BAM: Certified Reference Materials for the Analysis of Gold-Alloys

BAM Federal Institute for Materials Research and Testing certified three gold-based reference materials (CRMs) suitable for X-ray fluorescence spectrometry (rose gold, white gold, yellow gold). The mass fractions of the alloying elements gold, silver and partially copper, nickel and zinc were certified. Characterization of the CRMs was performed using wet chemical methods and fire assay.

Due to their high material value special requirements are placed on the analysis of precious metals. Since both the purity as well as the composition of the alloying elements determine the value of the material, for both the determination of impurities and traces as well as of major and minor components precise and accurate analytical methods are necessary.

For the determination of trace elements atomic spectrometric methods like ICP-OES are suitable. Normally the test sample has to be dissolved before analysis which, in case of precious metals is not always easy because of their chemical inertness.

Analytical Methods

For the determination of gold in gold alloys, various methods exist that differ in accuracy and in effort. A relatively simple and therefore widely used method is the assay by the touch needle [1]. With this method it is possible to analyze precious metals goods with minimum sample intake (ca. 0.5 mg) quickly and easily. However, the accuracy and precision of the assay by the touch needle is worse than that of the other, more elaborated methods.

More complex but also more precise is the fire assay method, the classical and worldwide used standard method for precise gold determination. The method is based on the fact that precious metals as opposed to nonprecious metals practically do not oxidize at high temperatures. Therefore the test sample is melted together with lead at a temperature of 1,150 °C. The nonprecious metals from the alloy and the lead react with oxygen. The metal oxides are absorbed by the porous crucible
The precious metals remain selectively as metallic grain. In a following step gold is separated from silver using nitric acid [2].

Mostly used for the analysis of precious metal alloys is energy dispersive X-ray fluorescence spectrometry (EDRFA).

EDXRF allows a direct investigation of solid samples, which is, in contrast to methods that require a previous dissolution of the sample, non-destructive [3].

For calibration and quality control of the precious metal analysis using EDXRF certified reference materials are necessary, e.g. to avoid errors caused by matrix effects. A few years ago a set of 16 gold alloy reference materials with gold grades between 33.3% and 99.99% was launched by the Polish Institute of Non-Ferrous Metals [4]. However, the nine materials with lower gold grades (33.3% to 57.8%) are now no longer available.

In general, today calibration functions implemented in mobile spectrometers already give very good results. Therefore, instead of a calibration set BAM prepared, based on commonly used gold alloys, the three certified reference materials ERM-EB506 rose gold, ERM-EB507 white gold and ERM-EB508 yellow gold. These three certified reference materials are especially intended for the use with mobile X-ray fluorescence spectrometers.

**Certification of Reference Materials**


First, a suitable candidate material was procured and examined for sufficient homogeneity using wavelength dispersive X-ray fluorescence spectrometry. From
each of the three different gold alloys ca. 270 slices with 15.75 mm diameter and 250 to 300 µm thickness were produced. The slices were embedded in acrylic glass discs with 40 mm diameter and 5 mm thickness:

- 585‰ rose gold (Au 585.9‰, Ag 39.5‰, Cu 357.0‰, Zn 19.1‰)
- 750‰ white gold (Au 751.8‰, Ag 30.7‰, Cu 147.5‰, Ni 49.5‰, Zn 20.9‰)
- 750‰ yellow gold (Au 751.6‰, Ag 249.7‰)

The materials were characterized by nine selected, highly experienced laboratories operating in the precious metals industry (working group "Precious Metals" of GDMB: [http://bit.ly/GIT-GDBM](http://bit.ly/GIT-GDBM)) using wet chemical methods. The participating laboratories were free to choose any suitable analytical method for analysis. The determination of gold was normally done using the fire assay method; only one laboratory used ICP-OES after dissolution of the material with aqua regia. All other elements (Ag, Cu, Ni and Zn) were determined using ICP-OES.

**Results and Discussion**

The certified mass fractions were calculated as mean values from the laboratories' means, normally based on four individual values. Table 1 shows the certified values together with their uncertainties. The calculation of the uncertainties was done taking into account contributions from the certification inter-laboratory comparison (scatter between laboratories) and from the homogeneity investigation.

As an example figure 1 shows the results of the certification inter-laboratory comparison for gold in rose gold. It can be seen that the results obtained with ICP-OES are in good agreement with those obtained with fire assay. Only the intra-laboratory spread is higher, here the precision of the fire assay method is still better. Also noteworthy is the very good agreement of the laboratory means which makes it possible to specify the certified values with very small uncertainties. To check for plausibility the results of five laboratories which determined all elements of interest, were summarized. The mean values of the sums for the three reference materials were between 99.98% and 100.03%.

**Literature**

[3] „Es ist nicht alles Gold was glänzt" Die Analyse von Edelmetallen; Spectro-Ametek, Whitepaper

Authors

Sebastian Recknagel, BAM Federal Institute for Materials Research and Testing, Germany

Contact

Bundesanstalt für Materialforschung und -prüfung (BAM)
Unter den Eichen 87
12205 Berlin
Germany
Phone: +49 30 8104 0
Telefax: +49 30 8112029