X-RADIOMETRIC ASSAYING IN THE MINING OF MOLYBDENUM-WOLFRAM ORES

by

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Translated by

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The possibility of separately determining molybdenum and wolfram in complex ores by X-ray radiometric assaying was set forth in Reference(1), but the proposed method needs improvement. Its chief shortcoming consists of the impossibility of the simultaneous estimation of wolfram and molybdenum percentages in the walls of mines. The use of two analyzers, of different construction and having different detectors and sources for the determination of each element, reduces the output and efficiency of the X-ray radiometric assaying and raises its cost.

The suggested method determines wolfram and molybdenum in ores by use of the L-series characteristic of the X-radiation of wolfram (8.5 keV) and the K-series of molybdenum (17.5 keV); these are simultaneously and efficiently excited by the isotope Cd-109 (22.5 keV) and are detected by a xenon proportional counter.

The spectra shown in Figure 1 are obtained by the use of the cadmium-109 source, activity 5 mCi, on the molybdenum and wolfram ore standards. In traces I and II, the secondary gamma spectra are obtained from the standards of molybdenum(I)- and of wolfram(II)-ores by the cadmium-109 source and the

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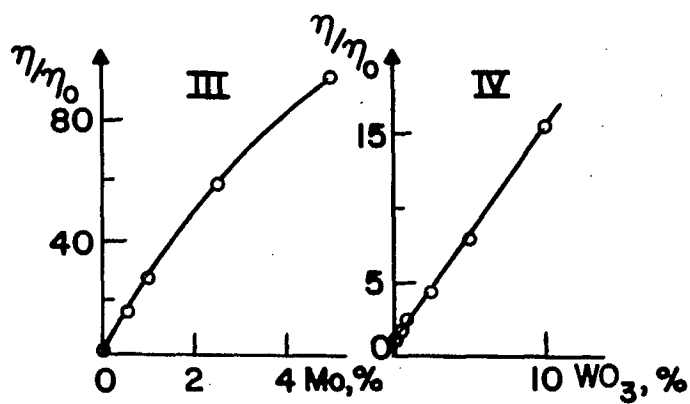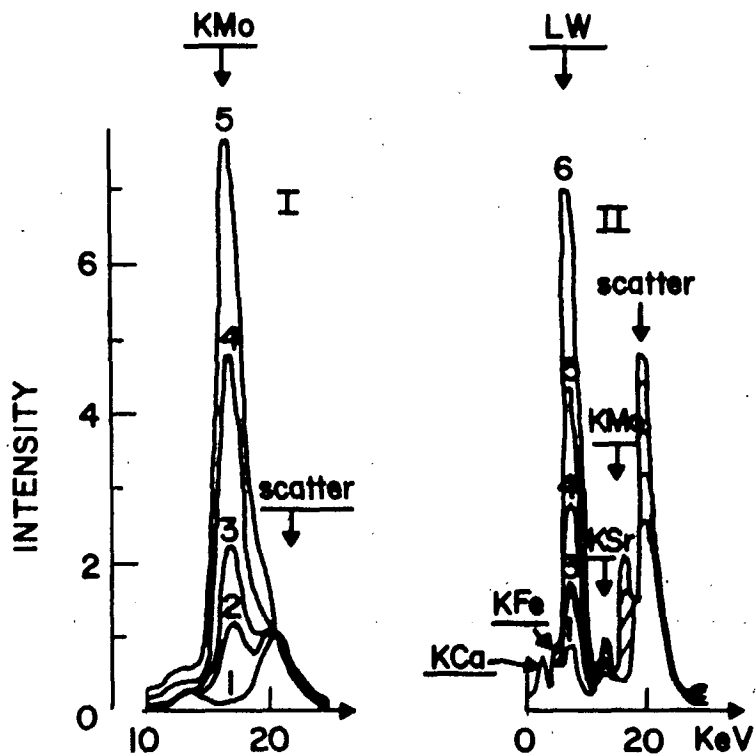   Mines and Resources, Ottawa, Canada.

Figure 1. Concerning the suggested method for the simultaneous determination of molybdenum and wolfram in complex ores by the X-radiometric method.

xenon proportional counter. The percentages of both Mo and WO_3 are: Trace $1 \equiv 0$; $2 \equiv 0.5$; $3 \equiv 1.0$; $4 \equiv 2.5$; $5 \equiv 5.0$; $6 \equiv 10\%$: in III and IV, are the relationships of the spectral values to the percentages of molybdenum(III) and wolfram(IV) in the master standards of the ore.

The recording of the spectra is accomplished in a spectrometer with xenon proportional counters that have 12 to 14% resolution for the 22.5 keV line. The standard ores are prepared from a mixture of gypsum and quartz with addition of molybdenum or wolfram (scheelite) concentrates.

The spectra obtained from the wolfram ore standards are very complex in that, besides the wolfram L-series and scattered source-radiation, the K-lines of molybdenum from the scheelite concentrate and the K-lines of calcium and strontium from the gypsum stand out clearly. Further, the iron K-lines appear in the spectra, and, in the barren-rock standard, also the copper K-lines.

