Supporting Information

Open-Framework Structures of Anhydrous $Sr(CF_3COO)_2$ and $Ba(CF_3COO)_2$

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EXPERIMENTAL

Laboratory Powder X-ray Diffraction. Polycrystalline products resulting from the decomposition of $Sr(CF_3COO)_2$ and $Ba(CF_3COO)_2$ during thermal analysis were characterized using a Bruker D2 Phaser powder diffractometer operated at 30 kV and 10 mA. Cu Kα radiation (λ = 1.5418 Å) was employed. A nickel filter was utilized to remove Cu Kβ. Diffractograms were collected in 10–60° 2θ range using a step size of 0.025° and a step time of 0.5 s.

Rietveld Analysis of $Sr(CF_3COO)_2$. Structural refinement of $Sr(CF_3COO)_2$ was carried out by means of Rietveld[1, 2] analysis of synchrotron X-ray diffraction (XRD) data. The General Structure

Analysis System (GSAS) with the Graphical User Interphase EXPGUI software suite was employed to that end.[3,4] The structural model provided by single crystal XRD studies was utilized. The following parameters were refined: (1) scale factor, (2) background, which was modeled using a shifted Chebyschev polynomial function, (3) peak shape, which was modeled using a modified Thompson–Cox–Hasting pseudo-Voight function,[5] (4) lattice parameters (*a* and *c*), and (5) one isotropic temperature factor for Sr (U_{Sr}) and one isotropic temperature factor for C, O, and F ($U_{C,O,F}$). The atomic positions were *not* refined. Atomic scattering factors were corrected for anomalous scattering and, for each composition, experimental data were corrected for absorption. The R_{wp} residual was employed to assess the quality of the refined structural model.

Crystallization of $Ca(CF_3COO)_2 \cdot H_2O$. $CaCO_3$ (99.99%, Sigma Aldrich) and anhydrous CF_3COOH (99%, Sigma Aldrich) were used as received. Double-distilled water was employed as the solvent. A mixture of 5 mL of double-distilled water and 1 mL of CF_3COOH was a prepared in a 50 mL two-neck round-bottom flask. Then, 2 mmol of $CaCO_3$ were added to this solution. Quantitative dissolution of the metal carbonate occurred within 1–3 mins and, as expected, was accompanied by vigorous gas evolution; a colorless, optically transparent solution was thus obtained. Crystallization of $Ca(CF_3COO)_2 \cdot H_2O$ was accomplished by solvent evaporation, which was induced by flowing dry nitrogen gas over the precursor solution. A needle-valve rotameter was employed to regulate the nitrogen flow rate. A constant flow rate of 200 mL/min was employed. The system was left undisturbed for 7 days at 25 °C under flowing nitrogen. After 7 days, white $Ca(CF_3COO)_2 \cdot H_2O$ powder was obtained at the bottom of the reaction flask. This powder was stored in a glove box (oxygen and water levels below 1 ppm) at room temperature for thermal analysis and single crystal X-ray diffraction studies.

Single Crystal X-ray Diffraction Study of $Ca(CF_3COO)_2 \cdot H_2O$. A colourless single crystal of $Ca(CF_3COO)_2 \cdot H_2O$ was recovered from the polycrystalline powder and used for structural determination. The crystal was irregular with approximate dimensions 0.16 × 0.24 × 0.44 mm. It was mounted on a Bruker D8 Venture single-crystal diffractometer and X-ray intensities were measured at 25 °C using Mo Kα radiation (λ = 0.71073 Å). Frames were integrated using the Bruker SAINT software. Experimental data were corrected for Lorentz, polarization, and absorption effects; for the latter, the multiscan method was employed. Structures were solved using direct methods and difference Fourier synthesis as embedded in the SHELXL–2014/7 software and SHELXLE graphical user interphase.[6,7] The $-CF_3$ groups in the three independent trifluoroacetate ligands (named A, B, and C) showed rotational disorder. These were modelled in two independent positions and allowed for rotational freedom and independent occupancies that refined to 53:47%, 45:56% and 49:51 for ligands A, B, and C, respectively. The hydrogen atoms of the two water molecules present in the structure were found in ΔF maps, and refined constrained with $U_{iso} = 1.5U_{eq}$ of the corresponding oxygen atom. A total of 150 restraints were applied to bond distances, bond angles, and atomic displacement parameters. The resulting crystal structure was visualized using the VESTA software. Crystal data of $Ca(CF_3COO)_2 \cdot H_2O$ was deposited in the Cambridge Crystallographic Data Centre with number 1421989.

