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A NEW VERSATILE HOLDER FOR THE CALIBRATING STANDARD USED FOR QUANTITATIVE ANALYSIS IN A JEOL 840 SEM

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Abstract

An improved holder has been designed for a cobalt calibrating standard used in an EDS Link 10,000 system attached to a JEOL 840 SEM. This holder allows the calibration of the EDS software for a wide range of samples differing in size, surface and height. These differences usually cause difficulties during quantitative analyses. The new holder allows the position of the cobalt to be adjusted according to the size and height of the sample.

Key Words: EDS, Cobalt standard, Holder, Calibrating element, Quantitative analysis, ZAF

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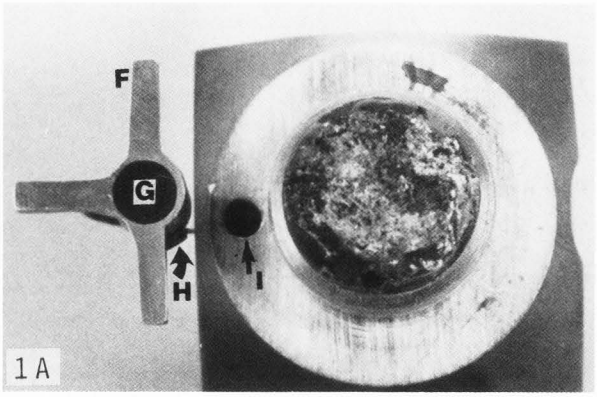
Introduction

The Link EDS AN10000 systems use a series of software programs (ZAF) to carry out chemical analyses. In particular the ZAF-4/FLS program is used for quantitative analysis of polished bulk specimens (1). The accuracy of the analyses depends strongly upon obtaining spectra from the sample, from a set of standards and from a calibration element (usually Co) under identical operating conditions. An advantage of this program is that in routine analyses, it is sufficient to obtain the spectra of the sample and that of the calibrating element. However one of the basic parameters that has to be kept constant is the distance from the sample/calibrating element to the detector. Furthermore it is important that both sample and calibrating element be perpendicular to the electron beam. It should be noted that if in the course of one type of analysis (e. g. quantitative) another type of analysis (such as digimap) is required, recalibration with the calibrating standard is necessary.

The calibrating element is embedded in the specimen holder recommended by the manufacturer. This allows it to be kept permanently in the microscope, but its distance from the detector is fixed and only samples of standard thickness can be accurately analysed. To overcome this difficulty, a more versatile type of holder has been designed whose height and position may be adjusted to those of the sample.

Description of the Holder

A holder for a cobalt calibrating standard must meet certain requirements which include: 1. reliability; 2. accuracy; 3. ease of operation, this includes that it be light and small, easy to repolish, and easy to move from one sample to another. 4. adaptability to a wide range of samples, varying in size and height. A holder which can only move along two horizontal directions does not meet the last requirement. The holder described here is attached to the sample holder with a flexible wire (fig. 1) and it can be moved in three perpendicular directions.



