X-ray powder diffraction data for $[C_6Trop]^+[PF_6]^-$, $C_{14}H_{27}NOPF_6$

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Experimental X-ray powder diffraction data, unit-cell parameters, and space group for $[C_6Trop]^+[PF_6]^-$, $C_{14}H_{27}NOPF_6$, are reported [$a = 16.1879(6)$ Å, $b = 11.4271(2)$ Å, $c = 10.3966(5)$ Å, $\alpha = 66.4949(3)°$, $\beta = 94.5589(0)°$, $\gamma = 93.3092(4)°$, unit-cell volume $V = 1923.17$ Å^3, $Z = 1$, and space group $P1$]. No detectable impurities were observed. © *2014 International Centre for Diffraction Data*. [doi:10.1017/S0885715614000098]

Key words: X-ray powder diffraction (XRD), $[C_6Trop]^+[PF_6]^-$, $C_{14}H_{27}NOPF_6$

I. INTRODUCTION

$[C_6Trop]^+[PF_6]^-$ ($C_{14}H_{27}NOPF_6$, see Figure 1), systematic name 8-hexyl-3-hydroxy-8-methyl-nortropanium hexafluorophosphate is a new potential ionic liquid (IL) synthesized by us recently, which is a white powders at room temperature. The skeleton of its cation originates from 3-α-tropanol, which is an important chemical and pharmaceutical raw material (Dewick *et al.*, 2009; Goodman *et al.*, 2010). The C_6 chain on the N atom lowers the melting point of the salt. Moreover, the solubility of the IL in certain reaction and extraction mixture could be changed greatly with a small-scale variation in temperature, similar to other reported ionic liquids (Li *et al.*, 2012) with temperature-sensitive properties and broad application prospects. Benzatropine and etybenzatropine are the famous derivatives of tropanol, which is also a building block of atropine, a cholinergic drug prototypical of the muscarinic antagonist class. The title compound $[C_6Trop]^+[PF_6]^-$ is also expected to have some biological activities and medicinal value.

At present, the crystal structure of $[C_6Trop]^+[PF_6]^-$ has not been reported.

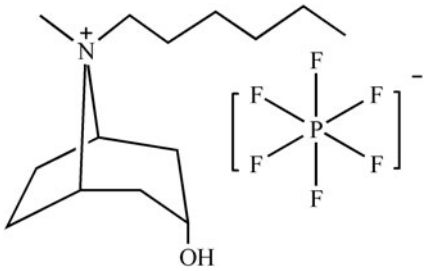

Figure 1. Structural formula of $[C_6Trop]^+[PF_6]^-$.

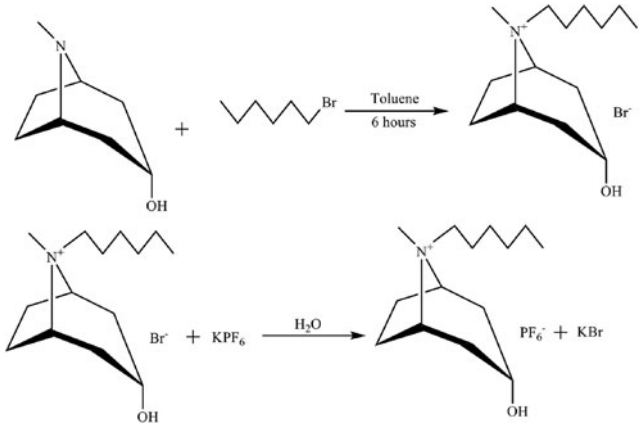

Figure 2. Synthesis procedure of $[C_6Trop]^+[PF_6]^-$.

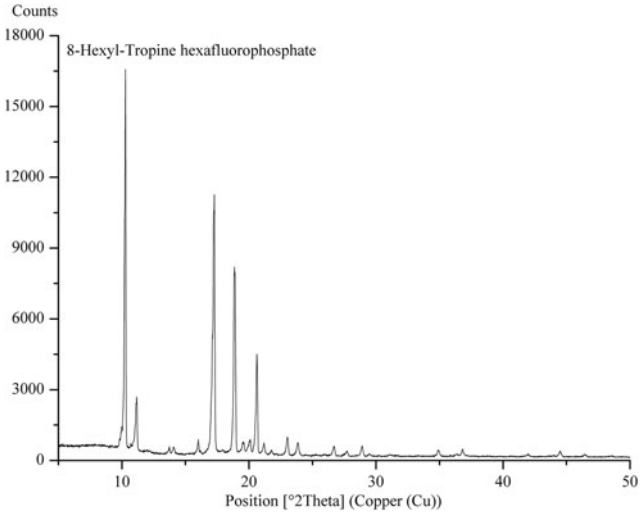

Figure 3. XRD pattern of the $[C_6Trop]^+[PF_6]^-$, using Cu$K\alpha_1$ radiation (λ = 1.54056 Å).

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TABLE I. Indexed XRD data of 8-hexyl-3-hydroxy-8-methyl-nortropanium hexafluorophosphate, $C_{14}H_{27}NOPF_6$.

