When thermal analysis leads to fundamental information in chemistry: the case of fluorinated materials

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The Bordeaux' Fluorine group has been deeply involved since the sixties in the synthesis of fluorinated materials using elemental F_2-gas and anhydrous HF as powerful fluorinating agents. For various applications related to reactivity, i.e. heterogeneous catalysis, environment and sustainable development, special attention should been drawn on the reaction mechanisms and kinetics, and also on the thermal stability of the obtained compounds. In order to go further in the investigation of mechanisms that occur during fluorination processes, an original experimental set-up has been designed to work under highly corrosive atmospheres. It allows the use *in-situ* of elemental F_2 (10% diluted in Ar) as a carrier gas up to 500°C and to follow the formation of a wide range of inorganic fluorides, especially those with transition metals in high oxidation states. This set is able to bring much information about synthesis processes and key parameters of the reactivity of numerous old and new inorganic fluorides[1].

Concerning the thermal stability of the obtained fluoro-compounds, the use of TGA measurement simultaneously coupled with Mass Spectrometry (MS) and Infrared Spectroscopy (FTIR) analysis appears to be a preferred technique. This hyphenated method has been therefore developed to characterize the nature of species which are formed during the degradation of fluorides and oxide-fluorides: elemental F_2-gas, HF, CF_x ... This technique allows to evaluate the experimental conditions for an optimum use of fluorides of rare earth elements such as Ce^{IV} in CeF_4, or transition metals such as Co^{III} in $CoF_3/KCoF_4$ or Ni^{IV} in K_2NiF_6, as fluorinating agents[2].

These originals set up will be introduced and some of the preliminary results will be presented on the occasion of this symposium.

1- A. Demourgues, N. Penin, D. Dambournet, R. Clarenc, A. Tressaud, E. Durand. *J Fluorine Chemistry*, **134** (2012), 35-43

2- J. Mizudako, Y. Matsukawa, H. Quan. *J Fluorine Chemistry*, **127** (2006), 79-84