TABLES AND FIGURES

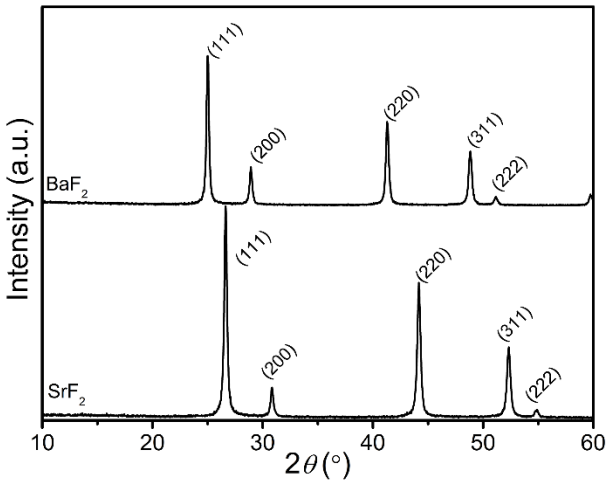

Figure S1. Powder XRD patterns of the thermal decomposition products of $Sr(CF_3COO)_2$ and $Ba(CF_3COO)_2$. All diffraction maxima can be indexed to the cubic SrF_2 (PDF No. 06–0262) and BaF_2 (PDF No. 04–0452) phases.

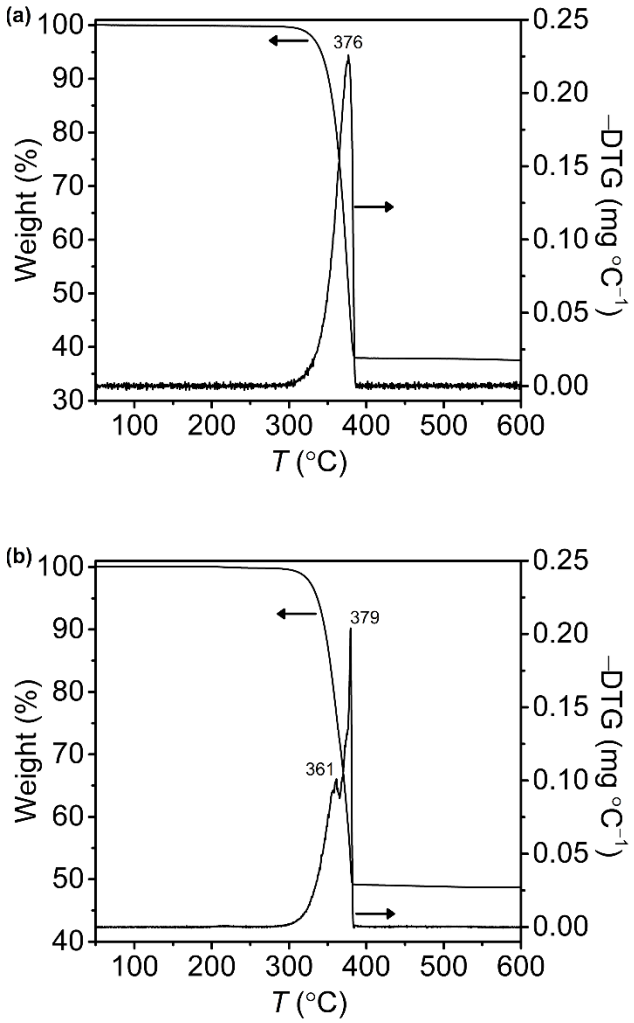

Figure S2. Thermograms and differential thermograms (first derivative) of (a) $Sr(CF_3COO)_2$ and (b) $Ba(CF_3COO)_2$.

Table S1 Fractional Atomic Coordinates and Isotropic Displacement Parameters of $Sr(CF_3COO)_2$

Atom	*x*	*y*	*z*	U_{eq} (Å^2)
Sr	0.39514(2)	0.41092(2)	0.49879(2)	0.00782(2)
C1	0.50970(4)	0.42456(4)	0.29853(9)	0.00992(13)
C2	0.56995(5)	0.43121(5)	0.20440(10)	0.01530(16)
C3	0.42518(4)	0.35464(4)	0.83027(9)	0.01013(13)
C4	0.50266(5)	0.39402(5)	0.78248(10)	0.01370(15)
O1	0.45089(3)	0.37092(3)	0.28795(7)	0.01250(11)
O2	0.52496(3)	0.47445(3)	0.38112(7)	0.01241(11)
O3	0.38247(3)	0.35884(4)	0.74821(7)	0.01301(11)
O4	0.41382(4)	0.32554(4)	0.94603(7)	0.01496(12)
F1	0.54930(4)	0.37820(4)	0.11491(7)	0.02437(14)
F2	0.59733(4)	0.49104(4)	0.13000(7)	0.02608(14)
F3	0.62131(3)	0.43370(4)	0.28406(8)	0.02358(13)
F4	0.50859(3)	0.40901(4)	0.64281(6)	0.01875(12)
F5	0.53637(4)	0.45530(3)	0.84995(8)	0.02509(14)
F6	0.53532(3)	0.35859(4)	0.80673(8)	0.02410(14)

