

## SUPPORTING INFORMATION

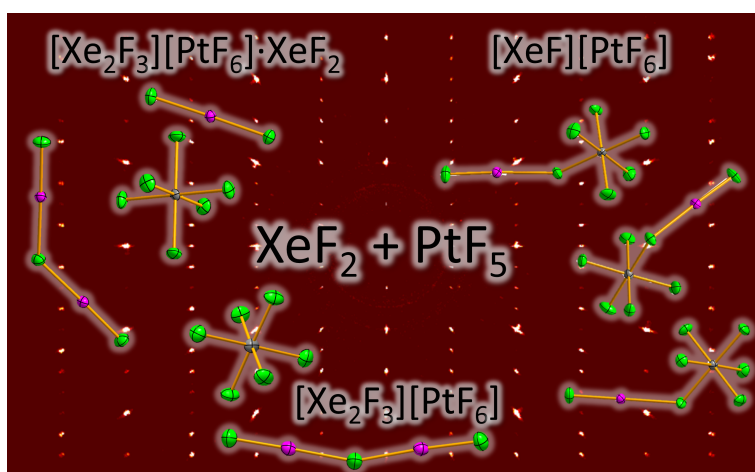
### Structural investigations of the $\text{XeF}_2\text{--PtF}_5$ system: crystal structures of $[\text{Xe}_2\text{F}_3][\text{PtF}_6]\cdot\text{XeF}_2$ , $[\text{Xe}_2\text{F}_3][\text{PtF}_6]$ , and $[\text{XeF}][\text{PtF}_6]$

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**Table S1.** Summary of crystallographic data and refinement results for [Xe<sub>2</sub>F<sub>3</sub>][PtF<sub>6</sub>]·XeF<sub>2</sub>(*mC120*) and [Xe<sub>2</sub>F<sub>3</sub>][PtF<sub>6</sub>]·XeF<sub>2</sub>(*oP120*).