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
9.9194	8.9096	7	1	−1	0	9.9285	8.9014	−0.0091
10.1952	8.6692	49	1	1	0	10.1909	8.6728	0.0043
10.3921	8.5054	100	1	0	−1	10.4897	8.4264	−0.0976
11.0618	7.9919	16	1	0	1	11.0739	7.9832	−0.0121
13.6616	6.4764	3	2	−1	0	13.6536	6.4801	0.0080
14.0423	6.3016	3	2	1	0	14.0366	6.3042	0.0057
14.0817	6.2840	3	2	−1	−1	14.0756	6.2867	0.0061
16.0119	5.5306	5	1	−1	1	15.9437	5.5541	0.0682
17.2199	5.1453	68	0	1	2	17.1318	5.1715	0.0881
18.6904	4.7436	8	2	−1	1	18.6847	4.7450	0.0057
18.8349	4.7076	49	3	1	0	18.7489	4.7290	0.0860
19.0975	4.6434	2	1	0	−2	19.1040	4.6418	−0.0065
19.6096	4.5233	5	2	−1	−2	19.6098	4.5232	−0.0002
20.4630	4.3365	7	2	2	0	20.4635	4.3364	−0.0005
21.1327	4.2006	4	2	1	2	21.1289	4.2013	0.0038
23.0234	3.8597	6	1	2	−1	23.0211	3.8601	0.0023
23.0497	3.8554	6	1	−2	1	23.0662	3.8527	−0.0165
23.8375	3.7297	4	4	1	0	23.8496	3.7279	−0.0121
26.7131	3.3344	4	3	2	2	26.7626	3.3283	−0.0495
28.9058	3.0863	4	3	−3	−2	28.9421	3.0825	−0.0363
34.9195	2.5673	3	2	−1	3	34.9376	2.5660	−0.0181
36.8890	2.4346	3	5	−2	−3	36.9015	2.4338	−0.0125
44.5046	2.0341	2	4	3	4	44.5089	2.0339	−0.0043

Only the peaks with I_{rel} of 1 or greater are presented [$a = 16.1879(6)$ Å, $b = 11.4271(2)$ Å, $c = 10.3966(5)$ Å, $\alpha = 66.4949(3)°$, $\beta = 94.5589(0)°$, $\gamma = 93.3092(4)°$, unit-cell volume $V = 1923.17$ Å^3, $Z = 1$, and space group $P1$]. All measured lines were indexed and are consistent with the $P1$ space group. The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.54056$ Å).

II. EXPERIMENTAL

A. Sample preparation

The title compound was synthesized as the following procedure (see Figure 2):

(1) 0.05 mol 3-α-tropanol (from Sigma-Aldrich, CAS No. 120-29-6, purity >97%) was dissolved in 50 ml toluene together with 0.05 mol 1-bromohexane. The solution was refluxed and reacted for 6 h, and was concentrated under vacuum to produce a white solid. The product was washed by ethyl acetate for three times to obtain $[C_6Trop]^+[Br]^-$.
(2) 0.01 mol $[C_6Trop]^+[Br]^-$ and 0.12 mol potassium hexafluorophosphate were dissolved in distilled water. Then the aqueous solution of potassium hexafluorophosphate was added to the aqueous solution of $[C_6Trop]^+[Br]^-$ and stirred thoroughly. Then the solid in the reaction system was filtrated and washed with distilled water to obtain a white powder of $[C_6Trop]^+[PF_6]^-$.

The sample was characterized by high-performance liquid chromatography, as well as by UV, IR, and NMR. UV (CH3CN, 200-400 nm) max: 204 nm. IR (KBr disc, cm-1): 3304 (-OH stretching vibration); 2960, 2931, 2925 (-CH3, -CH2 stretching vibration); 2886 (-CH stretching vibration); 1471, 1466 (-CH2 bending vibration); 1448, 1326 (-CH3 bending vibration); 1261, 1232 (C-N stretching vibration). PNMR (400M, DMSO) ppm: 1.680-1.738 (4H, m, 1,4-H, -CH2×2); 3.949 (2H, brs, 2,3-H, -CH×2); 2.134- 2.198 (4H, m, 5,6-H, -CH2×2); 3.173 (3H, s, 8-H, -CH3); 4.165 (1H, t, 9-H, -CH-O); 3.439 (1H, s, -OH); 3.268-3.310 (2H, t, 11-H, -CH2); 2.732-2.766 (2H, t, 12-H, -CH2); 1.346-1.450 (2H, t, 13-H, -CH2); 0.990 (3H, s, 14-H, -CH3). Eventually, the pure $[C_6Trop]^+[PF_6]^-$ was recrystallized in methanol, and its melting point was determined to be 185 °C.

B. Powder diffraction data collection and reduction

The diffraction pattern for the $[C_6Trop]^+[PF_6]^-$ powder was collected at room temperature using an X'Pert PRO diffractometer (PANalytical) with an PIXcel 1D detector and Cu$K\alpha_1$ radiation ($\lambda = 1.54056$ Å, generator setting: 40 kV and 40 mA). The diffraction data were collected in the angular range from 5° to 50° 2θ with a step size of 0.01313° 2θ and a counting time of 30 s/step. Data evaluation was performed using the software package Material Studio 4.2 (Accelrys Co., Ltd. USA).

The preliminary unit-cell parameters were obtained by analyzing the peak positions in the X-ray powder diffraction (XRD) pattern by the X-Cell method from the "Powder Indexing" tool. The indexing results were then refined with the type of Pawley, which involves assigning the Miller indices (h, k, l) to each observed peak in the experimental powder XRD pattern (Harris, 2012).

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 3. Indexing the results confirmed that $[C_6Trop]^+[PF_6]^-$ is triclinic with space group $P1$ and unit-cell parameters: $a = 16.1879(6)$ Å, $b = 11.4271(2)$ Å, $c = 10.3966(5)$ Å, $\alpha = 66.4949(3)°$, $\beta = 94.5589(0)°$, $\gamma = 93.3092(4)°$, unit-cell volume $V = 1923.17$ Å^3, $Z = 1$, and space group $P1$ (Table I).

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