VI. "FUSED" AMMONIUM FLUORIDE AND BIFLUORIDE IN THE ANALYSIS OF QUARTZ AND SILICATES

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(Abstract)

No melting points are recorded for $(NH_4)_2F_2$ or NH_4HF_2, except that it has been noted[1] that the former becomes liquid.

During experiments with the fluorides, it was observed by the writers, that such was the case, and it occurred to them that this property might facilitate the opening of silicates, owing to the higher temperature attainable in the melt (400° C-450° C), with the further advantage that the excess flux could be removed easily by elevating the temperature. Also, since NH_3 was given off in quantity at incipient fusion of the normal salt, and the acid salt seemed to melt more promptly and was more fluid, it seemed

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[1] Berzelius, J. J., "Lehrbuch Der Chemie 3,282 (1856).

reasonable to ascribe the observed properties to the latter salt and to postulate its formation from the normal salt according to the following equation:

$$(NH_4)_2F_2 + \text{heat} = NH_3 + NH_4HF_2$$

Experiments with finely ground quartz (1.0 gram) fused in platinum with either of these fluorides, (5.0 grams) for thirty minutes, showed rapid and complete decomposition.The same was found true for beryl, iron tourmaline, orthoclase, muscovite, sericite, and lepidolite. Cyanite and zircon proved refractory. The oxides resulting from the decomposition proved soluble in $HClO_4$, provided the oxides were not overheated, in which case moderate amounts can be rendered soluble again by refusion with added fluorides.

These satisfactory tests encourage the authors to attempt quantitative applications such as the determination of SiO_2 in glass sands by direct volatilization, the opening of silicates by the fluorides to replace the carbonate fusion enabling the alkalis to be determined in aliquots, thus superseding the J. Lawrence Smith method, etc.

The scheme above outlined is believed to offer marked advantages over those in which HF-$HC10_4$ mixtures have been proposed for opening special silicates, such as glasses and glazes, mainly due to the higher temperatures reach in the fusion. The superiority over the sodium carbonate fusion for this purpose lies in the fact that the ammonium fluoride fluxes are volatile and can therefore be removed, thus enabling the alkali metals to be determed in an aliquot of the solution. Also the temperature is lower with the fluorides, enabling gold dishes to be substituted for the more costly platinum.