XTrace

- High Performance Micro-Spot X-ray Source for SEM

Micro-XRF on SEM

Innovation with Integrity

Upgrading SEMs for Trace Element Analysis

XTrace is a micro-spot X-ray source for attachment to almost any SEM with a free inclined port on the specimen chamber. It adds the capabilities of a complete micro-XRF spectrometer to the microscope. Limits of detection are improved 20 to 50-fold in the mid to heavy element range. Additionally, larger sample volumes become accessible as X-rays have a higher information depth than electrons.

The use of polycapillary X-ray optics allows the generation of high fluorescence intensities on very small sample areas. The X-ray optics collect tube radiation from a large solid angle and concentrates the X-rays on spots down to 35 micrometers in diameter for Mo-K radiation.

The generated X-ray fluorescence spectrum is measured with the attached XFlash® silicon drift detector, belonging to the QUANTAX EDS* system. The use of an XFlash® SDD gives excellent energy resolution, as determined by the detector specifications. Count rates of about 40 kcps can be achieved in the analysis of metals, using a 30 mm² active area detector.

The high intensity produced by the polycapillary optics and the reduced spectral background of X-ray excitation leads to an enhanced sensitivity for trace elements. The improvement is approximately 20 to 50 times compared to electron excitation. Also, because X-ray excitation is more efficient for higher-Z elements, detection limits down to 10 ppm can be achieved. This makes XTrace most interesting for the trace analysis of heavy elements.

QUANTAX EDS and XTrace offer the possibility of a combined micro-XRF and EDS quantification under a single user interface providing optimum results that are better than the results for each method alone.

*The term EDS refers to electron excited energy-dispersive X-ray microanalysis in this document.

Designed for Ease-of-Use

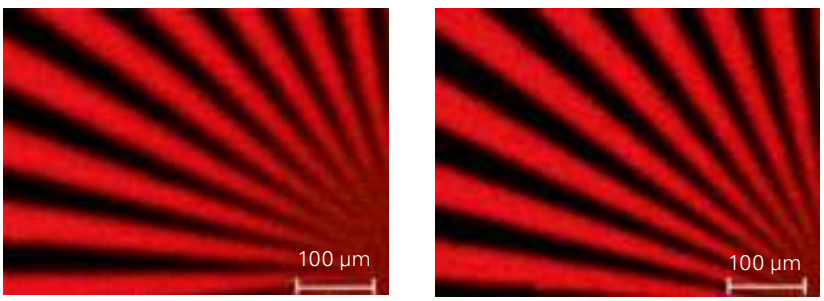
Concentrate on the task instead of system setup

- Distribution analysis with ESPRIT HyperMap collects complete data sets, supporting offline analysis
- Samples can be analyzed with EDS and micro-XRF without necessity of a position change
- Both measurement methods are implemented in the same analytical software suite – ESPRIT – allowing method change with a mouse click
- Does not interfere with standard SEM operation and EDS, XTrace can stay in a fixed position suitable for most tasks.

Providing the power of a micro-XRF spectrometer without the investment

- Analytical results are comparable to standalone systems
- Image tiling allows mapping large areas
- Three primary radiation filters to suppress diffraction peaks
- Uses the SEM stage, no separate stage needed
- Easy avoidance of diffraction peaks in XRF spectra through SEM stage rotation
- Allows sample tilt to produce minimum spot sizes.

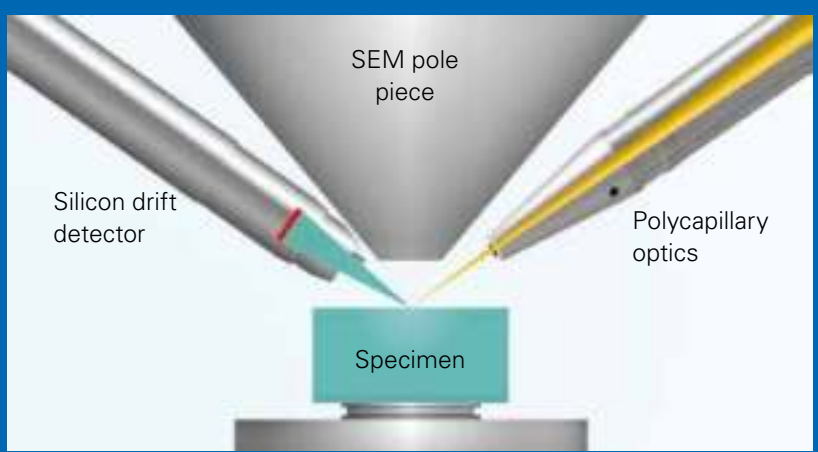
Resolution improvement through sample tilt



A chromium Siemens star mapped with a step size of 25 µm with untilted stage (left). The same star mapped with a step size of 25 µm and the specimen stage tilted towards the X-ray source by 30° (right), showing the difference in spatial resolution.