The appearance of these is due to the small concentration of iron and copper entering into the material of the beryllium capsule of the source. The K-series of iron and the L-series of wolfram are clearly resolved by the proportional counter; this makes possible the assay of wolfram and of iron that is always present in the scheelite ores. The copper K-lines are not resolved from the wolfram L-series but, in the molybdenum-wolfram ores, copper is not present in high concentrations. To eliminate the increase in background due to the characteristic copper radiation in the wavelength-range of the wolfram analytical lines, the use of Cd-109 sources in pure aluminum capsules is recommended.

Thus in the secondary spectra the following are clearly separated and resolved by the proportional counters --- the molybdenum K-series, the wolfram L-series, and the single peak of the constant radiation of the source;
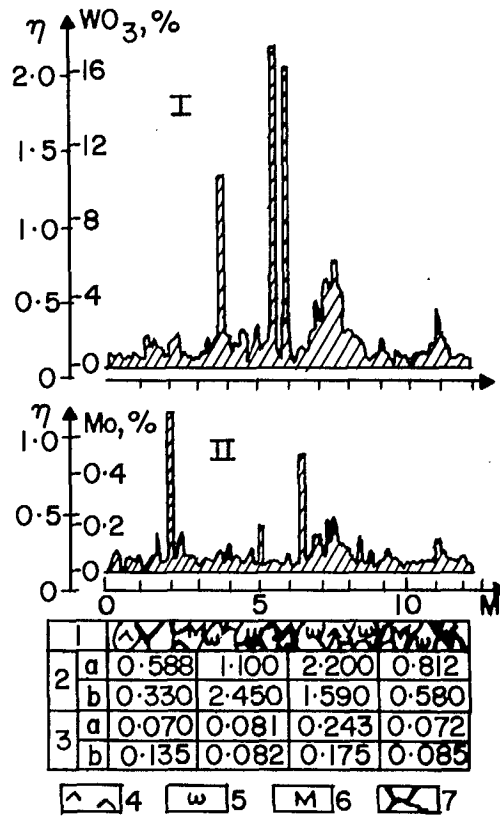
Figure 2. Separate quantitative determination of the percentage of wolfram(I) and molybdenum(II) in a complex skarn ore by X-radiometric assay of the wall of the mine workings:
1- Sketch of the channel; 2- Percentages of WO_3 by assay of the channel samples(a) and by XRM(b)%; 3- Percentages Mo assay of channel samples(a) and XRM(b)%; 4- Skarn; 5- Scheelite; 6- Molybdenite; 7- Quartz veins and veinlets.
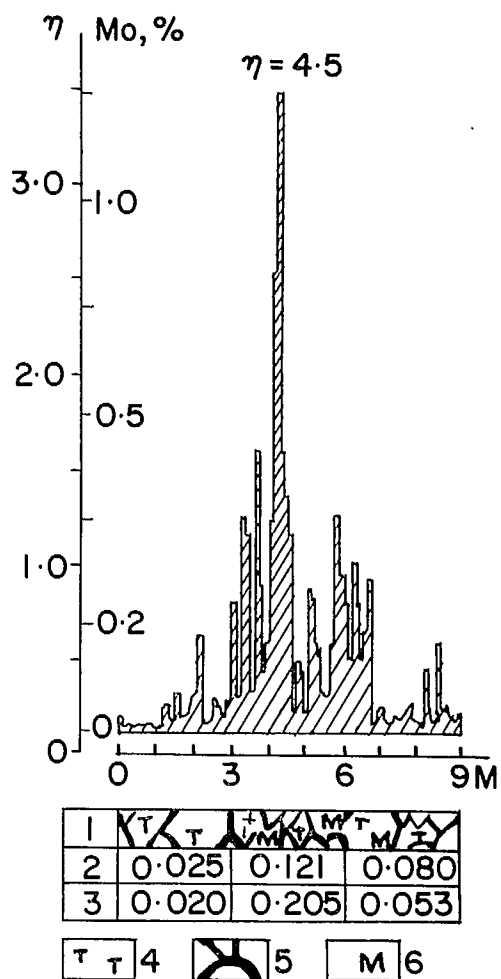
Figure 3. Determination of the percentage of molybdenum in chert ore by X-radiometric assay of the wall of mine workings:
1- Sketch of the channel; 2- Percentage of Mo from the results of channel assays; 3- The same for XRM; 4- Chert; 5- Quartz veins and veinlets; 6- Molybdenite.
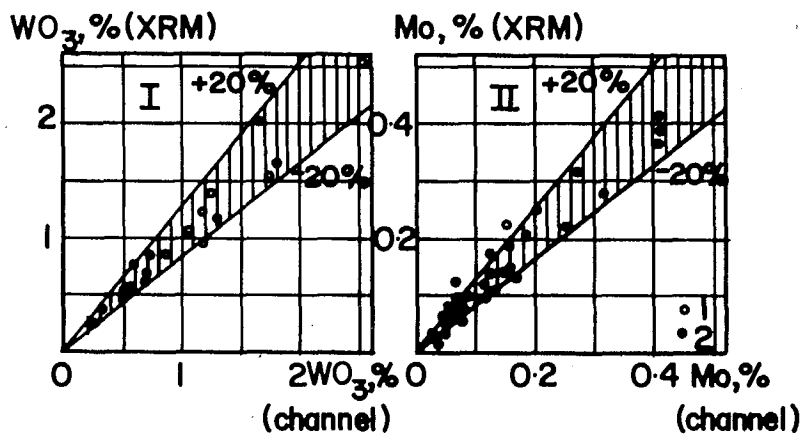
Figure 4. Comparison of the results of the determination of the percentages of WO_3(I) and Mo(II) by assaying channel samples and by XRM: 1- Skarn ore; 2- Chert ore.

these are suited to the X-radiometric method (XRM) and to determinations from spectral ratios.

