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Preliminary Testing of a Prototype Portable X-Ray Fluorescence Spectrometer

**By Lowell L. Patten, Neal B. Anderson,
and John J. Stevenson**

UNITED STATES DEPARTMENT OF THE INTERIOR

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CONTENTS

Page

Abstract.......... 1
Introduction.......... 2
  History.......... 2
  Previous Bureau of Mines work.......... 3
Acknowledgments.......... 3
X-ray theory and its application to the portable spectrometer.......... 3
Instrument design and description.......... 4
Analytical procedures and results.......... 6
  Laboratory testing.......... 7
    Interference.......... 9
    Matrix effect.......... 11
    Summary of results.......... 11
  Field testing.......... 12
Conclusions.......... 14
Recommendations.......... 15
Selected bibliography.......... 15
Appendix A.--Selected sections of PXRFS operation and maintenance manual.......... 17
Appendix B.--Tables of analytical results.......... 24
Appendix C.--Nuclear Regulatory Commission (NRC) licensing information.......... 33

ILLUSTRATIONS

1. Characteristics of X-ray fluorescence spectra.......... 4
2. Portable X-ray fluorescence spectrometer (PXRFS).......... 5
3. PXRFS as used in the field.......... 6
4. Periodic table of elements.......... 8
5. Sensor-head rack and pulverized sample in petri dish.......... 9
6. Spectra (CRT traces) of manganese ore and molybdenite.......... 10
7. Spectra (CRT traces) of erythrite and mercury ore.......... 10
8. Spectra (CRT traces) of samples containing iron, copper, calcium, and galena.......... 10
9. Spectra (CRT traces) of samples containing lead, zinc, and ferberite.......... 10
10. Spectra (CRT traces) illustrating interelement interference.......... 11
11. Calibration curves of five element standards having granitic matrixes.......... 12
12. High-impact plastic transport case for spectrometer.......... 13
13. Spectrometer in use on a mine dump.......... 13
14. Calibration spectrum of ^{109}Cd source under normal operating temperature and at 108° F.......... 14
A-1. Front panel and controls of PXRFS.......... 17

TABLES

B-1. X-ray excitation capabilities of ^{55}Fe and ^{109}Cd for selected elements.......... 24
B-2. Comparison of minus 200-mesh pulp sample analysis data with PXRFS test results.......... 25
B-3. Comparison of USGS sample standards with PXRFS test results.......... 31
B-4. PXRFS analysis of selected Bureau of Mines mineral display specimens.......... 32

PRELIMINARY TESTING OF A PROTOTYPE PORTABLE X-RAY FLUORESCENCE SPECTROMETER

By Lowell L. Patten,[1] Neal B. Anderson,[2] and John J. Stevenson[2]

ABSTRACT

The Federal Bureau of Mines participated with the National Aeronautics and Space Administration and Martin Marietta Aerospace in developing, building, and testing a portable X-ray fluorescence spectrometer for use as an analyzer in mineral-resource investigative work. The prototype battery-powered spectrometer, measuring 11 by 12 by 5 inches and weighing only about 15 pounds, was designed specifically for field use. The spectrometer has two gas-proportional counters and two radioactive sources, ^{109}Cd and ^{55}Fe. Preliminary field and laboratory tests on rock specimens and rock pulps have demonstrated the capability of the spectrometer to detect 33 elements, to date. Characteristics of the system present some limitations, however, and further improvements are recommended.

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INTRODUCTION

The principles of X-ray fluorescence spectrometry long have been known and studied intensively by numerous investigators (see bibliography). Laboratory model spectrometers have been available commercially for many years. The feasibility of a portable X-ray fluorescence spectrometer (PXRFS) was investigated in the 1960's, and several companies developed portable analyzers, some using X-ray generators and others using radioactive isotope sources. In general, however, these analyzers were heavy, cumbersome, complex to use, and lacking in versatility. Complications included the need to cool the X-ray generators, the use of filters to increase discrimination of the detectors, and the fact that most of these analyzers would detect only one or no more than a few elements at one time.

An improved portable spectrometer would greatly facilitate the Federal Bureau of Mines mineral-land assessment work, would substantially assist mineral exploration work in general, and could benefit both Government and industry as an aid in mineral identification and analysis.

This report summarizes results of a twofold project to apply principles of X-ray fluorescence spectrometry to mineral identification and element quantification. The first part of the project was to construct a portable, energy dispersive, X-ray fluorescence spectrometer. The second part was to test the prototype instrument in laboratory and field situations to determine its operating characteristics, its response, and its usefulness in mineral-resource investigative work.

Instrument response was observed and recorded in the Planetary Geology Laboratory of Martin Marietta Aerospace near Denver, Colo., in the Bureau's Intermountain Field Operations Center at the Denver (Colo.) Federal Center, in the mountains west of Denver, and near Tucson, Ariz.

During testing, a limited effort was made to compare the PXRFS capabilities with those of emission spectrometers. The PXRFS can be used in conjunction with, but not as a substitute for, a laboratory emission spectrometer.

History

The idea of a spectrometer that could be carried easily in the field and could quickly identify and quantify many elements with little or no sample preparation only recently became technologically possible.

In the 1970's, Martin Marietta (MM), under contract with National Aeronautic and Space Administration (NASA), developed a miniature X-ray fluorescence spectrometer, which employed ^{55}Fe and ^{109}Cd isotopes as X-ray sources, for use on the Viking Project Mars Lander.

The senior author of this report suggested that the Mars Lander technology might be followed in developing a portable spectrometer that could be useful as a field instrument in mineral exploration. Subsequently, Bureau personnel consulted with representatives of MM and NASA, and in 1975 a contract was negotiated for developing and constructing a portable X-ray fluorescence spectrometer. The work was funded mostly by the Bureau and it also provided a list of elements of interest in mineral exploration. Suggestions for design to facilitate field and laboratory use were contributed by both the Bureau and NASA.

The PXRFS was completed in mid-1980; subsequently, several minor adjustments and microprocessor program modifications were made. A testing program began in late 1980 and continued intermittently until the spring of 1981. A Nuclear Regulatory Commission (NRC) licensing (see appendix C for license requirements) problem delayed transfer of the spectrometer custody from the contractor to the

Bureau until July 1981 when the Bureau's source license was modified to include the PXRFS and its radioactive isotopes.

Previous Bureau of Mines Work

The Bureau of Mines has done considerable work in laboratory and field applications of X-ray fluorescence as shown by citations in the bibliography. Most of this research advanced the general technology but was directed mainly toward special applications, and, in particular, toward high quantitative accuracy in the laboratory.

ACKNOWLEDGMENTS

Those who technically contributed the most to the current project were Benton C. Clark, senior research scientist, and Ludwig Wolfert, staff engineer, both of Martin Marietta. Also instrumental in the development of the PXRFS were Warren C. Kelliher, contract manager, and Charles R. Eastwood, manager, environmental projects, both from NASA, and Sheldon P. Wimpfen, chief mining engineer, and Lee R. Rice, geologist, of the Bureau of Mines.

X-RAY THEORY AND ITS APPLICATION TO THE PORTABLE SPECTROMETER

X-rays can be generated by electrical apparatus or originate as gamma rays during the decay of radioactive isotopes of elements. The origin of characteristic X-ray fluorescence can briefly be described as follows: When sufficient energy is introduced into the atom, by X-rays or gamma rays, an electron is displaced from one of the inner shells. The atom is then in an excited (ionized) state. The place of the missing electron is filled immediately by an electron from a neighboring outer shell whose place, in turn, is filled by an electron from the next outer shell. The electron from a high energy level (outer shell) enters a lower energy level (inner shell), emitting excess energy in the form of X-ray fluorescence.

Each element has a characteristic emission energy for each electron shell, referred to as K, L, and M spectra (K-alpha, K-beta, etc.), indicating from which electron shell the fluorescence originates. Figure 1 illustrates these phenomena by showing the characteristic K, L, and M spectra and their corresponding energies (in kiloelectron volts, keV).

An X-ray fluorescence spectrometer, then, is used to record these characteristic spectra and their energies, thereby identifying and possibly quantifying the elements. Four functions, therefore, were required in the PXRFS to accomplish this: (1) generate X-rays, (2) transform fluorescent energies into electrical impulses, (3) process, interpret, and store these electrical impulses, and (4) display the interpreted data in a form the operator could use.

