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Hydrogen-bonded salt cocrystals of xenon difluoride and protonated perfluoroamides†

Erik Uran ^{ab} and Matic Lozinšek ^{*ab}

The hydrogen-bonding ability of XeF₂ is an important factor influencing its chemical properties and reactivity, yet structurally characterised examples of hydrogen-bonded xenon fluorides remain rare. In this work, three salt cocrystals containing hydrogen-bonded xenon difluoride and hexafluoroarsenate salts of protonated perfluoroamides—CF₃C(OH)NH₂[AsF₆].XeF₂, C₂F₅C(OH)NH₂[AsF₆].XeF₂, and C₃F₇C(OH)NH₂[AsF₆].XeF₂—were synthesised and structurally characterised. Diverse hydrogen-bonding motifs were observed, and the first crystallographically characterised examples of N–H⋯FXeF hydrogen bonds are presented. In total, eleven new crystal structures are reported, including two perfluoroamides, three protonated and two hemiprotonated perfluoroamides, and one salt cocrystal containing an oxonium ion. The XeF₂-containing cocrystals demonstrate that XeF₂ reliably functions as a hydrogen-bond acceptor and readily forms hydrogen-bonded cocrystals. These findings broaden the scope of noble-gas chemistry and highlight the potential of noble-gas fluorides for cocrystal formation.

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Introduction

Xenon difluoride (XeF₂) is the most common and extensively studied binary noble-gas fluoride and serves as a precursor to a wide range of xenon compounds.^{1,2} It is a nonpolar molecular compound with linear geometry. XeF₂ is a good fluoride-ion donor³ and thus forms a variety of Lewis acid–base adducts⁴ and a plethora of coordination compounds with metal cations.^{1,2,5}

The ability of XeF₂ to act as a hydrogen-bond acceptor strongly influences its physical and chemical properties. As a nonpolar molecule, it is highly soluble in the polar protic solvent anhydrous HF (aHF) (167 g/100 g at 30 °C).⁶ This unusually high solubility arises from the formation of FXe–F⋯HF hydrogen bonds.^{7–9} Furthermore, XeF₂ dissolved in aHF is a considerably more potent oxidiser than pure XeF₂, and even trace amounts of HF can catalyse its reactions with organic substrates through the hydrogen-bonding induced polarisation [FXe^{δ+}–F^{δ–}⋯HF].¹⁰ HF also facilitates fluorine exchange in XeF₂, enabling the synthesis of ¹⁸F-radiolabelled XeF₂.^{11,12} In certain cases, the influence of HF is so pronounced that it can unexpectedly alter reaction outcomes, even when inadvertently generated by reaction with the vessel material.¹³

Despite the ability of XeF₂ to act as a hydrogen-bond acceptor, systematic crystallographic investigations are absent, and reported solid-state examples remain scarce. To date, only a handful of crystallographically characterised examples of hydrogen-bonded XeF₂ have been described. These include O–H⋯FXeF hydrogen bonds observed in H₃O[AsF₆].2XeF₂ and in HNO₃.XeF₂ cocrystals,^{14,15} as well as an F–H⋯FXeF interaction observed in the coordination complex [Cd(HF)₂(XeF₂)(MF₆)₂] (M = Ta, Nb).^{5,16}

It has also been shown spectroscopically that protonated trifluoroacetamide (CF₃CONH₂) forms a hydrogen-bonded salt cocrystal¹⁷ with XeF₂, CF₃C(OH)NH₂[AsF₆].XeF₂.xHF.¹⁸ This cocrystal is particularly noteworthy, as it may feature both =OH⁺ and –NH₂ groups as hydrogen-bond donors,¹⁸ potentially offering insight into the hydrogen-bonding preferences of XeF₂.

To investigate the hydrogen-bonding propensity of XeF₂ in the solid state and its tendency to form cocrystals with NH and OH hydrogen-bond donors, the crystal structures of XeF₂ salt cocrystals with protonated CF₃CONH₂, C₂F₅CONH₂, and C₃F₇CONH₂ were studied in this work. The perfluoroamides were selected because of their anticipated resistance to oxidative-fluorination by XeF₂.

Results and discussion

Crystal structures of CF₃CF₂CONH₂ and CF₃CF₂CF₂CONH₂

The crystal structures of pentafluoropropionamide (C₂F₅CONH₂) and heptafluorobutyramide (C₃F₇CONH₂) were elucidated by low-temperature single-crystal X-ray diffraction (LT SCXRD)

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† Dedicated to Professor Resnati, celebrating a career in fluorine and noncovalent chemistry on the occasion of his 70th birthday.