Functional principle

The same sample region as excited by the SEM electron beam is irradiated with X-rays from a microfocus tube. Polycapillary X-ray optics concentrate a large solid angle of radiation to a small spot on the sample (yellow beam), providing good spatial resolution and high X-ray intensity. The silicon drift detector and signal processing chain installed for electron-induced energy-dispersive X-ray analysis is used to acquire, process and evaluate the produced X-ray fluorescence radiation (turquoise).



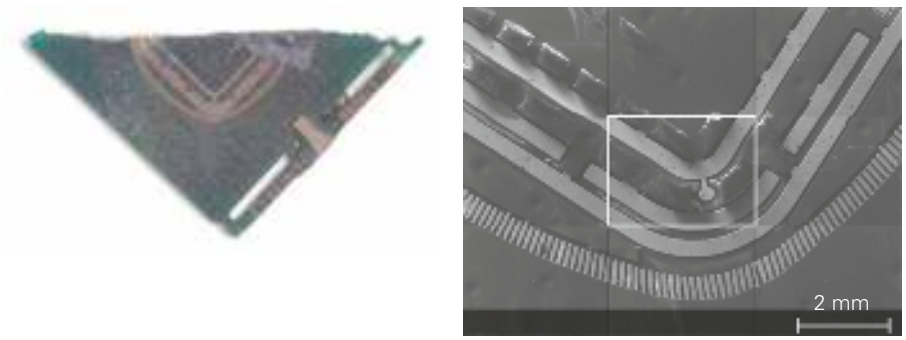
Application Examples

XTrace greatly increases the versatility of element analysis in the scanning electron microscope. It covers applications in materials analysis (e.g. metals, catalysts) forensics (e.g. paint, glass, gunshot residue), geoscience and many others.

Characterization of multilayer samples

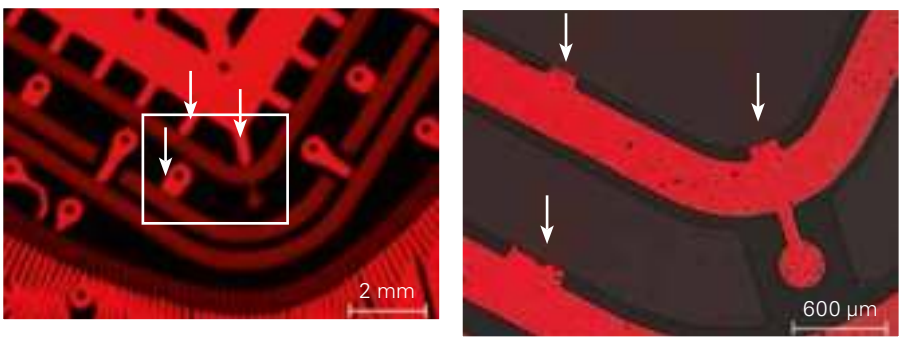
Multilayer samples are especially suitable for analysis with XTrace because of their sometimes complex internal structure, which may be partly "invisible" to EDS.

Sample images



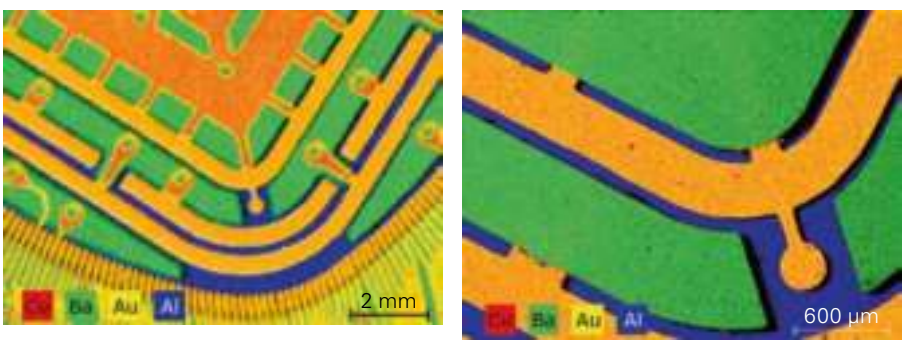
Optical (left) and secondary electron (SE) micrograph (right) of the piece of multi-layer PCB analyzed. The SE image is tiled from 9 single frames.

