

WAVELENGTH DISPERSIVE XRF FOR ANALYSIS OF SMALL AMOUNTS OF CATALYST – HOW SMALL CAN WE GO?

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Research and development efforts for new catalysts often require elemental analysis of much smaller sized samples (50-500 mg) than typically analyzed by conventional x-ray fluorescence (XRF) spectrometry. Rapid, accurate and precise analysis of elemental content is critical to meet timelines for process development, scale-up and catalyst performance investigations. X-ray fluorescence often has an advantage over other elemental analysis techniques such as inductively coupled plasma (ICP) emission spectroscopy and neutron activation analysis (NAA) in that sample preparation and/or analysis time can be much faster, reducing analysis turnaround times.

Modern wavelength dispersive XRF spectrometers have collimator masks which allow for definition of the sample area viewed by the detector (~ 6 to 40 mm in diameter). Small collimator masks used in conjunction with disposable micro XRF sample cups commercially available in several sizes (6 to 20 mm i.d.) enable easy and consistent measurement of small amounts of catalyst sample.

This presentation will focus on development and validation of an XRF method for determination of platinum and gallium supported on alumina for analysis of samples smaller than one gram. Calibrations were prepared with several different sample sizes and precision and accuracy data collected to determine the minimum amount of sample necessary to meet method requirements. Discussed will be preparation and verification of XRF standards by reference techniques (NAA, ICP), challenges of small sample analysis and comparison of the precision and accuracy of XRF methods developed using several different collimator masks and sample sizes.