Thus the single peak of the constant gamma-radiation of the source can be chosen instead of using the background as internal standard. The plot of the relationship of the value of the spectral ratio η (normalised to the value η_0 of the barren rock) to the percentages of wolfram and molybdenum obtained from the standard ores, is shown in Figure 1. From the figure, it is seen that, for wolfram, the nature of this relationship is linear, but for molybdenum, a gradual divergence occurs with increase in concentration. However, for small percentages of molybdenum (0 - 2%) this plot can be approximated as a straight line.

Investigations on standard ores and on powder specimens show that similar methods of measurement provide a threshold of sensitivity for the methods, of percentages of molybdenum from 0.01 - 0.02%, of wolfram 0.05% with a relative precision 20 - 30%.

Experiments on methods were done in the summer of 1970 on one of the deposits of wolfram-molybdenum ore from the North Caucasus. Depending on the site, the host rocks are divided into two basic types of ore: skarn and chert. The skarn ores contain complex scheelite-molybdenite mineralization, whereas the chert has only molybdenite mineralization. Because of their different XRM calibration graphs, the types of ores have to be defined through geological documentation of the mine workings.

The plotting of the calibration graphs is made from the XRM data and from the average assays of channel samples, cut either across or along the ore. These graphs permit, in the interpretation of the materials, automatic allowance for the effect of grain-structure features of the ores on the results of the X-radiometric assaying.

The measurements are made on portions of channel samples at spacings
of 5 or 10 cm. On the skarn ores, wolfram and molybdenum were assayed simul-
taneously. The work was done with a two-channel differential gamma-spectro-
meter developed from the AI-8 instrument and adapted for mine conditions. An
increase in the discrimination by the instrument was achieved by measurement
with a wide solid-angle without any collimation. To eliminate the effects of
mine wall irregularities on the instrument readings, inverted probes were
used: a probe 2 - 5 cm long guaranteed measurement uniformity in the values
of the spectral ratios with an accuracy of 3 - 4% for the variations of
distance between the plane of the instrument and the surface of the area
investigated within a 2-cm range.

Diagrams for XRM assaying of the mine walls and comparisons of XRM
assays for wolfram and molybdenum in skarn and chert ores with the assays of
channel samples are shown in Figures 2 and 3.

The XRM diagrams reflect the nature of the distribution of the min-
eralization profile of the wall surface of the mine-workings: in particular,
they clearly distinguish the quartz-molybdenite veins and the scheelite masses
from the background of lean general dissemination. In some places there are
marked discrepancies between the XRM assays and the assays of channel samples,
but, on the whole, the average assays are the same along and across the ore.

The XRM precision is illustrated in Figure 4 in which the average
XRM assay and the average assay of channel samples are compared across the
ore. It is seen that the XRM assay is within 20% of the assay of channel
samples, assuming the latter results are true. The latter measurements were
made by channel methods, and the discrepancy in the estimate of the percentages
of molybdenum and wolfram in this deposit may exceed 100% between original and
repeat assays, therefore the XRM error is due, for the most part, to the

uneven distribution of the mineralization. The XRM results permit the assumption that, for representativeness and precision, they are as good as the assays of channel samples.

The XRM method separately assays wolfram and molybdenum in complex ores with sensitivity and precision, suited to mine assay requirements, and may be recommended for use in mines. With a single instrument and with simultaneous determination of both elements, the XRM output can average 10 assays per hour, whereas the maximum output of a man cutting channel samples is only six samples per shift.

Translator's note: The author should have given the length, width, and depth of channelling (a difficult cut even by pneumatic tool). Or does he mean that, by wet method, a chemist can do only six assays a shift?

LITURATURE

1. E.P. Leman, V.V. Kotel'nikov, N.G. Bolotova and A.P. Ochkur. "X-radiometric assay of molybdenum-wolfram ores in underground drillings and mine-workings". Survey and protection of mineral resources No. 11, (1970).

2. V.A. Meyer, A.P. Ochkur, Yu.I. Kudryartsev, E.P. Leman, A.K. Bolshakov and V.C. Nakhabtsev. "Method of spectral ratios in the X-radiometric investigation of the elemental composition of ores under natural conditions". Problems in Ore Geophysics, No. 6L, Edition LGY, 1965.

Submitted by the professor of geochemical and radioactive research methods, L.G.I. to the editorial office February 1, 1971.