Single element maps of copper



Left: Micro-XRF stage map of copper over the area shown in the SE image above. The white rectangle indicates the area of the EDS map. Right: Overlay of the EDS map of copper with the SE image of the same area. The white arrows in both maps indicate solder pads that are visible in the XRF map and invisible in the EDS map. Their visibility in the XRF map is due to the higher information depth of X-rays.

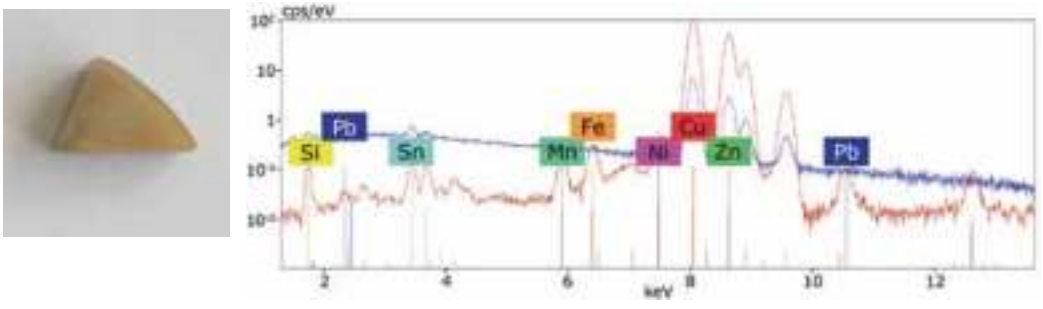
Multi-element maps of the sample



Micro-XRF (left) and EDS (right) multi-element maps of the same regions as before. The elements barium (Ba), gold (Au) and aluminum (Al) are displayed in addition to copper. Note that gold is obviously present in more locations than can be seen in the EDS map.

Versatility in SEM Analysis

Image and spectra of a copper alloy



Photograph of a piece of a copper alloy (left) and comparison of its XRF and EDS spectra (right) in logarithmic scaling. Note the presence of many trace elements that are visible in the red XRF spectrum. The alloy could be identified as yellow brass (CuZn33) through quantification of the XRF spectrum.

Reliable metals and alloy identification

The sensitivity of micro-XRF makes it very suitable for the identification and analysis of alloys, particularly for small particles, e.g. debris from engine wear and similar.

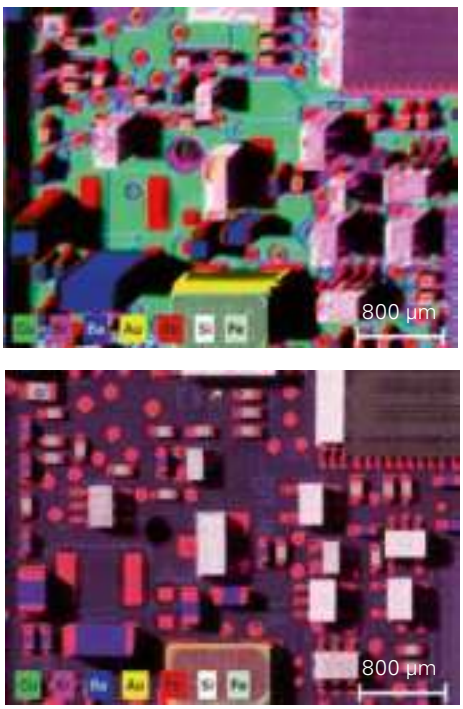
Analyzing components and circuits on PCBs

Both the trace element sensitivity and the information depth of XRF analysis are advantageous in investigating this sample type. PCBs may contain hazardous elements covered by the RoHS (Reduction of Hazardous Substances) regulation. These can be detected more reliably by micro-XRF, especially as RoHS and other regulations require low limits of detection.

