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Evaluation of Current Standardless Quantitative Analysis Programs using Energy Dispersive Spectrometry in the SEM

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The modern SEM is usually equipped with an Energy Dispersive X-ray detector, which is a convenient tool for performing both qualitative and quantitative chemical analysis. The manufacturers of these detectors provide with them computer programs which can be used to perform both the qualitative and the quantitative analysis automatically, with little input from the user. In the case of qualitative analysis, examination of the x-ray spectrum by a careful user (with a table of x-ray energies for all of the elements) can usually reveal errors made by the computer.

However, quantitative analysis, even on the ideal "flat polished homogeneous" specimen, encounters problems. Obviously, the heights of the elemental peaks on the spectrum are dependent on the concentrations of the elements in the specimen but unfortunately, the relationship between peak height and concentration is not simple due to various factors. These include effects due to Z, atomic number, as well as the possibility of absorption of the x-rays in the sample, which may in turn lead to fluorescence of other constituent elements. If standards (either of similar composition to the unknown specimen or of pure elements of the constituents of the unknown) are employed in the analysis, the quantitative results are usually very reliable. However, the ideal standards may not be easily available, and in any case the procedure is time-consuming. Very popular therefore are the "standardless quantitative analysis" programs which use calculated spectra stored in a "library" in the computer. These programs must include corrections for the factors of atomic number (Z), absorption (A) and fluorescence (F) in the unknown.

It has been demonstrated by Newbury (1999), using a suite of microanalysis standards, that standardless analysis is not as accurate as analysis with standards. At best the precision was reported to be 25%, compared with 2.5% when standards are used. However, new standardless programs have been developed and these need to be evaluated.

The current study is a comparison of two of the proprietary standardless quantitative analysis programs using three different SEMs. The samples used are a set of four standards produced by NIST– homogeneous binary alloys of copper and gold. The study tests the standardless quantitative analysis programs under different conditions of SEM accelerating voltages. For each set of conditions, the weight percentages are calculated by the program using all possible combinations of the peaks, LL, ML, MK and LK.

Tables I and II show the results for accelerating voltages of 30 and 15 kV respectively. At 30 kV, the results are quite accurate when the AuL and the CuK lines are used. However, at 15kV, where the overvoltage for the AuL line is relatively low (1.5), better accuracy is achieved using the AuM line with the CuK line. Generally, use of the lower-energy lines leads to less accurate results, which may be explained by the fact that the correction factors for these lines are not as well established.

Reference

Newbury, Dale E. (1999) Microscopy and Microanalysis, Vol.4, pp585-597

Table I

30 keV

20/80 Au/Cu

	Spot #1	Spot #2	Spot #3	**Average**
AuL CuL	26.15	26.50	26.85	**26.50**
AuM CuL	26.38	27.17	25.78	**26.44**
AuM CuK	20.70	22.22	20.90	**21.27**
AuL CuK	20.10	21.15	20.94	**20.73**

40/60 Au/Cu

	Spot #1	Spot #2	Spot #3	**Average**
AuL CuL	44.94	42.88	43.30	**43.71**
AuM CuL	46.14	45.51	45.25	**45.63**
AuM CuK	43.40	43.17	43.28	**43.28**
AuL CuK	41.64	39.66	40.64	**40.65**

60/40 Au/Cu

	Spot #1	Spot #2	Spot #3	**Average**
AuL CuL	62.63	62.47	63.39	**62.83**
AuM CuL	64.98	63.03	65.83	**64.61**
AuM CuK	63.98	64.06	64.69	**64.24**
AuL CuK	61.00	61.52	61.59	**61.37**

80/20 Au/Cu

	Spot #1	Spot #2	Spot #3	**Average**
AuL CuL	80.12	80.16	79.83	**80.04**
AuM CuL	82.00	81.62	81.52	**81.71**
AuM CuK	82.20	82.48	82.20	**82.29**
AuL CuK	80.09	80.93	80.37	**80.46**

Table II

15 keV

20/80 Au/Cu

	Spot #1	Spot #2	Spot #3	**Average**
AuL CuL	35.04	31.71	29.68	**32.14**
AuM CuL	24.20	24.41	23.25	**23.95**
AuM CuK	19.46	20.53	20.28	**20.09**
AuL CuK	31.47	28.67	27.55	**29.23**

40/60 Au/Cu

	Spot #1	Spot #2	Spot #3	**Average**
AuL CuL	50.65	48.96	49.53	**49.71**
AuM CuL	42.99	41.88	43.12	**42.66**
AuM CuK	41.47	40.85	41.99	**41.44**
AuL CuK	51.05	49.73	50.01	**50.26**

60/40 Au/Cu

	Spot #1	Spot #2	Spot #3	**Average**
AuL CuL	67.59	67.80	68.63	**68.01**
AuM CuL	60.70	60.44	61.42	**60.85**
AuM CuK	61.04	60.46	61.70	**61.07**
AuL CuK	69.38	69.39	70.40	**69.72**

80/20 Au/Cu

	Spot #1	Spot #2	Spot #3	**Average**
AuL CuL	83.19	83.44	83.31	**83.31**
AuM CuL	78.36	78.51	78.43	**78.43**
AuM CuK	79.53	80.24	80.23	**80.00**
AuL CuK	84.79	85.47	85.40	**85.22**

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