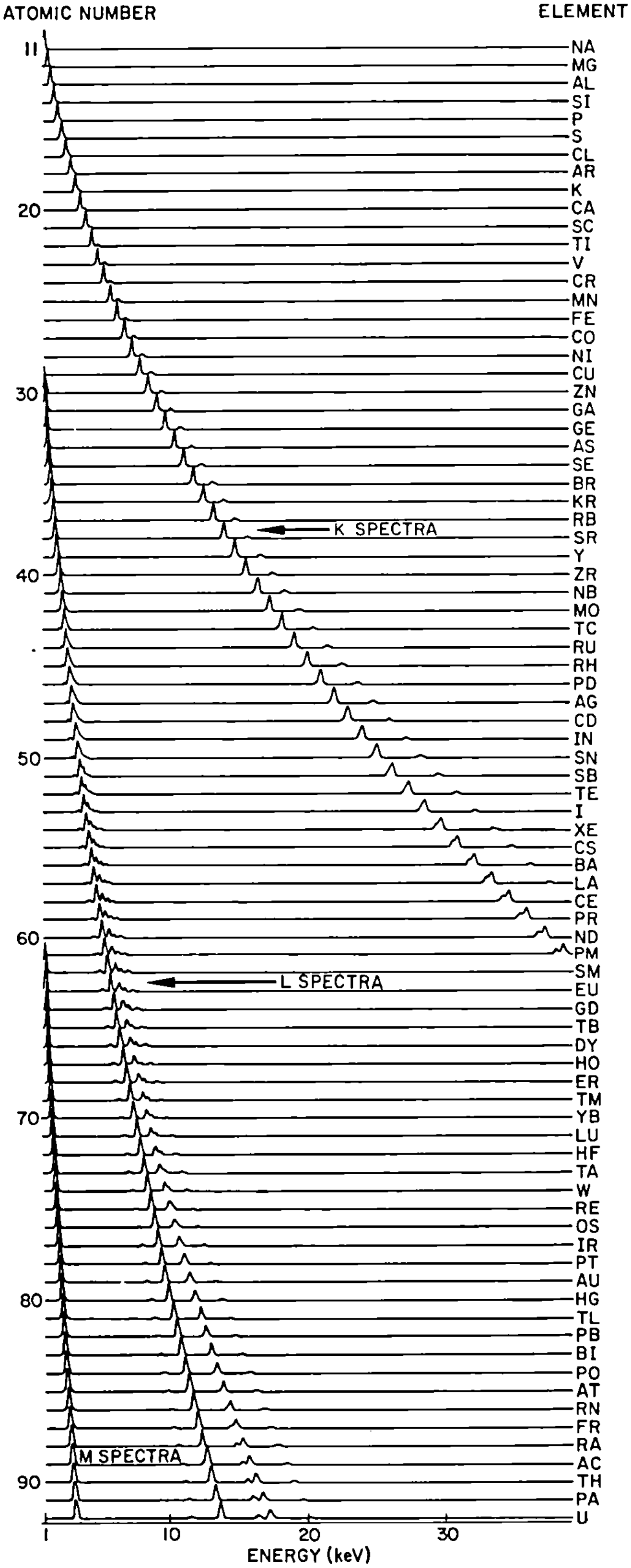

FIGURE 1. - Characteristics of X-ray fluorescence spectra. (Copyright, ASTM, 1916 Race Street, Philadelphia, Pa. 19103. Reprinted/adapted, with permission.)

INSTRUMENT DESIGN AND DESCRIPTION

Design objectives were to obtain an optimum combination of available technology and commercially available components, considering the factors of weight, size, operator training, speed of analysis, accuracy, versatility, and durability under field conditions. Within this philosophy, the optimum spectrometer design became a unit containing two X-ray sources (radioisotopes), in order to excite X-ray response from a wide range of elements, coupled with gas-proportional counters (detectors) that would feed electrical impulses (transformed fluorescent energies) to a microprocessor for processing, interpretation, and storage in memory; in turn, the microprocessor would feed data to a cathode ray tube (CRT) display and a liquid crystal display (LCD).

The PXRFS was designed for operators trained in mineral identification but lacking extensive technical knowledge of spectroscopy. The operator needs considerable experience in using the PXRFS before he/she fully understands the displays.

The PXRFS (fig. 2) consists of an analyzer unit, including a microprocessor, solid-state circuitry, batteries (power source ranges from 7 to 40 volts DC) and a control panel with a 1.3- by 1-inch CRT display, a 0.75- by 3.75-inch alphanumeric LCD, and a variety of switches and dials (described in appendix A), all connected to a sensor head by a 6-foot, spring-coiled cable. The sensor head contains two radioisotopes, 100 millicuries (mCi) of ^{55}Fe having a calibration target composed of sulfur and titanium, and 15 mCi of ^{109}Cd having a calibration target composed of titanium and zirconium, two collimators, and two gas-proportional counters. When its cable is unplugged, the sensor head can be stored in the lid of the analyzer unit.

Dimensions of the PXRFS, lid closed, are about 11 by 12 by 5 inches (sensor head is 10.5 by 2.5 by 1.5 inches). The complete spectrometer weighs about

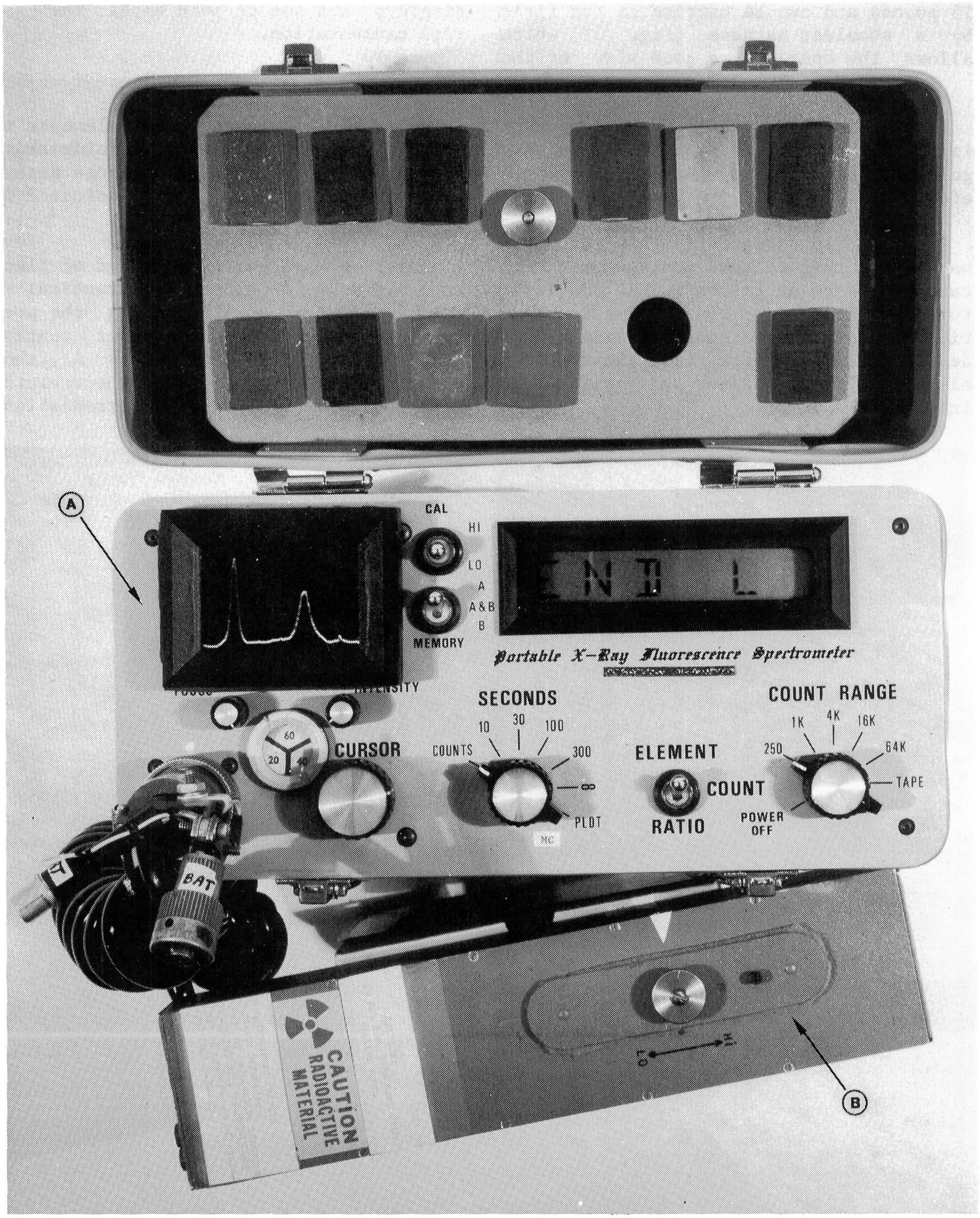

FIGURE 2. - Portable X-ray fluorescence spectrometer (PXRFS). A, Main analyzer unit; B, sensor head.

15 pounds and can be carried in the field by a shoulder harness (fig. 3) which allows the operator a good view of the displays and use of both hands for control manipulation.

ANALYTICAL PROCEDURES AND RESULTS

Information gathered during the tests is preliminary and is presented here as a guide to prospective portable spectrometer users.

Previous investigators (bibliography) have shown that factors having a significant influence on response include moisture, particle size, the geometric relationship between the source, sample, and detector, and effects from surrounding elements ("matrix effect and interelement interference").

The useful response from elements in certain combinations varied considerably; consequently, the success of these determinations varied widely, as indicated in table B-1.

