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3-[2-(Anilinocarbonyl)ethyl]-1-methyl-1*H*-imidazolium hexafluoridophosphate

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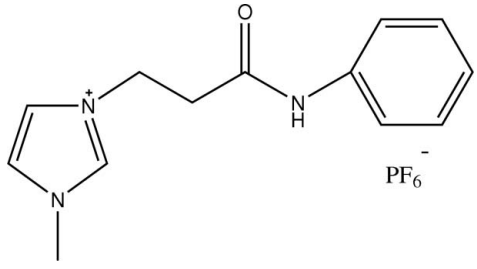
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.056; wR factor = 0.145; data-to-parameter ratio = 16.9.

The title compound, $C_{13}H_{16}N_3O^+\cdot PF_6^-$, which has an imide group in the imidazolium cation, is a new ionic liquid above its melting point. Two neighbouring molecules are connected by a weak non-classical C—H···O hydrogen bond with the formation of centrosymmetric 14-membered dimers.

Related literature

For the preparation of the compound, see: Yang *et al.* (2007).



Experimental

Crystal data

$C_{13}H_{16}N_3O^+\cdot PF_6^-$
M_r = 375.26
Monoclinic, $P2_1/c$
a = 9.6414 (4) Å
b = 19.4934 (10) Å
c = 8.8402 (4) Å
β = 103.6880 (11)°
V = 1614.27 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 296 (1) K
0.40 × 0.30 × 0.27 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.879, T_{max} = 0.937
3677 measured reflections
3677 independent reflections
2362 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.145
S = 1.01
3677 reflections
218 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O1^{i}	0.93	2.23	3.119 (3)	160

Symmetry code: (i) $-x+1, -y, -z+1$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); Larson (1970); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2076).

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Yang, W. L., Xu, D. Q., Liu, B. Y., Luo, S. P., Wang, B. T. & Xu, Z. Y. (2007). *J. Fine Chem.* **24**, 737–742.

supporting information

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3-[2-(Anilinocarbonyl)ethyl]-1-methyl-1*H*-imidazolium hexafluoridophosphate

Wen Pei, Xiaoyang Si, Dengxiang Ji and Jianbing Ji

S1. Comment

The phenol and aniline are two vital infectant in water. We prepared the new ionic liquid with a imide group in the imidazolium cations to extracted and seperate phenol and aniline from the water and achieved good result.

Two neighbour molecules are connected by a weak, non-classical, C12–H12···O1^{i} H-bonds with the formation centrosymmetrical 14-membered dimers (symmetry code: (i) 1 - *x*, -*y*, 1 - *z*).

S2. Experimental

The title compound was prepared according to the procedure of Yang *et al.* (2007). A mixture of 3-chloro-*N*-phenyl-propanamide (9.18 g, 0.05 mol) and 1-methylimidazole (4.1 g, 0.05 mmol) in 30 ml of acetonitrile was heated with stirring at 353 K for 9 h. The solvent was removed by distillation, and a gray liquid was obtained. Then 50 ml of water and KPF_6 (10.15 g, 0.055 mol) was added, the mixture was stirred at ambient temperature for 24 h. On completion, the water were filtered off and the title compound was obtained as a white solid in 61% yield. Diffraction quality crystals were obtained by slow evaporation of an ethylacetate solution at room temperature.

S3. Refinement

All H atoms were placed in calculated posistion for N–H = 0.86 Å, C–H = 0.97Å (for CH_2), C–H = 0.96Å (for CH_3), C–H = 0.93Å (for aromatic) and included in the final cycles of refinement as riding mode with U_{iso}(H) = 1.2U_{eq}(parent atom).