| Compound                                                                             | [Xe <sub>2</sub> F <sub>3</sub> ][PtF <sub>6</sub> ]·XeF <sub>2</sub> ( <i>mC120</i> ) | [Xe <sub>2</sub> F <sub>3</sub> ][PtF <sub>6</sub> ]·XeF <sub>2</sub> ( <i>oP120</i> ) |
|--------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------|
| Formula                                                                              | Xe <sub>3</sub> PtF <sub>11</sub>                                                      | Xe <sub>3</sub> PtF <sub>11</sub>                                                      |
| <i>F</i> <sub>w</sub>                                                                | 797.99                                                                                 | 797.99                                                                                 |
| <i>T</i> (K)                                                                         | 100.00(11)                                                                             | 100.00(11)                                                                             |
| Crystal system                                                                       | Monoclinic                                                                             | Orthorhombic                                                                           |
| Space group                                                                          | <i>C2/c</i>                                                                            | <i>Pbca</i>                                                                            |
| <i>a</i> (Å)                                                                         | 15.82016(16)                                                                           | 8.99866(8)                                                                             |
| <i>b</i> (Å)                                                                         | 9.44604(9)                                                                             | 13.29342(14)                                                                           |
| <i>c</i> (Å)                                                                         | 15.56492(16)                                                                           | 18.61809(18)                                                                           |
| $\alpha$ (°)                                                                         | 90                                                                                     | 90                                                                                     |
| $\beta$ (°)                                                                          | 109.7185(12)                                                                           | 90                                                                                     |
| $\gamma$ (°)                                                                         | 90                                                                                     | 90                                                                                     |
| <i>V</i> (Å <sup>3</sup> )                                                           | 2189.60(4)                                                                             | 2227.15(4)                                                                             |
| <i>Z</i>                                                                             | 8                                                                                      | 8                                                                                      |
| $\rho_{\text{calc}}$ (g/cm <sup>3</sup> )                                            | 4.841                                                                                  | 4.760                                                                                  |
| Crystal size (mm)                                                                    | 0.177 × 0.133 × 0.074                                                                  | 0.09 × 0.06 × 0.056                                                                    |
| Radiation type                                                                       | Ag <i>K</i> $\alpha$                                                                   | Ag <i>K</i> $\alpha$                                                                   |
| $\lambda$ (Å)                                                                        | 0.56087                                                                                | 0.56087                                                                                |
| $\mu$ (mm <sup>-1</sup> )                                                            | 11.850                                                                                 | 11.651                                                                                 |
| <i>F</i> (000)                                                                       | 2712                                                                                   | 2712                                                                                   |
| $\theta_{\text{max}}$ (°)                                                            | 36.935                                                                                 | 32.543                                                                                 |
| Index ranges                                                                         | -33 ≤ <i>h</i> ≤ 32<br>-20 ≤ <i>k</i> ≤ 20<br>-32 ≤ <i>l</i> ≤ 33                      | -16 ≤ <i>h</i> ≤ 16<br>-24 ≤ <i>k</i> ≤ 24<br>-33 ≤ <i>l</i> ≤ 31                      |
| Reflections collected                                                                | 145101                                                                                 | 130458                                                                                 |
| Independent reflections                                                              | 10756                                                                                  | 7539                                                                                   |
| Reflections with [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )]                               | 8908                                                                                   | 6434                                                                                   |
| <i>R</i> <sub>int</sub>                                                              | 0.0959                                                                                 | 0.0612                                                                                 |
| <i>R</i> <sub>sigma</sub>                                                            | 0.0318                                                                                 | 0.0251                                                                                 |
| Data/restraints/parameters                                                           | 10756/0/136                                                                            | 7539/0/136                                                                             |
| <i>S</i>                                                                             | 1.044                                                                                  | 1.043                                                                                  |
| <i>R</i> <sub>1</sub> , <i>wR</i> <sub>2</sub> [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] | 0.0268, 0.0663                                                                         | 0.0195, 0.0376                                                                         |
| <i>R</i> <sub>1</sub> , <i>wR</i> <sub>2</sub> [all data]                            | 0.0353, 0.0697                                                                         | 0.0276, 0.0391                                                                         |
| $\Delta\rho_{\text{min}}$ , $\Delta\rho_{\text{max}}$ (e/Å <sup>3</sup> )            | -4.079, 2.461                                                                          | -1.050, 1.529                                                                          |
| CSD deposition number <sup>[a]</sup>                                                 | 2529289                                                                                | 2529288                                                                                |

<sup>[a]</sup> These data can be obtained free of charge from FIZ Karlsruhe via [www.ccdc.cam.ac.uk/structures](http://www.ccdc.cam.ac.uk/structures)

**Table S2.** Summary of crystallographic data and refinement results for [Xe<sub>2</sub>F<sub>3</sub>][PtF<sub>6</sub>] and [XeF][PtF<sub>6</sub>](*mP108*).