Analytical procedure consisted of placing a sample as close as practical to the sensor-head port, opening the port by sliding the port-shutter control knob (as described in appendix A), thus irradiating the sample; then the operator recorded the lapsed time of irradiation,

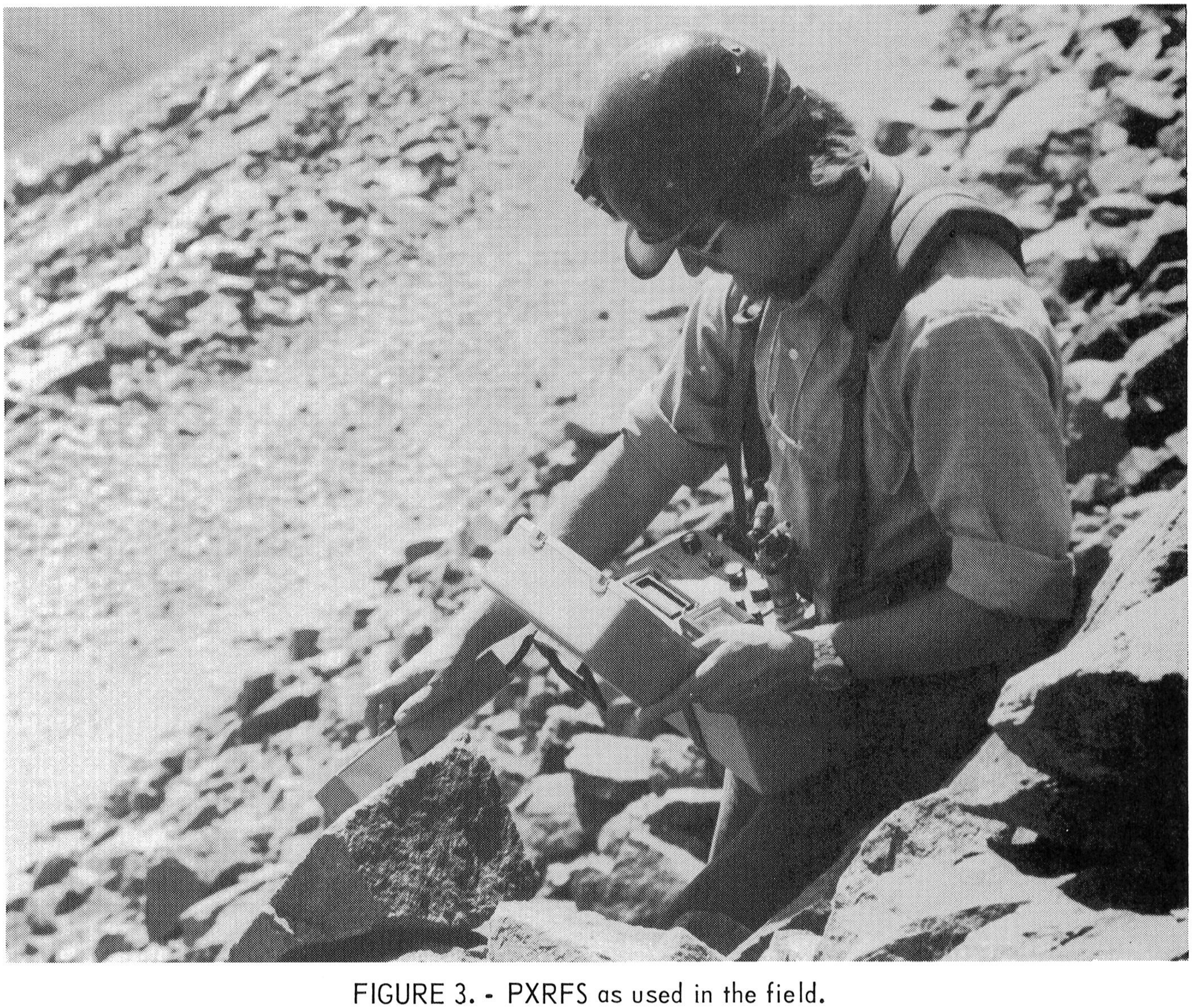

FIGURE 3. - PXRFS as used in the field.

total counts, elements detected, and the ratio values. The operator determines the length of analysis time by either presetting the time on the control panel or releasing the port-shutter control knob when a significant spectrum is displayed on the CRT. The spectrum is then studied and sometimes photographed by the operator.

The ratio value is derived by the microprocessor, and is a ratio between the fluorescent intensity (counts) of the element being measured and the backscatter (BS) peak intensity, a process intended to compensate automatically for sample grain size and the geometry of the source, sample, and detector. The BS peak is created by radioisotope X-rays that escape the fluorescence process, that is, reflected X-rays recorded by the detector and displayed as a peak at the high-energy end of the spectrum.

Laboratory Testing

Theoretically, 73 elements can be detected by the PXRFS tested by the Bureau (fig. 4A). However, as of this writing, only 33 elements have been detected, as shown in figure 4B. This is attributable in part to a lack of specimens or standards that contain the other elements. Moreover, the precious metals were not detected, owing partly to the lack of spectrometer sensitivity to low concentrations of precious metals normally found in natural occurrences and partly because the ^{109}Cd isotope decays to silver and does not excite fluorescence from silver atoms.

The PXRFS response to fluorescence (secondary X-ray emissions) of elements was investigated on 52 samples including 7 control standards loaned by Harlan Barton of the U.S. Geological Survey (USGS), Denver, Colo., 13 Bureau mineral display specimens, and 32 pulverized samples previously analyzed for a Bureau Indian-lands project (see tables B-2 through B-4).

Pulverized samples were poured into plastic 35-mm-diameter by 10-mm-deep petri dishes. Each dish was filled to heaping, tamped lightly for compaction, and leveled with a knife or straightedge, which provided a smooth sample surface for X-ray analysis. The petri dish was placed in a sample receptable in the base of a sensor-head rack (fig. 5), which has corner uprights designed to support the sensor head at an optimum analysis distance and position over the pulverized sample.

Pulverized samples in standard kraft paper envelopes were analyzed without removal from the envelopes. The response from the ^{55}Fe source was not usable because the envelope blocked the secondary, or perhaps, the primary X-rays; response from the ^{109}Cd source appeared normal. This rapid method of analysis may effectively identify the composition of pulverized samples without removing them from the envelopes.

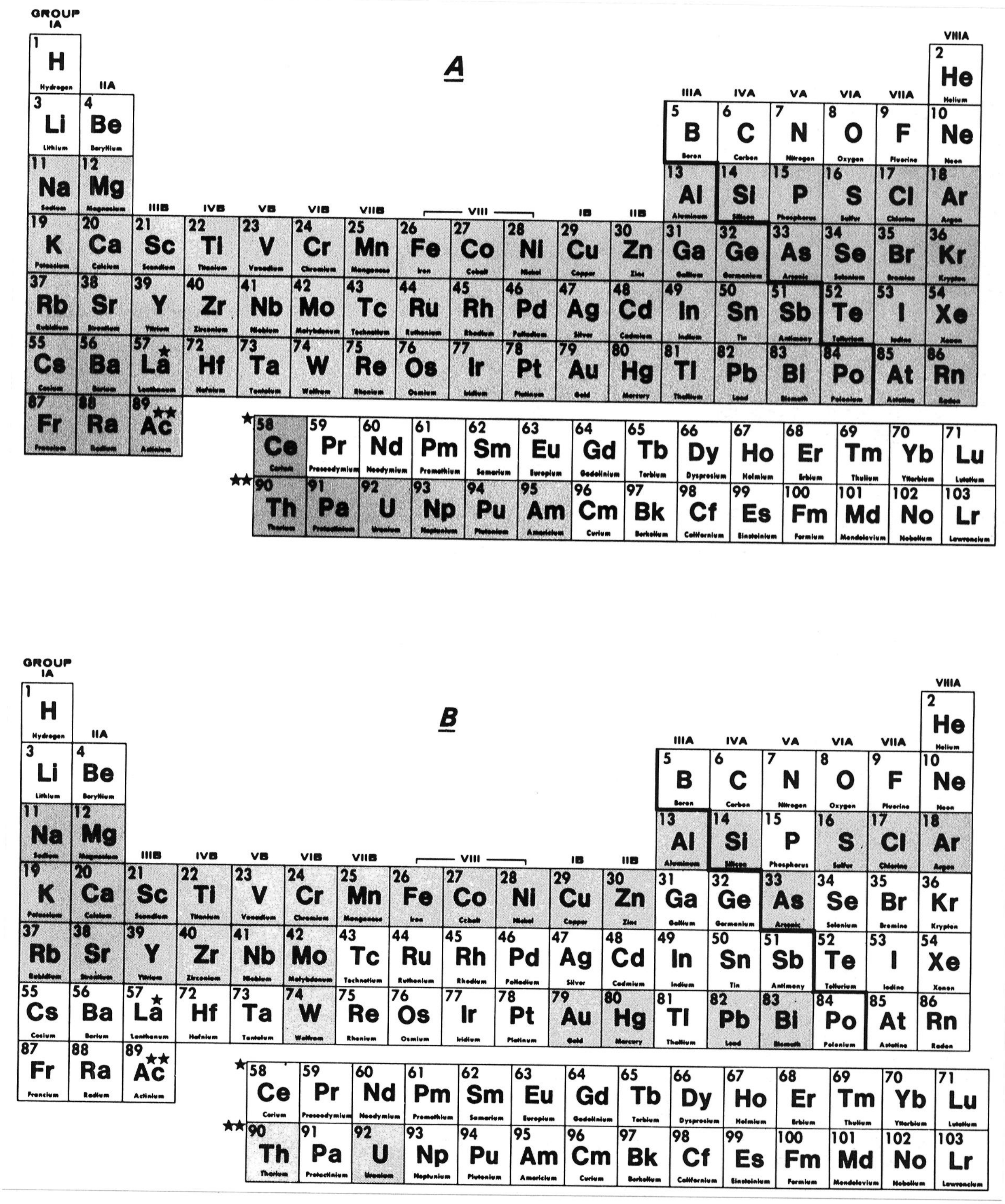

FIGURE 4. - Periodic table of elements. Shaded areas show *A*, elements theoretically detectable by PXRFS; *B*, elements actually detected in Bureau of Mines tests.

