

IDENTIFICATION OF GUMs AND SCREENING FOR CATALYST RESIDUES IN PHARMACEUTICAL END PRODUCTS USING XRF SPECTROMETRY

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X-ray fluorescence spectrometry (XRF) is a non-destructive technique with high elemental sensitivity and simple or no sample preparation. XRF can be used in the production and quality control to quantify major and minor elements in fillers, binders, lubricants, and other excipients. Traces of toxic compounds in excipients and final drug products can be detected and quantified down to ppb levels. Also traces of catalyst residues in final drug products can be quantified with high precision using XRF.

This paper presents two solutions for typical pharmaceutical applications: fingerprinting GUMs using a benchtop EDXRF spectrometer and screening catalysts residues in pharmaceutical end products using a high-end EDXRF spectrometer.

General Use Materials (GUMs) used in manufacturing are routinely analyzed by time consuming and complex instruments (such as titration and ion chromatography) for identity testing. It is desirable to simplify and speed up this process and make it more efficient. A PANalytical MiniPal 4 EDXRF spectrometer was evaluated for this purpose using several inorganic compounds commonly used on the GUMs list. This instrument is a relatively small, simple, low cost spectrometer. The XRF technique is inherently quick and simple, and requires virtually no sample preparation. The compounds tested can be identified qualitatively by comparison with reference spectra but this requires some expert knowledge of interpreting the spectra. The main technical objective of the evaluation was to establish the basis of a method that could reliably distinguish between compounds using a quantitative approach. This method should be suitable for relatively inexperienced operators with very little need to interpret the spectra. The evaluation also included the general suitability of the technique to a manufacturing environment.

XRF can also be used for screening and monitoring pharmaceutical end products concerning catalysts residues. Typical amounts for catalysts in end products should be below 10 µg/g and can be quantified at this concentration level with high precision.