Table S2 Anisotropic Atomic Displacement Parameters (Å^2) of $Sr(CF_3COO)_2$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Sr	0.00680(3)	0.00874(3)	0.00654(3)	–0.00069(2)	0.00020(2)	0.00286(2)
C1	0.0095(3)	0.0109(3)	0.0086(3)	–0.0004(2)	–0.0001(2)	0.0045(3)
C2	0.0112(3)	0.0177(4)	0.0143(4)	–0.0052(3)	0.0013(3)	0.0052(3)
C3	0.0093(3)	0.0113(3)	0.0099(3)	0.0005(2)	0.0007(2)	0.0052(3)
C4	0.0106(3)	0.0183(4)	0.0117(3)	0.0041(3)	0.0010(3)	0.0068(3)
O1	0.0094(2)	0.0109(3)	0.0144(3)	–0.0031(2)	–0.0002(2)	0.0029(2)
O2	0.0123(3)	0.0111(3)	0.0112(3)	–0.0032(2)	0.0011(2)	0.0038(2)
O3	0.0152(3)	0.0187(3)	0.0120(3)	0.0020(2)	–0.0007(2)	0.0071(2)
O4	0.0152(3)	0.0187(3)	0.0106(3)	0.0045(2)	0.0023(2)	0.0082(3)
F1	0.0190(3)	0.0296(3)	0.0223(3)	–0.0156(3)	0.0010(2)	0.0105(3)
F2	0.0241(3)	0.0265(3)	0.0201(3)	0.0055(3)	0.0121(3)	0.0071(3)
F3	0.0129(3)	0.0304(3)	0.0291(3)	–0.0099(3)	–0.0052(2)	0.0121(3)
F4	0.0166(3)	0.0286(3)	0.0123(3)	0.0083(2)	0.0056(2)	0.0122(2)
F5	0.0167(3)	0.0200(3)	0.0249(3)	–0.0019(2)	–0.0034(2)	–0.0011(2)
F6	0.0172(3)	0.0360(4)	0.0269(3)	0.0123(3)	0.0050(2)	0.0192(3)

Table S3 Fractional Atomic Coordinates and Isotropic Displacement Parameters of $Ba(CF_3COO)_2$

Atom	*x*	*y*	*z*	U_{eq} (Å^2)
Ba	0.38399(2)	0.40876(2)	0.46111(2)	0.01440(8)
C1	0.5243(2)	0.4321(2)	0.3017(3)	0.0164(8)
C2	0.5878(2)	0.4431(2)	0.2151(4)	0.0213(9)
C3	0.4350(2)	0.3774(2)	0.8004(4)	0.0189(9)
C4	0.5189(3)	0.4177(3)	0.8002(4)	0.0260(10)
O1	0.46179(15)	0.39120(16)	0.2608(2)	0.0206(6)
O2	0.54022(15)	0.46697(15)	0.4038(2)	0.0205(6)
O3	0.40396(15)	0.37828(16)	0.7010(2)	0.0224(7)
O4	0.40650(16)	0.34866(16)	0.9027(2)	0.0225(7)
F1	0.56555(15)	0.39269(15)	0.1245(2)	0.0339(7)
F2	0.61785(14)	0.50839(14)	0.1580(2)	0.0322(6)
F3	0.63992(13)	0.44022(14)	0.2807(2)	0.0292(6)
F4	0.54711(14)	0.44803(15)	0.6861(2)	0.0317(6)
F5	0.54551(15)	0.47180(15)	0.8845(2)	0.0374(7)
F6	0.54427(14)	0.37409(15)	0.8283(3)	0.0384(7)

