

## ***Analysis of Catalysts for Chemical Composition***

### **Introduction**

Heterogeneous catalysts typically comprise multiple nano-crystalline components made up from what often starts as a mixture of mixed inorganic compounds. Information on bulk and spatially-resolved chemical composition is vital to the researcher, producer and user of catalysts.

While other flyers cover surface and spatially-resolved chemistry, this flyer concentrates upon what Intertek MSG can do on bulk analysis, trace analysis and fouling in connection with the practice of heterogeneous catalysis.

### **Bulk Chemical Analysis**

**X-Ray Fluorescence analysis (XRF)** is a convenient way of checking that elemental composition is within specified ranges. It is also used to monitor for possible catalyst poisons in new and spent material. XRF provides a rapid and sensitive method of measuring most elements of the periodic table at levels in the range 0.02 to 100% in catalysts. This also applies to the raw materials used in manufacturing catalysts.

XRF is useful to identify poisoning by elements such as sulphur, chlorine or unwanted heavy metals which may have caused catalyst failure in service. Similarly, support materials may be screened for impurities.



If there is a requirement to check that actual concentrations present match those in a specification, then quantitative analysis by XRF is appropriate for the accurate determination of major and minor concentrations. XRF is an effective tool for examining catalysts for the elements Na to U in the approximate ranges of 0.01 to 100% with results expressed as elements or oxides (calculated).

For the determination of trace levels of most elements it may be more appropriate to use **ICP techniques** (OES or MS), for which it is usually necessary to bring the sample into aqueous solution using microwave digestion with acids, or alkaline fusions. The extra sensitivity offered by ICP-OES and particularly ICP-MS more than compensates for the dilution effect of bringing the sample into solution. With these techniques very low concentrations may be observed in one catalyst compared to another e.g. for comparing competitor's formulations. Similarly it is possible to examine the distribution of heavy metal poisoning throughout a catalyst bed (see below).



The determination of some non-metals is poor using XRF or ICP techniques, though some laboratories may estimate their concentrations using these techniques. **Combustion analysers** are a much better solution for the determination of carbon, hydrogen, nitrogen, sulphur, halogens and oxygen in suitable matrices. Intertek MSG has several instruments with different detection methods depending upon the levels of analytes expected.

## Bed Profiles and Poisoning

Intertek MSG uses XPS and SIMS as sensitive tools providing there is relatively little catalyst fouling. In some cases analytical STEM is able to identify the growth of new structures. Poison fronts may be determined from cross-sections.

Many catalysts are sensitive to even small concentrations of certain impurities, and these can arise in the feedstock or in catalyst manufacture. The active surfaces of these solids are very sensitive to traces of chemical compounds that include Cl, S, As, Pb, P, Fe, Hg and Zn. The presence of such impurities can cause loss of activity and/or reduce the specificity of the reaction. These effects on the catalyst may be temporary; then it may be possible to regenerate the material. In some cases the poisoning could be permanent and this may require the catalyst to be discharged and replaced, usually a costly exercise. By sub sampling from different areas of the bed and performing analyses, a profile of the extent of poisoning can be developed. Intertek MSG uses XRF semi-quantitative scans to monitor levels of poisons through a catalyst bed to help determine what section requires replacing and so reduce unnecessary expenditure.

Lead, arsenic and many other elements including phosphorus can be determined by ICP techniques after bringing the catalyst into solution. Detection limits for **ICP-OES** are ppb level in solution, whereas for **ICP-MS** are ppt level in solution for many elements.

The sulphur content of new and used catalysts can be examined after combustion in a stream of oxygen, using infra red detection of the combustion products. At the same time carbon determination is performed on the same instrument. Major to minor levels of sulphur and carbon are determined. Should there be a need to measure trace levels of sulphur then **microcoulometric detection** on a different instrument can be applied.

Chlorine deposition can be determined by **oxidative microcoulometry**, whereas water soluble anions can often be leached from catalysts and determined by ion chromatography.

Nitrogen contamination can be determined either by a CHN analyser, or for low levels a **combustion analyser** with chemiluminescence detection.

## Mass Balance Studies on Chemical Plants

Today's chemical processes invariably involve the presence of a catalyst. When the catalyst is expensive, or contains an expensive component such as a platinum group element, it is important to understand where losses of the element could occur. Analyses of the fresh catalyst, process streams, plant deposits and spent catalyst can be used to determine the mass balance for the component.

To learn more about chemical analysis in Intertek MSG and how it can be applied to your interests in catalysis, please contact:

Dr Jeff Franks

Tel: (44) (0)1642 435784

email: [jeff.franks@intertek.com](mailto:jeff.franks@intertek.com)

*All the techniques described above are used by Intertek MSG in characterising catalyst materials. In addition, crystalline phases may be examined and quantified by XRD, morphology by electron microscopy, pore systems and surface area by nitrogen BET methods. Please contact us for more information.*

For other MSG capabilities, please contact Dr Allan Stewart on:

Telephone: (44) (0)1642-435788 or

Facsimile: (44) (0)1642-435777

E-mail: [msgenquiry@intertek.com](mailto:msgenquiry@intertek.com)

Intertek MSG, The Wilton Centre, Redcar  
Cleveland TS10 4RF United Kingdom

Intertek MSG is part of ITS Testing Services (UK) Limited  
Registered Office: 25 Savile Row, London W1S 2ES