| Compound                                                                             | [Xe <sub>2</sub> F <sub>3</sub> ][PtF <sub>6</sub> ] <sup>[a]</sup>  | [XeF][PtF <sub>6</sub> ]( <i>mP108</i> )                             |
|--------------------------------------------------------------------------------------|----------------------------------------------------------------------|----------------------------------------------------------------------|
| Formula                                                                              | Xe <sub>2</sub> PtF <sub>9</sub>                                     | XePtF <sub>7</sub>                                                   |
| <i>F</i> <sub>w</sub>                                                                | 628.69                                                               | 459.39                                                               |
| <i>T</i> (K)                                                                         | 100.01(11)                                                           | 100.00(10)                                                           |
| Crystal system                                                                       | Monoclinic                                                           | Monoclinic                                                           |
| Space group                                                                          | <i>Ia</i>                                                            | <i>P2<sub>1</sub>/n</i>                                              |
| <i>a</i> (Å)                                                                         | 10.8860(2)                                                           | 7.91407(6)                                                           |
| <i>b</i> (Å)                                                                         | 8.09069(16)                                                          | 10.79245(8)                                                          |
| <i>c</i> (Å)                                                                         | 9.82822(19)                                                          | 21.17145(16)                                                         |
| $\alpha$ (°)                                                                         | 90                                                                   | 90                                                                   |
| $\beta$ (°)                                                                          | 92.1157(16)                                                          | 91.6071(7)                                                           |
| $\gamma$ (°)                                                                         | 90                                                                   | 90                                                                   |
| <i>V</i> (Å <sup>3</sup> )                                                           | 865.03(3)                                                            | 1807.59(2)                                                           |
| <i>Z</i>                                                                             | 4                                                                    | 12                                                                   |
| $\rho_{\text{calc}}$ (g/cm <sup>3</sup> )                                            | 4.827                                                                | 5.064                                                                |
| Crystal size (mm)                                                                    | 0.187 × 0.14 × 0.077                                                 | 0.192 × 0.08 × 0.074                                                 |
| Radiation type                                                                       | Ag <i>K</i> $\alpha$                                                 | Ag <i>K</i> $\alpha$                                                 |
| $\lambda$ (Å)                                                                        | 0.56087                                                              | 0.56087                                                              |
| $\mu$ (mm <sup>-1</sup> )                                                            | 12.933                                                               | 15.603                                                               |
| <i>F</i> (000)                                                                       | 1068                                                                 | 2340                                                                 |
| $\theta_{\text{max}}$ (°)                                                            | 27.860                                                               | 36.5510                                                              |
| Index ranges                                                                         | $-18 \leq h \leq 18$<br>$-13 \leq k \leq 13$<br>$-16 \leq l \leq 16$ | $-16 \leq h \leq 16$<br>$-22 \leq k \leq 22$<br>$-44 \leq l \leq 44$ |
| Reflections collected                                                                | 24636                                                                | 234850                                                               |
| Independent reflections                                                              | 4202                                                                 | 17702                                                                |
| Reflections with [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )]                               | 3949                                                                 | 15591                                                                |
| <i>R</i> <sub>int</sub>                                                              | 0.0688                                                               | 0.0885                                                               |
| <i>R</i> <sub>sigma</sub>                                                            | 0.0343                                                               | 0.0277                                                               |
| Data/restraints/parameters                                                           | 4202/2/110                                                           | 17702/0/245                                                          |
| <i>S</i>                                                                             | 1.076                                                                | 1.026                                                                |
| <i>R</i> <sub>1</sub> , <i>wR</i> <sub>2</sub> [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] | 0.0360, 0.0927                                                       | 0.0228, 0.0503                                                       |
| <i>R</i> <sub>1</sub> , <i>wR</i> <sub>2</sub> [all data]                            | 0.0393, 0.0943                                                       | 0.0281, 0.0519                                                       |
| $\Delta\rho_{\text{min}}$ , $\Delta\rho_{\text{max}}$ (e/Å <sup>3</sup> )            | -1.359, 2.346                                                        | -2.913, 2.275                                                        |
| CSD deposition number <sup>[b]</sup>                                                 | 2529287                                                              | 2529290                                                              |

<sup>[a]</sup> Refined as a two-component inversion twin with twin ratio of 0.576(14):0.424(14).

<sup>[b]</sup> These data can be obtained free of charge from FIZ Karlsruhe via [www.ccdc.cam.ac.uk/structures](http://www.ccdc.cam.ac.uk/structures)

**Table S3.** Experimental (SCXRD) geometrical parameters for [Xe<sub>2</sub>F<sub>3</sub>][PtF<sub>6</sub>]·XeF<sub>2</sub>(*mC120*).

