

Preface

X-ray fluorescence, which is an analytical method for determination of the elemental composition of bulk material and for characterization of coating systems, has a long-time history and is used in many laboratories. Conventionally, large sample areas are analyzed with XRF. This requires the preparation of homogeneous and flat samples. During the preparation process, the sample material often needs to be deformed or damaged, i.e., the preparation is destructive. But often the composition of final products needs to be determined and this should be done nondestructive. This means the complete sample needs to be positioned in the instrument, a homogenization or polishing to get a flat sample area is not possible. In that case, micro-X-ray fluorescence (μ -XRF) can be used because this investigates only small sample areas due to the concentration of excitation radiation by X-ray optics to these small areas. Then, even irregular shaped samples can be analyzed because for the small excitation spot a flat sample area can be found.

μ -XRF has shown an exciting and strong development in the last 10–15 years mainly due to the availability of new and improved X-ray optics and driven by the high request for position-sensitive analytical methods. The array of different applications for μ -XRF is continuously growing. At the beginning the excitation intensity of synchrotron sources was required to get sufficient fluorescence intensity from small sample areas. In this case, the radiation was only collimated. But nowadays, with the availability of focusing optics μ -XRF can also be performed with laboratory instruments. This allows the use of the method for a larger user community and also a further enlargement of interesting applications.

Therefore, a summary of the state of the art of μ -XRF seems to be helpful for the actual user of the method for better understanding of instrument design and its influence on the analytical performance, but also to give suggestions for the use of this powerful method for different purposes.

The method also offers a few new interesting questions for understanding the interaction of X-rays in small sample volumes and the influence of the sample environment to the measured intensities. This is a new situation for XRF which was scientifically nondescript. XRF in general is physically very good understood and mathematically described that even standardless quantifications are possible. But for μ -XRF this situation is changed—both the physical models for the analysis of small sample areas and the new methodological possibilities offer interesting fields for research.

The author had the privilege to be associated with the development of μ -XRF instruments during the last 20 years in different companies and was involved in the introduction of new instruments into the market. This enabled to accumulate experiences on the instrumentation as well as about the application.

There are already several monographs available that give a detailed description of the interaction of X-rays with matter. Therefore, here these topics are described briefly and summarize only the basic facts required for the understanding of the main functions of both the different components of a micro-X-ray fluorescence spectrometer and the quantification procedures. In this way, the described content should be easily understandable even for beginners but gives instrument users the necessary knowledge for an efficient use of the method.

The [Chap. 1](#) of the book describes the interaction of high energetic electromagnetic radiation with matter and the basic design of X-ray spectrometers, while [Chap. 2](#) describes in detail the different components of the spectrometer, in particular, the components of micro-X-ray spectrometers like different X-ray optics and their applicability for μ -XRF instruments, the different possibilities and requirements for sample positioning, and the energy-dispersive detectors mostly used in μ -XRF instruments. In [Chap. 3](#) the different geometric arrangements for μ -XRF, the corresponding instrument types, and the measurement modes as well as the possibilities for the presentation and interpretation of elemental distributions are discussed.

Quantification for small areas can be different from large areas because sample homogeneity and also the environment of the analyzed area influence the analytical result. An important application of μ -XRF is the examination of layer systems. Thickness and composition are of interest. The corresponding quantification models are discussed together with general considerations about analytical errors in [Chap. 4](#).

This is followed by a discussion of different sample preparation methods that can be used for μ -XRF. Also if sample preparation is easy or even not necessary or possible, it is necessary to consider the analyzed volume and its relation to the material that should be characterized.

In [Chap. 6](#), a comparison is given with other analytical methods with spatial resolution including a discussion of possibilities for the combination of μ -XRF with these methods.

Finally, different examples for the application of μ -XRF are presented. The predominant part of measurements were performed with instruments which were developed in groups under the responsibility of the author. This was for measurements with collimators, mainly the M1 Ora and M1 Mistral from Bruker Nano GmbH, but also instruments of the series Compact of Roentgenanalytik GmbH, for measurements with polycap optics, mainly the M4 Tornado from Bruker Nano GmbH and also the Eagle from EDAX. Because the measurements were performed over a long period of time the instrumental configuration was different for most of the measurements. Only few applications were performed with other instruments, though this will be separately mentioned.

These examples are arranged according to the different measurement modes—i.e., point, multi-point, and distribution analysis and for distribution analysis also for the different scientific disciplines where μ -XRF actually is used.

Berlin, 2013

Michael Haschke

<http://www.springer.com/978-3-319-04863-5>

Laboratory Micro-X-Ray Fluorescence Spectroscopy
Instrumentation and Applications

Haschke, M.

2014, XVIII, 356 p. 254 illus., 107 illus. in color.,

Hardcover

ISBN: 978-3-319-04863-5