Electronic Supplementary Material (ESI) for CrystEngComm.

Supporting information

Crystal Structures of Hydrated Rare-Earth Bis(trifluoromethylsulfonyl)imide Salts

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Full experimental details:

Scandium(III) bis(trifluoromethylsulfonyl)imide: Scandium(III) oxide (10 mmol, 1.404 g) and a solution of 80 wt% hydrogen bis(trifluoromethylsulfonyl)imide (29 mmol, 10.160 g) in water (30mL) heated for two hours. Crystals of $[Sc(H_2O)_7][Tf_2N]_3{\cdot}H_2O$ grown at room temperature on a Schlenk line.

Lanthanum(III) bis(trifluoromethylsulfonyl)imide: Lanthanum(III) oxide (4.95 mmol, 1.614 g) and a solution of 80 wt% hydrogen bis(trifluoromethylsulfonyl)imide (30.0 mol, 10.56 g) in water (30 mL) heated for one hour. Crystals of $La(H_2O)_3(Tf_2N)_3$grown at room temperature on a Schlenk line. Crystals of $La(H_2O)_3(Tf_2N)_3{\cdot}2.5H_2O$ obtained if HTf_2N remained from the initial reaction.

Cerium(III) bis(trifluoromethylsulfonyl)imide : $Ce_2(CO_3)_3$ (3.68 g, 6.12 mmol) and a solution of 80 wt% hydrogen bis(trifluoromethylsulfonyl)imide (4.30 g, 12.2 mmol)in water (30 mL) were mixed under reduced pressure (20 mbar) at 60 °C. The remaining $Ce_2(CO_3)_3$ was filtered off after one hour. Crystals of $Ce(H_2O)_3(Tf_2N)_3$grown at room temperature on a Schlenk line.

Praseodymium(III) bis(trifluoromethylsulfonyl)imide: Praseodymium(III,IV)oxide (Pr_6O_{11}) (3.81 mmol, 3.90 g) and a solution of 80 wt% hydrogen bis(trifluoromethylsulfonyl)imide (30.0 mmol, 10.56 g) in water (30 mL) heated for one hour. Crystals of $Pr(H_2O)_3(Tf_2N)_3$grown at room temperature on a Schlenk line.

Neodymium(III) bis(trifluoromethylsulfonyl)imide: Neodymium(III) oxide (4.99 mmol, 1.68 g) and a solution of 80 wt% hydrogen bis(trifluoromethylsulfonyl)imide (30.0 mmol, 10.56 g) in water (30mL) heated for one hour. Crystals of $Nd(H_2O)_3(Tf_2N)_3$grown at room temperature on a Schlenk line.

Samarium(III) bis(trifluoromethylsulfonyl)imide: Samarium(III) oxide (6.28 mmol, 2.19 g) was added to a solution of 80 wt% hydrogen bis(trifluoromethylsulfonyl)imide (0.030 mol, 10.56 g) in water (30mL) and heated for one hour. Crystals of $Sm(H_2O)_5(Tf_2N)_3$grown at room temperature on Schlenk line.

Gadolinium(III) bis(trifluoromethylsulfonyl)imide: Gadolinium(III) oxide (6.02 mmol, 2.18 g) and a solution of 80 wt% hydrogen bis(trifluoromethylsulfonyl)imide (12.0 mmol, 4.23 g) in water (30mL) heated for one hour. Crystals of fully hydrated $Gd(Tf_2N)_3$grown at room temperature on Schlenk line. Crystals of $Gd(H_2O)_3(Tf_2N)_3$grown at 60°C on the Schlenk line.

Terbium(III) bis(trifluoromethylsulfonyl)imide: Terbium(III) oxide (6.12 mmol, 2.19 g) and a solution of 80 wt% hydrogen bis(trifluoromethylsulfonyl)imide (12.0 mmol, 4.22 g) in water (30mL) and heated for one hour. Crystals of $Tb(H_2O)_5(Tf_2N)_3$grown at room temperature on Schlenk line.

Dysprosium(III) bis(trifluoromethylsulfonyl)imide: Dysprosium(III) oxide (5.17 mmol, 1.930 g) and a solution of 80 wt% hydrogen bis(trifluoromethylsulfonyl)imide (30.0 mmol, 10.62 g) in water (30mL) heated for 1 hour. Crystals of fully hydrated $Dy(Tf_2N)_3$grown at room temperature on Schlenk line. Crystals of $Dy(H_2O)_5(Tf_2N)_3$grown at 60°C on the Schlenk line.

Holmium(III) bis(trifluoromethylsulfonyl)imide: Holmium(III) oxide (10.33 mmol, 3.905 g) and a solution of 80 wt% hydrogen bis(trifluoromethylsulfonyl)imide (63.3 mmol, 22.250 g) in water (30mL) and heated for one hour. Crystals of fully hydrated $Ho(Tf_2N)_3$grown at room temperature on Schlenk line.

Erbium(III) bis(trifluoromethylsulfonyl)imide: Erbium(III) oxide (9.68 mmol, 3.706 g) and a solution of 80 wt% hydrogen bis(trifluoromethylsulfonyl)imide (55.1 mmol, 19.365 g) in water (30mL) heated for one hour. Crystals of fully hydrated $Er(Tf_2N)_3$grown at room temperature on Schlenk line. Crystals of $Er(H_2O)_5(Tf_2N)_3$grown at 60°C on the Schlenk line.

Thulium(III) bis(trifluoromethylsulfonyl)imide: Thulium(III) oxide (5.95 mmol mol, 2.29 g) and a solution of 80 wt% hydrogen bis(trifluoromethylsulfonyl)imide (11.9 mmol, 4.18 g) in water and (30mL) heated for one hour. A solid material could be grown at room temperature on Schlenk line but no crystals suitable for X-ray diffraction could be isolated. On one occasion a crystal of probable formula formula $[Tm_6(O)(OH)_8(OH_2)_{16}(Tf_2N)_2][Tf_2N]_6{\cdot}xH_2O$ was found after several days at 60°C on the Schlenk line.

Ytterbium (III) bis(trifluoromethylsulfonyl)imide: Ytterbium(III) oxide (5.91 mmol, 2.31 g) and a solution of 80 wt% hydrogen bis(trifluoromethylsulfonyl)imide (11.9 mmol, 4.19 g) in water (50mL) stirred under reflux conditions (100 °C) for two hours. Crystals of fully hydrated $Yb(Tf_2N)_3$grown at room temperature on Schlenk line. Crystals of $Yb(H_2O)_5(Tf_2N)_3$grown at 60°C on the Schlenk line.

Lutetium(III) bis(trifluoromethylsulfonyl)imide: Lutetium(III) oxide (5.91 mmol, 2.35 g) and a solution of 80 wt% hydrogen bis(trifluoromethylsulfonyl)imide (11.8 mmol, 4.15 g) in water (50mL) stirred under reflux conditions (100 °C) for two hours. Crystals of fully hydrated $Lu(Tf_2N)_3$grown at room temperature on Schlenk line.

Yttrium(III) bis(trifluoromethylsulfonyl)imide: Yttrium(III) oxide (5.49 mmol, 1.240 g) and a solution of 80 wt% hydrogen bis(trifluoromethylsulfonyl)imide (30.5 mmol, 10.709 g) in water (50mL) stirred under reflux conditions (100 °C) for two hours. The reaction mixture turned into a clear solution after approximately 10 minutes. Water and acid were removed by rotary evaporation in vacuo to obtain white crystals. Crystals of fully hydrated $Y(Tf_2N)_3$grown at room temperature on Schlenk line.

Additional Figures:

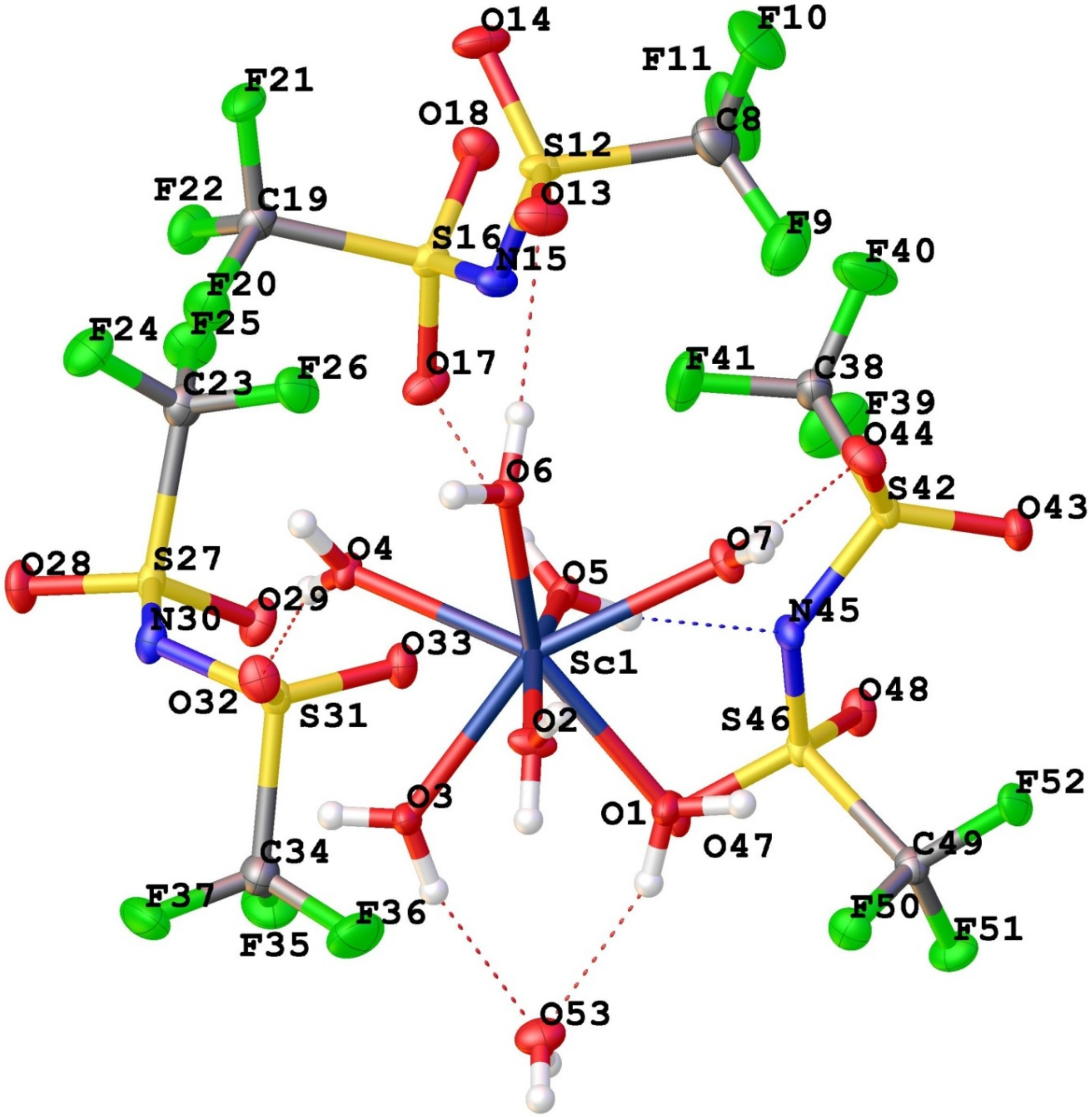

Figure S1. View of the crystal structure of $[Sc(H_2O)_7][Tf_2N]_3 \cdot H_2O$ showing the crystallographic asymmetric unit and the displacement ellipsoids at the 50% probability level.

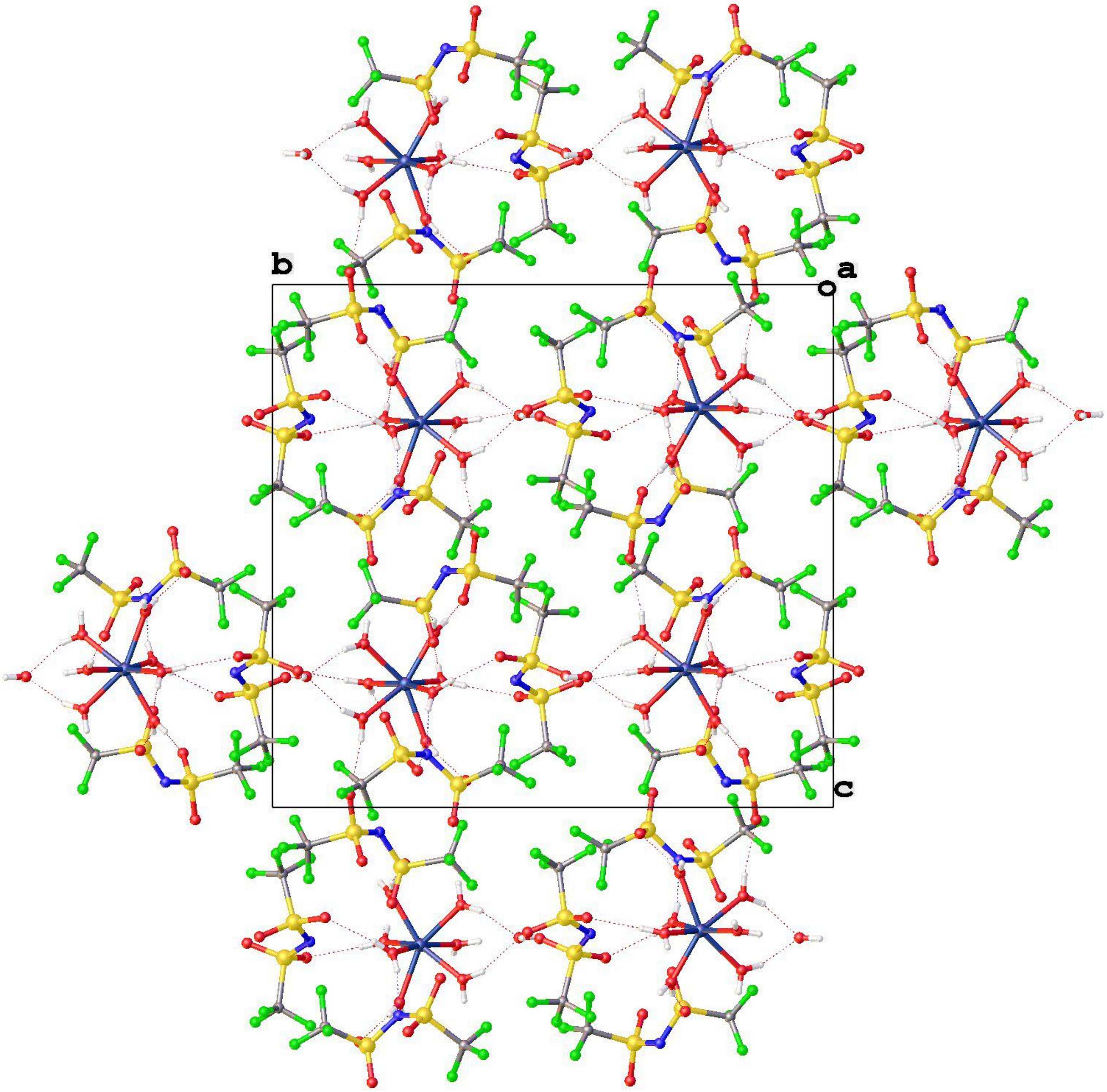

Figure S2. View of the packing in the crystal structure of $[Sc(H_2O)_7][Tf_2N]_3 \cdot H_2O$.

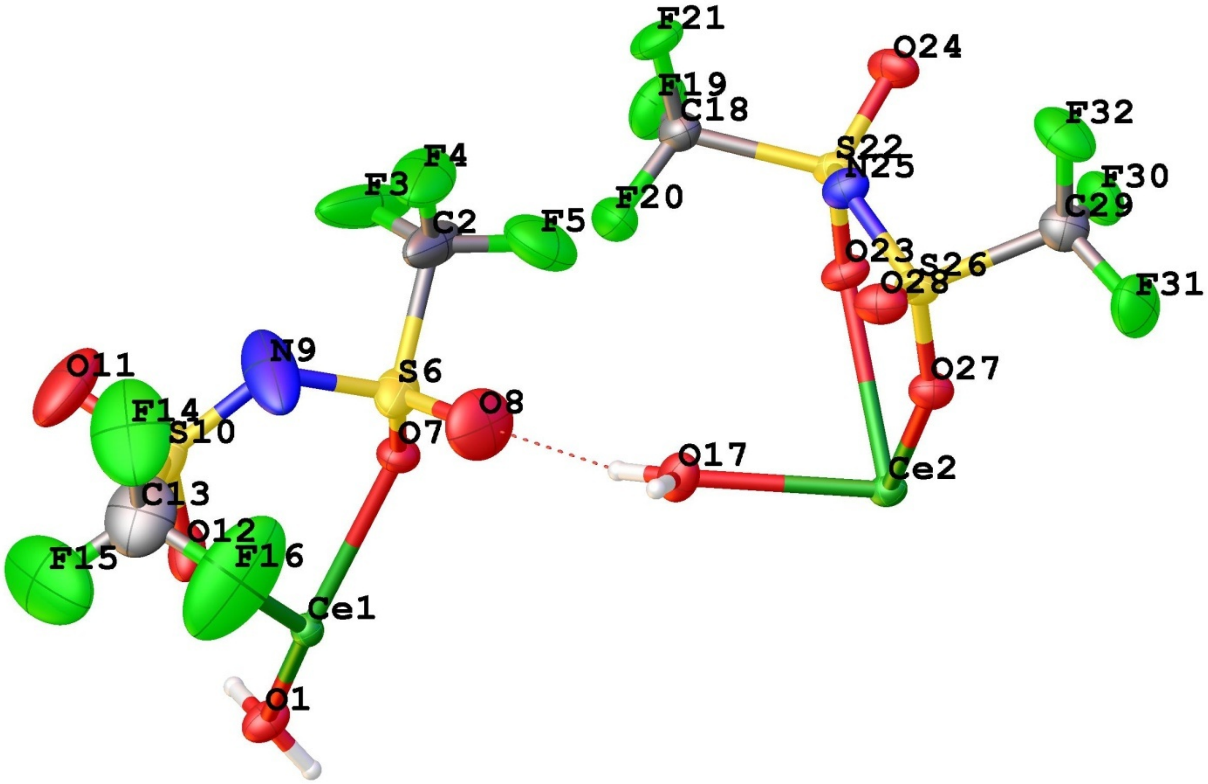

Figure S3. View of the crystal structure of $Ce(H_2O)_3(Tf_2N)_3$ showing the crystallographic asymmetric unit and the displacement ellipsoids at the 50% probability level.

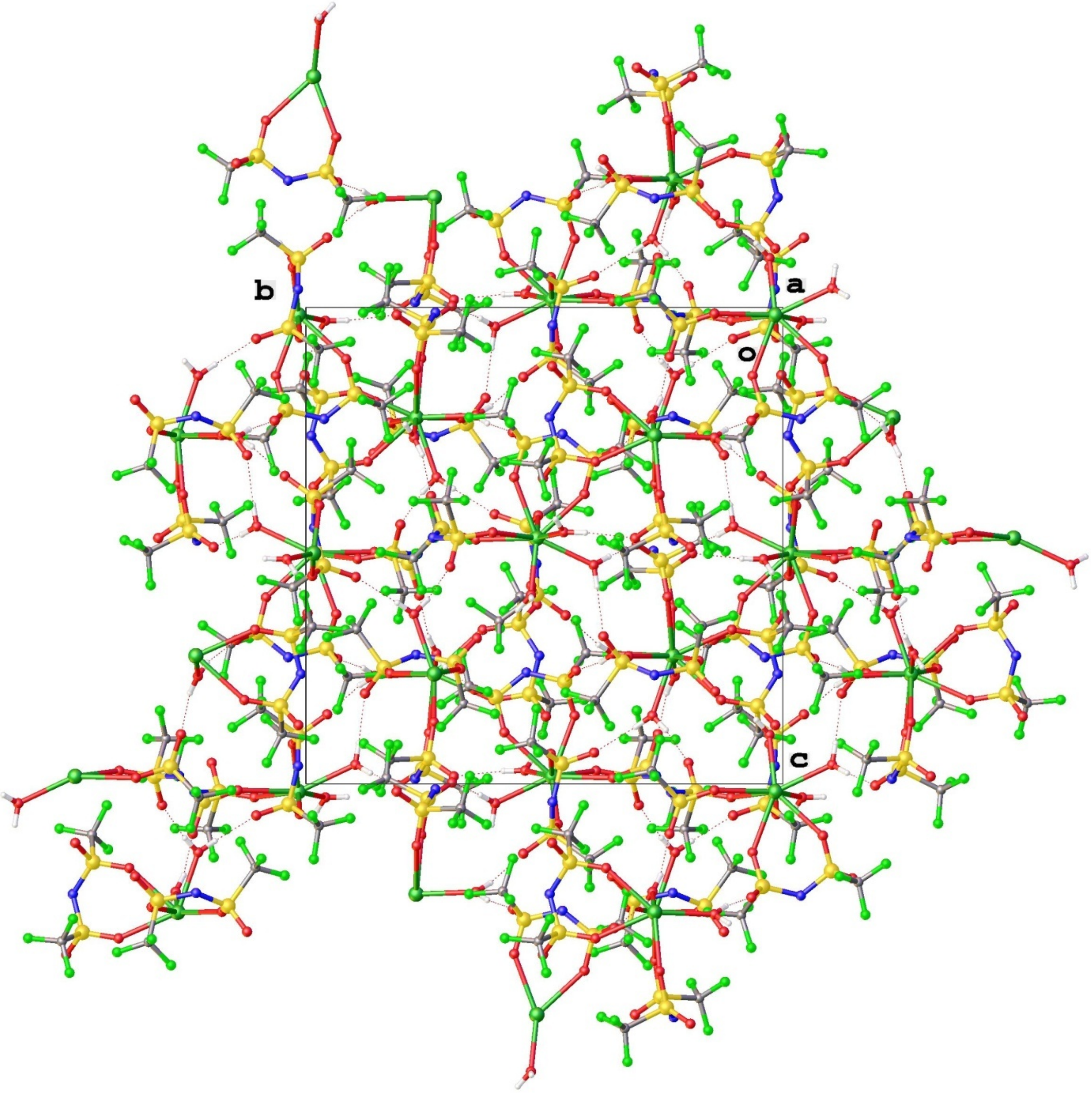

Figure S4. View of the packing in the crystal structure of $Ce(H_2O)_3(Tf_2N)_3$.

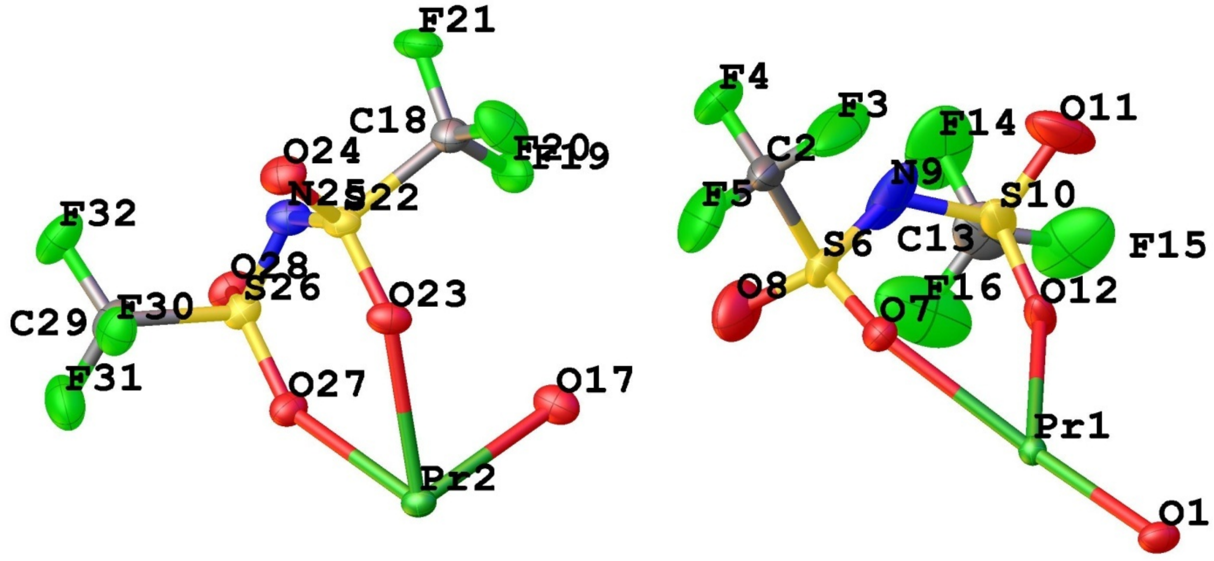

Figure S5. View of the crystal structure of $Pr(H_2O)_3(Tf_2N)_3$ showing the crystallographic asymmetric unit and the displacement ellipsoids at the 50% probability level.

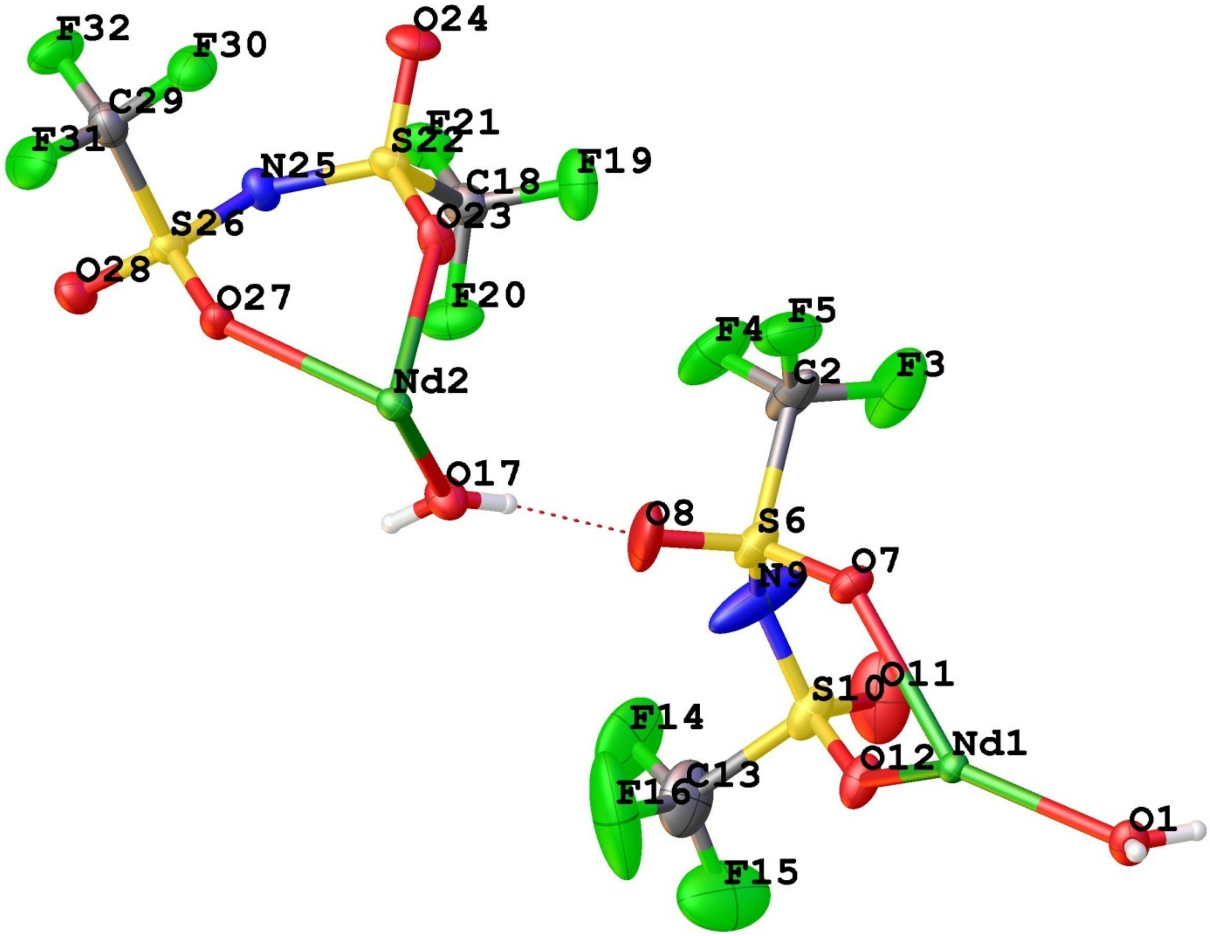

Figure S6. View of the crystal structure of $Nd(H_2O)_3(Tf_2N)_3$ showing the crystallographic asymmetric unit and the displacement ellipsoids at the 50% probability level.

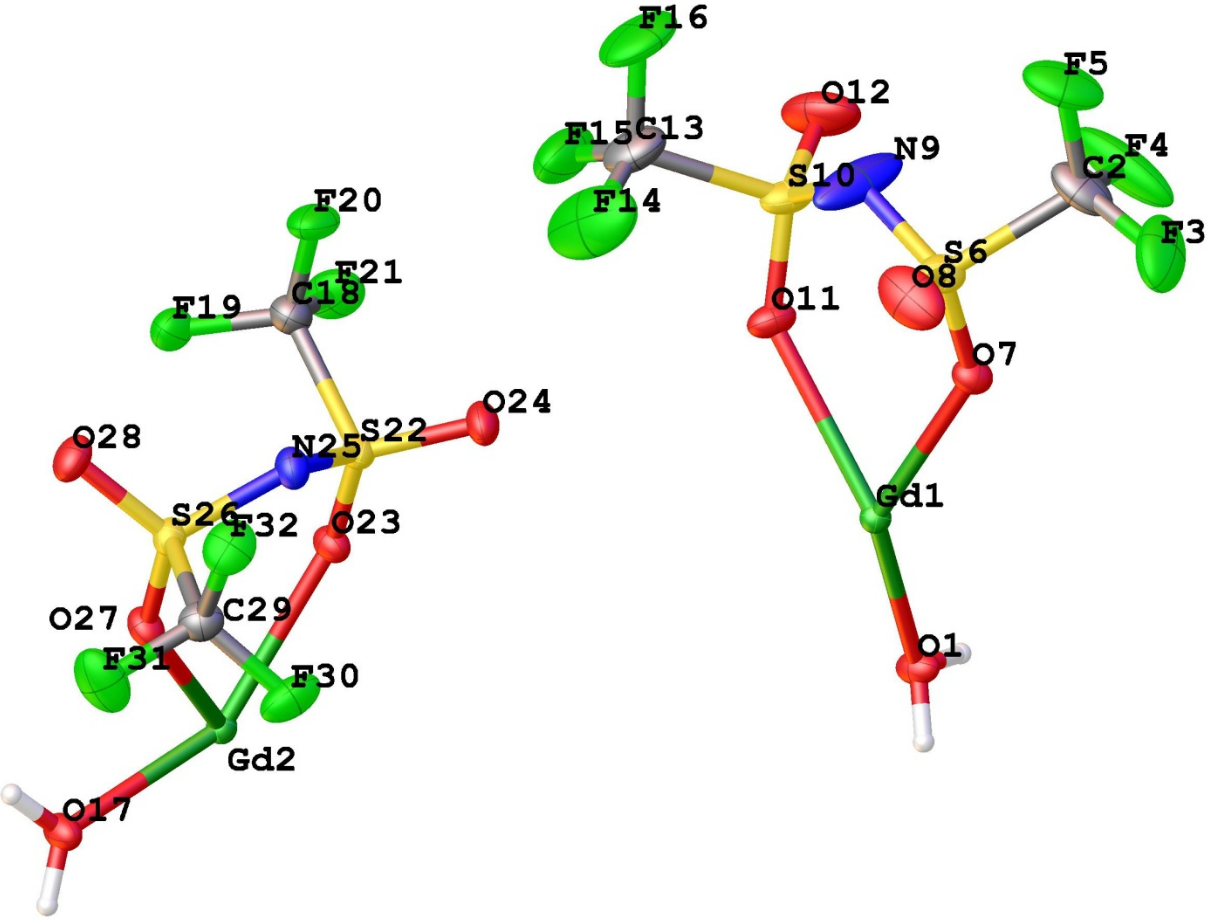

Figure S7. View of the crystal structure of Gd(H_2O)$_3$(Tf_2N)$_3$ showing the crystallographic asymmetric unit and the displacement ellipsoids at the 50% probability level.

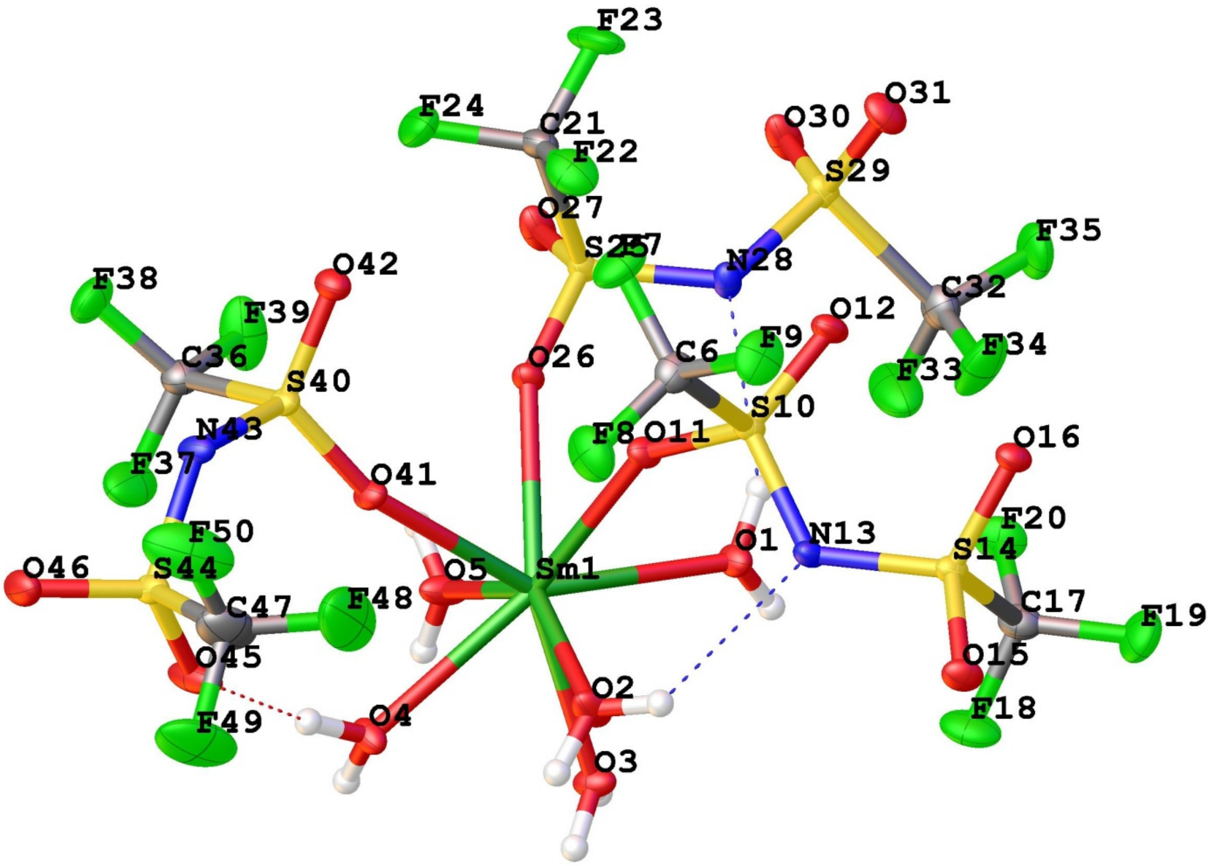

Figure S8. View of the crystal structure of Sm(H_2O)$_5$(Tf_2N)$_3$ showing the crystallographic asymmetric unit and the displacement ellipsoids at the 50% probability level.

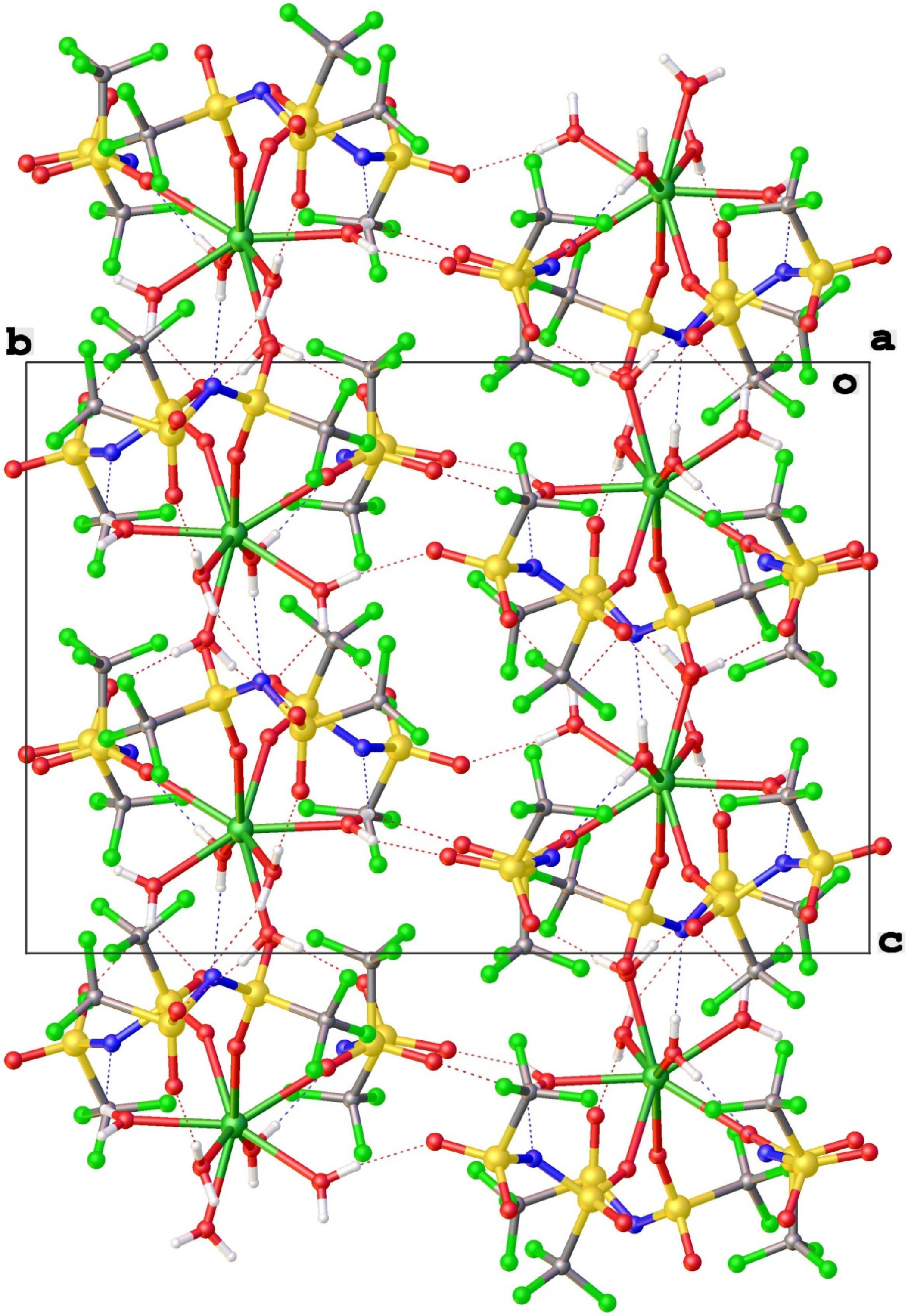

Figure S9. View of the packing in the crystal structure of $Sm(H_2O)_5(Tf_2N)_3$.

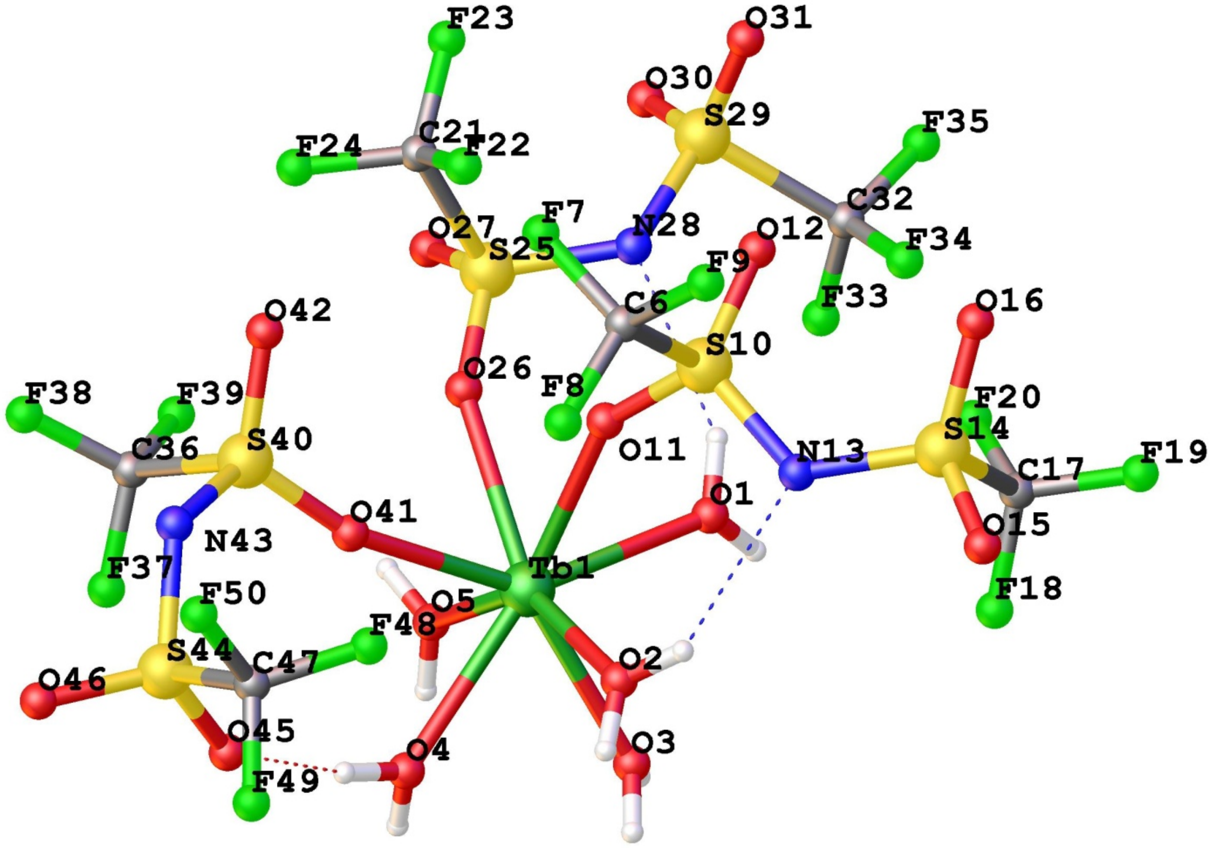

Figure S10. View of the crystal structure of $Tb(H_2O)_5(Tf_2N)_3$ showing the crystallographic asymmetric unit and the displacement ellipsoids at the 50% probability level.

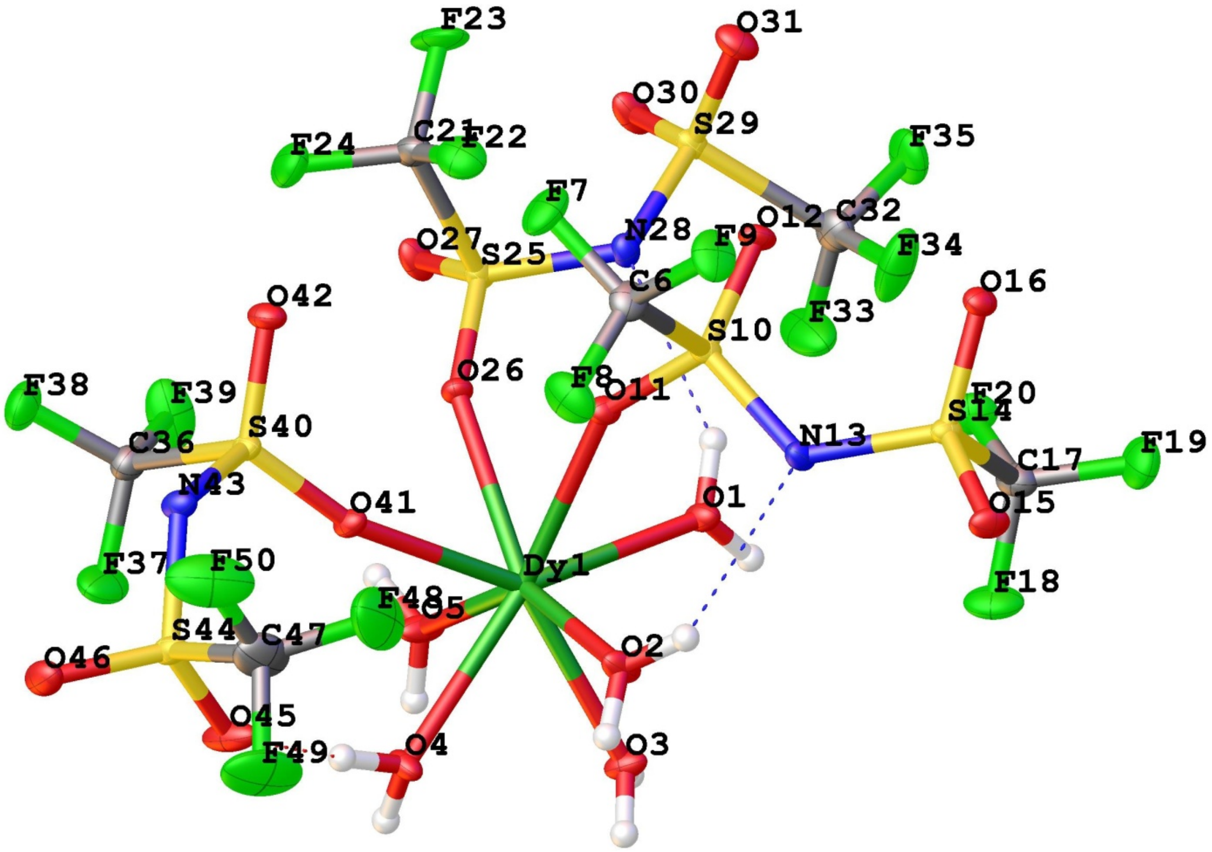

Figure S11. View of the crystal structure of $Dy(H_2O)_5(Tf_2N)_3$ showing the crystallographic asymmetric unit and the displacement ellipsoids at the 50% probability level.

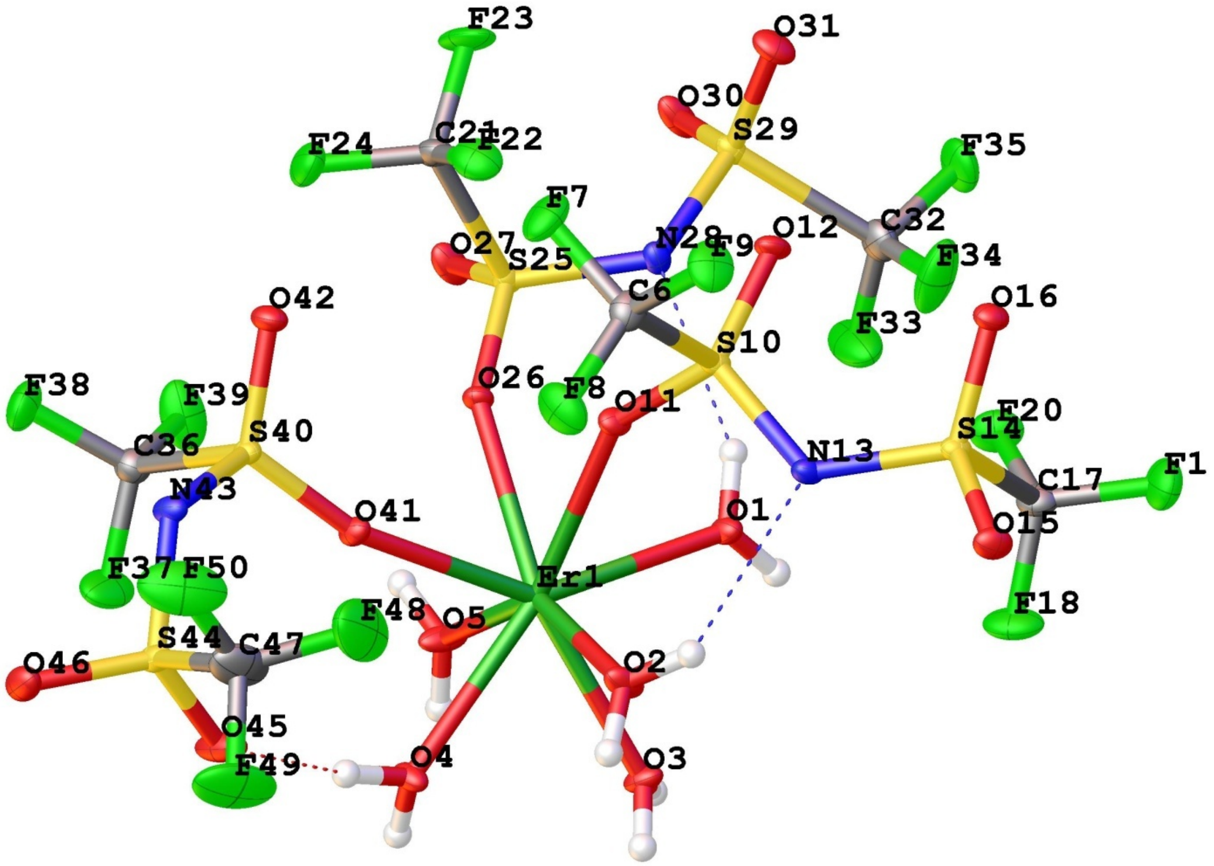

Figure S12. View of the crystal structure of Er(H$_2$O)$_5$(Tf$_2$N)$_3$ showing the crystallographic asymmetric unit and the displacement ellipsoids at the 50% probability level.

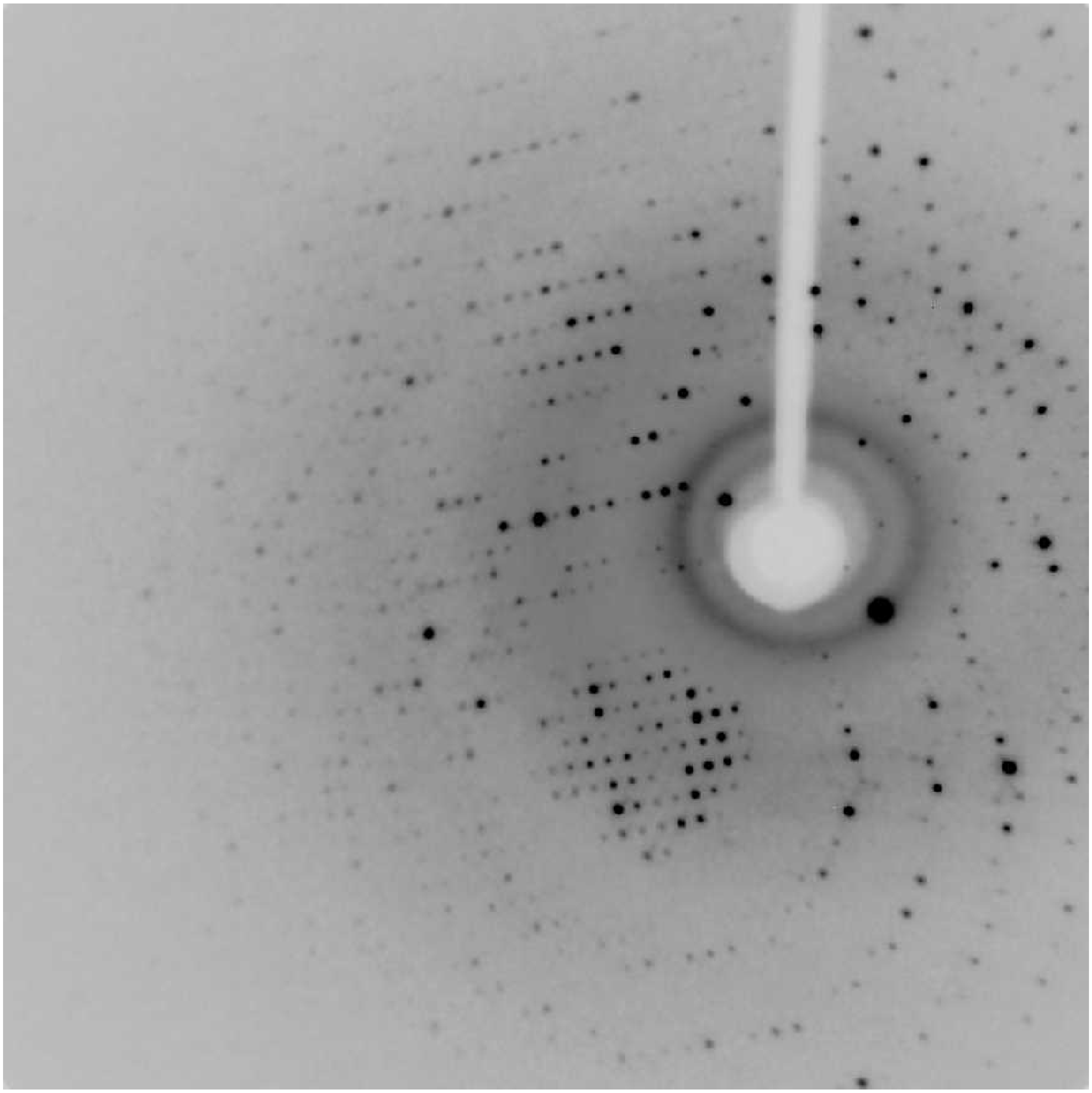

Figure S13. Example of a diffraction image for the fully hydrated structure of $Gd(Tf_2N)_3$.

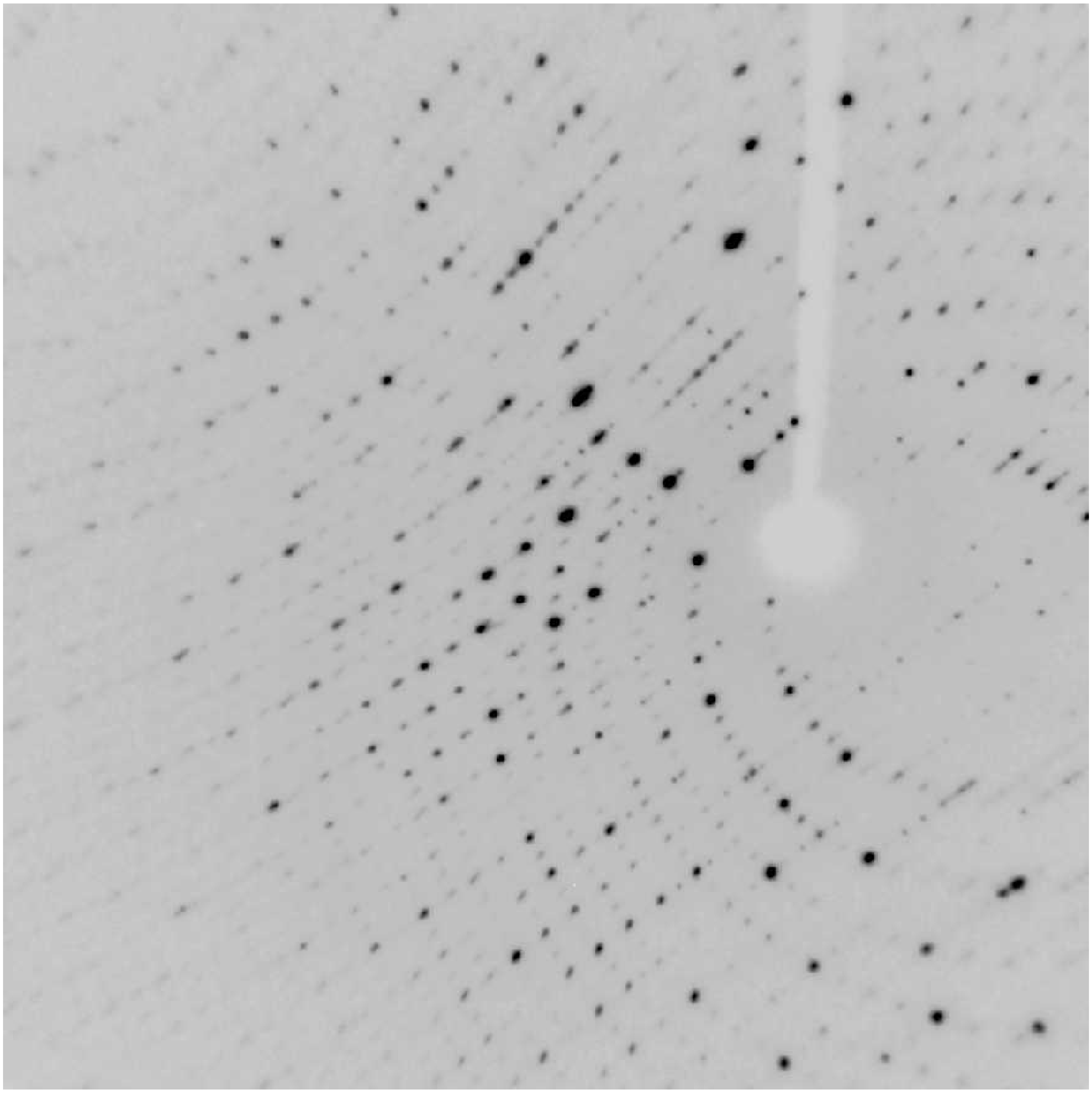

Figure S14. Example of a diffraction image for the fully hydrated structure of Dy(Tf_2N)$_3$.

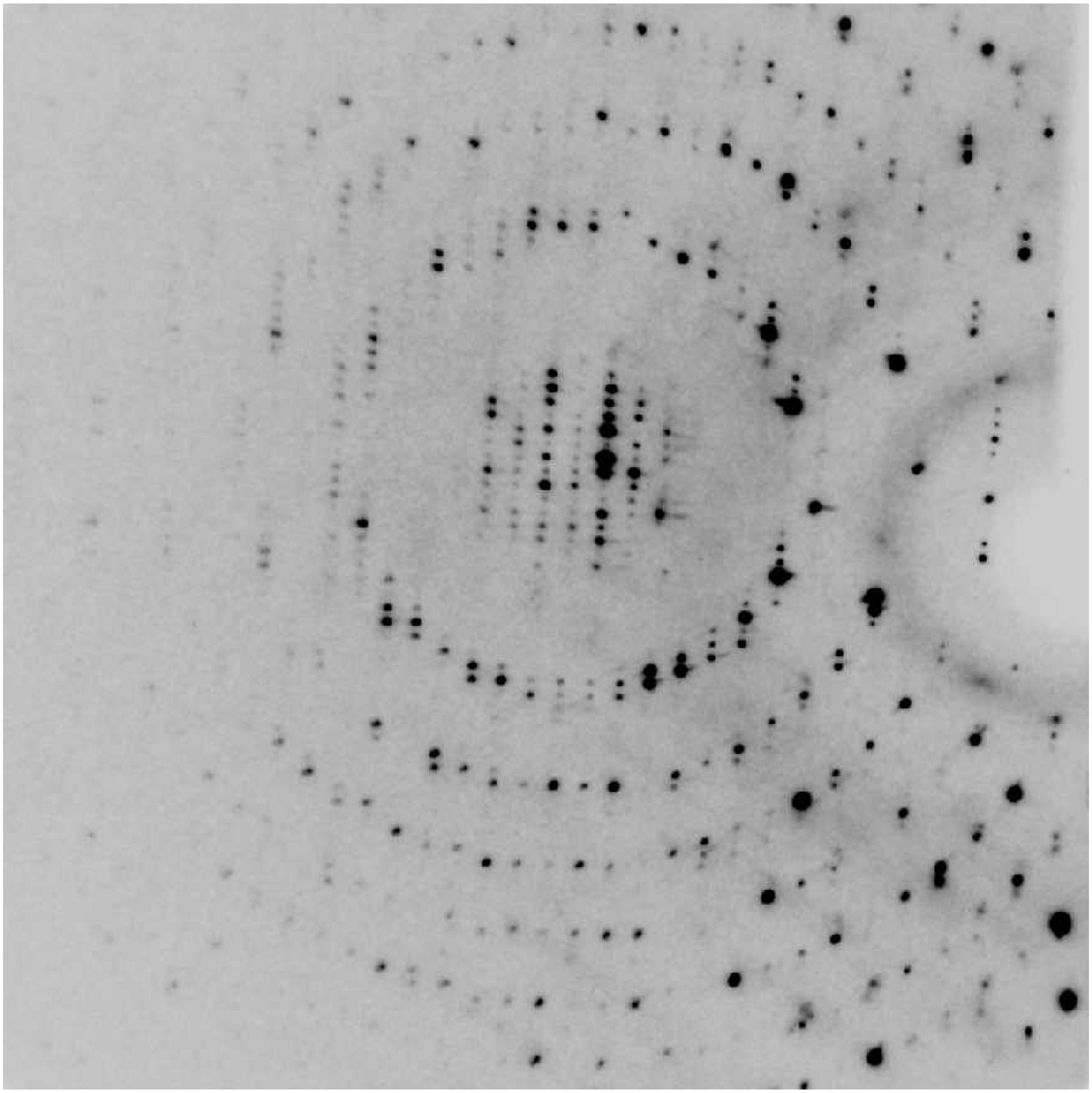

Figure S15. Example of a diffraction image for the fully hydrated structure of $Ho(Tf_2N)_3$.

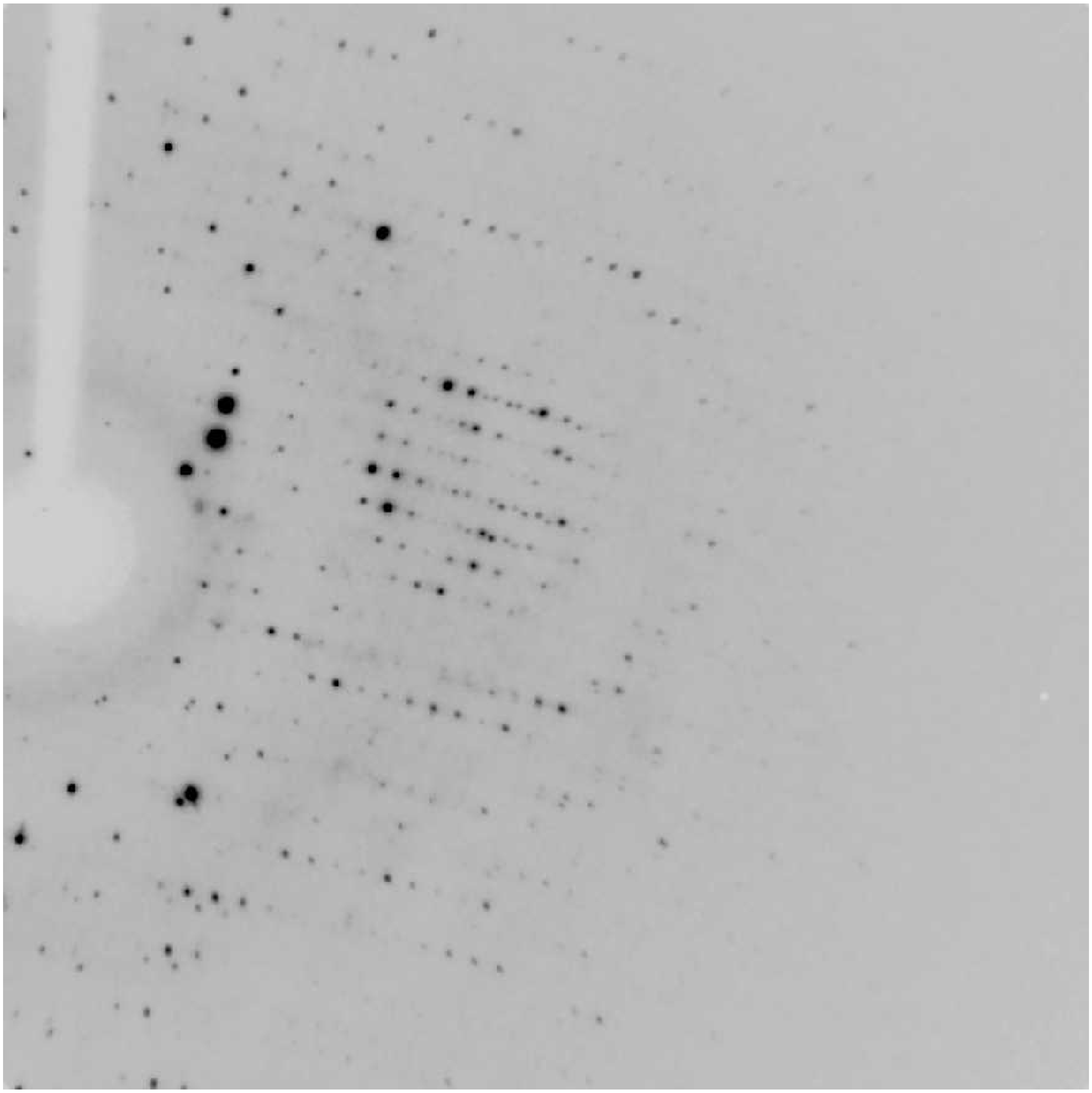

Figure S16. Example of a diffraction image for the fully hydrated structure of $Er(Tf_2N)_3$.

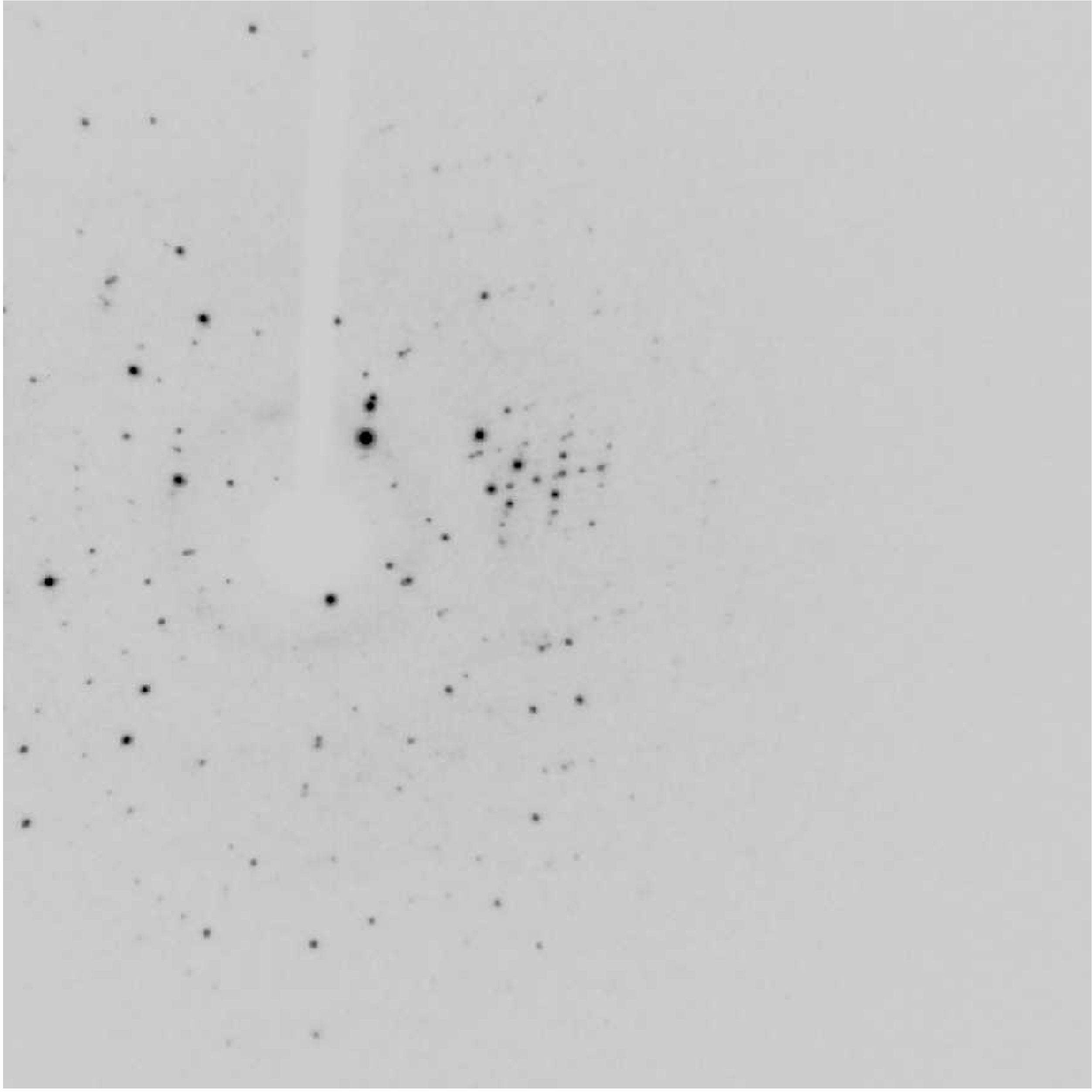

Figure S17. Example of a diffraction image for the fully hydrated structure of $Yb(Tf_2N)_3$.

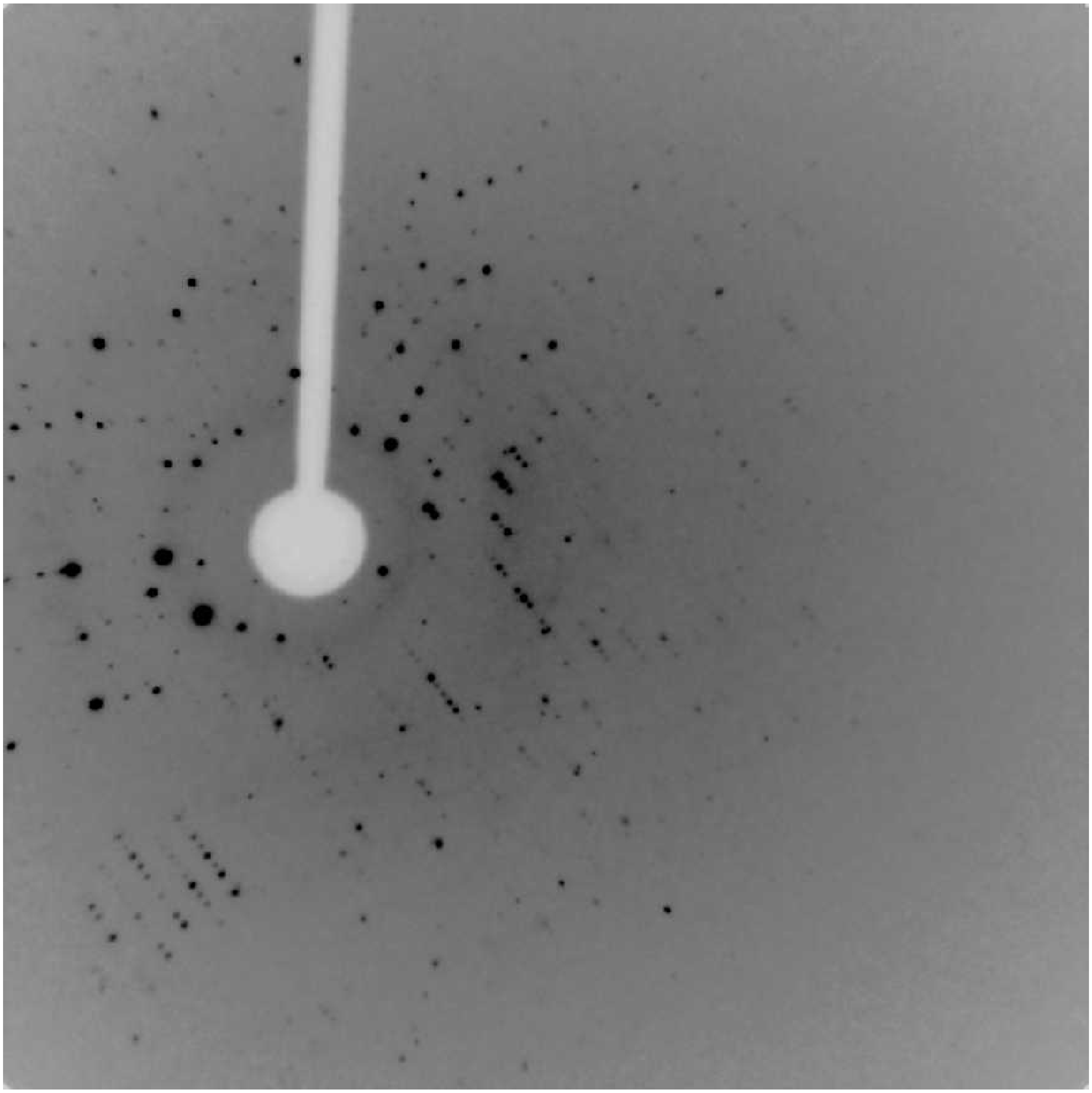

Figure S18. Example of a diffraction image for the fully hydrated structure of $Y(Tf_2N)_3$.

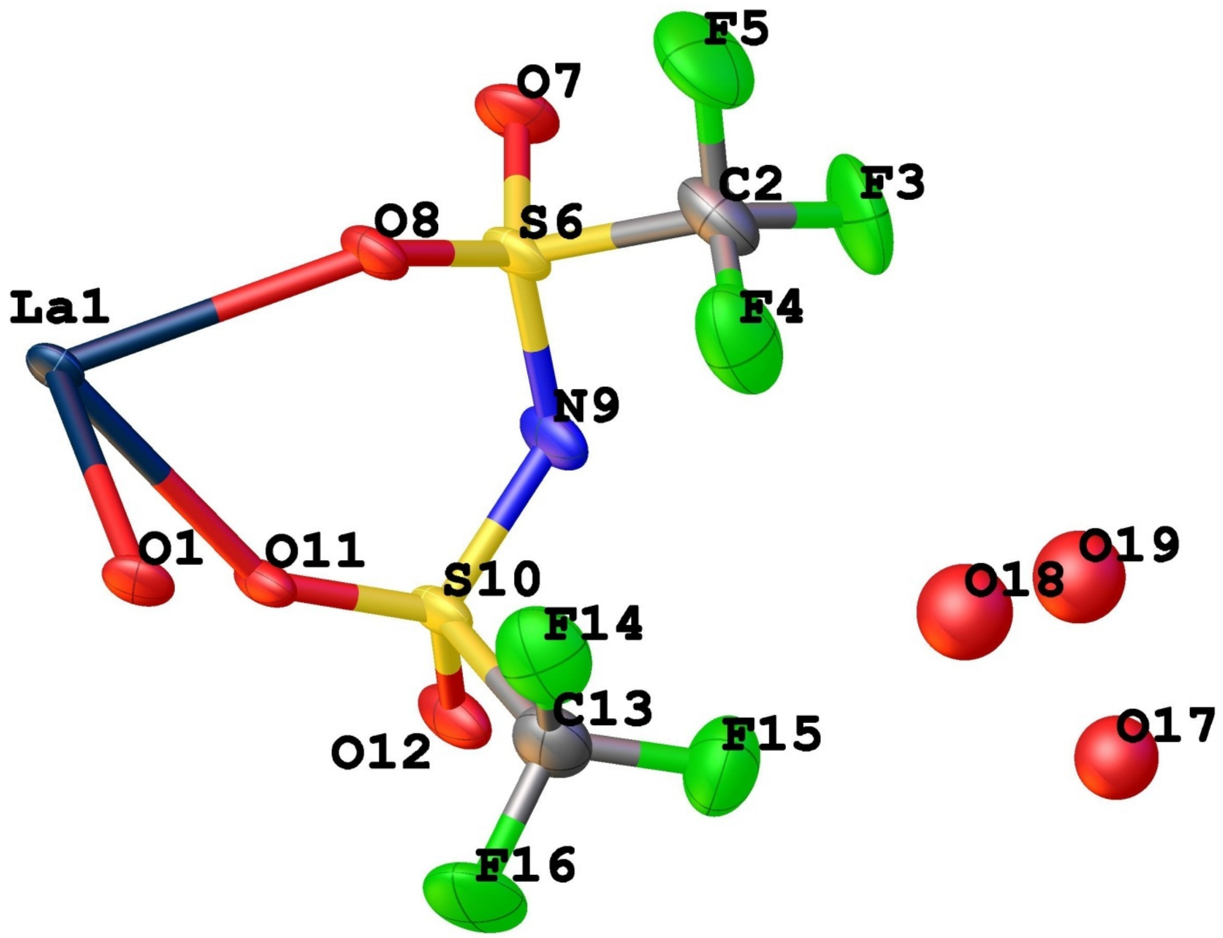

Figure S19. View of the crystal structure of $La(H_2O)_3(Tf_2N)_3 \cdot 2.5H_2O$ showing the crystallographic asymmetric unit and the displacement ellipsoids at the 50% probability level.