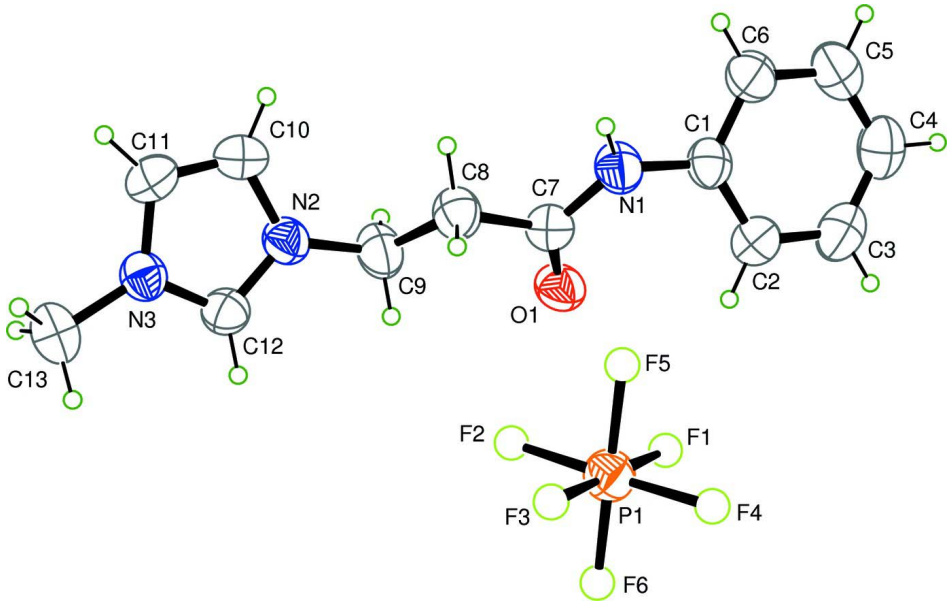

Figure 1

View of the title molecule with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

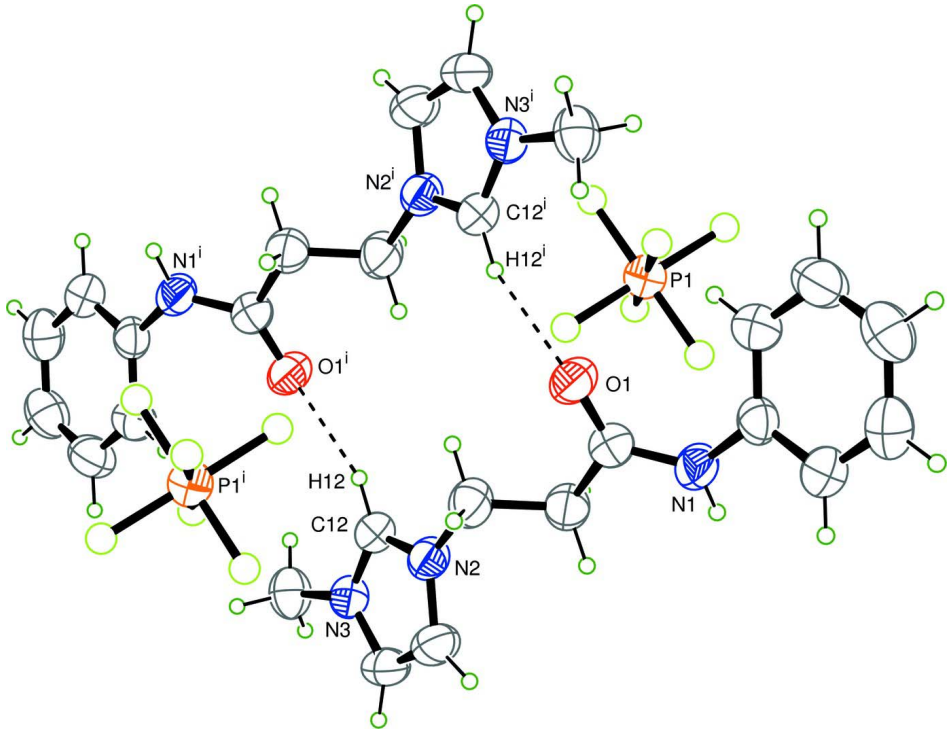

Figure 2

View of a hydrogen-bonded (dashed lines) dimers with the symmetry code: (i) 1 - *x*, -*y*, 1 - *z*.

3-[2-(Anilinocarbonyl)ethyl]-1-methyl-1*H*-imidazolium hexafluorophosphate

Crystal data

$C_{13}H_{16}N_3O^+\cdot PF_6^-$
M_r = 375.26
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 9.6414 (4) Å
b = 19.4934 (10) Å
c = 8.8402 (4) Å
β = 103.6880 (11)°
V = 1614.27 (13) Å^3
Z = 4
$F(000)$ = 768.00
D_x = 1.544 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 9621 reflections
θ = 3.0–27.4°
μ = 0.24 mm^{-1}
T = 296 K
Block, colourless
0.40 × 0.30 × 0.27 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Detector resolution: 10.00 pixels mm^{-1}
ω–scan
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.879, T_{max} = 0.937
3677 measured reflections
3677 independent reflections
2362 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.037
θ_{max} = 27.4°
h = −12→12
k = −25→0
l = 0→11

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.145
S = 1.01
3677 reflections
218 parameters
0 restraints
H-atom parameters constrained
$w = 1/[0.0013F_o^2 + 5.0000\sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 192 (26)

Special details

Refinement. Refinement using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (*S*) are based on F^2. *R*-factor (gt) are based on *F*. The threshold expression of $F^2 > 2.0\sigma(F^2)$ is used only for calculating *R*-factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.66835 (6)	0.13474 (4)	0.87558 (8)	0.0527 (2)
F1	0.80918 (19)	0.10215 (11)	0.8443 (2)	0.0979 (7)
F2	0.57794 (19)	0.09494 (9)	0.7276 (2)	0.0832 (5)
F3	0.5293 (2)	0.16754 (12)	0.9074 (2)	0.1199 (8)
F4	0.7630 (2)	0.17460 (10)	1.0203 (2)	0.0966 (6)
F5	0.6809 (2)	0.19681 (9)	0.7656 (2)	0.0946 (6)
F6	0.6574 (2)	0.07272 (11)	0.9846 (2)	0.1142 (8)
O1	0.78438 (18)	0.06145 (9)	0.4653 (2)	0.0562 (5)
N1	0.8257 (2)	0.17224 (11)	0.4113 (2)	0.0540 (6)
N2	0.3528 (2)	0.07578 (10)	0.2310 (2)	0.0486 (5)
N3	0.1253 (2)	0.06435 (10)	0.1961 (2)	0.0461 (5)
C1	0.9762 (2)	0.17472 (12)	0.4617 (2)	0.0477 (6)

C2	1.0511 (2)	0.13719 (13)	0.5867 (3)	0.0604 (8)
C3	1.1984 (3)	0.14078 (16)	0.6293 (3)	0.0685 (9)
C4	1.2713 (3)	0.18264 (16)	0.5499 (3)	0.0659 (9)
C5	1.1969 (3)	0.22013 (14)	0.4270 (3)	0.0671 (9)
