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A MULTINUCLEAR MAGNETIC RESONANCE STUDY OF $\text{TeF}_x(\text{OTeF}_5)_{4-x}$,
 $[\text{TeF}_x(\text{OTeF}_5)_{3-x}]^+[\text{AsF}_y(\text{OTeF}_5)_{6-y}]^-$ AND $\text{Cs}^+[\text{As}(\text{OTeF}_5)_6]^-$

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The $[\text{OTeF}_5]^-$ and F^- acceptor properties of AsF_5 and $\text{As}(\text{OTeF}_5)_5$ have been investigated for the series $\text{TeF}_x(\text{OTeF}_5)_{4-x}$, resulting in the mixed series $[\text{TeF}_x(\text{OTeF}_5)_{3-x}]^+[\text{AsF}_y(\text{OTeF}_5)_{6-y}]^-$. The latter anions and cations have been studied in solution using ^{19}F and ^{125}Te NMR spectroscopy. Pure $\text{Te}(\text{OTeF}_5)_4$ also reacts with $\text{As}(\text{OTeF}_5)_5$ to give $[\text{Te}(\text{OTeF}_5)_3]^+[\text{As}(\text{OTeF}_5)_6]^-$ which has also been characterized by Raman spectroscopy. The interaction of $\text{Cs}^+[\text{OTeF}_5]^-$ or $[(\text{CH}_3)_4\text{N}]^+[\text{OTeF}_5]^-$ with $\text{As}(\text{OTeF}_5)_5$ in CH_3CN yields the $[\text{As}(\text{OTeF}_5)_6]^-$ anion. The quadrupolar ^{75}As atom ($I = 3/2$, 100% natural abundance) $[\text{As}(\text{OTeF}_5)_6]^-$ possesses cubic local symmetry, permitting the observation of the ^{75}As resonance and a two-bond ^{125}Te - ^{75}As spin-spin coupling in the ^{75}As and ^{125}Te spectra. The $[\text{As}(\text{OTeF}_5)_6]^-$ anion represents one of only a few examples with suitable symmetry which can be observed by ^{75}As NMR spectroscopy.