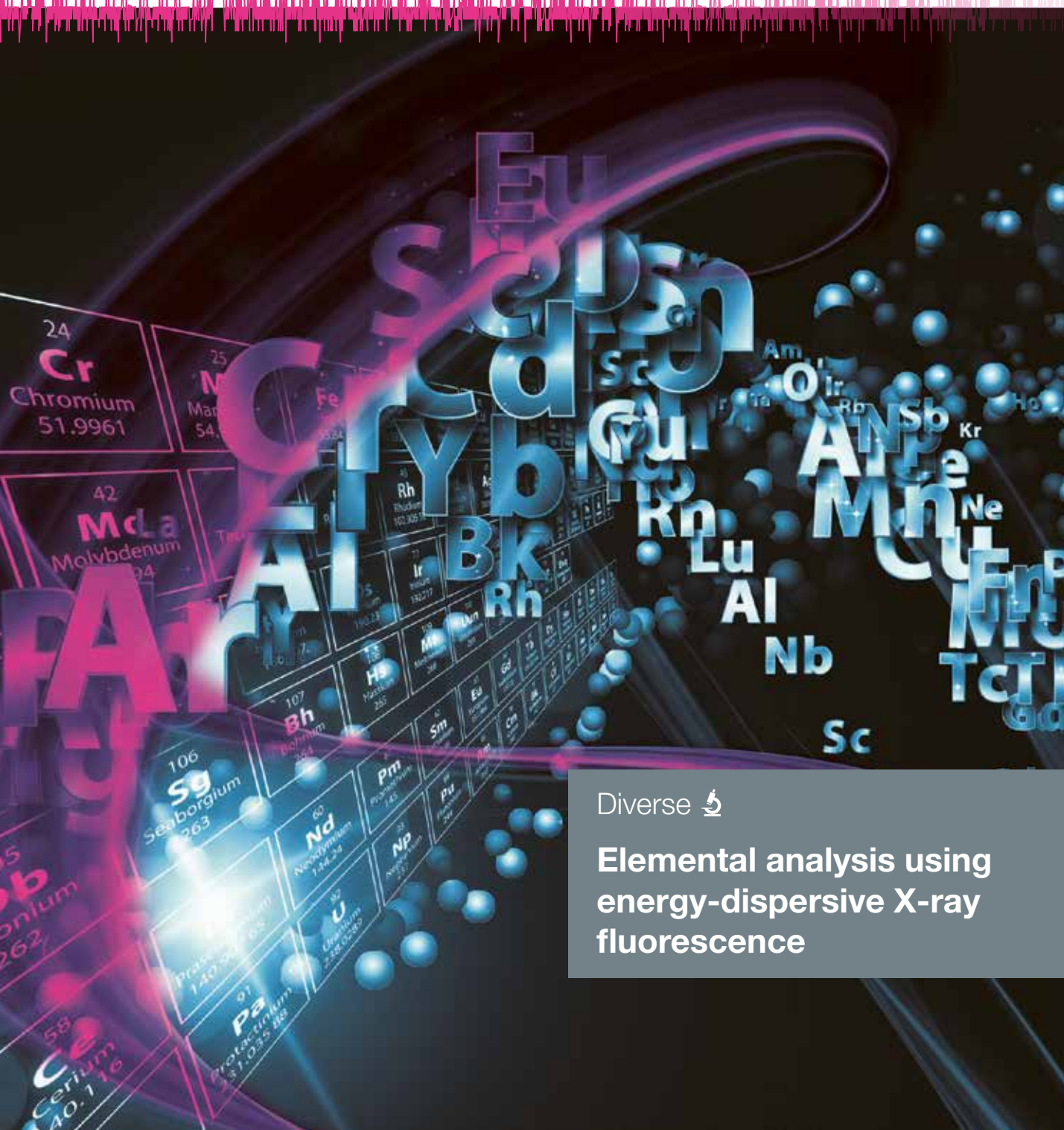


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# ANALYTICS

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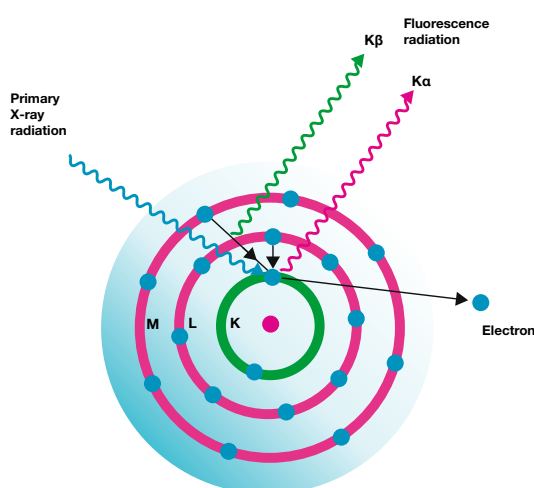
**Elemental analysis using  
energy-dispersive X-ray  
fluorescence**

# Elemental analysis using energy-dispersive X-ray fluorescence

Author: Andreas Fankhauser

Many popular crime series suggest to the viewer that in an analytical laboratory both accurate qualitative and quantitative results can be generated within seconds, without the sample or piece of evidence suffering any damage. In laboratory reality, however, time-consuming, destructive sample preparation procedures are often necessary to solve complex analytical problems. One exception is elemental analysis using energy-dispersive X-ray fluorescence (ED-XRF). As in a television thriller, the spectroscopic method enables a qualitative statement on the elementary composition of a material to be made within a very short time.

## 1 Function principle photoelectric effect and ED-XRF



Energy-dispersive X-ray fluorescence is based on the photoelectric effect. The excitation of the sample material takes place by means of short-wave, high-energy X-rays. Due to the high energy, electrons close to the nucleus are also ejected from the sample atoms. The resulting free spaces are directly occupied by electrons farther from the nucleus, while the energy released during this stabilisation process is emitted as fluorescence radiation (see 1).

The energy difference between electrons of different nuclear shells depends on the atomic number. Consequently, the detected fluorescence signal is characteristic for each element<sup>1</sup>.