| Bond distances (Å) |            |            |            |
|--------------------|------------|------------|------------|
| Pt1–F6             | 1.8771(13) | Xe1–F1     | 1.9118(12) |
| Pt1–F7             | 1.8798(16) | Xe1–F2     | 2.1825(12) |
| Pt1–F8             | 1.8802(16) | Xe2–F2     | 2.1690(12) |
| Pt1–F9             | 1.8819(13) | Xe2–F3     | 1.9133(12) |
| Pt1–F10            | 1.8904(12) | Xe3–F4     | 1.9779(12) |
| Pt1–F11            | 1.8909(13) | Xe3–F5     | 1.9993(12) |
| Angles (°)         |            |            |            |
| F10–Pt1–F11        | 89.48(8)   | F8–Pt1–F9  | 90.82(7)   |
| F6–Pt1–F11         | 179.89(8)  | F7–Pt1–F11 | 90.34(7)   |
| F6–Pt1–F10         | 90.62(7)   | F7–Pt1–F10 | 90.86(7)   |
| F6–Pt1–F9          | 89.34(8)   | F7–Pt1–F9  | 89.20(7)   |
| F6–Pt1–F8          | 90.29(7)   | F7–Pt1–F8  | 179.97(7)  |
| F6–Pt1–F7          | 89.68(7)   | F1–Xe1–F2  | 179.28(5)  |
| F9–Pt1–F11         | 90.55(7)   | F4–Xe3–F5  | 178.48(6)  |
| F9–Pt1–F10         | 179.93(7)  | Xe2–F2–Xe1 | 136.77(6)  |
| F8–Pt1–F11         | 89.68(7)   | F3–Xe2–F2  | 178.75(5)  |
| F8–Pt1–F10         | 89.11(7)   |            |            |

**Table S4.** Experimental (SCXRD) geometrical parameters for [Xe<sub>2</sub>F<sub>3</sub>][PtF<sub>6</sub>]·XeF<sub>2</sub>(*oP120*).

| Bond distances (Å) |            |            |            |
|--------------------|------------|------------|------------|
| Pt1–F6             | 1.8744(12) | Xe1–F1     | 1.9187(13) |
| Pt1–F7             | 1.8787(13) | Xe1–F2     | 2.1757(12) |
| Pt1–F8             | 1.8836(13) | Xe2–F2     | 2.1612(12) |
| Pt1–F9             | 1.8839(13) | Xe2–F3     | 1.9232(12) |
| Pt1–F10            | 1.8851(14) | Xe3–F4     | 1.9855(12) |
| Pt1–F11            | 1.8871(12) | Xe3–F5     | 1.9900(13) |
| Angles (°)         |            |            |            |
| F7–Pt1–F10         | 179.47(6)  | F6–Pt1–F9  | 89.75(7)   |
| F7–Pt1–F11         | 89.65(6)   | F6–Pt1–F8  | 90.03(6)   |
| F7–Pt1–F9          | 90.19(6)   | F8–Pt1–F10 | 89.88(6)   |
| F7–Pt1–F8          | 90.11(6)   | F8–Pt1–F11 | 90.37(6)   |
| F10–Pt1–F11        | 89.82(6)   | F8–Pt1–F9  | 179.63(6)  |
| F9–Pt1–F10         | 89.82(6)   | F3–Xe2–F2  | 178.16(5)  |
| F9–Pt1–F11         | 89.85(6)   | F4–Xe3–F5  | 179.23(6)  |
| F6–Pt1–F7          | 90.79(6)   | F1–Xe1–F2  | 177.05(6)  |
| F6–Pt1–F10         | 89.74(6)   | Xe2–F2–Xe1 | 134.03(6)  |
| F6–Pt1–F11         | 179.41(6)  |            |            |

