

EVALUATION OF PORTABLE XRF AND XRD ANALYZERS FOR IDENTIFICATION OF COUNTERFEIT PHARMACEUTICAL PRODUCTS

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Portable XRF devices are becoming more widely used for an ever growing number of applications and can detect some elements down to single ppm levels. Although lab-based XRD can be used to identify the major components in a sample, portable XRD devices are a relatively recent advance. These techniques are rarely used for identifying counterfeit products, particularly in a regulatory environment. The focus of this presentation is the development, evaluation, and comparison of portable XRF and XRD methods to definitively answer the question as to whether an erectile dysfunction (ED) medication is authentic or not.

A Bruker Tracer III handheld XRF analyzer was used to analyze both authentic and counterfeit ED samples. Here, the samples were analyzed “as is”, with no prior sample preparation or homogenization, and XRF measurement times of one minute. Spectra were acquired under optimized conditions (i.e., kV, current, and filter) to improve signal to noise ratios for the light elements in these samples. Replicate spectra were acquired for the same pill and different pills to evaluate run-to-run and pill-to-pill variability, respectively. The results were evaluated by a number of statistical methods. Using the t test, f test, or principal component analysis, spectra of the suspected counterfeit products were proven to be statistically different from the authentic product at the 95% confidence level.

A InXitu BTX II portable powder XRD analyzer was used to analyze the same products. Here, the samples were ground and sieved prior to analysis. A unique feature of this analyzer is its use of a piezoelectric sample vibrator to effectively randomize the positions of the sample particles relative to the X-ray beam. Spectra were acquired for time periods varying between 5-100 minutes to study the effect of improved signal to noise ratios on the diffractograms. The results clearly showed significant difference between the authentic and suspected counterfeit products. Although the major ingredients of these products can be identified, the presence of amorphous or unknown materials can render this task more difficult in some cases.

Whether using XRF or XRD to identify fake pharmaceutical products, one must have either a sample of the authentic product for comparison or access to the reference spectra or diffractogram. The XRF method is simpler to use, requires minimal or no sample preparation, and can be accomplished in a minute or less (*assuming* that the elemental profiles of the authentic and counterfeit product are indeed different). The XRD method is more attractive insofar that it allows the possibility of identifying the composition of the product in question (i.e., qualitative and quantitative data). Either method would be highly useful in identifying counterfeit pharmaceutical and other products which are becoming more widespread with the tremendous growth in global trade, and might find use for widely counterfeited medications such as anti-malarial drugs, Lipitor™, and Tamiflu™.