## Applications

The description of the field of application allows parallels to be drawn to the role of infrared spectroscopy in organic structure analysis. ED-XRF is also a fast and relatively inexpensive analysis method. However, instead of functional organic groups, the main elements of a sample are identified and on this basis initial conclusions on identity are possible. ED-XRF is therefore also an important addition to the ICP-MS technique, which is predestined for trace analysis up to the ppt range due to its high sensitivity, but not for non-destructive analyses. The key characteristics of both methods are compared in Table 1.

As a fast and non-destructive analysis method, ED-XRF is already routinely used in various industries. Interlabor Belp AG focuses on troubleshooting and special projects in the pharmaceutical industry.

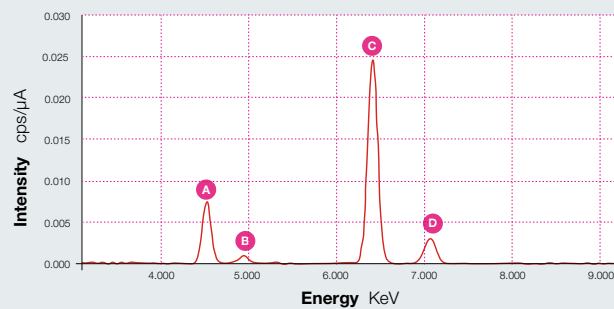
An interesting application of ED-XRF is the determination of the qualitative composition of tablets. Especially the inorganic excipients titanium dioxide, zinc oxide or magnesium in magnesium stearate, which are frequently used in tableting, can be detected without any problems. Consequently, ED-XRF can be used as part of bioequivalence studies to show that generic drugs have the same composition as the original preparations (see 2). The corresponding measurement data can be used to support approval or any bio-availability studies. The already mentioned inorganic minerals titanium dioxide and zinc oxide are equally popular ingredients in skin and sun creams due to their effect as UV filters. Classical methods for determining the zinc oxide content in the end product are usually based on complexometric titration. In this context, ED-XRF turns out to be a time- and cost-efficient analytical alternative. In the building materials industry, the exact determination of the composition of the materials used and the identifica-