Metals and hazardous elements in polymers

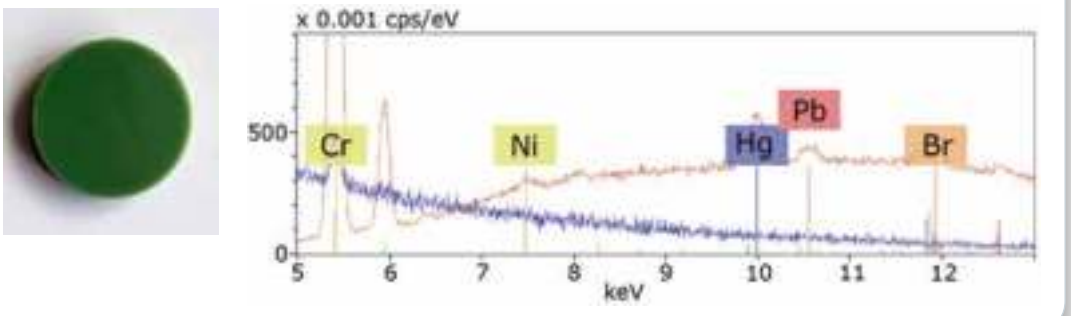
Polymers are often engineered to suit a specific purpose. This includes the use of metals and minerals as additives. XTrace can be used to both detect elements and map their distribution in polymers. Additionally, RoHS analysis is also important, especially for toys.

Micro-XRF and EDS analysis of a PCB



Tiled micro-XRF (top) and EDS (bottom) maps of the same region of a PCB with components. The same colors were used for the same elements in both maps. The noticeably different color mix in both is a product of the higher penetration depth of X-rays. Copper is obviously concentrated in deeper layers. The apparent tilt of the features in the XRF map is an effect of the inclined X-ray source and the sample topography. It can be reduced by tilting the sample towards the source.

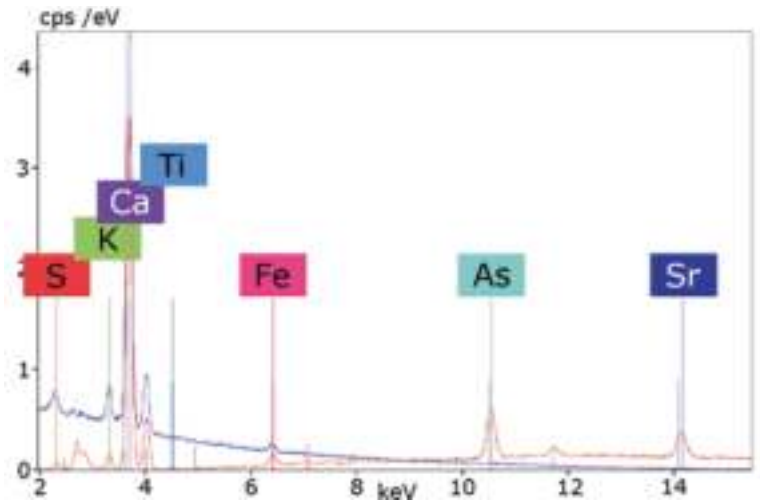
Image and spectra of a polymer



Optical micrograph (left) and spectra (right) of a polymer standard for metals and hazardous element detection testing. The micro-XRF spectrum (red) shows the trace elements nickel (Ni), mercury (Hg), lead (Pb) and bromine (Br), which cannot be detected with EDS. Both spectra were acquired within 300 s at an input count rate of 6 kcps.

A Single Interface for Micro-XRF and EDS

Micro-XRF and EDS spectra of a glass standard



Comparing micro-XRF (red)and EDS (blue) spectra of a NIST 620 glass standard. The unlabeled peaks in the low energy range of the Micro-XRF spectrum are scattered tube radiation (rhodium L lines).