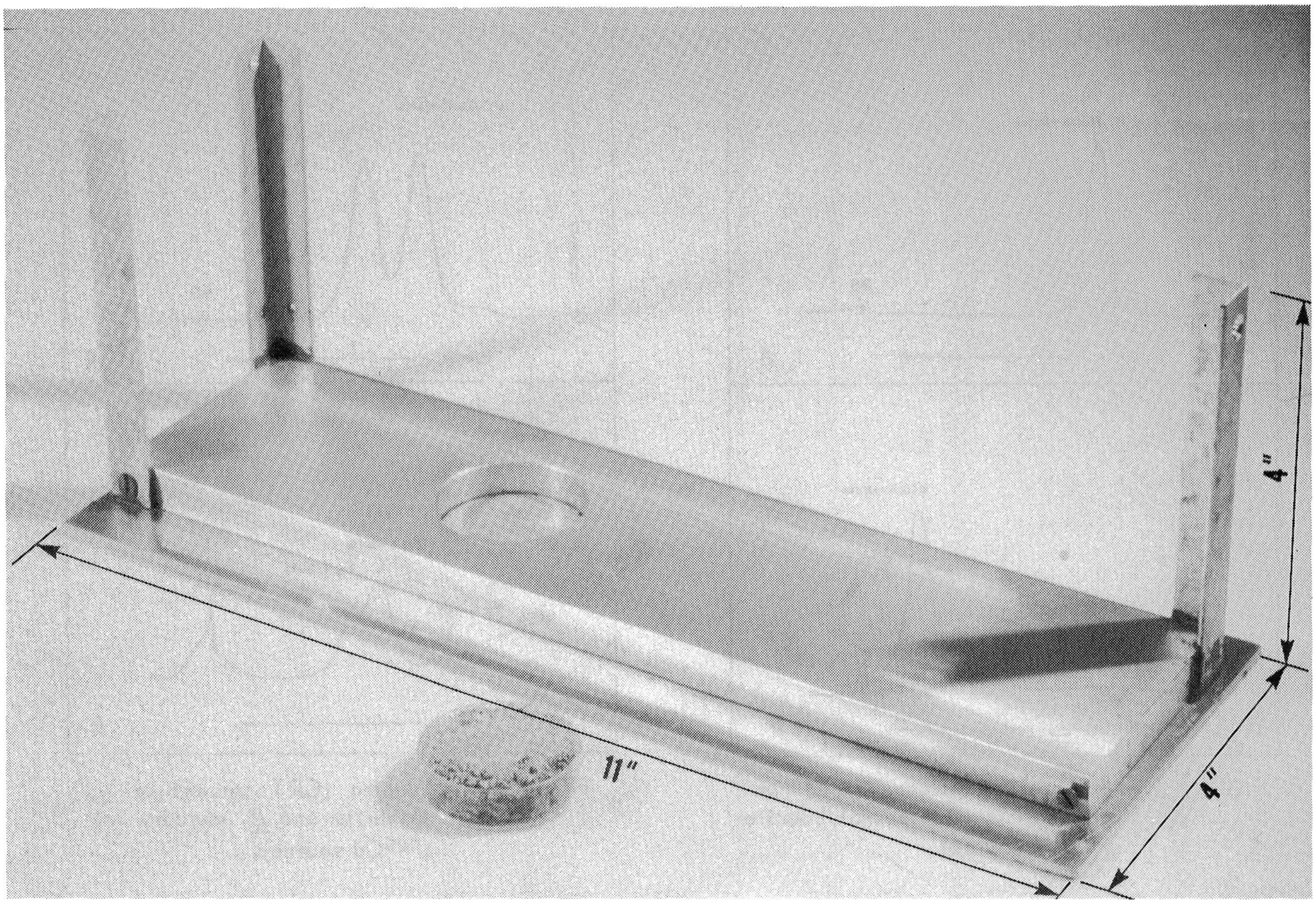

FIGURE 5. - Sensor-head rack and pulverized sample in petri dish.

Figures 6 through 9 are spectral traces from photographs of the CRT display. Each figure shows the response of certain elements or minerals. These spectral traces illustrate the fluorescent intensity (counts) plotted in relation to the element emission energies as interpreted by the PXRFS microprocessor. Shown are the element X-ray emission peaks and their spectral line classifications and the position of element emissions relative to backscatter emissions.

Interference

Because X-ray emission energies (kiloelectron volts) of many elements (fig. 1) are close in value, and because the PXRFS uses a proportional counter as a detector, fine resolution of the element emission energies was difficult. The problem was interference between elements, recorded as distortions of spectra.

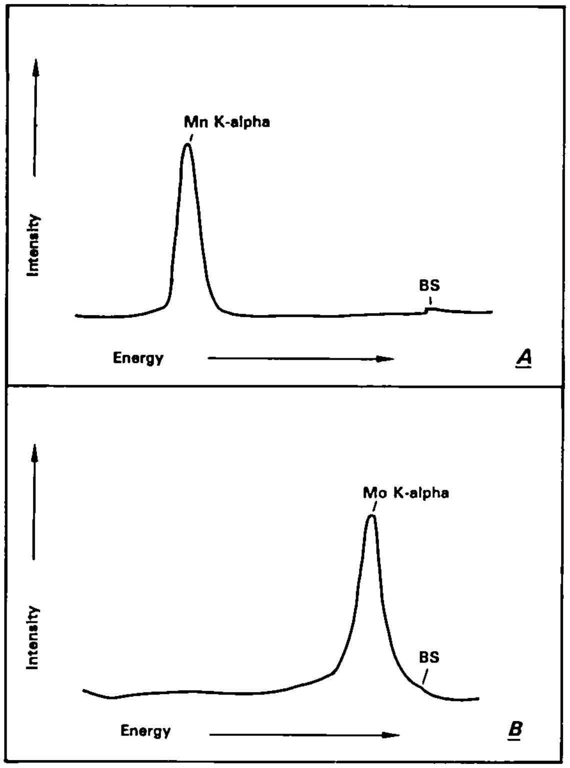

FIGURE 6. - Spectra (CRT traces) of *A*, manganese ore and *B*, molybdenite (^{109}Cd source).

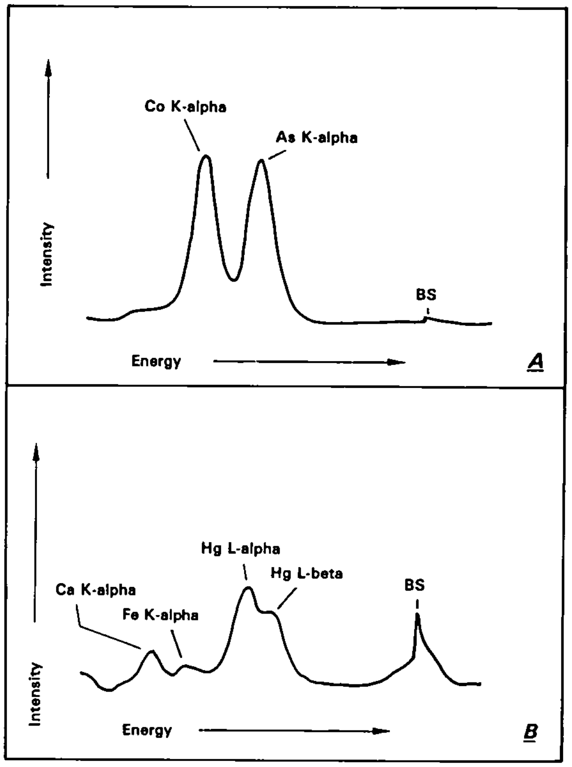

FIGURE 7. - Spectra (CRT traces) of *A*, erythrite and *B*, mercury ore (^{109}Cd source).