**Table 1**

Key characteristics	ED-XRF	ICP-MS
Measurable sample types	<ul style="list-style-type: none"> <li>• Powder</li> <li>• Metals</li> <li>• Aqueous solutions</li> <li>• Organic solutions</li> <li>• Whole objects</li> </ul>	<p>Only aqueous solutions without organic matrix</p> <p>All other samples are mineralised, e.g. by acid digestion</p>
Measuring range	<p>Upper limit: 100 %</p> <p>Lower limit: approx. 10 ppm</p>	<p>Upper limit: approx. 10 ppm (in the test sample after sample dilution up to 100 %)</p> <p>Lower limit: approx. 10 ppt</p>
Qualitative analytics	possible	possible
Quantitative analytics	Possible if matrix-identical standards exist	possible
Limitations	Light elements not determinable (hydrogen to neon)	Increased interference in some elements (sulphur, silicon, calcium, potassium, iron, vanadium)

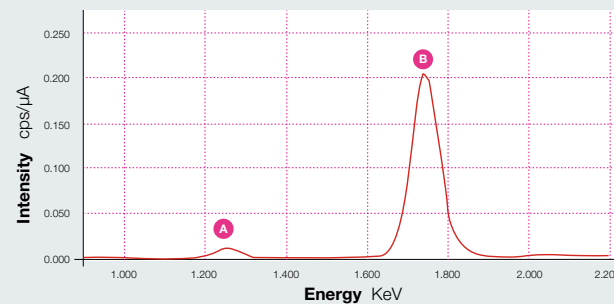
**2 ED-XRF Tablet shell analysis**

- A** Titanium K- $\alpha$  Line
- B** Titanium K- $\beta$ 1 Line
- C** Iron K- $\alpha$  Line
- D** Iron K- $\beta$ 1 Line



**2 ED-XRF Tablet core analysis**

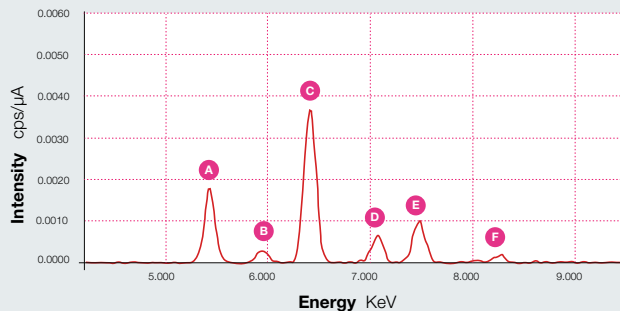
- A** Magnesium K- $\alpha$  Ligne
- B** Silicon K- $\alpha$  Ligne



tion of any impurities represent an essential component of process and quality control<sup>2</sup>. Since the materials are almost always present as inorganic solids, they are predestined for non-destructive analysis using ED-XRF. In this way, it was possible to clearly specify sodium sulphate as the cause of concrete efflorescence during a troubleshooting. The alternative analysis strategy, identification by wet chemical precipitation reactions, is much more labour intensive and does not allow any quantitative statements. Furthermore, due to the matrix effects of precipitation reactions, it is not possible to clearly interpret the results for each sample.

### 3 ED-XRF Drill sample analysis from inside the oven.

- A Chrome K- $\alpha$  Line
- B Chrome K- $\beta$ 1 Line
- C Iron K- $\alpha$  Line
- D Iron K- $\beta$ 1 Line
- E Nickel K- $\alpha$  Line
- F Nickel K- $\beta$ 1 Line



Another special project involved the analysis of a metal furnace from the high-tech components industry. Since the composition of steel and other iron alloys can be determined by ED-XRF without any sample preparation, a drilling sample from the interior of the furnace was analysed directly and the main elements have been identified (see **3**).

### Conclusion

Energy-dispersive X-ray fluorescence is a fast and non-destructive analytical method that enables the qualitative and quantitative determination of almost all elements relevant in industry. It can be used universally for everything from the determination of UV filters and tablet compositions to material testing and troubleshooting in the metal and building materials industries. □

### Literature

1. Shackley, M. Steven. „X Ray Fluorescence Spectrometry (XRF).“ *The Encyclopedia of Archaeological Sciences* (2011): 1-5.
2. Giurlani, Walter, Massimo Innocenti, and Alessandro Lavacchi. „X-ray microanalysis of precious metal thin films: Thickness and composition determination.“ *Coatings* 8.2 (2018): 84.

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