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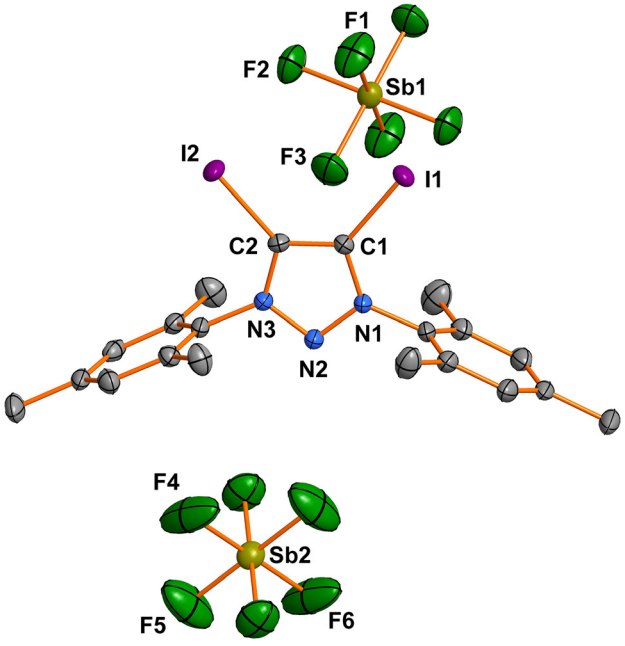
The crystal structure of 4,5-diiodo-1,3-dimesityl-1*H*-1,2,3-triazol-3-ium hexafluoridoantimonate(V), $C_{20}H_{22}F_6I_2N_3Sb$



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Abstract

$C_{20}H_{22}F_6I_2N_3Sb$, triclinic, $P\bar{1}$ (no. 2), a = 10.2268(10) Å, b = 10.8850(9) Å, c = 12.1413(10) Å, α = 101.810(2)°, β = 90.465(3)°, γ = 103.194(3)°, V = 1285.9(2) Å^3, Z = 2, $R_{gt}(F)$ = 0.0291, $wR_{ref}(F^2)$ = 0.0717, T = 200 K.

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The molecular structure is shown in the figure (the asymmetric unit is labelled). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.39 × 0.36 × 0.26 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	3.53 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.6°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	23,644, 5906, 0.038
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 5292
$N(param)_{refined}$:	298
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

Source of material

1,3-dimesityl-1*H*-1,2,3-triazol-3-ium tetrafluoroborate 0.45 g (1 mmol) and potassium tert-butoxide 0.25 g (2.2 mmol) and I_2 (510 mg, 2 mmol) were added in Schlenk tube under dry nitrogen. Then THF (20 mL) was added at –78°C. The mixture was stirred for 3 h. After evaporation of THF, dichloromethane (100 mL) was added and inorganic salts were removed by filtration. The dichloromethane solution and silver hexafluoroantimonate 0.35 g (1 mmol) were added in a Schlenk tube under dry nitrogen and then stirred for 3 h. Precipitated AgI was removed by filtration and a white solid was got by evaporation of dichloromethane. Yield: 81%.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
I1	0.92024 (2)	0.58457 (2)	0.14730 (2)	0.03131 (7)
I2	0.67083 (3)	0.58409 (2)	0.39986 (2)	0.03804 (7)
N1	0.8452 (3)	0.3132 (2)	0.1927 (2)	0.0229 (5)
N2	0.7785 (3)	0.2378 (2)	0.2574 (2)	0.0248 (5)
N3	0.7200 (3)	0.3158 (2)	0.3281 (2)	0.0223 (5)
C1	0.8299 (3)	0.4369 (3)	0.2221 (2)	0.0224 (6)
C2	0.7480 (3)	0.4383 (3)	0.3105 (3)	0.0236 (6)
C3	0.6487 (3)	0.2674 (3)	0.4189 (2)	0.0234 (6)
C4	0.5130 (3)	0.2074 (3)	0.4012 (3)	0.0267 (6)
C5	0.4502 (3)	0.1607 (3)	0.4906 (3)	0.0291 (7)
H5	0.357594	0.117867	0.481370	0.035*
C6	0.5199 (3)	0.1751 (3)	0.5932 (3)	0.0298 (7)
C7	0.6554 (4)	0.2370 (3)	0.6061 (3)	0.0313 (7)
H7	0.702718	0.247712	0.676352	0.038*
C8	0.7240 (3)	0.2838 (3)	0.5189 (3)	0.0260 (6)
C9	0.9237 (3)	0.2576 (3)	0.1059 (3)	0.0257 (6)
C10	0.8585 (4)	0.1920 (3)	0.0031 (3)	0.0301 (7)

