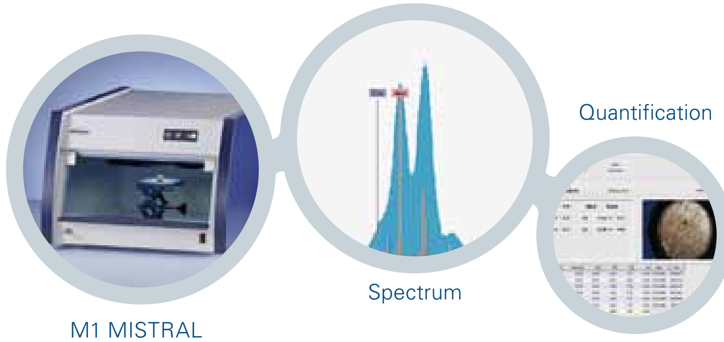

Lab Report XRF 432

Repeatability and stability tests on the M1 MISTRAL spectrometer

The performance of an analytical instrument is determined by parameters like repeatability, reproducibility, stability and accuracy. These parameters describe the instrument behavior in certain situations.

Repeatability is the probability that an analytical result remains the same for repeated measurements in a short period of time. If measured for a long period of time, this property is called stability. Reproducibility is the capability to repeatedly deliver the same result for the same sample but for different users or in different laboratories. Finally, accuracy is the ability to obtain the correct result.

For this lab report, various measurements were performed to determine the repeatability and stability in dependence of different measurement parameters.

Instrumentation

All measurements were performed using the M1 MISTRAL spectrometer equipped with a large area proportional counter. It features following technical parameters:

Excitation	W-tube (max. 40 kV, 40 W) glass side window
Detection	prop-counter with 1100 mm^2 sensitive area 900 eV energy resolution (Mn Kα) 30,000 cps maximum count rate
Dimensions	size (WxDxH): 450x550x420 mm, 46 kg

Analysis

Since one of the main applications of the M1 MISTRAL spectrometer is the analysis of jewelry alloys, the measurements were performed on a series of gold alloys.

Working principle

When excited by the radiation of an X-ray tube, the sample emits characteristic radiation. This radiation is detected by an energy-dispersive detector that delivers energy-proportional signals. For this analysis, a proportional counter was used. The energy distribution of the detected radiation is determined by a pulse height analysis. Special quantification models are necessary to calculate the concentration of different elements in the sample. The complete instrument is controlled by a special software package on a laptop computer that is connected to the device via USB.

Results

Stability

The stability of the instrument was determined by repeated measurements of pure Au over a long period of time. The variation of intensity over a period of more than 40 hours (more than 2400 single measurements of 60 seconds each) was 0.168 %. The theoretically expected value calculated by statistics is 0.164 %, i.e. only insignificantly lower.

Temperature dependence

The temperature dependence of intensity over a large temperature range is shown in Figure 1. The room temperature changed from 23° to 35° C. The instrument temperature, displayed on the diagram, changed from 29° to 43° C. The peak intensity is stable for all these temperature changes. The instrument temperature is 8 degrees higher than the room temperature. This shows the very low power consumption of the device.

The temperature dependence of peak position is marginal. Changes are smaller than 0.1 % for this large temperature range. However, the detector's energy resolution is influenced by temperature. The FWHM slightly increases with temperature, but also only less than 0.1 % per degree.

Repeatability of quantification

In order to determine repeatability, four samples were measured 10 times for 60 seconds each. Since the measurement results are influenced by the element concentration, the measurements were carried out on samples with different compositions. The delivered results regarding repeatability are shown in Table 1.

Tolerance against fluctuations in excitation intensity

The quantification procedure also shows high tolerance against changes in excitation conditions. In order to illustrate this feature, the same sample was measured with the same tube voltage but different tube currents. For every excitation parameter, measurements were repeated five times. The results are presented in Table 2.

Conclusion

Changes in temperature, excitation intensity or other factors have only very little influence on the repeatability and stability of the instrument. They are lower than statistical fluctuation, which means that they can be disregarded.

Temperature dependence of intensity

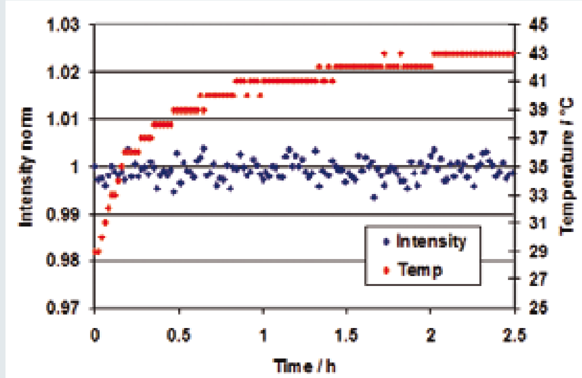

Fig. 1 Diagram showing the temperature dependence of intensity over a large temperature range

Repeatability

Gold content	Stand. deviation for 10 measurements for			
	Au (wt.%)	Au statistic error (%)	Ag (wt.%)	Cu (wt.%)
22 karat (91.6 %)	0.09	0.30	0.05	0.07
18 karat (75 %)	0.05	0.33	0.08	0.07
14 karat (58.5 %)	0.11	0.37	0.33	0.06
10 karat (41.7 %)	0.09	0.50	0.11	0.10

Tab. 1 Repeatability analysis results

Tolerance

Tube current	Average concentrations with standard deviation		
	Au	Ag	Cu
Given	64.5	14.9	15.1
800 µA	64.54 ± 0.122	14.94 ± 0.080	15.18 ± 0.040
780 µA	64.53 ± 0.103	14.93 ± 0.046	15.19 ± 0.072
650 µA	64.50 ± 0.078	15.00 ± 0.089	15.15 ± 0.129
500 µA	64.64 ± 0.104	14.99 ± 0.127	15.01 ± 0.090

Tab. 2 Tolerance analysis results

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