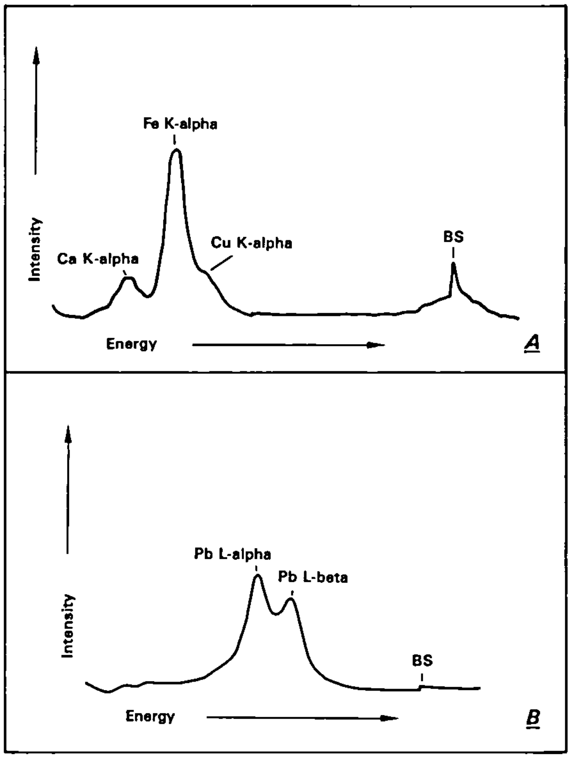

FIGURE 8. - Spectra (CRT traces) of *A*, sample containing iron, copper, and calcium, and *B*, galena (^{109}Cd source).

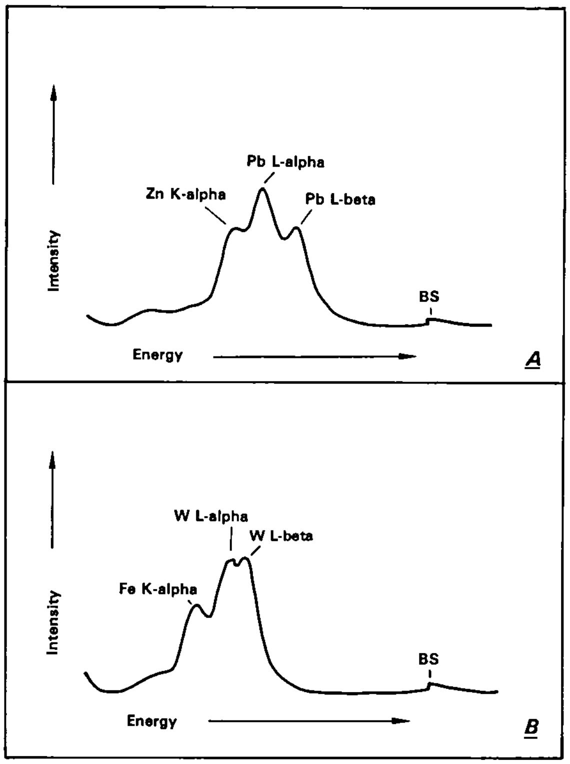

FIGURE 9. - Spectra (CRT traces) of *A*, sample containing lead and zinc and *B*, ferberite (^{109}Cd source).

Figure 10A is a spectral trace from a pulverized sample of a quartz vein in granite, which chemical analysis indicated to contain 23.7% As, 5.2% Cu, and 3.05% Pb. This spectral trace illustrates the interference created when copper, iron, and arsenic occur together. The copper emissions were interpreted as having an additive effect to the area of the spectrum between the iron and arsenic emission peaks. Also, the arsenic emissions appear to mask the lead responses; however, lead emissions have an additive effect on the arsenic emission peak. Figure 10B, a spectral trace of a pulverized sample of galena in an igneous rock, which contains 1.32% Pb and 1.95% Zn, illustrates further the interference problem by showing the distortion of the predominant iron emission peak by the lead and zinc emissions.

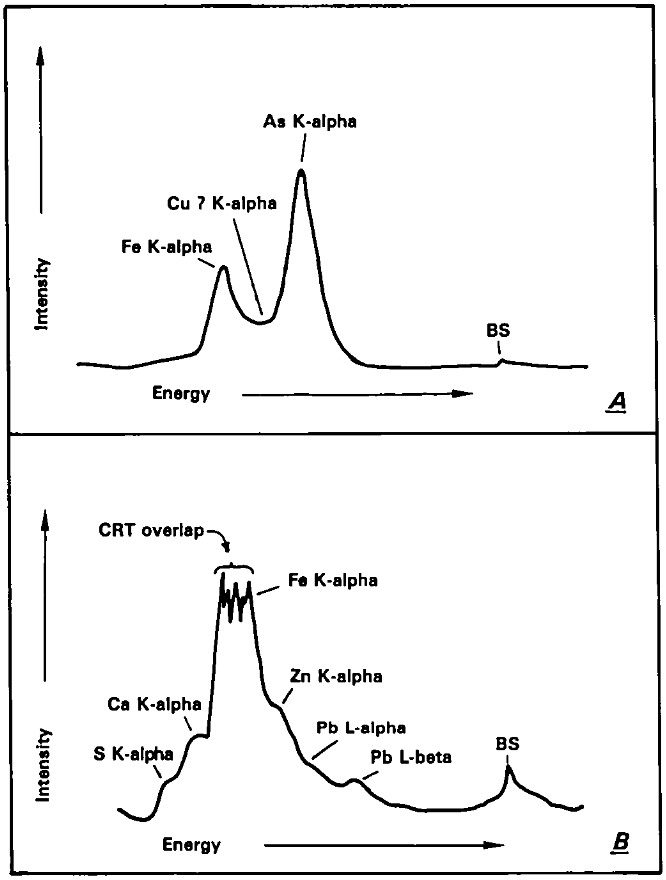

FIGURE 10. - Spectra (CRT traces) illustrating interelement interference. *A*, Sample containing As, Cu, Pb, and Fe; *B*, high-Fe sample containing Zn and Pb (^{109}Cd source).

Iron was the most significant source of interference, owing to its natural abundance and strong X-ray emission response. Many elements of economic interest occur with iron; because iron interfered with the X-ray responses of these elements, iron interference became the major problem. In tests on certain clays (ceramic raw materials) even minor amounts of iron impurities were detected; the PXRFS could be used for the selection of low-iron clays for industrial purposes.

With experience, the PXRFS operator was able to recognize characteristic emission peaks of elements even though the peaks were modified by interference.

Matrix Effect

The matrix effect is a form of interference simply defined as the effect matrix elements have on the X-ray response of other elements. Matrix effect is due to an interaction between the emission and absorption characteristics of the matrix elements and the emission energies (or wavelengths) of the elements being analyzed.

Rock-forming or gangue minerals (matrix) can have a significant effect on the X-ray responses from elements of possible economic interest. If rock composition changes, the matrix effect also changes. That is, as the relative proportion of matrix elements changes, their effect on economic elements changes in the form of an increase or decrease in secondary radiations or emission absorptions.

Summary of Results

Positive identification and semiquantitative analyses of elements were possible for many of the samples tested. The limitations created by the detection system used in the PXRFS, however, precluded utilization of the instrument's full potential.

Element identification was possible for most of the elements tested and was affected only by the presence of interfering elements for element concentrations less than 0.10%.

Quantitative analyses were hindered by both interelement interference and matrix effect, the matrix effect especially affecting the results when element concentrations were low (generally less than 0.10%). Semiquantitative results probably can be obtained by use of the PXRFS, but it will be necessary to use samples (standards) that have known but varying percentages of element concentration for comparison with samples of unknown composition. Five element calibration curves (fig. 11), constructed from USGS standardized samples, are plots of element concentrations relative to the ratio values calculated by the PXRFS. Quantification of elements in a sample of unknown composition can be estimated by using calibration curves if the composition of the sample approximates that of the standards used to make the calibration curves.

The overall quantification results, based on spectral emission data as compared to analyses of the samples, varied from useful to nearly useless, depending on the relative percentages of interfering elements as well as the X-ray response characteristics of a particular element and those elements adjacent to it in the spectrum.

Field Testing

On short field trips to selected mining districts west of Denver, Colo., and on a field trip to southwestern Arizona, the PXRFS was tested for its durability and its practicability in addition to its analytical accuracy and response capabilities.

The PXRFS was transported in a four-wheel-drive vehicle to selected test sites over varying types of roads. During transportation, the instrument was carried in a high-impact plastic case (fig. 12).

Hand specimens collected from outcrops or mine dumps and outcrop faces were analyzed during the field tests (fig. 13). Some hand specimens and outcrop faces were difficult to analyze with the PXRFS because of their surface irregularities. One-handed operation of the sensor head during field tests while climbing on rock outcrops proved to be difficult because of the sensor head shape and the tension on the port shutter control return spring.

