MS27-P06 | The Benefits of $Cu-K_B$ Radiation in Elucidating the Molecular Structure of Polypnictogen Cations

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Cationic polypnictogen frameworks, which are not completely saturated with organic substituents, are a relatively rare class of compounds, with metal fragment stabilised $[P_{10}]^2+,[1]$ $[E_4]^{2^+}$ (E = Pnictogen) [2] and the homoleptic $[P_9]^+$ being some of the most prominent representatives [3]. In depth crystallographic studies of compounds containing such species are often hampered by the presence of so called WCAs (WCA = weakly coordinating anion) which are needed for their stabilisation. One of the least coordinating, yet crystallographically often problematic WCAs is the $[TEF]^-$ anion ($[TEF]^-$ = $[Al(OC(CF_3)_3)_4]^-$). Its sphere-like topology can lead to very high degrees of disorder within the Perfluorptertbutyl – groups which often cannot be resolved properly by using standard wavelengths. Compared to $Cu-K_\alpha$ radiation, $Cu-K_\beta$ radiation has the advantage of a better resolution limit (0.72 Å), while still allowing for relatively high intensity measurements compared to $Mo-K_\alpha$ radiation. Thus, utilisation of $Cu-K_\beta$ radiation is beneficial for the X-ray crystallographic investigation and allows a better structural solution of compounds containing highly disordered WCAs such as $[TEF]^-$. This will be demonstrated by a comparison of X-ray crystallographic data of compounds of the general formulae $[Cp'''Ni(\eta^3-P_4R_2)][TEF]$ obtained by using $Cu-K_\alpha$, $Cu-K_\beta$ and $Mo-K_\alpha$ radiation.

[1] M. V. Butovskiy, G. Balazs, M. Bodensteiner, E. V. Peresypkina, A. V. Virovets, J. Sutter and M. Scheer, Angew. Chem. Int. Ed. 2013, 52, 2972 – 2976.

[2] L. Dütsch, M. Fleischmann, S. Welsch, G. Balazs, W. Kremer, and M. Scheer, Angew. Chem. Int. Ed. 2018, 57, 3256 – 3261.

[3] T. Köchner, T. A. Engesser, H. Scherer, D. A. Plattner, A. Steffani, and I. Krossing, Angew. Chem. Int. Ed. 2012, 51, 6529 – 6531.