**Table S5.** Experimental (SCXRD) geometrical parameters for [Xe<sub>2</sub>F<sub>3</sub>][PtF<sub>6</sub>].

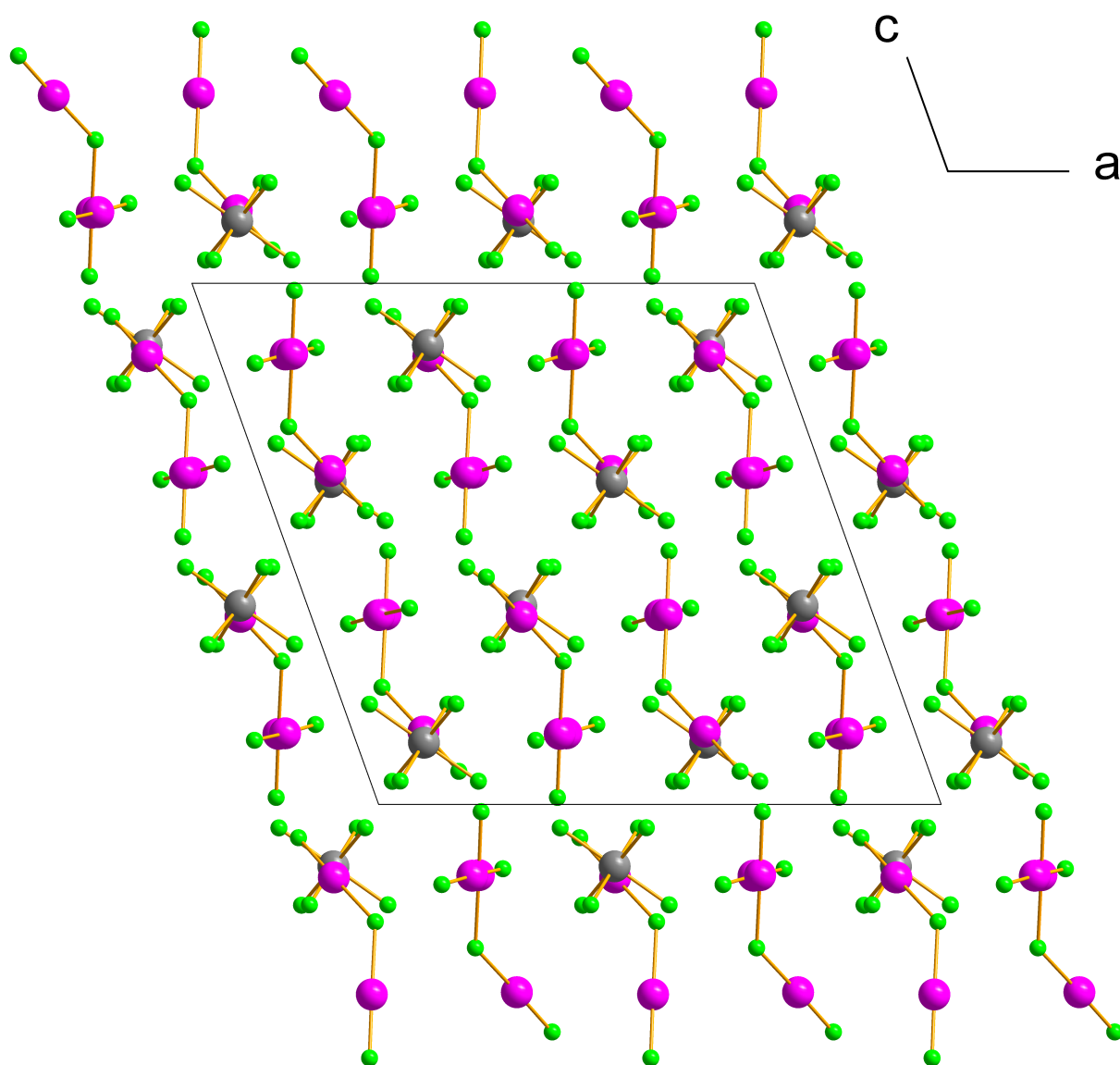
| Bond distances (Å) |          |            |          |
|--------------------|----------|------------|----------|
| Pt1–F4             | 1.877(7) | Pt1–F9     | 1.894(8) |
| Pt1–F5             | 1.879(9) | Xe1–F1     | 1.914(9) |
| Pt1–F6             | 1.884(8) | Xe1–F2     | 2.143(9) |
| Pt1–F7             | 1.892(8) | Xe2–F2     | 2.143(9) |
| Pt1–F8             | 1.894(9) | Xe2–F3     | 1.917(9) |
| Angles (°)         |          |            |          |
| F5–Pt1–F6          | 179.0(5) | F4–Pt1–F8  | 179.1(4) |
| F5–Pt1–F8          | 90.4(4)  | F4–Pt1–F7  | 89.8(4)  |
| F5–Pt1–F7          | 88.6(4)  | F4–Pt1–F9  | 91.2(4)  |
| F5–Pt1–F9          | 91.2(4)  | F7–Pt1–F8  | 89.4(4)  |
| F6–Pt1–F8          | 88.8(4)  | F7–Pt1–F9  | 179.0(4) |
| F6–Pt1–F7          | 90.7(4)  | F9–Pt1–F8  | 89.7(4)  |
| F6–Pt1–F9          | 89.5(4)  | F3–Xe2–F2  | 176.6(4) |
| F4–Pt1–F5          | 89.4(4)  | F1–Xe1–F2  | 178.7(4) |
| F4–Pt1–F6          | 91.4(4)  | Xe1–F2–Xe2 | 158.2(4) |

