Hydro Aluminium Reference Samples

The Hydro Aluminium Research & Development in Bonn produces reference samples for the calibration of automated instrumentation used in the direct analysis of solid samples. The most important applications are optical (atomic) emission spectrometry with spark excitation (S-OES) and x-ray fluorescence spectrometry (XRF). The reference samples are also suitable for other analytical methods. Long standing experience in casting practice and analysis insure the reliability and quality of the reference samples.

The samples which are listed in the catalogue are available at the time of publication. They were produced only in limited quantities. When individual reference samples are sold out they are replaced by new batches if further demand exists. This leads to small deviations from the analyses given in the catalogue. When any standard ordered or requested is out of stock or unavailable an effort will be made to recommend an appropriate substitute.

Production and application

Reference samples for the analysis of aluminum and aluminum alloys are produced by means of DC-casting which is especially suitable for producing homogeneous samples. The radial segregation is reduced due to this casting procedure and a removal of the casting skin is not necessary. The billets obtained are cut in discs of 20 mm thickness. Setting-up samples (or recalibration samples) for the control and correction of the instrumentation drift are also offered in greater lengths.

The structure and the morphology of the reference samples widely correspond to those of
the cylindrical analytical test samples of 38 mm diameter and 30 mm height which are taken (sampled) in the cast houses from liquid metal melts. The comparable structure of reference and solidified test samples is especially advantageous for analytical methods which are sensitive to structure influences.

The homogeneity of the reference samples is checked by S-OES and XRF. The chemical compositions are tested if possible by two independent analytical procedures corresponding to the accepted state of the art of analytical techniques. Gravimetry, photometry, atomic absorption spectrometry (AAS), optical emission spectrometry with inductively coupled plasma source (ICP-OES), mass spectrometry with inductively coupled plasma source (ICP-MS),

X-ray fluorescence spectrometry (XRF) and neutron activation analysis (NAA) are used. These procedures are subjected to further control by means of comparative analyses with other experienced analytical laboratories.

The valid certificate of analysis is delivered with each sample. It refers to the homogeneous ring zone between 3 mm and 20 mm measured from the edge, the normally used spark zone in S-OES. In a few samples slight segregation was observed between the ring zone generally used for sparking and the center zone of 20 mm diameter. Therefore only these ring zones have to be used for all analytical procedures. In XRF the mask of the sample holder has to be arranged such that only the certified area is measured. For the calibration of wet chemical methods drillings must be taken only from the described area.

The properties of the samples are optimized with regard to their main use, the spark emission spectrometry (S-OES). Reference samples are also recommended for calibration and control of other analytical methods, e.g. optical emission spectrometry with glow discharge (GDOS) excitation, X-ray fluorescence spectrometry (XRF), optical emission spectrometry with ICP-source (ICP-OES), mass spectrometry with ICP-source (ICP-OES), atomic absorption spectrometry (AAS) and mass spectrometry with spark source or glow discharge source (SSMS, GDMS).

Special samples are offered for the daily control of instruments used for multi element analyses, i.e. setting-up or recalibration samples. They contain element contents which give for each element a low and a high signal in the useful measuring range. The regular measurement of setting-up samples which are constant in their properties gives actual values, which allow a drift correction of the analytical instrumentation by comparison with the nominal values recorded on these setting-up samples during the basic calibration.

For setting-up samples no certified element contents are necessary. Typical data are sufficient for the selection. A basic requirement, however, is a good homogeneity of the element distributions, which is guaranteed by the casting procedure used. The contribution of the sample homogeneity to the whole variation of measuring procedure shall be lower than the reproducibility of the measurement itself. Some elements, despite the use of the most modern casting techniques, can not be homogeneously distributed over the length of a billet, e.g. 1 m. Setting-up samples having a content gradient over the length of the billet must be cut in pieces of 50 mm in maximum. The nominal

value referring to the basic calibration must be determined for each piece before use.

The known influence of the statistical error on a single measurement affects the whole recalibration period. After each drift correction, which is normally performed in each shift, therefore, a test by means of a reference sample is recommended to guarantee a reliable correction of the instrumentation drift. The reliability of the results is assured if a reference sample is used for which the composition is comparable to those of the test sample.