C6	1.0501 (2)	0.21689 (12)	0.3810 (3)	0.0589 (8)
C7	0.7400 (2)	0.11745 (12)	0.4140 (2)	0.0467 (7)
C8	0.5835 (2)	0.13090 (12)	0.3475 (3)	0.0536 (7)
C9	0.5065 (2)	0.06525 (12)	0.2934 (3)	0.0587 (8)
C10	0.2893 (2)	0.11482 (13)	0.1046 (2)	0.0606 (8)
C11	0.1480 (2)	0.10802 (14)	0.0839 (2)	0.0596 (8)
C12	0.2500 (2)	0.04552 (12)	0.2837 (2)	0.0469 (6)
C13	−0.0136 (2)	0.03930 (13)	0.2138 (3)	0.0607 (8)
H1	0.7839	0.2098	0.3749	0.065*
H2	1.0024	0.1094	0.6424	0.072*
H3	1.2486	0.1147	0.7124	0.082*
H4	1.3703	0.1854	0.5795	0.079*
H5	1.2462	0.2485	0.3732	0.081*
H6	1.0010	0.2427	0.2967	0.071*
H10	0.3359	0.1412	0.0442	0.073*
H11	0.0783	0.1291	0.0073	0.071*
H12	0.2638	0.0159	0.3685	0.056*
H81	0.5726	0.1621	0.2600	0.064*
H82	0.5427	0.1515	0.4270	0.064*
H91	0.5201	0.0338	0.3808	0.070*
H92	0.5466	0.0454	0.2126	0.070*
H131	−0.0048	0.0238	0.3187	0.073*
H132	−0.0446	0.0020	0.1430	0.073*
H133	−0.0823	0.0758	0.1912	0.073*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0535 (4)	0.0500 (4)	0.0539 (4)	−0.0018 (3)	0.0113 (3)	0.0002 (3)
F1	0.0664 (11)	0.1231 (17)	0.1046 (14)	0.0181 (11)	0.0211 (10)	−0.0216 (12)
F2	0.0852 (12)	0.0635 (11)	0.0860 (11)	−0.0082 (8)	−0.0097 (9)	−0.0124 (9)
F3	0.0853 (14)	0.162 (2)	0.1203 (17)	0.0382 (14)	0.0409 (13)	−0.0086 (15)
F4	0.1166 (15)	0.0890 (14)	0.0679 (11)	0.0010 (11)	−0.0106 (10)	−0.0213 (10)
F5	0.1389 (17)	0.0636 (11)	0.0753 (11)	−0.0261 (11)	0.0136 (11)	0.0114 (9)
F6	0.1306 (18)	0.1012 (16)	0.1067 (15)	−0.0211 (13)	0.0199 (13)	0.0490 (12)
O1	0.0532 (10)	0.0470 (10)	0.0682 (11)	0.0004 (8)	0.0141 (8)	0.0127 (8)
N1	0.0485 (11)	0.0400 (12)	0.0690 (14)	0.0004 (9)	0.0050 (10)	0.0066 (10)
N2	0.0452 (11)	0.0438 (11)	0.0566 (12)	−0.0013 (9)	0.0114 (9)	0.0057 (9)
N3	0.0449 (11)	0.0423 (11)	0.0513 (11)	−0.0017 (9)	0.0115 (9)	−0.0019 (9)
C1	0.0485 (13)	0.0406 (13)	0.0524 (14)	−0.0029 (11)	0.0086 (11)	−0.0069 (11)
C2	0.0622 (17)	0.0636 (17)	0.0514 (15)	−0.0057 (13)	0.0059 (13)	0.0049 (13)
C3	0.0626 (18)	0.073 (2)	0.0603 (17)	0.0050 (15)	−0.0033 (14)	−0.0012 (15)
C4	0.0497 (15)	0.076 (2)	0.0702 (18)	0.0004 (14)	0.0102 (15)	−0.0162 (16)
C5	0.0625 (18)	0.0656 (19)	0.0763 (19)	−0.0155 (14)	0.0227 (16)	−0.0096 (16)