Table S4 Anisotropic Atomic Displacement Parameters (Å^2) of $Ba(CF_3COO)_2$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Ba	0.01447(14)	0.01716(14)	0.00877(11)	–0.00162(9)	0.00021(9)	0.00579(11)
C1	0.020(2)	0.016(2)	0.0136(18)	0.0012(16)	–0.0011(16)	0.0099(19)
C2	0.015(2)	0.023(2)	0.021(2)	–0.0055(17)	0.0017(17)	0.0056(19)
C3	0.022(2)	0.019(2)	0.0167(19)	–0.0009(16)	–0.0007(17)	0.0110(19)
C4	0.035(3)	0.034(3)	0.016(2)	0.0062(18)	0.0011(18)	0.023(2)
O1	0.0177(16)	0.0236(16)	0.0181(14)	–0.0031(12)	–0.0018(12)	0.0086(14)
O2	0.0214(16)	0.0192(16)	0.0153(13)	–0.0064(11)	–0.0001(11)	0.0060(13)
O3	0.0213(16)	0.0292(18)	0.0176(14)	0.0029(12)	–0.0024(12)	0.0132(14)
O4	0.0248(17)	0.0232(17)	0.0173(14)	0.0050(12)	0.0042(12)	0.0104(14)
F1	0.0339(16)	0.0400(17)	0.0266(13)	–0.0158(12)	0.0019(11)	0.0175(14)
F2	0.0335(16)	0.0312(16)	0.0294(14)	0.0109(11)	0.0135(11)	0.0143(13)
F3	0.0211(14)	0.0341(16)	0.0339(13)	–0.0005(12)	0.0010(11)	0.0148(13)
F4	0.0215(14)	0.0384(16)	0.0272(13)	0.0132(12)	0.0095(11)	0.0090(13)
F5	0.0302(16)	0.0348(17)	0.0313(14)	–0.0071(12)	–0.0048(12)	0.0043(13)
F6	0.0233(15)	0.0361(17)	0.0591(18)	0.0155(14)	0.0001(13)	0.0174(14)

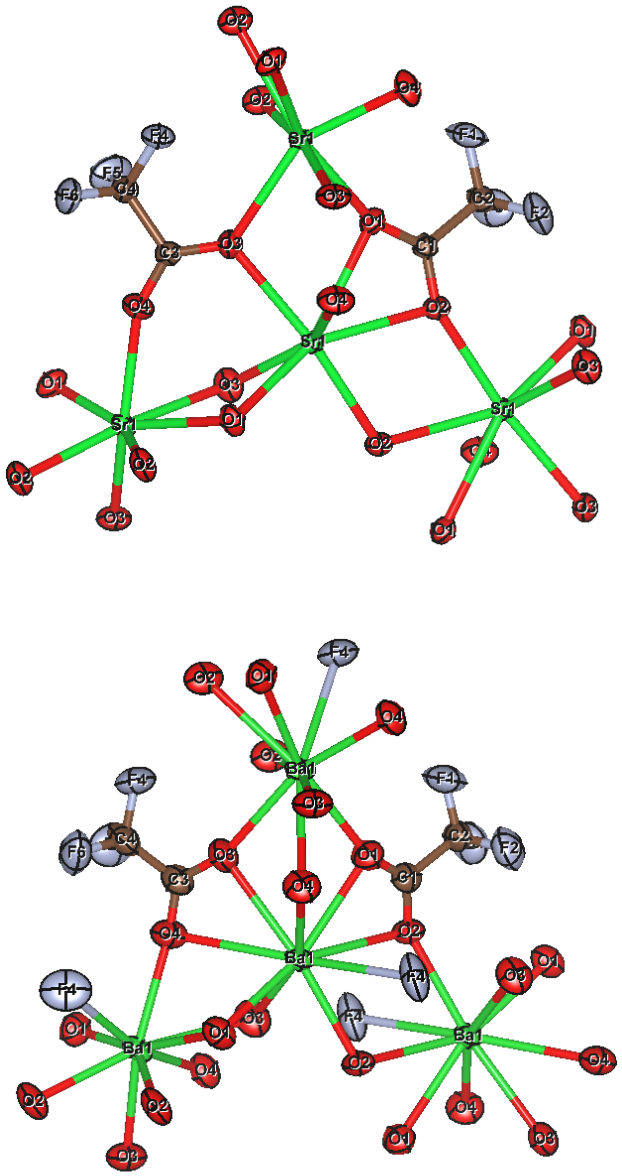

Figure S3. Thermal ellipsoids (90% probability) in $Sr(CF_3COO)_2$ (top) and $Ba(CF_3COO)_2$ (bottom).