Table 1 Summary of crystal data and refinement results for crystal structures of amides and protonated amides

| Compound | C ₂ F ₅ CONH ₂ | C ₃ F ₇ CONH ₂ | CF ₃ C(OH)NH ₂ [AsF ₆] | C ₂ F ₅ C(OH)NH ₂ [AsF ₆] | C ₃ F ₇ C(OH)NH ₂ [AsF ₆] |
|--|---|---|--|--|--|
| Space group | <i>C2/c</i> | <i>P</i> $\bar{1}$ | <i>P2</i> ₁ / <i>c</i> | <i>Pccn</i> | <i>P2</i> ₁ / <i>c</i> |
| <i>a</i> (Å) | 21.7871(5) | 5.11713(18) | 9.81910(18) | 8.12957(13) | 6.17592(15) |
| <i>b</i> (Å) | 5.11704(12) | 5.27137(14) | 7.90095(13) | 25.2768(4) | 7.94187(19) |
| <i>c</i> (Å) | 10.0754(3) | 12.7768(3) | 20.5015(4) | 9.34322(16) | 21.7914(5) |
| α (°) | 90 | 95.467(2) | 90 | 90 | 90 |
| β (°) | 98.140(2) | 91.890(3) | 98.6498(18) | 90 | 96.014(2) |
| γ (°) | 90 | 105.584(3) | 90 | 90 | 90 |
| <i>V</i> (Å ³) | 1111.94(5) | 329.847(18) | 1572.42(5) | 1919.93(5) | 1062.95(4) |
| <i>M</i> | 163.06 | 213.07 | 302.97 | 352.98 | 402.99 |
| <i>Z</i> | 8 | 2 | 8 | 8 | 4 |
| <i>T</i> (K) | 100 | 100 | 100 | 100 | 100 |
| <i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)] | 0.048 | 0.028 | 0.033 | 0.027 | 0.027 |
| <i>wR</i> (<i>F</i> ²) | 0.138 | 0.076 | 0.087 | 0.058 | 0.068 |

(Tables 1 and S1), whereas the crystal structure of CF₃CONH₂ has been previously reported at 295 K and 110 K.^{19,20} For comparison of bond lengths (Table S2), only the structure obtained at 110 K was considered.²⁰

C₂F₅CONH₂ (Fig. 1a and S1) crystallizes in the monoclinic space group *C2/c* with *Z* = 8. The C=O bond length (1.2323(19) Å) is comparable to the distances observed in the crystal structures of other primary amides, and the same applies to the C–N bond (1.317(2) Å).²¹ Two N–H⋯O hydrogen bonds (2.912(2) Å, 171(2)°; 2.8396(17) Å, 149(2)°; Table S3) in the crystal structure form R₂²(8) and R₆⁴(16) hydrogen-bonding motifs,²² which assemble into a corrugated layer parallel to the *bc* plane (Fig. S2 and S3).

C₃F₇CONH₂ (Fig. 1b and S4) crystallizes in the triclinic space group *P* $\bar{1}$ with *Z* = 2. The C=O bond distance (1.2293(15) Å) is essentially identical to that in C₂F₅CONH₂, as is the C–N bond (1.3162(16) Å). These bond distances are shorter than the corresponding ones observed in non-fluorinated secondary amides, such as capsaicin.²³ Two N–H⋯O hydrogen bonds

(Table S4) are present in the crystal structure (2.9313(14) Å, 174.3(16)°; 2.8495(14) Å, 140.4(15)°), which fall within the typical range for amide molecules.²¹ The R₂²(8) and R₄⁴(8) hydrogen-bond motifs link the molecules into a ladder along the *a*-crystallographic axis (Fig. S5).

Protonation of amides in superacidic media HF–AsF₅

All amides are soluble in aHF and readily undergo protonation upon addition of AsF₅. In all cases, protonation occurs at the oxygen atom, consistent with previous observations.^{18,24–26} Low-temperature crystallisation from aHF afforded crystals of suitable quality for SCXRD.

CF₃C(OH)NH₂[AsF₆][−] (Tables 1, S1 and S2; Fig. S6) crystallises in the monoclinic space group *P2*₁/*c* with *Z* = 8 and *Z'* = 2. Upon protonation, the C=O bonds (1.2795(19), 1.282(2) Å) lengthen and the C–N bonds (1.279(2), 1.281(2) Å) shorten relative to those in CF₃CONH₂ (1.2304(12) and 1.3164(13) Å, respectively).²⁰ These changes in the C=O and C–N bond lengths are consistent with previous crystallographic studies of protonated amides.^{25–27} The O–H⋯F hydrogen bonds (2.5860(16) Å, 2.6530(18) Å; Table S5) bracket the value observed in CF₃C(OH)NH₂[SbF₆][−] (2.600(1) Å), whereas the N–H⋯F hydrogen bonds (2.8236(18)–3.0797(18) Å) are comparable to those in CF₃C(OH)NH₂[SbF₆][−] (2.884(2), 2.933(2) Å).²⁶ All hydrogen-bond angles (121(2)–179(3)°) fall within the typical range. The [AsF₆][−] anions deviate from ideal octahedral geometry, with the longest As–F bonds (1.7524(10), 1.7557(10) Å) participating in hydrogen bonding with =OH⁺ group. In the crystal structure, cations and anions are linked through O–H⋯F and N–H⋯F hydrogen-bonded chains (Fig. 2a and S7).

