Electronic supplementary materials *Mendeleev Commun.*, 2022, **32**, 212–214

Synthesis, crystal structure and thermal properties of $Na_2Ti(CF_3COO)_6(CF_3COOH)_2$

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Experimental. The precursors were synthesized just before the synthesis of the target product. Trifluoroacetic acid anhydride $(CF_3CO)_2O$ was synthesized in the course of the study by refluxing CF_3COOH (99%) with P_2O_5 for 12 h followed by distillation of $(CF_3CO)_2O$ with P_2O_5.

A CF_3COONa solution was synthesized by the reaction of a calculated amount of $NaHCO_3$ with $CF_3COOH/(CF_3CO)_2O$ (1:1 vol.%) without isolation of the dry product.

A solution of $TiCl_4$ (2.0 ml, 18.2 mmol) in 10 ml of CCl_4 and 10 ml of a $CF_3COOH/(CF_3CO)_2O$ mixture (1:1 vol%) was refluxed in an argon atmosphere until the evolution of HCl ceased. Then a solution of CF_3COONa (2.47 g, 18.2 mmol) in 10 ml of a $CF_3COOH/(CF_3CO)_2O$ mixture (1:1 vol%) was added with heating. The resulting solution was concentrated in a vacuum desiccator with P_2O_5. After 5-10 days, transparent crystals unstable in air and suitable for single-crystal X-ray diffraction analysis formed. The crystals were filtered off and dried in a flow of argon. The yield was 60%.

Figure S1 A crystallographically independent fragment of the $Na_3Ti_{1.5}(TFA)_9(HTFA)_3$ chain in $Na_2Ti(CF_3COO)_6(CF_3COOH)_2$ presented as thermal ellipsoids. The fluorine atoms of trifluoromethyl groups are not shown.

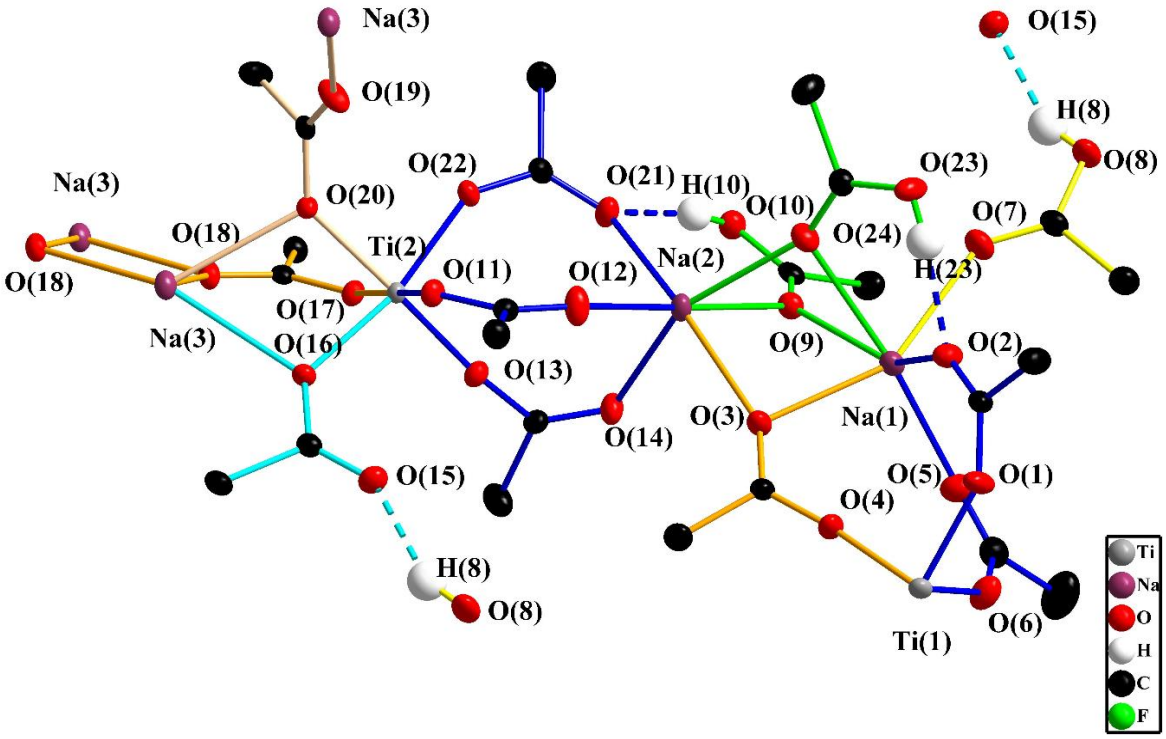

Table S1 Bond lengths (Å).

Ti(1)	O(1)	**A**^{5}	1.962(2)	Ti(2)	O(11)	**A**^{1}	1.9075(19)
					O(13)	**A**^{2}	1.8402(19)
	O(4)	**B**^{1}	1.8867(19)		O(16)	**D**	1.9860(19)
					O(17)	**B**^{2}	1.9780(19)
	O(6)	**A**^{4}	1.926(2)		O(20)	**C**	1.9596(18)
					O(22)	**A**^{3}	1.9617(19)

Na(1)	O(2)	**A**^{5}	2.423(2)	Na(2)	O(3)	**B**^{1}	2.285(2)	Na(3)	O(16)	**D**	2.571(2)
	O(3)	**B**^{1}	2.390(2)		O(9)	**E**^{2}	2.390(2)		O(18)	**B**^{2}	2.394(2)
	O(5)	**A**^{4}	2.258(2)		O(12)	**A**^{1}	2.246(2)		O(18)’	**B**^{2}**’**	2.397(2)
	O(7)	**F**	2.357(2)		O(14)	**A**^{2}	2.375(2)		O(19)	**C**	2.317(2)
	O(9)	**E**^{2}	2.417(2)		O(21)	**A**^{3}	2.342(2)		O(20)	**C**	2.506(2)
	O(24)	**E**^{1}	2.598(2)		O(24)	**E**^{1}	2.409(2)		F(22)	**B**^{2}	2.748(2)
									F(25)	**B**^{2}	2.628(2)
									F(30)	**B**^{2}	2.855(5)

Table S2 Crystal Data and Structure Refinement for compounds **1**.

Compound	**1**
CCDC entry no.	2108171
Empirical formula	$C_{48}H_6F_{72}Na_6O_{48}Ti_3$
Formula weight	3000.17
Temperature (K)	100(2)
Space group	$P\bar{1}$
a (Å)	8.5999(4)
b (Å)	12.4742(6)
c (Å)	24.1371(11)
α (°)	101.057(2)
β (°)	92.957(2)
γ (°)	109.916(2)
Volume (Å^3)	2370.04(19)
Z	1
D_{calc} (Mg m^{-3})	2.102
Absorption coeff. (mm^{-1})	0.511
F(000)	1458
refl	
Collected refl./unique refl.	33712/ 11437
Data/restraints/parameters	11437 / 271 / 830
GOF on F^2	1.033
R_1 / wR_2 [$I > 2\sigma(I)$]	0.0525/0.1184
R_1 / wR_2 (all data)	0.0702/0.1256
Largest peak/hole (e/A^3)	1.179 and -0.792