two methyl groups (H112 and H123) also points towards O1. There are also intermolecular hydrogen bonds to a pair of symmetry-equivalent bromide anions from the H atoms of the water molecule (Table 1).



Experimental

The title compound was prepared by the quarternization of 1-*tert*-butylimidazole with picolyl bromide in methanol. The crude product

was recrystallized by cooling a saturated solution of (I) in dried acetone to 238 K. 1H NMR (300 MHz, DMSO- d_6) δ 1.6 (9H, s, tBu), 5.6 (2H, s, CH_2), 7.6 and 8.6 (2 \times 1H, d, 4,5-imidazolium-CH), 7.5, 7.9, 8.0 and 8.1 (4 \times 1H, m, 3,4,5,6-picolyl-CH), 9.5 (H, s, 2-imidazolium-CH). LRMS (ESIPOS) m/z 216 (M^+). Analysis found: C 49.68, H 6.43, N 13.38%; $C_{13}H_{20}BrN_3O$ requires: C 49.69, H 6.42, N 13.37%.

Crystal data

$C_{13}H_{18}N_3^+ \cdot Br^- \cdot H_2O$
 $M_r = 314.23$
Monoclinic, $P2_1/c$
 $a = 9.7083$ (19) Å
 $b = 13.214$ (3) Å
 $c = 11.574$ (2) Å
 $\beta = 92.11$ (3)°
 $V = 1483.8$ (5) Å³
 $Z = 4$

$D_x = 1.407$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 12 783 reflections
 $\theta = 0.998$ – 26.373°
 $\mu = 2.764$ mm⁻¹
 $T = 150$ (2) K
Prism, colorless
 $0.20 \times 0.20 \times 0.13$ mm

Data collection

Enraf–Nonius KappaCCD area-detector diffractometer
 φ and ω scans to fill Ewald sphere scans
Absorption correction: multi-scan (SORTAV; Blessing, 1995)
 $T_{min} = 0.608$, $T_{max} = 0.724$
30 583 measured reflections

3021 independent reflections
2608 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.048$
 $\theta_{max} = 26.35^\circ$
 $h = -12 \rightarrow 12$
 $k = -16 \rightarrow 16$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.071$
 $S = 1.065$
3021 reflections
243 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0362P)^2 + 0.7258P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.41$ e Å⁻³
 $\Delta\rho_{min} = -0.64$ e Å⁻³

Table 1

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C13-H132 \cdots O1^i$	0.97 (3)	2.34 (3)	3.292 (3)	165 (2)
$O1-H2O \cdots Br1^{ii}$	0.98 (4)	2.33 (4)	3.305 (2)	175 (3)
$O1-H1O \cdots Br1$	0.98 (4)	2.33 (4)	3.301 (2)	171 (3)

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

All H atoms were located by difference Fourier synthesis and refined isotropically [$C-H = 0.89$ (2)– 1.05 (4) Å]. No constraints or restraints were applied to the structural models.

Data collection: COLLECT (Hooft, 1998); cell refinement and data reduction: SCALEPACK (Otwinowski & Minor, 1997); program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); software used to prepare material for publication: PLATON (Spek, 1990).

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