C₂F₅C(OH)NH₂[AsF₆][−] (Tables 1, S1 and S2) crystallises in the orthorhombic space group *Pccn* with *Z* = 8 and features a disordered –C₂F₅ moiety (Fig. S8). Perfluorinated alkyl chains frequently exhibit disorder in the crystalline state,²⁸ as F⋯F interactions are relatively weak,²⁹ and can therefore adopt various conformations. The C=O (1.2821(15) Å) and C–N (1.2772(16) Å) bonds are longer and shorter, respectively, than those in C₂F₅CONH₂. The [AsF₆][−] anion deviates from ideal octahedral geometry, with the *mer*-As–F bonds involved



Fig. 1 R₂²(8) hydrogen-bonding motifs in the crystal structures of (a) C₂F₅CONH₂ and (b) C₃F₇CONH₂. Hydrogen bonds are shown as dashed orange lines. Displacement ellipsoids are drawn at the 50% probability level, and hydrogen atoms are represented as spheres of arbitrary radius.





Fig. 2 Crystal structure of (a) the hydrogen-bonded chain in $\text{CF}_3\text{-C(OH)NH}_2[\text{AsF}_6]$ and (b) the discrete hydrogen-bonded cluster in $\text{C}_2\text{F}_5\text{C(OH)NH}_2[\text{AsF}_6]$ (only one orientation of the disordered $-\text{C}_2\text{F}_5$ unit is shown). Hydrogen bonds are shown as dashed orange lines. Displacement ellipsoids are drawn at the 50% probability level, and hydrogen atoms are represented as spheres of arbitrary radius.

in hydrogen bonding being longer (1.7253(8)–1.7453(8) Å) than the remaining As–F bonds (1.6976(8)–1.7111(8) Å). The hydrogen bonds (Table S6) between the $=\text{OH}^+$ and $-\text{NH}_2$ groups and the $[\text{AsF}_6]^-$ anions ($\text{O(H)}\cdots\text{F}$, 2.6006(12) Å, $172(2)^\circ$; $\text{N(H)}\cdots\text{F}$, 2.8309(13) Å, $174(2)^\circ$ and 2.8316(14) Å, $161.3(19)^\circ$) lead to the formation of discrete units (Fig. 2b, S8 and S9), exhibiting $\text{R}_2^2(8)$ and $\text{R}_4^4(12)$ hydrogen-bonding motifs.

$\text{C}_3\text{F}_7\text{C(OH)NH}_2[\text{AsF}_6]$ (Tables 1, S1 and S2) crystallises in the monoclinic space group $P2_1/c$ with $Z = 4$, with the $[\text{AsF}_6]^-$ anion disordered over two positions (Fig. S10). The C=O bond (1.2797(14) Å) is elongated, and the C–N bond (1.2841(16) Å) is shortened compared to those in $\text{C}_3\text{F}_7\text{CONH}_2$. A similar C=O(H) bond distance (1.274(2) Å) was observed in the crystal structure of $(\text{C}_6\text{F}_5)_2\text{COH}[\text{AsF}_6]$.³⁰ Hydrogen bonds (Table S7) are formed between the $=\text{OH}^+$ group (2.541(3), 2.557(3) Å; $157(3)$, $165(3)^\circ$) or the $-\text{NH}_2$ group (2.737(4)–3.179(5) Å, $118.0(19)$ – $168.1(19)^\circ$) and the $[\text{AsF}_6]^-$ anions. The $\text{O}\cdots\text{F}$ hydrogen bond is the shortest among the protonated amides in this study, and also shorter than those in $\text{CH}_3\text{C(OH)NH}_2[\text{AsF}_6]$ ²⁵ and $\text{CF}_3\text{C(OH)NH}_2[\text{SbF}_6]$.²⁶ The $[\text{AsF}_6]^-$ anion deviates from ideal octahedral geometry (1.642(3)–1.795(2) Å). The $\text{C}_3\text{F}_7\text{C(OH)NH}_2^+$ cations and $[\text{AsF}_6]^-$ anions are linked into a hydrogen-bonded ribbon (Fig. 3 and S11), exhibiting conjoined $\text{R}_4^4(12)$, $\text{R}_2^2(8)$ and $\text{R}_2^2(8)$ motifs.

Two hemiprotonated salts, $(\text{CF}_3\text{CONH}_2)_2\text{H}[\text{AsF}_6]$ and $(\text{C}_3\text{F}_7\text{CONH}_2)_2\text{H}[\text{AsF}_6]$ (Fig. 4 and S12–S17; Tables 2, S1 and S2), were also crystallographically characterised. The former was inadvertently found during the low-temperature crystal selection and mounting of the $\text{CF}_3\text{C(OH)NH}_2[\text{AsF}_6]\cdot\text{XeF}_2$ sample, whereas the latter was identified as an impurity in

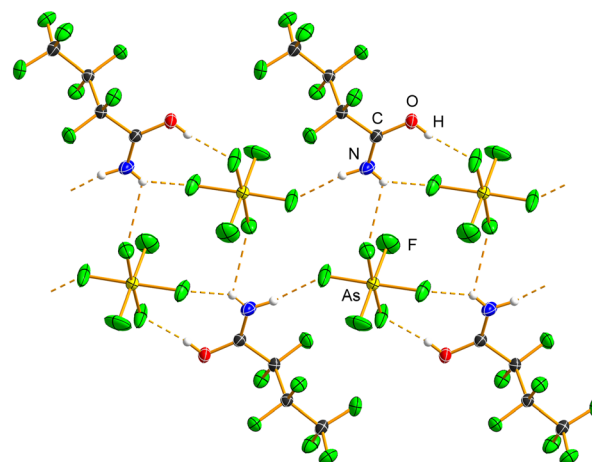


Fig. 3 Hydrogen-bonded ribbon in $\text{C}_3\text{F}_7\text{C(OH)NH}_2[\text{AsF}_6]$ (only one orientation of the disordered $[\text{AsF}_6]^-$ anion is shown). Hydrogen bonds are shown as dashed orange lines. Displacement ellipsoids are drawn at the 50% probability level, and hydrogen atoms are represented as spheres of arbitrary radius.

the sample of $\text{C}_3\text{F}_7\text{C(OH)NH}_2[\text{AsF}_6]\cdot\text{XeF}_2$ salt cocrystals. Both compounds crystallise in the triclinic space group $P\bar{1}$ with $Z = 2$. In both structures, the C=O bonds are elongated (1.283(6) Å in $(\text{CF}_3\text{CONH}_2)_2\text{H}[\text{AsF}_6]$; 1.2652(9), 1.2459(9) Å in $(\text{C}_3\text{F}_7\text{CONH}_2)_2\text{H}[\text{AsF}_6]$), whereas the C–N bonds are shortened (1.274(7) Å in $(\text{CF}_3\text{CONH}_2)_2\text{H}[\text{AsF}_6]$; 1.2942(10), 1.3027(10) Å in $(\text{C}_3\text{F}_7\text{CONH}_2)_2\text{H}[\text{AsF}_6]$) compared to the non-protonated amides.²⁰ The values for one of the amide molecules in $(\text{CF}_3\text{CONH}_2)_2\text{H}[\text{AsF}_6]$ fall within the



Fig. 4 Hydrogen-bonded dimers in the crystal structure of (a) $(\text{CF}_3\text{CONH}_2)_2\text{H}[\text{AsF}_6]$ and (b) $(\text{C}_3\text{F}_7\text{CONH}_2)_2\text{H}[\text{AsF}_6]$. The short $\text{O}\cdots\text{H}\cdots\text{O}=\text{C}$ hydrogen bonds are shown as dashed orange lines. Displacement ellipsoids are drawn at the 50% probability level, and hydrogen atoms are represented as spheres of arbitrary radius.



Table 2 Summary of crystal data and refinement results for hemiprotonated amides and $\text{H}_3\text{O}[\text{AsF}_6]\cdot 2\text{CF}_3\text{CONH}_2$ salt cocrystal

| Compound | $(\text{CF}_3\text{CONH}_2)_2\text{H}[\text{AsF}_6]$ | $(\text{C}_3\text{F}_7\text{CONH}_2)_2\text{H}[\text{AsF}_6]$ | $\text{H}_3\text{O}[\text{AsF}_6]\cdot 2\text{CF}_3\text{CONH}_2$ |
|-------------------------|--|---|---|
| Space group | $P\bar{1}$ | $P\bar{1}$ | $Pnma$ |
| a (Å) | 5.2815(3) | 5.32051(4) | 11.5349(2) |
| b (Å) | 10.1517(6) | 10.45222(9) | 14.0649(3) |
| c (Å) | 12.4911(6) | 16.10170(12) | 7.78994(14) |
| α (°) | 108.936(5) | 90.5740(6) | 90 |
| β (°) | 93.107(5) | 90.8760(6) | 90 |
| γ (°) | 102.904(5) | 103.0402(7) | 90 |
| V (Å ³) | 611.63(6) | 872.156(12) | 1263.82(4) |
| M | 416.02 | 616.06 | 434.04 |
| Z | 2 | 2 | 4 |
| T (K) | 100 | 100 | 100 |
| $R[F^2 > 2\sigma(F^2)]$ | 0.047 | 0.022 | 0.037 |
| $wR(F^2)$ | 0.125 | 0.055 | 0.085 |

range for neutral amide,²⁰ owing to the relatively high standard uncertainties of the bond lengths. The $\text{O}(\text{H})\cdots\text{O}$ hydrogen bond length in $(\text{CF}_3\text{CONH}_2)_2\text{H}[\text{AsF}_6]$ (2.426(5) Å, 170(8)°) is essentially identical to that in $(\text{C}_3\text{F}_7\text{CONH}_2)_2\text{H}[\text{AsF}_6]$ (2.4174(9) Å, 172(2)°) (Tables S8 and S9), and comparable to literature values for such hydrogen-bonded systems.²¹ The nearly equidistant position of the hydrogen atom ($\text{O}-\text{H}$, $\text{H}\cdots\text{O}$: 1.13(9), 1.31(9) Å in $(\text{CF}_3\text{CONH}_2)_2\text{H}[\text{AsF}_6]$; 1.06(2), 1.36(2) Å in $(\text{C}_3\text{F}_7\text{CONH}_2)_2\text{H}[\text{AsF}_6]$), together with the relatively short $\text{O}\cdots\text{O}$ distances, indicates strong, positive charge-assisted hydrogen bonding, (+) CAHB.³¹ These structures represent rare examples of proton sharing between two primary amide molecules,^{21,32} a motif more commonly observed in secondary and tertiary amides.²¹ The $-\text{NH}_2$ groups are hydrogen-bonded to $[\text{AsF}_6]^-$ anions ($\text{N}\cdots\text{F}$, 2.644(5)–3.005(5) Å in $(\text{CF}_3\text{CONH}_2)_2\text{H}[\text{AsF}_6]$; 2.8168(9)–3.1046(9) Å in $(\text{C}_3\text{F}_7\text{CONH}_2)_2\text{H}[\text{AsF}_6]$) (Fig. S13–S17), resulting in the formation of ribbons that are further interconnected by the anions into layers parallel to the ab plane.

A crystal of $\text{H}_3\text{O}[\text{AsF}_6]\cdot 2\text{CF}_3\text{CONH}_2$ (Tables 2, S1 and S2; Fig. 5 and S18–S20) was fortuitously found during the low-temperature crystal selection and mounting of the $\text{CF}_3\text{C}(\text{OH})\text{NH}_2[\text{AsF}_6]\cdot\text{XeF}_2$ sample. It crystallises in the orthorhombic space group $Pnma$ with $Z = 4$. The amide molecule is not protonated, resulting in $\text{C}=\text{O}$ (1.236(4) Å) and $\text{C}-\text{N}$ (1.304(4) Å) bond lengths that are close to those in CF_3CONH_2 .²⁰ The amide molecule acts as both a hydrogen-bond donor and acceptor (Table S10, Fig. S19), forming $\text{N}-\text{H}\cdots\text{F}(\text{As})$ and $\text{N}-\text{H}\cdots\text{O}(\text{C})$ hydrogen bonds. An $\text{R}_2^2(8)$ motif is observed between two amide molecules, with the $\text{N}\cdots\text{O}$ hydrogen bond (2.959(4) Å, 171(4)°) comparable to that found in CF_3CONH_2 .²⁰ The H_3O^+ cation forms three hydrogen bonds: two symmetrically equivalent $\text{O}-\text{H}\cdots\text{O}(\text{C})$ (2.525(3) Å, 167(4)°) and one $\text{O}-\text{H}\cdots\text{F}$ hydrogen bond (2.657(5) Å, 173(7)°) with the $[\text{AsF}_6]^-$ anion. Together, these hydrogen bonds form a hydrogen-bonded cluster represented by $\text{R}_6^6(20)$, $\text{R}_4^4(14)$ and $\text{R}_2^2(8)$ graph-set motifs²² (Fig. 5), which further extend into a layer parallel to the bc plane (Fig. S20).

Hydrogen-bonded salt cocrystals of XeF_2

The reaction of amides with equimolar amounts of $[\text{XeF}][\text{AsF}_6]$ at temperatures down to -30 °C leads to the formation of $\text{RC}(\text{OH})\text{NH}_2[\text{AsF}_6]\cdot\text{XeF}_2$ salt cocrystals. This indicates that a proton from HF is transferred to the amide, generating a protonated amide, while the resulting fluoride anion reacts with $[\text{XeF}]^+$ to form XeF_2 . This behaviour was also reported in a previous study of the $\text{CF}_3\text{CONH}_2-[\text{XeF}][\text{AsF}_6]$ system.¹⁸

The salt cocrystals (Tables 3, S1 and S2) thus feature protonated amides cocrystallised with XeF_2 and exhibit a rare $\text{O}-\text{H}\cdots\text{FXeF}$ hydrogen bond, as well as the first crystallographically characterised examples of $\text{N}-\text{H}\cdots\text{FXeF}$ hydrogen bonds.

$\text{CF}_3\text{C}(\text{OH})\text{NH}_2[\text{AsF}_6]\cdot\text{XeF}_2$ (Fig. 6 and S21) crystallises in the monoclinic space group $P2_1/n$ with $Z = 4$. The XeF_2 molecule exhibits slight asymmetry in $\text{Xe}-\text{F}$ bond distances (1.9669(10), 2.0237(9) Å) compared to pure XeF_2 (1.999(4) Å),³³ and it remains linear (178.10(5) Å). The asymmetry of XeF_2 is slightly smaller than that observed in $\text{XeF}_2\cdot\text{HNO}_3$ (1.9737(8), 2.0506(8) Å).¹⁵



Fig. 5 $\text{R}_4^4(14)$ hydrogen-bonded cluster in the crystal structure of the $\text{H}_3\text{O}[\text{AsF}_6]\cdot 2\text{CF}_3\text{CONH}_2$ salt cocrystal. Hydrogen bonds are shown by dashed orange lines. Displacement ellipsoids are drawn at the 50% probability level, and hydrogen atoms are represented as spheres of arbitrary radius.



Table 3 Summary of crystal data and refinement results for salt cocrystals of protonated amides with XeF₂

| Compound | CF ₃ C(OH)NH ₂ [AsF ₆] ⁻ ·XeF ₂ | C ₂ F ₅ C(OH)NH ₂ [AsF ₆] ⁻ ·XeF ₂ | C ₃ F ₇ C(OH)NH ₂ [AsF ₆] ⁻ ·XeF ₂ |
|----------------------------|---|---|---|
| Space group | <i>P</i> 2 ₁ / <i>n</i> | <i>Aea</i> 2 | <i>Pnna</i> |
| <i>a</i> (Å) | 7.41785(9) | 8.67561(10) | 8.62011(14) |
| <i>b</i> (Å) | 9.84875(11) | 31.0125(4) | 35.5418(5) |
| <i>c</i> (Å) | 14.90113(17) | 8.65174(9) | 8.71910(12) |
| α (°) | 90 | 90 | 90 |
| β (°) | 99.4517(11) | 90 | 90 |
| γ (°) | 90 | 90 | 90 |
| <i>V</i> (Å ³) | 1073.85(2) | 2327.77(4) | 2671.31(7) |
| <i>M</i> | 472.27 | 522.28 | 572.29 |
| <i>Z</i> | 4 | 8 | 8 |
| <i>T</i> (K) | 100 | 100 | 100 |
| $R[F^2 > 2\sigma(F^2)]$ | 0.025 | 0.018 | 0.026 |
| $wR(F^2)$ | 0.067 | 0.038 | 0.066 |

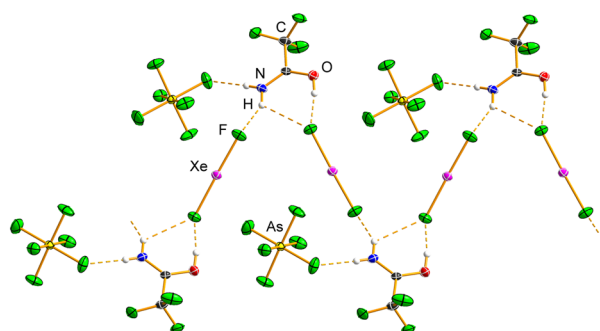


Fig. 6 Hydrogen-bonded ribbon in the crystal structure of the salt cocrystal CF₃C(OH)NH₂[AsF₆]⁻·XeF₂. Only one orientation of the disordered -CF₃ moiety is shown. Hydrogen bonds are shown as dashed orange lines. Displacement ellipsoids are drawn at the 50% probability level, and hydrogen atoms are represented as spheres of arbitrary radius.

CF₃C(OH)NH₂[AsF₆]⁻·XeF₂ is the only salt cocrystal in this series that exhibits both O–H···F(Xe) and N–H···F(Xe) hydrogen bonds. One fluorine atom of XeF₂ is acting as a bifurcated acceptor (Fig. 6 and S21; Table S11). The O–H···F(Xe) hydrogen bond (2.5467(14) Å, 171(3)°) is shorter than that in H₃O[AsF₆]⁻·2XeF₂ (2.571(3) Å)¹⁴ and HNO₃·XeF₂ (2.690(1) Å).¹⁵ It is also significantly shorter than the O–H···F(As) hydrogen bonds in CF₃C(OH)NH₂[AsF₆]⁻ and C₂F₅C(OH)NH₂[AsF₆]⁻, but comparable to that in C₃F₇C(OH)NH₂[AsF₆]⁻. The N–H···F(Xe) hydrogen bonds (2.7865(15) Å, 151(3)°; 3.0894(16), 124(2)°), which involve a single bifurcated donor, are longer than those observed in the other two salt cocrystals described in this study. The C=O (1.2773(15) Å) and C–N (1.2772(16) Å) bond lengths are essentially identical to those in the protonated salts,²⁶ indicating a negligible influence of hydrogen bonding on the overall geometry of the CF₃C(OH)NH₂⁺ cation. The -CF₃ moiety is disordered, as also observed in the crystal structure of CF₃CONH₂.²⁰ The [AsF₆]⁻ anion participates in hydrogen bonding with the -NH₂ group (2.8270(18) Å, 176(3)°; 3.0594(15) Å, 111(2)°), resulting in a slight deviation from ideal octahedral geometry (As–F, 1.7006(12)–1.7417(11) Å).

Hydrogen bonds between CF₃C(OH)NH₂⁺ and XeF₂ form a zigzag chain parallel to the *b*-crystallographic axis, with pendant [AsF₆]⁻ anions connected to the chain *via* N–H···F(As) hydrogen bonds, giving rise to a ribbon-like structure (Fig. 6 and S22).

Both C₂F₅C(OH)NH₂[AsF₆]⁻·XeF₂ and C₃F₇C(OH)NH₂[AsF₆]⁻·XeF₂ (Tables 3, S1 and S2; Fig. S23–S26) crystallise in orthorhombic space groups, *Aea*2 and *Pnna*, respectively, with *Z* = 8. The asymmetry of the Xe–F bond lengths in C₂F₅C(OH)NH₂[AsF₆]⁻·XeF₂ (1.9734(14), 2.0061(15) Å) and in C₃F₇C(OH)NH₂[AsF₆]⁻·XeF₂ (1.9674(15), 2.0135(16) Å) is comparable. The shorter Xe–F bonds are similar to that observed in the trifluoroacetamide analogue, whereas the longer Xe–F bonds are significantly shorter. In both cocrystals, the F–Xe–F angle is essentially linear (179.88(9)°; 179.57(7)°).

The N–H···F(Xe) hydrogen bonds (Tables S12 and S13) in C₂F₅C(OH)NH₂[AsF₆]⁻·XeF₂ (2.688(3), 2.729(3) Å) and C₃F₇C(OH)NH₂[AsF₆]⁻·XeF₂ (2.692(3), 2.737(3) Å) are comparable and are significantly shorter than the corresponding hydrogen bonds in CF₃C(OH)NH₂[AsF₆]⁻·XeF₂. The C=O (1.289(2), 1.285(3) Å) and C–N (1.279(3), 1.280(3) Å) bond lengths in C₂F₅C(OH)NH₂[AsF₆]⁻·XeF₂ and C₃F₇C(OH)NH₂[AsF₆]⁻·XeF₂ are almost identical to those observed in the corresponding protonated salts.

The protonated oxygen atom acts as a hydrogen-bond donor towards the [AsF₆]⁻ anions, forming bifurcated hydrogen bonds (Fig. 7, S23 and S25), which are longer than the O–H···F(As) hydrogen bonds observed in the parent protonated salts.

The packing in both C₂F₅C(OH)NH₂[AsF₆]⁻·XeF₂ and C₃F₇C(OH)NH₂[AsF₆]⁻·XeF₂ consists of hydrogen-bonded ribbons composed of alternating protonated amide and XeF₂ molecules, similar to those observed in CF₃C(OH)NH₂[AsF₆]⁻·XeF₂. These ribbons are further connected by O–H···F(As) hydrogen bonds, and in the case of C₃F₇C(OH)NH₂[AsF₆]⁻·XeF₂, also by N–H···F(As) hydrogen bonds (Fig. S24 and S26).

The relatively small difference in Xe–F bond lengths in the present XeF₂ cocrystals suggests that hydrogen bonding has only a minor influence on XeF₂ ionisation (XeF₂ → XeF⁺ +





Fig. 7 Hydrogen bonds (dashed orange lines) in the crystal structures of the salt cococrystals (a) $C_2F_5C(OH)NH_2[AsF_6] \cdot XeF_2$ and (b) $C_3F_7C(OH)NH_2[AsF_6] \cdot XeF_2$. The OH groups are bifurcated hydrogen-bond donors; however, two $[AsF_6]^-$ anions have been omitted for clarity. Displacement ellipsoids are drawn at the 50% probability level, and hydrogen atoms are represented as spheres of arbitrary radius.

F^-).³⁴ In particular, the shorter Xe–F bonds (1.9669(10)–1.9734(14) Å) are considerably longer than those found in $[XeF]^+$ tight ion pairs^{33,35,36} and in $[Xe_2F_3]^+$ salts.^{35,37} They are comparable to the shortest Xe–F bond lengths in XeF_2 adduct-salts with $[BrOF_2]^+$ (1.956(5), 1.960(4) Å)³⁸ and $[BrO_2]^+$ cations (1.970(4)–1.978(3) Å).³⁹ Nevertheless, the distortion of hydrogen-bonded XeF_2 observed in the present salt cococrystals is significant when compared with Xe–F bond distances observed in the crystal structures containing cococrystallised XeF_2 , e.g., $3XeF_2 \cdot 2MnF_4$ (1.9933(7) Å),³⁶ and in the molecular cococrystals $XeF_2 \cdot XeF_4$ (1.9940(9) Å)³⁷ and $XeF_2 \cdot XeOF_4$ (2.014(5) Å),⁴⁰ in which XeF_2 is centrosymmetric.

Vibrational spectroscopy

To corroborate the findings from LT SCXRD and to gain further insight into the ionisation of XeF_2 , low-temperature Raman spectra were measured (Fig. 8 and S27–S40). Two bands at 457–475 and 528–535 cm^{-1} are observed in all XeF_2 salt cococrystals in this study, corresponding to the elongated and shortened Xe–F bond, respectively. These bands are



Fig. 8 Raman spectra of XeF_2 salt cococrystals with protonated amides recorded at low temperatures ($-90^\circ C$). The green dashed line is placed at the position of free XeF_2 (497 cm^{-1})⁴¹ which was observed as an impurity in the reactions.

significantly shifted from that of pure XeF_2 (497 cm^{-1})⁴¹ and from values observed when cococrystallised XeF_2 does not participate in significant intermolecular interactions, such as in $XeF_2 \cdot XeOF_4$ ($494, 503\text{ cm}^{-1}$),⁴⁰ $XeF_2 \cdot XeF_4$ (505 cm^{-1}),⁴² $3XeF_2 \cdot 2MnF_4$ (508 cm^{-1}),³⁶ and $XeF_2 \cdot N_2O_4$ (509 cm^{-1}).¹⁵ The value of the higher-frequency band is comparable to the Raman shifts reported for the adduct salts $[BrOF_2]^- [AsF_6]^- XeF_2$ ($531, 543, 559\text{ cm}^{-1}$),³⁸ $[BrO_2]^- [AsF_6]^- nXeF_2$ ($n = 1, 2; 516\text{--}546\text{ cm}^{-1}$),³⁹ and for the hydrogen-bonded cococrystals $H_3O[AsF_6] \cdot 2XeF_2$ (552 cm^{-1})¹⁴ and $HNO_3 \cdot XeF_2$ (529 cm^{-1}).¹⁵ However, these shifts are significantly smaller than those observed in $[XeF]^+$ tight-ion pair salts ($>600\text{ cm}^{-1}$) and $[Xe_2F_3]^+$ cations ($580\text{--}600\text{ cm}^{-1}$).^{1,33,35,36,43,44} The band around 535 cm^{-1} is particularly noteworthy, as this value coincides with that observed for XeF_2 dissolved in aHF, which has been attributed to the $FXe-F \cdots HF$ hydrogen bonds.^{7–9}

In addition to the bands attributed to XeF_2 , those arising from $[AsF_6]^-$ anions are observed around 375 and 680 cm^{-1} .^{18,25,26,45} Vibrations from the protonated amide molecules are also present, including an intense band around 800 cm^{-1} corresponding to $\nu(C-C)$,^{25,26} and peaks near 1100 cm^{-1} attributed to C–F vibrations.^{18,26,46} In all protonated amides, the N–H stretching vibrations were observed in $3150\text{--}3400\text{ cm}^{-1}$ range.^{18,26,46}



Experimental

Caution! Anhydrous HF, AsF₅, XeF₂, [XeF][AsF₆] and the compounds prepared in this study are highly reactive and hazardous. The amides used may cause skin, eye, and respiratory irritation. Contact with the skin must be avoided, and all compounds should be handled exclusively in a well-ventilated fume hood.

Appropriate safety precautions must be observed at all times, and working with minimal quantities is strongly recommended.

Materials and methods

Reactions were carried out in fluorinated ethylene propylene (FEP) vessels equipped with Kel-F or PTFE valves. All vessels were passivated with fluorine prior to use. Volatile substances were handled using a fluorine-resistant metal vacuum line, whereas solids were manipulated inside an N₂-filled glovebox. Detailed synthetic procedures are provided in the SI. Characterisation was performed by low-temperature single-crystal X-ray diffraction and low-temperature Raman spectroscopy. Single-crystal selection and mounting were carried out using a low-temperature crystal-mounting apparatus, as described previously (SI).^{30,36,47} Low-temperature Raman spectra were recorded directly on the aluminium trough used for mounting single crystals for X-ray diffraction measurements.

Conclusions

In this work, the perfluoroamides trifluoroacetamide (CF₃-CONH₂), pentafluoropropionamide (C₂F₅CONH₂), and heptafluorobutyramide (C₃F₇-CONH₂), were protonated in superacidic medium HF-AsF₅, and the crystal structures of the resulting salts, CF₃C(OH)NH₂[AsF₆], C₂F₅C(OH)NH₂[AsF₆], and C₃F₇C(OH)NH₂[AsF₆] were elucidated. Protonation at the carbonyl oxygen atom is consistently observed. In addition, the crystal structures of the amides C₂F₅CONH₂ and C₃F₇CONH₂, the hemiprotonated salts (CF₃CONH₂)₂H[AsF₆] and (C₃F₇CONH₂)₂H[AsF₆], and the oxonium salt cocrystal H₃O[AsF₆]·2CF₃CONH₂ were determined. Low-temperature reactions of the perfluoroamides with [XeF][AsF₆] in aHF yielded rare XeF₂-containing salt cocrystals: CF₃C(OH)NH₂[AsF₆]·XeF₂, C₂F₅-C(OH)NH₂[AsF₆]·XeF₂ and C₃F₇C(OH)NH₂[AsF₆]·XeF₂. Their crystal structures reveal a rare example of O-H···FXeF and the first crystallographically characterised cases of N-H···FXeF hydrogen bonding. The XeF₂ molecule is slightly polarised, as indicated by the differences observed in Xe-F bond lengths compared with those in free XeF₂; this finding is corroborated by low-temperature Raman spectroscopy. The reported crystal structures display diverse hydrogen-bonding motifs involving O-H···F(Xe), N-H···F(Xe), O-H···F(As) and N-H···F(As) interactions. The salt cocrystals prepared and structurally characterised in

this study demonstrate that XeF₂ readily forms hydrogen-bonded cocrystals and serves as a reliable hydrogen-bond acceptor. These results open new possibilities for the exploration of cocrystal formation with noble-gas fluorides and the expansion of noble-gas chemistry.

Author contributions

Conceptualization, data curation, formal analysis, investigation, visualization, writing – original draft: EU; funding acquisition, methodology, project administration, resources, supervision: ML; validation, writing – review & editing: EU, ML. Both authors agreed on the final version of the article.

Conflicts of interest

There are no conflicts to declare.

Data availability

Supplementary information: crystallographic details, Raman spectra, experimental details. See DOI: <https://doi.org/10.1039/d5ce00956a>.

Crystallographic data for all reported crystal structures has been deposited at the Cambridge Crystallographic Data Centre (CCDC) under deposition numbers 2493130–2493140.^{48a-k}

Data for this article, including SCXRD datasets and Raman spectra are available at Zenodo open repository at <https://doi.org/10.5281/zenodo.17432981>.

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