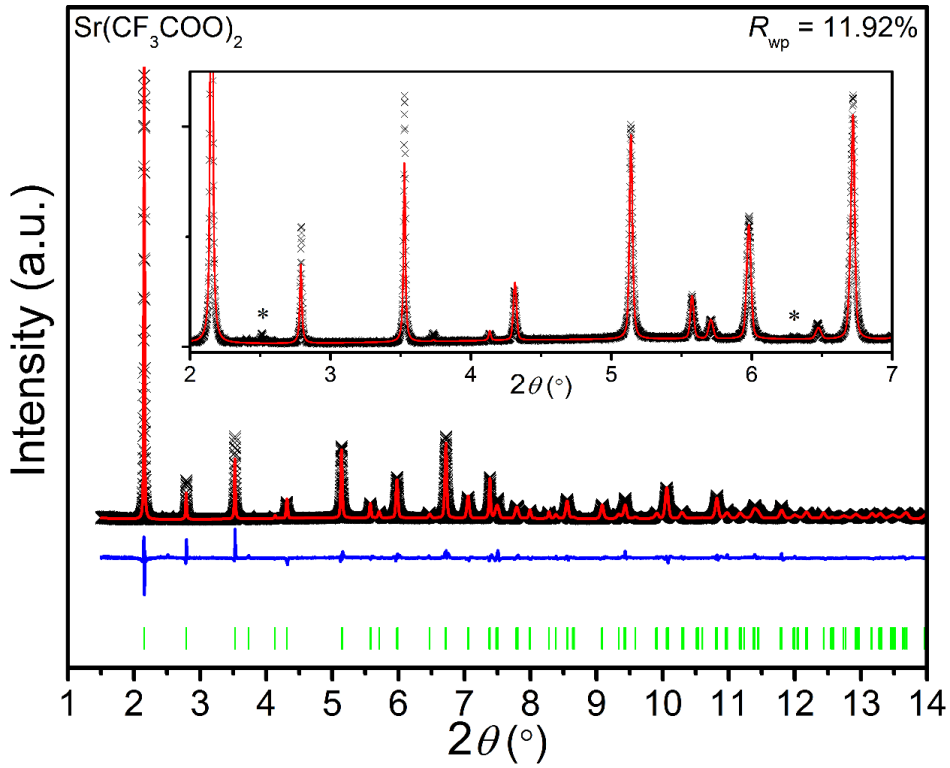

Figure S4. Rietveld analysis of the synchrotron XRD pattern of $Sr(CF_3COO)_2$. The experimental (×) and calculated (—) patterns are shown along with the difference curve (—) and the tick marks (|). Low-intensity diffraction maxima appearing at $2\theta \sim 2.51$ and 6.28° and belonging to trace secondary phases are shown in the inset. The following structural parameters were extracted from Rietveld analysis: $a = 21.9878(4)$ Å, $c = 9.4911(2)$ Å, $100 \times U_{Sr} = 3.31(6)$, $100 \times U_{C,O,F} = 6.28(11)$. Atomic positions were *not* refined.

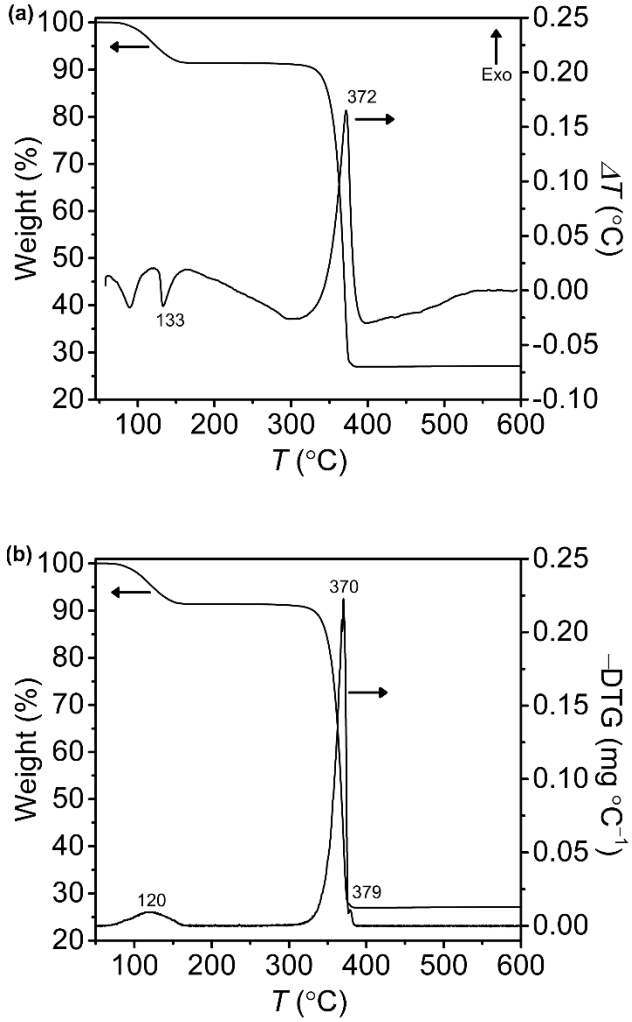

Figure S5. Thermal analysis of $Ca(CF_3COO)_2 \cdot H_2O$. (a) Thermogravimetric and differential thermal analysis. (b) Thermogram and differential thermogram (first derivative).