C6	0.0641 (17)	0.0466 (15)	0.0630 (16)	−0.0047 (12)	0.0093 (14)	0.0008 (13)
C7	0.0483 (14)	0.0455 (14)	0.0477 (14)	0.0031 (11)	0.0141 (11)	−0.0002 (11)
C8	0.0479 (14)	0.0491 (15)	0.0623 (16)	0.0013 (11)	0.0099 (12)	0.0012 (12)
C9	0.0437 (13)	0.0504 (15)	0.0793 (19)	−0.0014 (11)	0.0091 (12)	0.0053 (13)
C10	0.0631 (17)	0.0627 (17)	0.0556 (15)	−0.0049 (13)	0.0133 (13)	0.0175 (13)
C11	0.0589 (17)	0.0611 (17)	0.0529 (15)	0.0034 (13)	0.0016 (13)	0.0161 (13)
C12	0.0503 (14)	0.0405 (13)	0.0489 (13)	0.0002 (10)	0.0100 (11)	0.0024 (10)
C13	0.0470 (14)	0.0621 (17)	0.0745 (18)	−0.0067 (12)	0.0172 (13)	−0.0082 (14)

Geometric parameters (Å, º)

P1—F1	1.581 (2)	C5—C6	1.378 (3)
P1—F2	1.5920 (18)	C7—C8	1.508 (3)
P1—F3	1.569 (2)	C8—C9	1.500 (3)
P1—F4	1.5878 (18)	C10—C11	1.337 (4)
P1—F5	1.5745 (19)	N1—H1	0.860
P1—F6	1.565 (2)	C2—H2	0.930
O1—C7	1.220 (2)	C3—H3	0.930
N1—C1	1.414 (3)	C4—H4	0.930
N1—C7	1.354 (3)	C5—H5	0.930
N2—C9	1.469 (3)	C6—H6	0.930
N2—C10	1.371 (3)	C8—H81	0.970
N2—C12	1.329 (3)	C8—H82	0.970
N3—C11	1.363 (3)	C9—H91	0.970
N3—C12	1.319 (2)	C9—H92	0.970
N3—C13	1.468 (3)	C10—H10	0.930
C1—C2	1.378 (3)	C11—H11	0.930
C1—C6	1.391 (3)	C12—H12	0.930
C2—C3	1.383 (4)	C13—H131	0.960
C3—C4	1.373 (4)	C13—H132	0.960
C4—C5	1.363 (3)	C13—H133	0.960
F1—P1—F2	89.22 (10)	N3—C11—C10	107.2 (2)
F1—P1—F3	179.54 (12)	N2—C12—N3	108.8 (2)
F1—P1—F4	89.07 (10)	C1—N1—H1	116.3
F1—P1—F5	90.36 (11)	C7—N1—H1	116.4
F1—P1—F6	89.02 (12)	C1—C2—H2	120.0
F2—P1—F3	91.21 (10)	C3—C2—H2	120.0
F2—P1—F4	178.00 (12)	C2—C3—H3	119.8
F2—P1—F5	88.52 (9)	C4—C3—H3	119.8
F2—P1—F6	91.50 (10)	C3—C4—H4	120.3
F3—P1—F4	90.50 (11)	C5—C4—H4	120.3
F3—P1—F5	89.49 (12)	C4—C5—H5	119.3
F3—P1—F6	91.13 (13)	C6—C5—H5	119.3
F4—P1—F5	90.45 (10)	C1—C6—H6	120.3
F4—P1—F6	89.51 (10)	C5—C6—H6	120.3
F5—P1—F6	179.38 (13)	C7—C8—H81	109.2
C1—N1—C7	127.3 (2)	C7—C8—H82	109.2

C9—N2—C10	126.9 (2)	C9—C8—H81	109.2
C9—N2—C12	125.2 (2)	C9—C8—H82	109.2
C10—N2—C12	107.8 (2)	H81—C8—H82	109.5
C11—N3—C12	108.7 (2)	N2—C9—H91	108.8
C11—N3—C13	126.36 (19)	N2—C9—H92	108.8
C12—N3—C13	124.9 (2)	C8—C9—H91	108.8
N1—C1—C2	122.6 (2)	C8—C9—H92	108.8
N1—C1—C6	118.1 (2)	H91—C9—H92	109.5
C2—C1—C6	119.3 (2)	N2—C10—H10	126.3
C1—C2—C3	120.1 (2)	C11—C10—H10	126.3
C2—C3—C4	120.5 (2)	N3—C11—H11	126.4
C3—C4—C5	119.3 (2)	C10—C11—H11	126.4
C4—C5—C6	121.4 (2)	N2—C12—H12	125.6
C1—C6—C5	119.4 (2)	N3—C12—H12	125.6
O1—C7—N1	123.4 (2)	N3—C13—H131	109.5
O1—C7—C8	122.1 (2)	N3—C13—H132	109.5
N1—C7—C8	114.5 (2)	N3—C13—H133	109.5
C7—C8—C9	110.5 (2)	H131—C13—H132	109.5
N2—C9—C8	112.3 (2)	H131—C13—H133	109.5
N2—C10—C11	107.5 (2)	H132—C13—H133	109.5

C1—N1—C7—O1	1.5 (4)	C13—N3—C12—N2	177.3 (2)
C1—N1—C7—C8	−178.3 (2)	N1—C1—C2—C3	179.0 (2)
C7—N1—C1—C2	−34.4 (4)	N1—C1—C6—C5	−179.7 (2)
C7—N1—C1—C6	145.6 (2)	C2—C1—C6—C5	0.3 (3)
C9—N2—C10—C11	177.3 (2)	C6—C1—C2—C3	−1.0 (4)
C10—N2—C9—C8	61.7 (3)	C1—C2—C3—C4	1.3 (4)
C9—N2—C12—N3	−177.1 (2)	C2—C3—C4—C5	−0.8 (4)
C12—N2—C9—C8	−122.1 (2)	C3—C4—C5—C6	−0.0 (4)
C10—N2—C12—N3	−0.3 (2)	C4—C5—C6—C1	0.2 (4)
C12—N2—C10—C11	0.7 (2)	O1—C7—C8—C9	−23.5 (3)
C11—N3—C12—N2	−0.1 (2)	N1—C7—C8—C9	156.3 (2)
C12—N3—C11—C10	0.5 (3)	C7—C8—C9—N2	178.7 (2)
C13—N3—C11—C10	−176.8 (2)	N2—C10—C11—N3	−0.7 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O1^{i}	0.93	2.23	3.119 (3)	160

Symmetry code: (i) −*x*+1, −*y*, −*z*+1.