Combined quantification results of the glass standard

Element	At. No.	Line S.	EDS Mass [%] Norm.	XRF Mass [%] Norm.	Comb. Mass [%] Norm.	Certified Val. /M%
Oxygen	8	K-Series	45.71	45.58	46.03	46.82
Sodium	11	K-Series	10.54	10.32	10.61	10.68
Magnesium	12	K-Series	2.32	2.27	2.33	2.22
Aluminium	13	K-Series	1.34	0.89	0.89	0.95
Silicon	14	K-Series	33.94	34.99	34.18	33.70
Sulfur	16	K-Series	0.16	0.12	0.12	0.11
Potassium	19	K-Series	0.37	0.35	0.35	0.34
Calcium	20	K-Series	5.30	5.34	5.34	5.08
Titanium	22	K-Series	0.00	0.01	0.01	0.01
Iron	26	K-Series	0.28	0.03	0.03	0.03
Arsenic	33	K-Series	0.04	0.06	0.06	0.04
Strontium	38	L-Series	0.00	0.04	0.04	–

XRF: Oxygen quantification by stoichometry

Quantification results table of the glass standard, showing the concentrations calculated from EDS data, micro-XRF data and the combination of both. The certified concentrations for this standard have been added on the right for comparison.

XTrace is controlled by Bruker's ESPRIT software suite that is also part of the other analytical tools for electron microscopy, including EDS, WDS and EBSD. This provides the user with a set of advantages:

- Operation of all analytical tools under a single interface
- Switching between methods with a mouse click
- Direct analysis of identical sample positions with different methods
- Easy combination of analytical results gained through different methods.

The added benefit for XTrace users is that EDS and micro-XRF quantification results can be used for mutual enhancement of quantification reliability.

The best of both worlds – combining XRF and EDS quantification for most accurate results

ESPRIT employs an advanced standardless Fundamental Parameter (FP) approach for accurate and reliable quantitative analysis of micro-XRF spectra. If desired, this can be further refined by using calibration standards.

ESPRIT provides the option to use micro-XRF and EDS quantification results simultaneously and the advantages of both methods come into effect. EDS provides reliable light element results, while micro-XRF has its strengths in the medium to heavy element range with limits of detection down to 10 ppm. This means that the combined concentration calculation from EDS and micro-XRF results, as offered by the analytical software suite, is of the highest quality attainable by energy-dispersive analysis.

Analytical Flexibility Built in

Apart from spot analysis and line scans using the SEM stage, ESPRIT supports single and multi-frame XRF mapping. Maps are saved in hyperspectral databases (ESPRIT HyperMap), where a complete spectrum is stored at every point of the map. This permits analysis at any time during or after measurement and provides different options for data evaluation.

Point analysis

Placing a crosshair over a point in the map will display the spectrum from that point under the Spectrum Tab of the ESPRIT software. This provides a fast overview of the composition at the current position.

Line scan

The line scan tool allows placing an arbitrary line in the HyperMap and to plot the element concentrations along this line, both qualitatively and quantitatively.

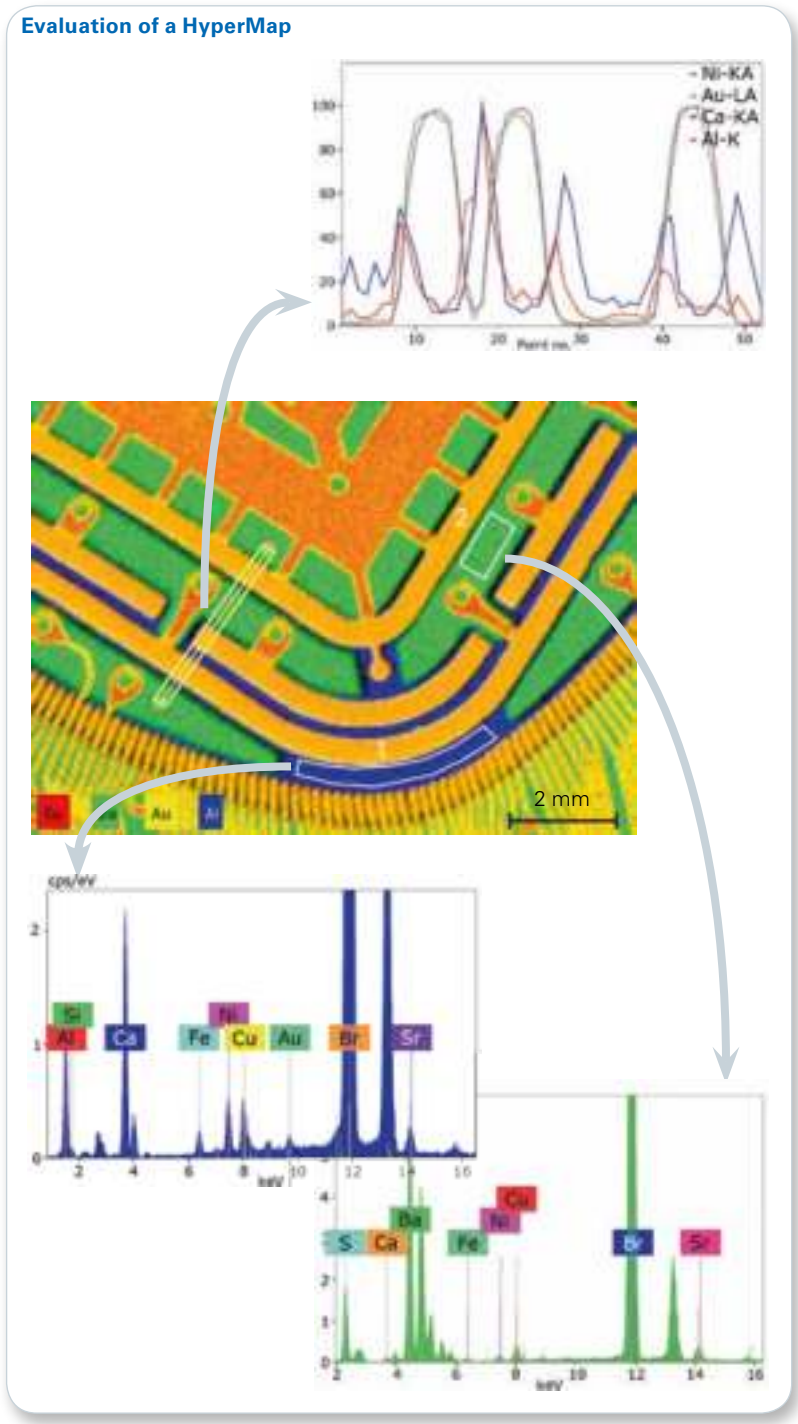
Object analysis

Several different shapes – rectangles ellipses and polygons – can be drawn in the HyperMap. The sum spectra of these objects can be inspected under the Spectrum Tab. This option is useful for the analysis of objects of a single composition and especially for the improvement of count statistics.

Phase analysis

Maps can sometimes be difficult to interpret, especially when many elements are present. The ESPRIT Autophase tool can identify areas of similar composition and attribute them to different chemical phases present in the sample. Phases can be either defined by the user as objects in a HyperMap or calculated by histogram analysis.

Evaluation of a HyperMap



HyperMaps allow easy extraction and processing of data. Several different tools were used to analyze data from the HyperMap in the center of the illustration:
Top: A line scan along the yellow line defined in the center left of the map. The rectangle around the center line indicates that the line was broadened to improve statistics. The graphics show the distribution of Ni, Au, Ca and Al.
Bottom: Sum spectra of the areas "1" and "2" in the map, showing differences in composition.

Technical Specifications

Parameter	Description
Sample types	Solids, particles
Excitation	High brilliance X-ray tube with polycapillary X-ray optics
Excitation parameters Target material Tube parameters Spot size Polycapillary optics length Filters	 Rh, optional Mo, W 50 kV, 600 µA (maximum 30 W, depending on X-ray tube) Less than 40 µm for Mo K 400 mm Up to 3 primary X-ray beam filters, standard configuration 100 µm Al, 20 µm Ti, 10 µm Ni (other filters according to customer requirements possible)
Detection	XFlash® silicon drift detector*
Instrument control	no additional PC required, QUANTAX EDS PC can be used (recommended)*
Instrument software	Bruker ESPRIT
Instrument control functions	Complete control of tube parameters and filters
Spectra evaluation	XRF peak identification, artifact and background correction, peak area calculation, standardless quantification, combined XRF and EDS quantification, layer analysis
Distribution analysis	HyperMap capability (Hyperspectral database)
Result presentation	Quantification results, statistical evaluation, element distribution (line scan, mapping)
Power requirements	100 - 240 VAC (1P), 50/60 Hz
Dimensions	300 mm x 250 mm x 140 mm
Weight	11 kg
Quality and safety	ISO 9001:2008, CE certified Fully radiation protected system, radiation < 1 µSv/h

*XTrace requires a pre-installed QUANTAX energy-dispersive X-ray spectrometer (EDS), consisting of XFlash® silicon drift detector, SVE signal processing unit and system PC.