Table 2: (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C11	0.9364 (4)	0.1356 (3)	−0.0764 (3)	0.0353 (8)
H11	0.895654	0.090838	−0.148437	0.042*
C12	1.0712 (4)	0.1423 (3)	−0.0544 (3)	0.0354 (8)
C13	1.1310 (4)	0.2103 (3)	0.0501 (3)	0.0347 (7)
H13	1.223943	0.216934	0.065380	0.042*
C14	1.0587 (3)	0.2688 (3)	0.1328 (3)	0.0291 (7)
C15	1.1262 (4)	0.3418 (4)	0.2458 (3)	0.0449 (9)
H15A	1.111096	0.429251	0.261601	0.067*
H15B	1.223026	0.346570	0.244486	0.067*
H15C	1.088331	0.296839	0.304609	0.067*
C16	1.1513 (5)	0.0797 (4)	−0.1429 (4)	0.0506 (11)
H16A	1.170973	0.131512	−0.200509	0.076*
H16B	1.099317	−0.007244	−0.177914	0.076*
H16C	1.235802	0.073934	−0.107953	0.076*
C17	0.7117 (5)	0.1805 (5)	−0.0221 (4)	0.0527 (11)
H17A	0.659311	0.145196	0.036261	0.079*
H17B	0.683315	0.122776	−0.095716	0.079*
H17C	0.696626	0.265962	−0.023296	0.079*
C18	0.8712 (4)	0.3487 (4)	0.5328 (3)	0.0376 (8)
H18A	0.919494	0.302575	0.475214	0.056*
H18B	0.906363	0.347537	0.607831	0.056*
H18C	0.883701	0.438228	0.524509	0.056*
C19	0.4502 (4)	0.1250 (4)	0.6896 (3)	0.0433 (9)
H19A	0.428566	0.196682	0.743745	0.065*
H19B	0.509618	0.085232	0.727154	0.065*
H19C	0.367091	0.060526	0.660699	0.065*
C20	0.4380 (4)	0.1916 (4)	0.2897 (3)	0.0437 (9)
H20A	0.475204	0.135478	0.230876	0.066*
H20B	0.447465	0.276306	0.270616	0.066*
H20C	0.342528	0.152752	0.295265	0.066*
Sb1	0.500000	0.500000	0.000000	0.03456 (9)
F1	0.6002 (3)	0.6575 (3)	0.0804 (3)	0.0769 (9)
F2	0.3504 (3)	0.5266 (3)	0.0798 (2)	0.0629 (7)
F3	0.5512 (3)	0.4170 (3)	0.1076 (2)	0.0757 (9)
Sb2	1.000000	0.000000	0.500000	0.03798 (9)
F4	0.8504 (3)	0.0349 (3)	0.4368 (3)	0.0777 (9)
F5	0.8934 (4)	−0.1160 (5)	0.5734 (4)	0.1232 (17)
F6	0.9978 (6)	−0.1310 (4)	0.3778 (3)	0.137 (2)

Single crystals were obtained under ambient conditions via solvent evaporation in the solvents of dichloromethane. **^{1}H NMR** (400 MHz, chloroform-d): δ (ppm) 7.12 (s, 4H), 2.40 (s, 6H), 2.01 (s, 12H). **^{13}C NMR** (100 MHz, chloroform-d): δ (ppm) 143.8, 134.4, 130.3, 105.6, 21.4, 17.3.

Experimental details

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å) and were included in the refinement in the riding model approximation, with d(C–H) = 0.98 Å (methyl), U_{iso}(H) = 1.5 U_{eq}(C), and 0.95 Å (aromatic), U_{iso}(H) = 1.2U_{eq}(C).

Comment

The halogen bond is a noncovalent interaction between electrophilic halides (σ-hole) and Lewis base or electron rich regions [5]. It is widely applied in crystal engineering, anion recognition, supramolecular chemistry, organocatalysis, and materials science, even tuning of biomolecular systems [6]. The 5-iodo-1,2,3-triazolium is a strong halogen bond donor which has been applied in many fields such as anion recognition [7]. Further development was achieved using 4,5-diiodo-1,3-dimesityl-1,2,3-triazolium salts with different anions. Even tetrafluoroborate shows halogen bonding *via* a trimer with Chinese lantern shape conformation. The distances of iodine atom and fluorine atom show that weak halogen bonding exists [8]. So a weaker anion hexafluoroantimonate was considered to form a new crystal.

The ORTEP-type diagram is presented in the Figure. There is one organic cation and two halfs of a hexafluoroantimonate anions in the asymmetric unit. The hexafluoroantimonate anions do not show halogen bonding in the crystal. The two iodine atoms and benzene rings show weak π-I interactions, connecting the cations to form a chain.

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