X-Ray Fluorescence for Analysis of Fluorine in Aluminosilicate Material

Lora L. Brehm*, The Dow Chemical Company, Midland, MI
Reinhard H. Hinz, The Dow Chemical Company, Schkopau, Germany

Development of easy, safe routine method for the analysis of fluorine (F) is challenging especially for solid materials. Methods using ion selective electrode potentiometry (ISE) or ion chromatography (IC) for detection of fluoride in aqueous solutions involve laborious and potentially hazardous sample preparation procedures in order to remove the fluorine from the solid and get it in solution. Combustion ion chromatography systems require that fluorine be completely released from the solid during combustion. Neutron activation can be used for fluorine analysis but has limitations based on sample composition. Another option for fluorine analysis in solids is x-ray fluorescence (XRF) spectrometry for which developed methods are readily adapted for routine use. Fluorine can be analyzed in a solid sample after preparation as a pressed powder pellet. Analysis of fluorine by XRF does pose a challenge as sensitivity of the technique is limited by several factors, one being the poor fluorescence yield of the characteristic F Kα line and the other being its low energy. Infinite thicknesses are on the order of a few microns so the analysis is greatly influenced by particle size, surface variations and at times migration of fluorine in the surface region of a prepared specimen.

This paper will present the results of the development and validation of an x-ray fluorescence method for the analysis of fluorine in aluminosilicate samples at concentrations from about 0.01 to 1.5 weight percent. The optimization of sample preparation, measurement conditions and calibration as well as preparation and validation of calibration and accuracy check standards using an alternative method will be discussed. The stability of the prepared specimen for measurement of F Kα was also evaluated and will be presented.