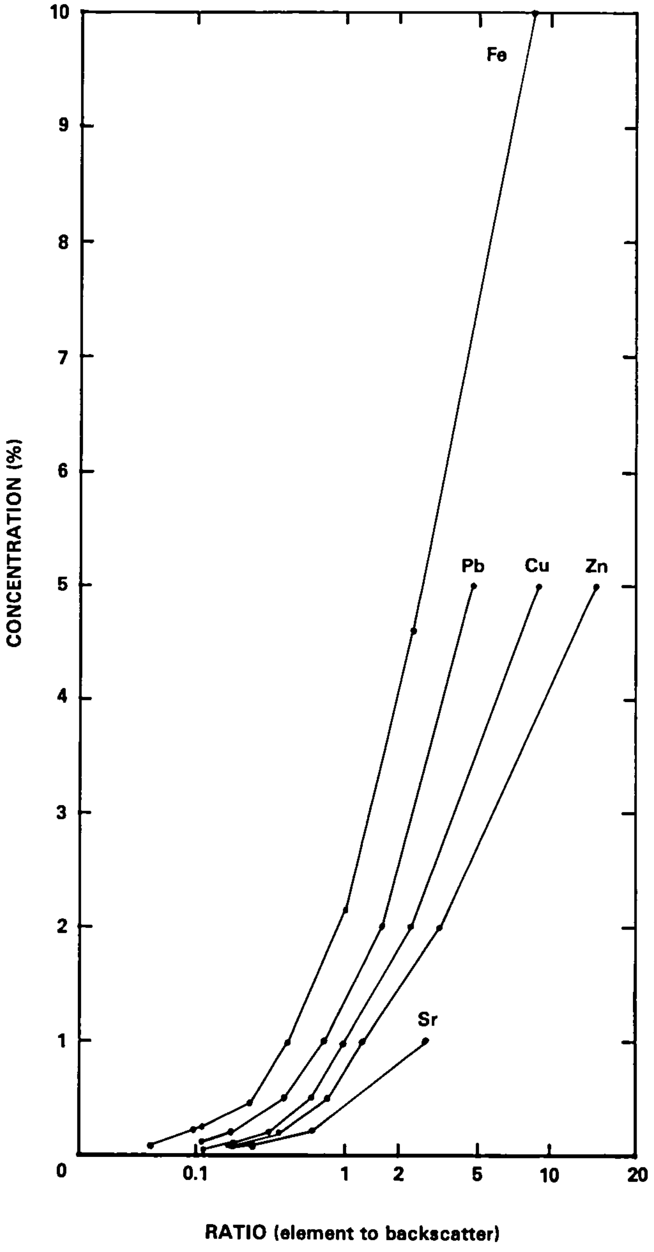

FIGURE 11. - Calibration curves of five element standards having granitic matrixes.

FIGURE 12. - High-impact plastic transport case for spectrometer.

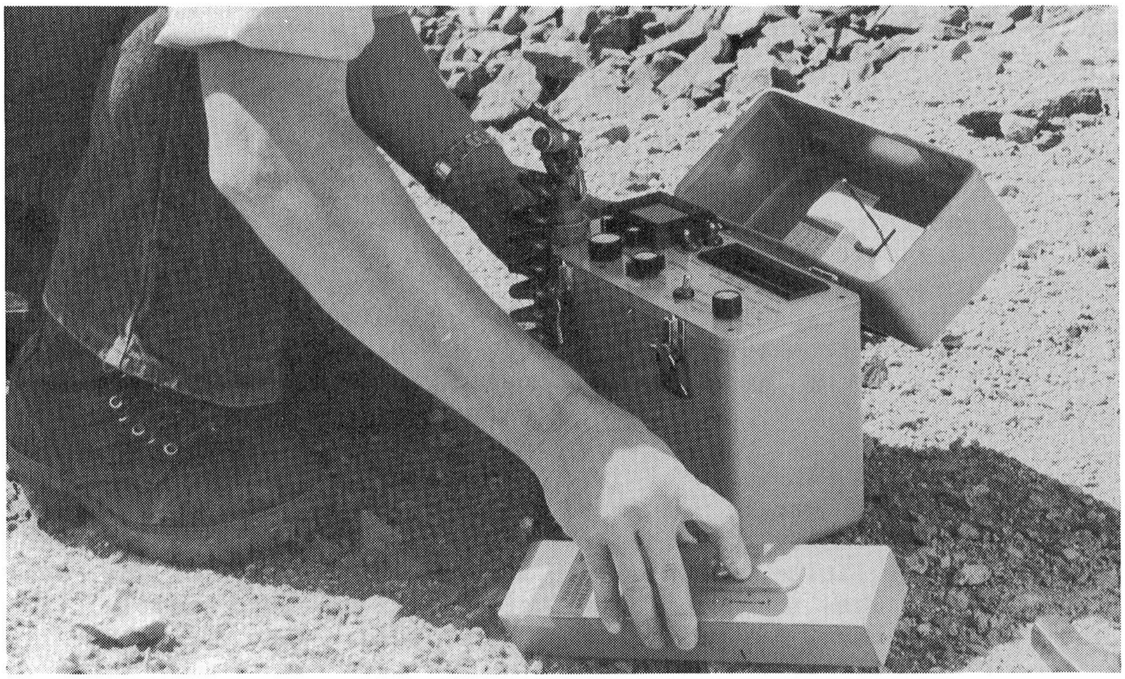

FIGURE 13. - Spectrometer in use on a mine dump.

The PXRFS response was compared with visual, physical, and chemical mineral-identification techniques; siderite, sphalerite, ferberite, and powellite were among the minerals identified during the field tests.

The tests disclosed that air temperatures between 50° and 90° F had little effect on the PXRFS; however, the instrument did undergo a spectral shift toward the low-energy end of the spectrum at 108° F. Figure 14*A* shows the ^{109}Cd calibration spectrum (trace) produced by the PXRFS within the operating temperature range; figure 14*B* illustrates the spectral shift that occurs to the same calibration spectrum at 108° F. At air temperatures less than 50° F, the spectrum appeared to shift slightly toward the high-energy end of the spectrum. The spectral shift phenomenon made element identification difficult and necessitated calibration adjustments in the sensor head that were time consuming and not always successful.

Durability of the PXRFS was somewhat less than expected. A fuse holder attached to the chassis broke loose; wires in the sensor head broke twice from flexure; a beryllium window in the ^{109}Cd-coupled detector shattered, which made the detector and the PXRFS inoperable; and the analyzer unit lid hinges were sprung from the weight of the sensor head.

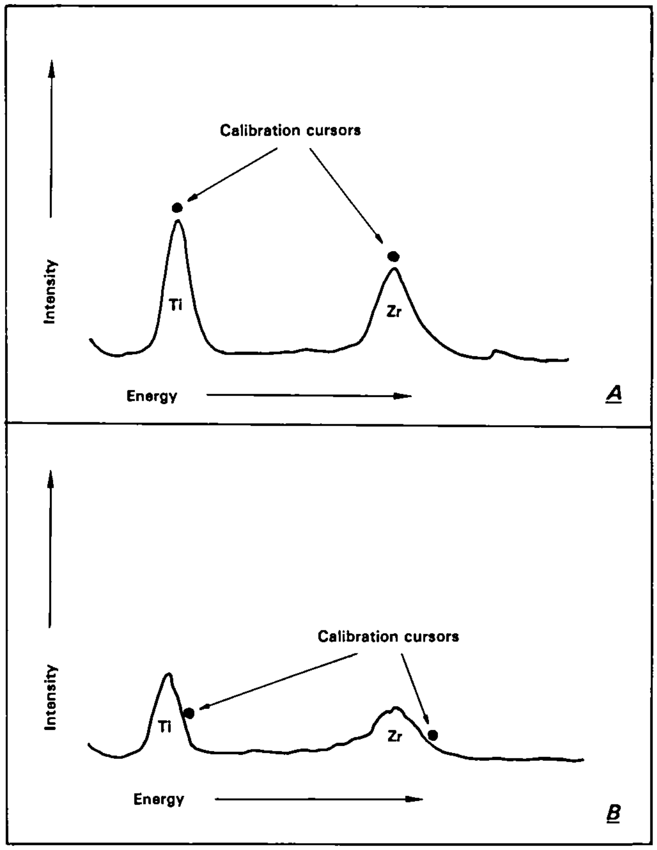

FIGURE 14. - Calibration spectrum of ^{109}Cd source. *A*, under normal operating temperature; *B*, at 108° F.

CONCLUSIONS

This project involved limited testing of the PXRFS for field use in mineral-resource investigative work, and results presented in this report should be considered preliminary. Testing demonstrated a portion of the capabilities and limitations of the spectrometer in mineral identifications and quantitative determinations.

The advantages of the PXRFS are (1) it is portable and can be carried in the field with little difficulty, (2) it has repeat analysis capabilities, (3) analysis time is generally less than 1 minute (if the radioisotopes have not undergone a decay of one half-life), (4) all data are available instantly on the displays, (5) no sample preparation is necessary in the field, and (6) it is relatively maintenance free.