Table S5 Crystal and Structural Determination Data of $Ca(CF_3COO)_2 \cdot H_2O$

	$Ca(CF_3COO)_2 \cdot 4/3H_2O$
Chemical formula	$Ca_{0.75}H_2C_3O_4F_{4.50}$
Formula weight (g)	217.61
Temperature (°C)	25(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space-group	$P2_1/n$
Lattice parameters	a = 9.4572(3) Å b = 9.3568(3) Å c = 16.5456(5) Å $\alpha = \gamma = 90°$ β = 90.710(2)°
Volume (Å^3)	1463.99(8)
Z	8
Calculated density (g cm^{-3})	1.979
μ (mm^{-1})	0.747
$F(000)$	862
θ range (°)	3.06 to 37.60° (0.58 Å resolution)
Index ranges	$-16 \le h \le 16$ $-16 \le k \le 16$ $-28 \le l \le 28$
Reflections collected	133652
Independent reflections	133652
Independent reflections coverage	98.1%
Refinement method	Full-matrix least-squares on F^2
Function minimized	$\Sigma\, w(F_o^2 - F_c^2)^2$
Weighting scheme	$w = 1 / [\sigma^2(F_o^2) + (0.0887P)^2 + 4.3049P]$ where $P = (F_o^2 + 2F_c^2) / 3$
Data, restraints, parameters	133652, 150, 321
Largest difference peak and hole (e Å^{-3})	2.112 and −2.043
r.m.s. deviation from mean (e Å^{-3})	0.230
R	9.25% (84283 data with $I > 2\sigma(I)$) 17.37% (all data)
R_w	23.72% (84283 data with $I > 2\sigma(I)$) 26.28% (all data)
Goodness-of-fit on F^2	1.057

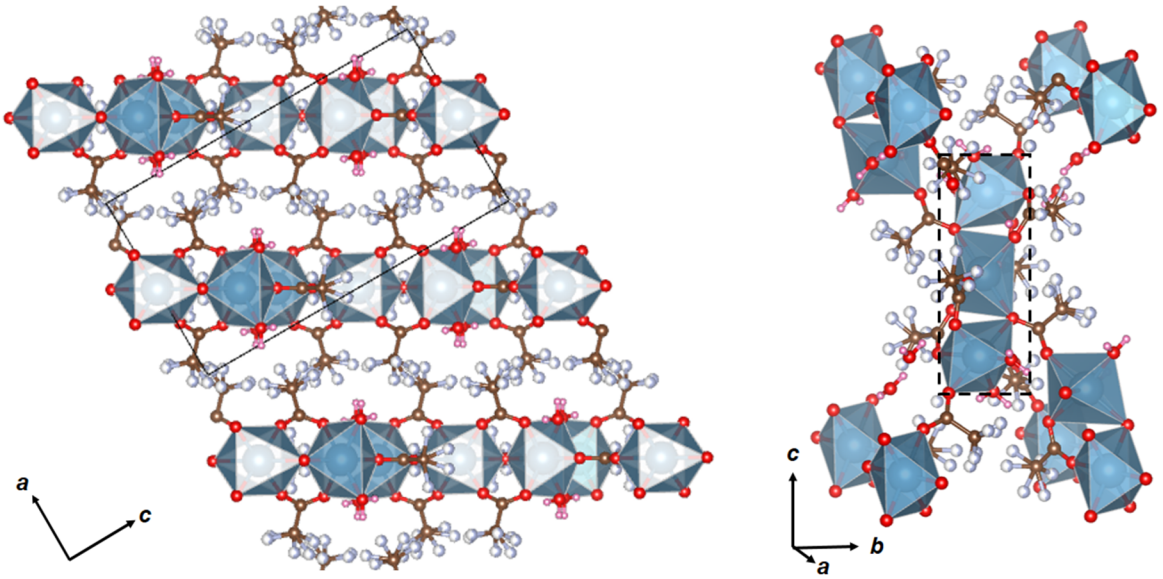

Figure S6. Crystal structure of $Ca(CF_3COO)_2{\cdot}H_2O$. Left panel: layered structure seen along the *b* axis in the (101) planes, with the layers running parallel to the *b* axis and the trifluoromethyl groups directed outwards. The trifluoromethyl groups are rotationally disordered and occupy two different positions. Right panel: trimer of calcium–oxygen edge-sharing octahedra (indicated with a dashed line). The central calcium atom is coordinated to six oxygen atoms belonging to carboxylate groups, whereas the terminal calcium atoms are coordinated to four oxygen atoms belonging to carboxylate groups and two oxygen atoms from two water molecules. The two coordinating water molecules are *cis* positioned. The crystal structure is in excellent agreement with that reported by Khristov et al.[8] (Ca: blue; C: brown; O: red: F: light blue; H: pink).