**Table S6.** Experimental (SCXRD) geometrical parameters for [XeF][PtF<sub>6</sub>](*mP108*).

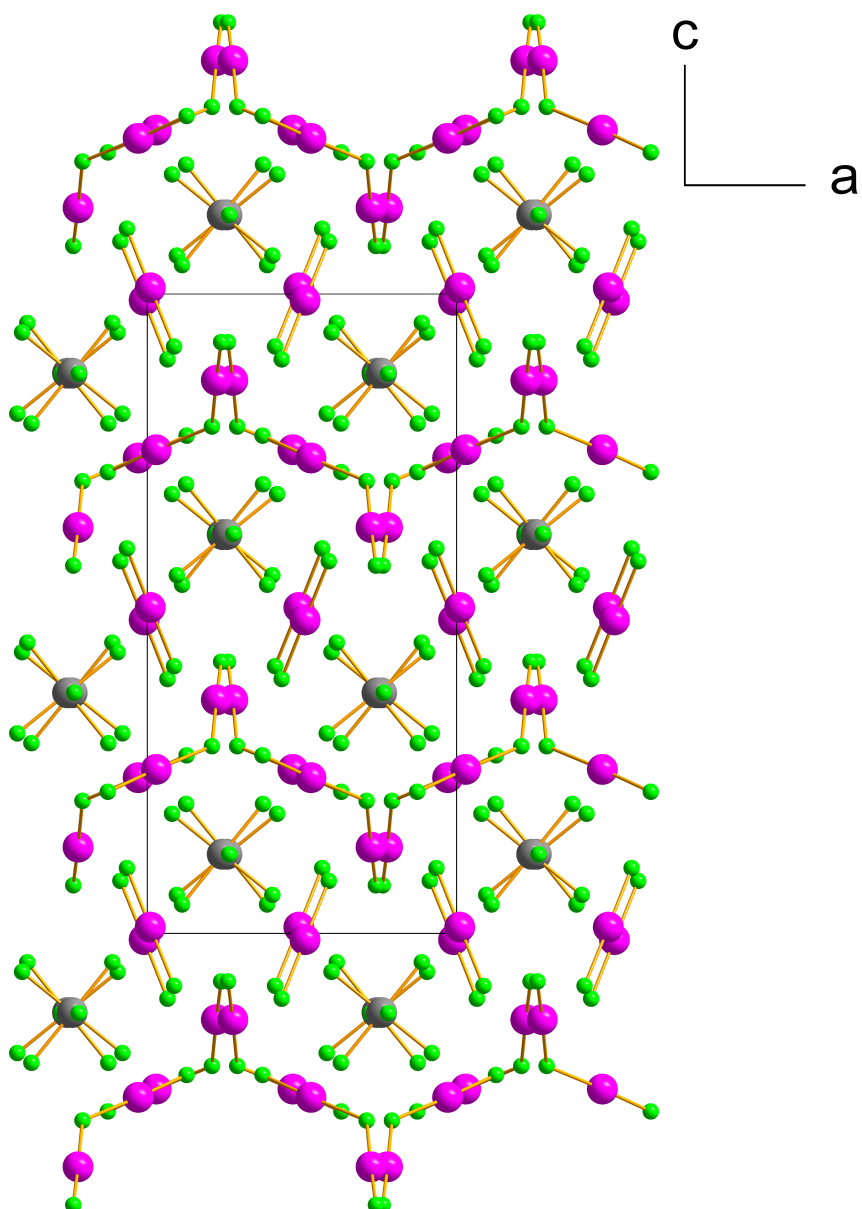
| Bond distances (Å) |            |         |            |
|--------------------|------------|---------|------------|
| Pt1–F2             | 1.9552(14) | Pt3–F16 | 1.9590(14) |
| Pt1–F3             | 1.8637(14) | Pt3–F17 | 1.8666(14) |
| Pt1–F4             | 1.8666(14) | Pt3–F18 | 1.8693(14) |
| Pt1–F5             | 1.8695(13) | Pt3–F19 | 1.8711(14) |
| Pt1–F6             | 1.8716(14) | Pt3–F20 | 1.8735(13) |
| Pt1–F7             | 1.8744(14) | Pt3–F21 | 1.8809(13) |
| Pt2–F9             | 1.9557(14) | Xe1–F1  | 1.8934(16) |
| Pt2–F10            | 1.8656(13) | Xe1–F2  | 2.2514(14) |
| Pt2–F11            | 1.8686(16) | Xe2–F8  | 1.8946(15) |
| Pt2–F12            | 1.8701(15) | Xe2–F9  | 2.2741(14) |
| Pt2–F13            | 1.8742(14) | Xe3–F15 | 1.9023(15) |
| Pt2–F14            | 1.8791(15) | Xe3–F16 | 2.2255(14) |

**Table S6.** Continued

| Angles (°)  |           |             |           |
|-------------|-----------|-------------|-----------|
| F4–Pt1–F5   | 89.84(7)  | F17–Pt3–F19 | 90.21(8)  |
| F4–Pt1–F6   | 89.34(7)  | F19–Pt3–F16 | 179.42(6) |
| F4–Pt1–F2   | 178.99(7) | F19–Pt3–F21 | 90.51(7)  |
| F4–Pt1–F7   | 91.80(8)  | F19–Pt3–F20 | 91.96(7)  |
| F5–Pt1–F6   | 90.77(8)  | F14–Pt2–F9  | 89.03(7)  |
| F5–Pt1–F2   | 90.32(6)  | F12–Pt2–F14 | 178.78(7) |
| F5–Pt1–F7   | 88.61(8)  | F12–Pt2–F13 | 90.39(8)  |
| F6–Pt1–F2   | 89.66(7)  | F12–Pt2–F9  | 89.91(7)  |
| F6–Pt1–F7   | 178.70(8) | F13–Pt2–F14 | 88.98(8)  |
| F3–Pt1–F4   | 90.27(8)  | F13–Pt2–F9  | 87.87(7)  |
| F3–Pt1–F5   | 179.74(9) | F10–Pt2–F14 | 91.16(7)  |
| F3–Pt1–F6   | 89.46(9)  | F10–Pt2–F12 | 89.46(7)  |
| F3–Pt1–F2   | 89.57(8)  | F10–Pt2–F13 | 179.07(8) |
| F3–Pt1–F7   | 91.16(9)  | F10–Pt2–F11 | 89.87(8)  |
| F7–Pt1–F2   | 89.21(7)  | F10–Pt2–F9  | 91.22(7)  |
| F18–Pt3–F16 | 89.90(7)  | F11–Pt2–F14 | 90.32(9)  |
| F18–Pt3–F21 | 179.28(7) | F11–Pt2–F12 | 90.73(9)  |
| F18–Pt3–F20 | 90.82(7)  | F11–Pt2–F13 | 91.05(7)  |
| F18–Pt3–F19 | 89.58(7)  | F11–Pt2–F9  | 178.74(7) |
| F21–Pt3–F16 | 90.02(7)  | F8–Xe2–F9   | 178.14(7) |
| F20–Pt3–F16 | 88.32(7)  | F15–Xe3–F16 | 179.26(7) |
| F20–Pt3–F21 | 88.46(7)  | F1–Xe1–F2   | 175.97(7) |
| F17–Pt3–F18 | 89.91(7)  | Pt3–F16–Xe3 | 124.20(7) |
| F17–Pt3–F16 | 89.52(7)  | Pt1–F2–Xe1  | 138.42(8) |
| F17–Pt3–F21 | 90.80(7)  | Pt2–F9–Xe2  | 117.33(6) |
| F17–Pt3–F20 | 177.72(8) |             |           |



**Figure S1.** Crystal packing in  $[\text{Xe}_2\text{F}_3][\text{PtF}_6] \cdot \text{XeF}_2$  (mC120), viewed along the  $b$ -crystallographic axis.



**Figure S2.** Crystal packing in  $[\text{Xe}_2\text{F}_3][\text{PtF}_6] \cdot \text{XeF}_2$  ( $oP120$ ), viewed along the  $b$ -crystallographic axis.

**Table S7.** Observed bands in the low-temperature Raman spectra of  $[\text{Xe}_2\text{F}_3][\text{PtF}_6]\cdot\text{XeF}_2(mC120)$ ,  $[\text{Xe}_2\text{F}_3][\text{PtF}_6]\cdot\text{XeF}_2(oP120)$ ,  $[\text{Xe}_2\text{F}_3][\text{PtF}_6]$ , and  $[\text{XeF}][\text{PtF}_6](mP108)$  (Figure 6), with relative intensities given in parentheses.

| $[\text{Xe}_2\text{F}_3][\text{PtF}_6]\cdot\text{XeF}_2(mC120)$ | $[\text{Xe}_2\text{F}_3][\text{PtF}_6]\cdot\text{XeF}_2(oP120)$ | $[\text{Xe}_2\text{F}_3][\text{PtF}_6]$ | $[\text{XeF}][\text{PtF}_6](mP108)$ |
|-----------------------------------------------------------------|-----------------------------------------------------------------|-----------------------------------------|-------------------------------------|
| 66 (26)                                                         | 65 (29)                                                         | 60 (5)                                  | 56 (3)                              |
| 81 (25)                                                         | 81 (26)                                                         | 72 (8)                                  | 128 (8)                             |
| 154 (13)                                                        | 154 (8)                                                         | 160 (24)                                | 146 (4)                             |
| 215 sh                                                          | 163 (6)                                                         | 174 (12)                                | 155 (3)                             |
| 231 (50)                                                        | 231 (37)                                                        | 229 sh                                  | 224 (14)                            |
| 236 (50)                                                        | 236 (37)                                                        | 237 (52)                                | 229 (17)                            |
| 285 (2)                                                         | 285 (3)                                                         | 278 (1)                                 | 236 (12)                            |
| 352 (2)                                                         | 351 (1)                                                         | 579 (98)                                | 244 (15)                            |
| 504 sh                                                          | 505 sh                                                          | 591 (65)                                | 249 (22)                            |
| 509 (40)                                                        | 509 (32)                                                        | 635 (100)                               | 260 (4)                             |
| 525 (26)                                                        | 525 (19)                                                        |                                         | 508 (15)                            |
| 577 sh                                                          | 576 sh                                                          |                                         | 515 (5)                             |
| 581 (36)                                                        | 580 (25)                                                        |                                         | 522 (2)                             |
| 587 (52)                                                        | 587 (39)                                                        |                                         | 580 (11)                            |
| 589 (57)                                                        | 589 (46)                                                        |                                         | 589 (4)                             |
| 598 (100)                                                       | 598 (100)                                                       |                                         | 599 (31)                            |
| 633 (81)                                                        | 633 (65)                                                        |                                         | 607 (23)                            |
| 654 (22)                                                        | 654 (15)                                                        |                                         | 609 (21)                            |
|                                                                 |                                                                 |                                         | 626 (40)                            |
|                                                                 |                                                                 |                                         | 651 sh                              |
|                                                                 |                                                                 |                                         | 655 (100)                           |
|                                                                 |                                                                 |                                         | 667 (4)                             |

sh: shoulder