Limitations of the PXRFS are (1) poor resolution of emission data owing to use of a gas-proportional detector that causes interference problems, (2) short half-life (^{109}Cd = 1.2 years, and ^{55}Fe = 2.6 years) of the radioisotopes results in periodic replacement expense, (3) durability is somewhat less than expected, (4) the calibration adjustment screws, located in one end of the sensor head, are relatively inaccessible, (5) spectral shifts occur when the PXRFS is operated in air temperatures less than 50° F or more than 90° F, (6) the CRT display is hard to read because of its miniature size, or because of bright daylight in the field, and (7) one-handed operation of the sensor head is difficult because of its shape and port shutter control spring tension.

At this time the PXRFS falls short in achieving the desired objective of reliable quantitative analytical results.

RECOMMENDATIONS

Recommendations for modification of the PXRFS mainly involve the detectors and the radioactive sources. In order to improve spectral resolution and reduce operating expenses of the PXRFS, the following modifications are suggested:

1. Replace the gas-proportional counters with a single mercuric iodide detector to enhance spectral resolution.

2. Replace the ^{109}Cd and ^{55}Fe radioisotopes with an americium (^{241}Am) source, thereby increasing the element excitation range and reducing isotope replacement costs (the ^{241}Am source has a half-life of 458 years).

A miniature X-ray generator may be an alternative to radioactive isotopes as an X-radiation source.

Other modifications to the PXRFS, for special-use purposes (such as ore control sampling) and to further reduce cost and complexity, might include the following: Use only one radioactive source; test a weaker radioactive source (at lower cost); eliminate the CRT display; use a three- or four-digit LCD and a manual element selector (cursor control in present PXRFS) with a meter (analog) readout.

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APPENDIX A.--SELECTED SECTIONS OF PXRFS OPERATION AND MAINTENANCE MANUAL[1]

Figure A-1 provides a display of the front panel and controls of the PXRFS as a reference for the instructions contained in this section.

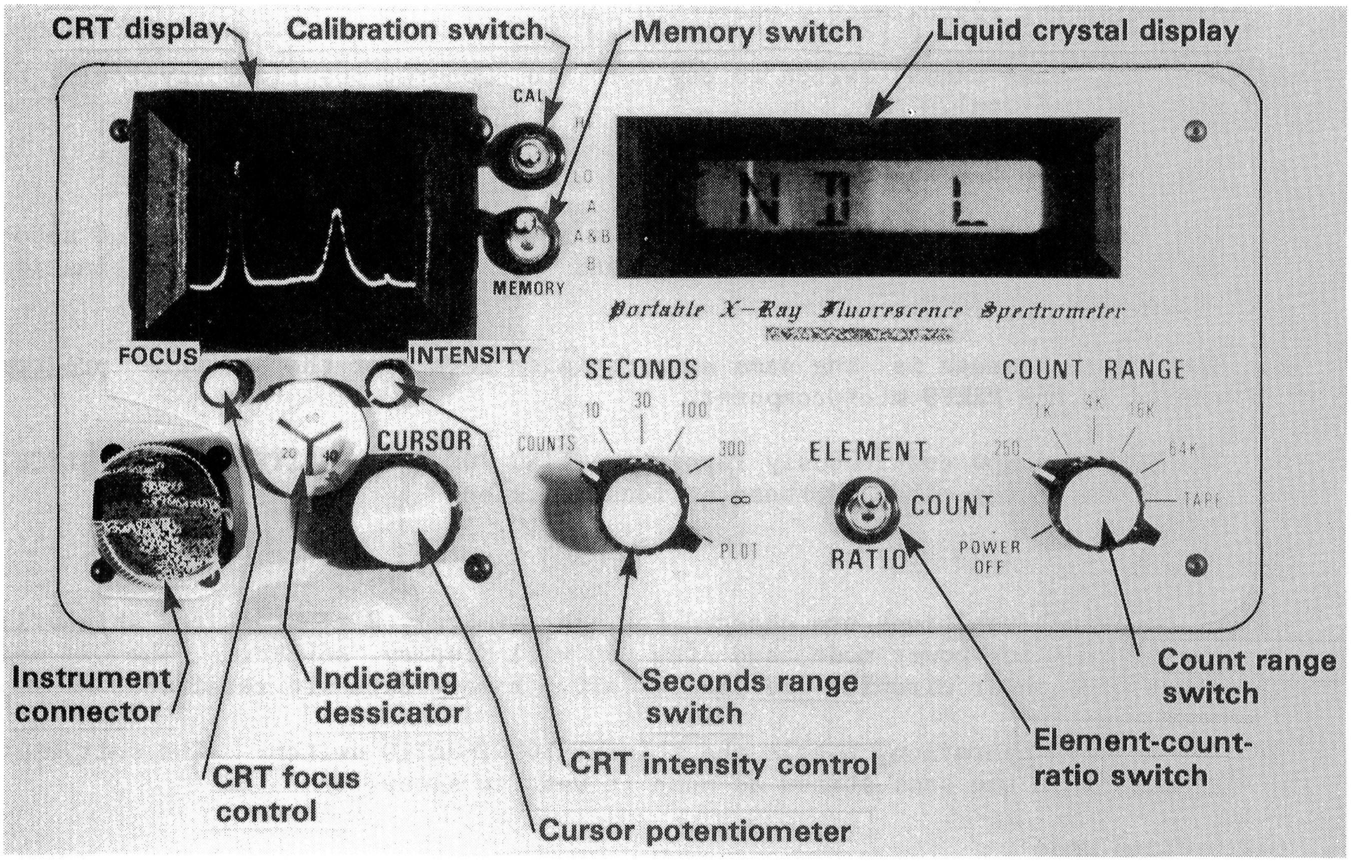

FIGURE A-1. - Front panel and controls of PXRFS.

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[1] Martin Marietta Corp., rev. July 11, 1980.

OPERATING INSTRUCTIONS

1. Power on

   A. Connect sensor head to instrument by pushing the sensor head cable connector onto the face panel connector (aline before plugging in) and then turning clockwise for approximately one-third turn.

   B. Turn the COUNT RANGE switch to any of its numbered positions (typically to the 250 position).

   C. Set the SECONDS switch to 30.

   D. The liquid crystal display (LCD) shows the results of the instrument automatic self-test. It will display either "SELF TEST PASSED" or a combination of warning messages.

   E. The next message is the name and revision level of the software program stored in the PXRFS microcomputer.

   F. The LCD will now continuously repeat "WAITING FOR OPEN SHUTTER OR CAL SWITCH" until one or the other of these actions is taken.

2. Sleep Mode

   A. If no switch positions are changed for approximately 2 minutes, the unit will switch to a low-power mode, and the LCD will display "ASLEEP." The CRT and other high power circuits are powered off. Memory data are retained.

   B. To resume operation, toggle the ELEMENT-COUNT-RATIO switch. The unit will wake up with the same status as when it went to sleep.

3. Calibration Mode

   A. Calibration should be performed before starting a new series of analyses.

   B. After the power-on step, with the LCD repeating the "WAITING..." message, set the SECONDS switch to either COUNTS or to one of the numbered seconds positions.

   C. Set COUNT RANGE switch to desired scale (typically 250 for calibration).

   D. Verify sensor head shutter is closed.

   E. Toggle CAL switch momentarily to LO. LCD displays "Fe-55" for 2 seconds, then begins displaying elapsed analysis time as the spectrum is accumulated.

   F. The unit is now analyzing the Fe-55 calibration sample (sulfur and titanium target) built into the sensor head.

   G. Adjust CRT focus and intensity knobs for best viewing of the spectrum.

   H. When analysis is complete the LCD will display "CAL," and the CRT display will be continuous with two flashing cursors. The left cursor marks where the sulfur peak should appear, and the right cursor where the titanium peak should appear.

NOTE: At any time during or after analysis the COUNT RANGE switch may be used as desired to adjust the vertical expansion of the spectrum on the CRT display.

I. If the cursors are not on the two peaks, adjust the LO trimpot on the sensor head and repeat the analysis as necessary. After adjusting the potentiometer, restart the spectrum by operating the CAL switch again.

J. After the Fe-55 channel is calibrated, repeat the above procedure for Cd-109. Substitute CAL HI for CAL LO and use the HI trimpot on the sensor head. For Cd-109 the left cursor marks titanium and the right cursor marks zirconium.

4. Normal Operation

A. After power-on and calibration steps, the unit is ready to analyze samples.

B. To analyze, adjust the SECONDS and COUNT RANGE switches for the desired type of analysis. Set the MEMORY switch to either A or B, depending upon which half of memory the spectrum is to be stored. Use the A and B setting only if there is no need to compare two different spectra.

C. Place the sensor head port against the sample of interest. If this is not possible, place the port as close to the sample as possible (see "Safety" section at the end of the appendix).

D. Using an index finger, press and hold the shutter control either forward for a Cd-109 analysis, or backward for an Fe-55 analysis. The LCD will first identify which radiation source is in use and then the elapsed analysis time (in seconds). The beeper in the sensor head will sound to warn that a radioactive source is exposed through the open shutter.

NOTE: If the beeper doesn't sound when the shutter is opened, notify the instrument manufacturer.