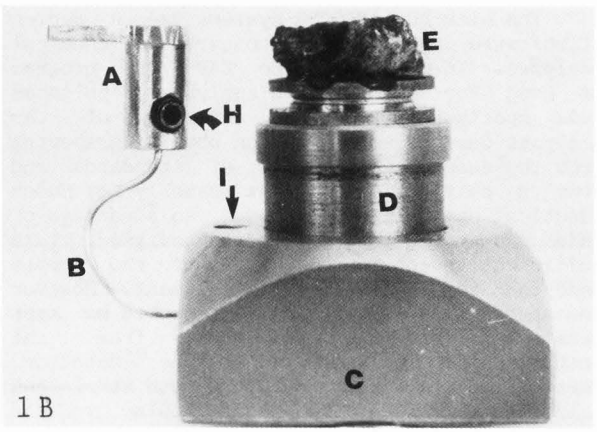
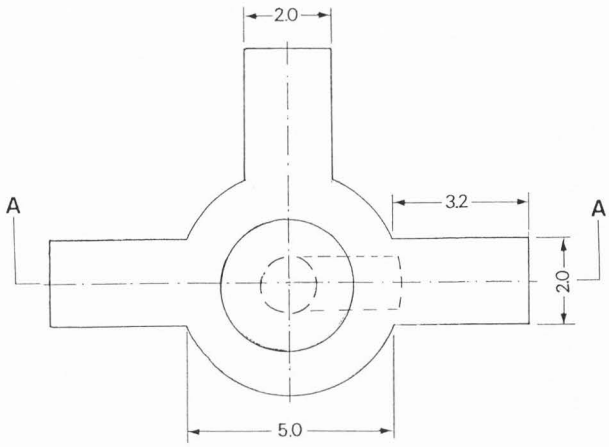
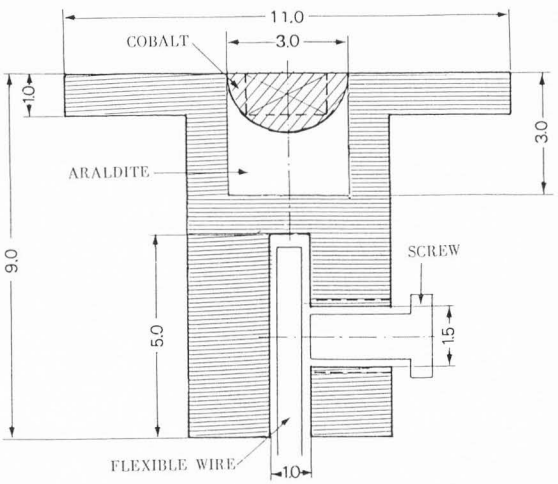


Fig. 1 - Cobalt and specimen holders
The arrows indicate the Allen screws
A - Holder B - Flexible wire
C - Specimen holder D - Cylinder
E - Sample F - Wings G - Cobalt
H - Allen screw I - Tightening screw

The holder consists of an aluminum cylinder and weighs 0.65 grams. (fig. 2). Aluminum is preferred to bronze because of the high intensity of the Cu spectrum and its proximity to Co. The cobalt standard at the top of the cylinder embedded in araldite is surrounded by three "wings" (fig. 1A); These "wings" have a twofold purpose: a) They help to position the cobalt standard at the correct height and angle which have to be similar to those of the sample (both have to be perpendicular to the electron beam). The height of these wings should be as close as possible to 25 mm from the base of the holder. The sample has also to be positioned at the same height. This is done by adjusting the height of the cylinder which holds the sample (Fig. 1B). The whole assembly is brought to an exact focus (working distance of 39mm) with the Z-axis micrometer of the specimen stage. b) They help to position the cobalt during polishing. The small size of the cobalt calibrating element does not allow the use of an automatic polishing machine and polish has to be done by hand. The highly polished cobalt surface is then cleaned with petroleum ether (ultrasound for 10 seconds). Finally a light carbon coating is applied to the holder. The cobalt used for standard should be dense and non-porous.



Plan view



Cross section A-A
Fig. 2 - Drawing of the cobalt holder
(dimensions in mm)

The holder is connected to the sample by a flexible copper wire (fig. 1B) 0.3 to 0.5 mm in diameter. A smaller diameter allows greater flexibility but takes longer to stabilize after moving. The wire length may be varied according to the size of the sample. A 35 mm wire is long enough for most samples, up to those measuring 66 x 66 mm and for a 39 mm working distance. A sample of this size cannot be inserted through the exchange chamber, it requires the opening of the whole stage.

An Allen screw (fig. 1B) clamps the wire in the aluminum body of the holder and a tightening screw (fig. 1A) clamps the wire in a hole drilled through the specimen holder. The frequent changes in the position of the cobalt standard cause stress in the wire, and eventually induce its break. The hole enables the removal of wire remnants after breaking.

A problem that arises is linked to the specimen exchange-rod assembly(2); at the time when samples are exchanged, the high vacuum tends to suck the holder towards the specimen door, thus damaging the cobalt holder. The damage can be avoided by slightly tightening the spring leaf, C24-1 in fig. 3. The tightening holds the holder securely.

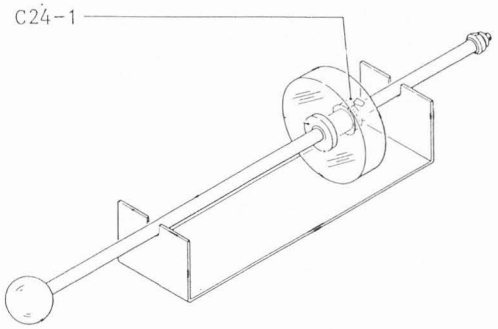


Fig. 3 - Exchange-rod assembly (2).
(Reproduced by permission of JEOL)

Operating Conditions

A first step of the analysis, after the cobalt calibration is to analyse a known sample. The easiest to use is the cobalt itself. The result for 15kV, a working distance of 39 mm, 100 seconds acquisition time and 0 degree tilt was 100.07% (per weight without normalization). The cobalt standard was further used to detect deviations in the analysis with different operating conditions. Tables 1 and 2 summarize the results. Table 3 gives the result of analysis of standard minerals using the cobalt calibration.

Conclusions

The holder for a cobalt calibrating element proposed in this work has been shown to be simple and easy to operate.

It can be seen from table 2 that the working distance is one of the most critical factors in the working conditions in order to obtain good quantitative results. It also shows that a decrease of the working distance is more damaging to the results than a similar increase. On the other hand, at high magnification even a 20 degree tilt of the sample does not make a sensible difference in the results (Table 1).

Table 1
Variations in the Co analysis results with changes of the sample tilt (towards the EDS detector).

Tilt (degrees)	Weight (%)
0	99.4
10	99.3
15	99.0
20	99.1
30	96.3
40	95.4

High Voltage 15 kV; Working distance 39 mm; Acquisition time, 60 seconds; Magnification, x40K.

Table 2
Variations in the Co analysis results with changes in the working distance. (Same working conditions as in table 1).

Working distance (mm)	Weight (%)
36	81.8
37	87.7
38	92.2
39	99.5
40	100.1
41	102.0
42	100.0

High Voltage 15 kV; Tilt 0 deg.; Acquisition time, 60 seconds; Magnification, x40K.

Table 3
EDS analysis of standard minerals against recommended standard values.

Specimen	Element	EDS Wt %	Standard Wt %*	Remarks
Jadeite	Si	27.8	27.71	
	Al	12.9	12.91	
	Na	10.3	10.97	
	Ca	0.1	0.44	
	Fe	0.2	0.18	
	O	46.9	47.57	Stoichiometry
		98.2	99.78	Total
Wolla-stonite	Si	23.5	23.80	
	Ca	34.7	34.30	
	Fe	0.2	0.05	
	O	40.7	41.30	Stoichiometry
		99.1	99.45	Total

High Voltage 15 kV; Working distance 39 mm; Tilt 0 deg.; Acquisition time, 60 seconds; Magnification, x40K.

*Recommended value from Micro Analysis Consultants Ltd. Standard Number 1691.

Acknowledgements

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References

1. JEOL (U.K.) Ltd. (1984). Instructions for JSM-840 scanning microscope. Mechanical parts list. p. 38. JEOL House, Grove Park, Colindale, London, England NW9 0JN.

2. Link Analytical Ltd. (1983). Link Systems. ZAF-4/FLS operating instructions SR2-500-ZAF-1083. Halifax Road, High Wycombe, Bucks, England HP12 3SE.

Discussion with Reviewers

Reviewer 1: How are the problems with tilt and working distance (evident from Tables 1 and 2) overcome with the new holder?
Author:The problems with tilt and working distance are not overcome with the new holder, but with it, it is much easier to position any sample at the correct height. At high magnifications, tilt up to 20 degrees does not create any serious problems (see table 1) with the new holder.

Reviewer 2:Why not make a standard holder from a plastic material to minimize extraneous X-rays?
Author:Plastic material does not conduct electricity and will therefore cause charge to accumulate. The aluminum holder minimizes extraneous X-ray emission (the aluminum X-ray spectra has a low intensity).

Reviewer 2:Where can a suitably pure Co standard be obtained?
Author:Johnson Mathey, Materials Technology, U.K. Orchard Road, Royston, Herts SG8 SHE England, sells cobalt wire of appropriate purity (catalog no. 944151).

Reviewer 2:Why not use a multi-element standard in place of cobalt? The cobalt correction may be different for low and high energy x-rays.
Author:Multi-element standards are used and their profiles and data are already on file. The cobalt is used as a calibrating element and not as a standard. The cobalt measurements allow a correction for small variations in the environmental (e.g. room temperature) and instrumental (e.g. mains supply) conditions.