Table S6 Fractional Atomic Coordinates and Isotropic Displacement Parameters of $Ca(CF_3COO)_2 \cdot H_2O$

Atom	*x*	*y*	*z*	U_{eq} (Å^2)
Ca1	0.20398(8)	0.43373(8)	0.29379(4)	0.0290(2)
Ca2	0.0000	0.5000	0.5000	0.0356(3)
O1A	0.3193(4)	0.2825(4)	0.39005(18)	0.0567(9)
C1A	0.2984(5)	0.3044(5)	0.4613(3)	0.0409(9)
O2A	0.2048(5)	0.3720(5)	0.4925(3)	0.0974(16)
C2A	0.4036(5)	0.2438(5)	0.5178(3)	0.0774(19)
F1A	0.4916(9)	0.1513(10)	0.4815(4)	0.095(3)
F2A	0.4881(12)	0.3451(9)	0.5467(9)	0.178(6)
F3A	0.3575(10)	0.1724(13)	0.5814(6)	0.152(5)
F1A'	0.5319(7)	0.2989(14)	0.5124(6)	0.120(4)
F2A'	0.3628(11)	0.2738(18)	0.5927(4)	0.153(5)
F3A'	0.4173(17)	0.1037(6)	0.5149(9)	0.168(6)
O1B	–0.0972(5)	0.3730(4)	0.3945(3)	0.0826(13)
C1B	–0.0953(4)	0.3017(5)	0.3330(3)	0.0402(9)
O2B	0.0024(3)	0.2820(4)	0.2866(2)	0.0527(9)
C2B	–0.2306(4)	0.2269(4)	0.3121(2)	0.0571(13)
F1B	–0.3216(11)	0.2296(17)	0.3722(6)	0.138(5)
F2B	–0.2941(13)	0.2986(11)	0.2520(7)	0.110(5)
F3B	–0.2237(10)	0.0937(7)	0.2869(9)	0.096(4)
F1B'	–0.3435(6)	0.3095(7)	0.3113(7)	0.111(3)
F2B'	–0.2306(8)	0.1601(11)	0.2411(4)	0.104(3)
F3B'	–0.2610(8)	0.1226(8)	0.3649(4)	0.092(3)
O1C	0.2049(3)	0.7622(3)	0.29705(16)	0.0393(7)
C1C	0.1407(4)	0.7366(4)	0.3598(2)	0.0289(8)
O2C	0.1123(3)	0.6161(3)	0.38689(16)	0.0394(7)
C2C	0.0924(4)	0.8623(4)	0.4086(2)	0.0525(13)
F1C	0.0650(10)	0.9826(6)	0.3708(4)	0.090(3)
F2C	0.2009(6)	0.8983(8)	0.4600(4)	0.098(3)
F3C	–0.0128(7)	0.8356(7)	0.4576(4)	0.084(3)
F1C'	0.0753(9)	0.8371(7)	0.4859(2)	0.078(2)
F2C'	–0.0410(6)	0.8946(8)	0.3817(4)	0.089(3)
F3C'	0.1612(8)	0.9824(6)	0.4004(5)	0.089(3)
O1W	0.4289(3)	0.5319(4)	0.3005(2)	0.0554(9)
O2W	0.0760(4)	0.5346(4)	0.1868(2)	0.0544(9)
H11W	0.507(3)	0.502(5)	0.323(3)	0.083
H12W	0.456(5)	0.601(4)	0.269(3)	0.083
H21W	0.011(5)	0.484(5)	0.162(3)	0.082
H22W	0.098(5)	0.600(4)	0.152(2)	0.082

Table S7 Anisotropic Atomic Displacement Parameters (Å^2) of $Ca(CF_3COO)_2 \cdot H_2O$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Ca1	0.0306(4)	0.0290(4)	0.0275(4)	–0.0055(3)	0.0050(3)	0.0042(3)
Ca2	0.0524(7)	0.0257(6)	0.0293(5)	–0.0025(4)	0.0254(5)	0.0021(5)
O1A	0.081(2)	0.060(2)	0.0295(16)	0.0066(14)	–0.0068(15)	–0.0055(18)
C1A	0.048(2)	0.030(2)	0.045(2)	0.0051(18)	0.0023(18)	0.0043(17)
O2A	0.079(3)	0.054(3)	0.161(5)	0.001(3)	0.046(3)	0.029(2)
C2A	0.105(5)	0.086(5)	0.041(3)	–0.001(3)	–0.013(3)	0.018(4)
F1A	0.091(5)	0.107(6)	0.086(5)	0.002(4)	–0.012(4)	0.064(4)
F2A	0.170(8)	0.188(8)	0.173(8)	–0.068(6)	–0.098(6)	0.004(6)
F3A	0.168(7)	0.165(8)	0.123(6)	0.108(6)	0.034(5)	0.081(6)
F1A'	0.064(5)	0.181(9)	0.115(7)	–0.010(6)	–0.018(5)	0.018(5)
F2A'	0.214(8)	0.217(9)	0.029(4)	0.008(5)	–0.013(5)	–0.062(7)
F3A'	0.204(9)	0.136(8)	0.163(9)	0.048(6)	–0.080(7)	0.039(7)
O1B	0.122(4)	0.060(3)	0.066(3)	–0.023(2)	0.001(2)	–0.015(2)
C1B	0.041(2)	0.032(2)	0.048(2)	0.0043(18)	0.0034(18)	–0.0008(17)
O2B	0.0331(16)	0.053(2)	0.073(2)	0.0037(16)	0.0155(15)	–0.0025(14)
C2B	0.034(3)	0.076(4)	0.062(3)	0.002(3)	0.010(2)	0.000(2)
F1B	0.080(6)	0.201(9)	0.133(7)	–0.015(6)	0.064(6)	–0.022(6)
F2B	0.091(6)	0.114(7)	0.122(7)	0.003(6)	–0.057(5)	0.002(5)
F3B	0.065(5)	0.048(5)	0.174(9)	–0.012(5)	–0.016(6)	–0.012(4)
F1B'	0.044(3)	0.090(5)	0.199(8)	0.002(5)	–0.009(4)	0.025(3)
F2B'	0.089(5)	0.134(7)	0.089(5)	–0.044(5)	0.001(4)	–0.046(5)
F3B'	0.077(4)	0.085(5)	0.115(5)	0.040(4)	–0.011(4)	–0.038(4)
O1C	0.0476(17)	0.0396(17)	0.0312(14)	0.0074(11)	0.0193(12)	–0.0023(13)
C1C	0.0352(19)	0.0267(19)	0.0251(17)	0.0008(13)	0.0118(14)	–0.0020(14)
O2C	0.0585(18)	0.0237(14)	0.0366(15)	0.0026(11)	0.0251(13)	–0.0028(12)
C2C	0.079(4)	0.028(2)	0.051(3)	–0.001(2)	0.027(2)	0.002(2)
F1C	0.149(5)	0.037(3)	0.086(4)	0.000(3)	0.005(3)	0.020(3)
F2C	0.106(4)	0.093(4)	0.094(4)	–0.051(3)	–0.007(3)	0.000(3)
F3C	0.088(4)	0.078(4)	0.089(4)	–0.029(3)	0.060(3)	–0.002(3)
F1C'	0.122(4)	0.074(4)	0.038(3)	–0.009(2)	0.021(3)	0.021(3)
F2C'	0.088(4)	0.083(4)	0.097(4)	–0.015(3)	0.011(3)	0.040(3)
F3C'	0.104(4)	0.039(3)	0.124(4)	–0.012(3)	0.040(3)	–0.009(3)
O1W	0.0412(18)	0.046(2)	0.079(3)	0.0073(17)	–0.0019(16)	–0.0065(15)
O2W	0.062(2)	0.051(2)	0.051(2)	0.0124(16)	–0.0170(16)	–0.0041(16)

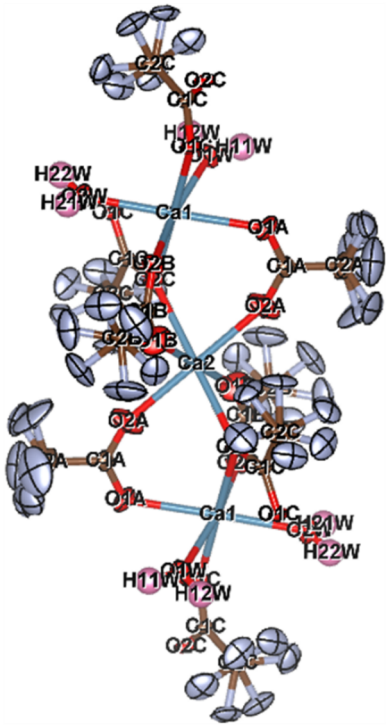

Figure S7. Thermal ellipsoids (30% probability) in $Ca(CF_3COO)_2 \cdot H_2O$.

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