E. While waiting for the analysis to complete, observe the spectrum building up on the CRT. When sufficient data have been taken, release the shutter control. Verify that the alinement circle is properly centered in the port window, indicating the spring-loaded shutter is in the fully closed position.

F. The COUNT RANGE switch may be used to expand the CRT display as desired.

CAUTION: Do not turn the COUNT RANGE switch to the 9 o'clock position, or the unit will be powered off and special data lost.)

G. To identify a peak, use the CURSOR knob to adjust the bright dot to the top of the peak of interest. When the cursor is adjusted to an element channel, it will flash once per second, and with the ELEMENT-COUNT-RATIO switch set to ELEMENT, the LCD will display the element's chemical symbol.

H. For a hard-copy record of the spectrum, connect the plot or tape output wires to a chart recorder or tape recorder, respectively. These wires are available from the connector at the front panel end of the sensor cable.

1. For a plot, set the SECONDS switch to PLOT. The LCD will display "PLOT REQUESTED" and "PLOT." When the "PLOT" message appears the unit will begin outputting 0- to -5-volt analog signals of spectral data at eight channels per second. The spectrum will repeat as long as the SECONDS switch is left on plot. Switching off the PLOT position at any time will terminate the output and return the spectrometer to the previous mode.

2. For recording the spectrum onto magnetic tape, connect the tape output plug to the auxiliary or microphone input of a recorder.

   NOTE: It may be necessary to adjust the drive circuit output impedance to match the recorder input impedance. Turning the TAPE trimpot, on the INTERFACE printed circuit board, clockwise will increase the impedance to 60 kilohms maximum; turning counter-clockwise will decrease it to its 10-kilohm minimum.

   Set the COUNT RANGE switch to TAPE and then put the recorded in the record mode. The LCD will display "TAPE DUMP REQUESTED" and "RECORD" (signals start of dump). The spectrum will be dumped only once, and the dump is not interruptable (takes approximately 2 minutes).

I. To perform more analyses, either leave the MEMORY switch in the same position it was, or change from A to B or from B to A to save the last spectrum taken. Repeat the steps under section 4.

5. Charging the Batteries

A. Be sure the COUNT RANGE switch is in the power-off position.

B. Unplug sensor head cable by twisting counter-clockwise for approximately one-third turn and pulling to disengage.

C. Plug in cable from the charging unit to the front panel connector.

D. Place the CHARGE switch on the charging unit to one of its three positions. Under standard conditions, the unit contains nickel-cadmium batteries, but lead-acid may also be installed. Use LO RATE for nickel-cadmium batteries if the time available for charging is 16 hours or more; if a fast charge is needed, or if the unit is to be operated during charging, use the HI RATE setting.

E. Connect the appropriate terminals to either the 110-volt-ac source, or automotive battery (12 volts dc).

ROUTINE MAINTENANCE

1. Changing Sensor Heads

A. Place the COUNT RANGE switch in the power-off position.

B. Unplug sensor head at the front panel connector of the main unit by twisting counter-clockwise for one-third turn and pulling off.

C. Plug in new sensor by alining connector, pushing to mate pins, and rotating connector clockwise to snap into locking position.

2. Changing the Desiccant Tube

A. Replace or rejuvenate desiccant whenever the 60% sector changes from its normal blue color to either pale pink or white.

B. Remove the cartridge from the front panel by unscrewing it with fingers or a suitable straightedge.

C. Screw in new unit, or rejuvenate removed cartridge by baking in an oven at 125° C (257° F) for 2 hours or more.

3. Replacing the Batteries

The unit is powered by seven D-size batteries. Either rechargeable batteries (for example, nickel-cadmium or lead-acid cells) or nonrechargeable cells (for example, carbon-zinc, mercury, or lithium types) may be used. Standard rechargeable batteries are of the nickel-cadmium type, but the lead-acid type is preferred for operation in very cold weather.

A. Turn power off and unplug the sensor head. Remove the hinged lid (optional).

B. Unscrew and remove the four mounting feet at the base of the instrument.

C. Carefully place the unit on the side where the four mounts were removed (front panel facing up).

D. Lift up on the front panel to pull the unit slowly out of the housing (assistance from a second person will be helpful and is a sensible precaution).

E. Set the unit down on a smooth surface.

F. Note the battery orientations. All cells are alined in the same direction, with negative terminals closer to the CRT than the positive terminals.

G. Remove batteries from holders by pushing end clips toward each other, and releasing tiedown clip with a slight twisting motion.

H. Place new battery in proper direction and again press end clips inward to facilitate installing the tiedown clip in its original orientation.

I. Replace one or more batteries as required.

J. Carefully place unit in housing, aline holes at bottom with ports, and screw in mounting feet.

TROUBLESHOOTING

The first thing to suspect in the event of improper operation of the unit is a weak or dead battery. Even when the unit is powered by an external electrical source, a malfunctioning cell can prevent proper operation. Therefore, first try changing the batteries (see step 3 of the "Routine Maintenance" section) to verify that is not the problem. During this operation, check for any obvious internal problems: a printed circuit board that is not seated into its connector, a broken wire, dirt or other contamination in critical locations, charred or burned spots, etc.

If this does not solve the problem, and the unit is known to have been exposed to high humidity or low temperatures, remove the desiccant tube and place the entire unit in a dry, warm environment to remove condensation. Replace the tube with a fresh or rejuvenated desiccant.

In the case that the calibration peaks do not line up properly and cannot be adjusted into position with the appropriate potentiometer, the unit will require resetting by the manufacturer. However, it is possible to make temporary use of the instrument for field purposes by using the calibration targets built into the lid to note the channel numbers into which the elements of interest fall. With this approach, the operator must disregard the element symbols given on the LCD since these peaks will now occur slightly upscale or downscale from the normal position.

SAFETY

The PXRFS instrument has been engineered with every consideration for safety. A number of special features are designed to minimize the possible risks to the operator and bystanders. It is extremely important, however, that all persons who are using, or have plans to use this instrument, read this section very carefully. THIS UNIT CONTAINS RADIOACTIVE MATERIALS and although these sources emit relatively low energy X-rays, and are carefully shielded, there is the possibility of unnecessary radiation exposure whenever the shutter is opened without the sensor head being placed flush against a suitable thick sample.

1. Radiation Hazard

The standard sensor head contains two radioactive materials: approximately 100 mCi of iron-55 isotope and 15 mCi of cadmium-109. These sources are electroplated onto a substrate and then hermetically sealed into a rugged holder. The possibility of leakage of radioactive material is extremely remote. The sources are in turn mounted in shielded collimators. When in the neutral closed position the safety shutter provides additional shielding to prevent any radiation from penetrating outside the sensor head. There is no hazard associated with handling or being near the sensor head when the shutter is closed (that is, when neither source is exposed). Of course, the sensor head should be kept secured from access by casual bystanders at all times.

During acquisition of a spectrum, one or the other of the two sources must necessarily be positioned to irradiate the samples with X-rays. To prevent unnecessary exposure to the operator and for maximum safety, the following rules must be followed:

A. Always place the sensor head analysis port up against the sample before opening the shutter.

B. If the sample surface is irregular or it is impractical to place the sensor head totally against the sample, be sure to position your hands, limbs, and body such that they are behind the sensor head.

C. Never look at or handle the port window, except when the shutter is fully closed.

D. If the warning beeper fails to operate when the shutter is opened, return the unit to the manufacturer.

E. Never attempt to repair or disassemble the sensor head. The only exception is simple replacement of the plastic film port window using a pre-prepared window kit. Make sure the shutter is in the neutral closed position before making this change.

F. Insure that the radiation sources are leak checked at required intervals (typically, every 6 months).

G. Have available a thin-window Geiger counter or other radiation monitoring device. Periodically check the radiation level during normal operation to make sure unsafe procedures are not being followed.

2. Shock Hazard

All circuits within the PXRFS sensor head operate at low voltages, with the exception of the detector bias supply. This supply provides 1,000 to 1,500 volts for operation of the proportional counters. Because this supply is potted, shielded by a metal housing, and contains a current-limiting resistor, the possibility of a malfunction causing serious shock is minimal. In the event of any indications of electronic problems within the sensor head, the unit should be returned to the manufacturer.

An additional high voltage circuit is located within the main unit to provide the electron beam accelerating voltage for CRT operation. Because of this supply, it is recommended the unit never be turned to power-on condition when removed from its housing--for example, during a battery change-out operation.

APPENDIX B.--TABLES OF ANALYTICAL RESULTS

TABLE B-1. - X-ray excitation capabilities of ^{55}Fe and ^{109}Cd for selected elements

Element	X-ray source used in analysis	Source excitation capabilities	Most likely interfering elements
Aluminum	^{55}Fe	Fair	Mg, Si
Arsenic	^{55}Fe	Good	Pb, Se, Hg
Calcium	^{55}Fe and ^{109}Cd	Fair	K
Chromium	^{55}Fe and ^{109}Cd	Good	Fe, V, Mn
Cobalt	^{109}Cd	Good	Mn, Fe, Ni, Cu
Copper	^{109}Cd	Good	Fe, Co. