Recalibration Samples (R)

When calibrating spectrometers (AES, XRF) with calibration samples, recalibration samples (setting-up-samples), are measured a number of times in order to obtain a reliable nominal value suitable for the calibration. The additive and/or multiplicative changes in spectrometer intensity increases variations of the calibration curve in the linear scale of the co-ordinate system. In order to trace (calculate) the actual intensities at any subsequent time for the nominal intensities produced during calibration, a low intensity (low point LP) and a high intensity (high point HP) has to be determined for each analysis channel. In metal analysis, low points for all analysis channels are often measured with pure metal (Fe, Al, Cu etc.). This may cause inconveniences.

The high points are usually measured with synthetically combined, highly homogeneous and highly precise samples with as many elements as possible. The synthetic combination is given as an approximate analysis. The samples in many cases are not located on the calibration curves.

The mathematical procedure of recalibration, being automatically calculated by modern spectrometers, is:

$$LP\ nom = a + b\ LP\ act$$
$$HP\ nom = a + b\ HP\ act$$

From these two linear equations both a and b are calculated and applied as follows to the measured intensity I act of the unknown sample:

$$I\ nom = a + b\ I\ act$$

When using recalibration samples it is not always guaranteed, even if the sample number is the same, that the new sample will correspond exactly to the sample which has to be replaced. It is therefore recommended that at least three sets of recalibration samples should be procured when buying a spectrometer. If recalibration samples are to be replaced with others with the same coding, new nominal recalibration values should be determined with the spectrometer as follows:

Very careful recalibration with new grinding paper, clean grinding wheel, sharp turning pool and reversing plates has to be carried out. Numerous sparkings (minimum six, preferably more) both on the recalibration samples in use as well as on those to be replaced have to be made. The new recalibration samples are then measured with minimum six sparkings using the „recalibrated intensity ratios“ program. The values obtained are the new nominal recalibration values. These have to be entered automatically in the corresponding chart and stored in the data base.

If recalibration samples have to be replaced with others having a different coding (e.g. C and D replaced with RN 13 and RN 14), the above mentioned procedure must be carried out in order to determine the nominal values for the new recalibration samples.

Recalibration frequency is specific to each instrument and its individual application:

- Instrument-specific recalibration is required to be carried out at different intervals. Even if instruments are of identical design, have the same application and stability limits. (Because of differences in the stability of phototubes, which the spectrometer manufacturer can only influence by selection.)

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- Application-specific is understood to be for the same stability, the recalibration frequency depends on preset quality limits. Thus the limits for metal producers (e.g. if the lower end of the concentration range is to be set for expensive alloy elements) will be tighter than for metal processors who often just wish to identify the type of material - its quality number.

We are able to manufacture recalibration samples with exactly identical composition in a limited quantity. „Rolling recalibration“ is then feasible, which significantly improves the long-time accuracy of the spectrometer.

Recalibration samples for mobile spectrometers

Mobile spectrometers are generally used for „front line“ identification testing. Usually the alloy elements are sufficient for such testing. Accompanying and trace elements are not required.

Stationary spectrometers are generally used to determine all relevant elements, including any accompanying and trace elements which - according to Quality Standard or agreement between producer and user - must not exceed maximum contents.

The number of elements in recalibration samples for mobile spectrometers is therefore less than for stationary spectrometers. However, homogeneity and precision may not be lower, particularly in the case of recalibration samples for mobile spectrometers with SDAR (spark discharge in argon).

As the alloy elements to be examined are the same for mobile as well as for stationary spectrometers, the contents of these elements must also be the same.

From recalibration samples required for stationary spectrometers we have „derived“ (mainly for low-alloy steel and Al base metal) recalibration samples for mobile spectrometers.

Type of sample	Stationary	Mobile
Low-alloy steel	RN 13 + RN 14	RN 16 + RN 17
Al-base	RA 18 + RA 19	RA16 + RA 17

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Fe comparison samples for mobile spectrometers

Mobile spectrometers, particularly those with arc or spark discharges in air, are mostly used for identification testing of metals. Determination of chemical elements in the usual sense, using calibration curves, is only occasionally carried out. Spectral intensities of test pieces are compared with those of comparison samples (within preset tolerances). If these are within the tolerances, quality is considered to be the same as that of comparison samples.

We have assembled a set of comparison samples of frequently used steels. The analysis were carried out on a number of spectrometers calibrated with international certified reference materials. The comparison samples are themselves reference samples in accordance with ISO 9000. The comparison samples are 40 mm in diameter and 30 mm high. Other heights are available on request.

Analysis of comparison samples:

Fe - Basis		Ni - Basis		Cu - Basis		Al - Basis	
1.0580	St 52	2.4608	LC-Ni 99	2.0070	E-Cu	3.3525	AlMg 2 Mn 0,3
1.5415	15 Mo 3	2.4360	Monel 400	2.1090	RG 7	3.1645	AlCuMgPb
1.7131	16 MnCr 5	2.4858	Icoloy 825	2.0401	CuZn 39 Pb 3	3.2315	AlMgSi 1
1.7225	42CrMo 4	2.4663	Inconel 617	2.1052		3.4345	
1.2080	X 210 Cr 12	2.4665	Hastelloy X				
1.4034	X 46 Cr 13						
1.4305	X 10 CrNiS 18 9						
1.4306	X 2CrNi 19 11						
1.4571	X 6 CrNiMoTi17 12 2						
1.4876	X 10 NiCrAlTi 32 20						
1.3243	EMo 5 Co 5						
1.0737	9 SMnPb 36						
1.2067							
1.2379							
1.2842							
1.0503							

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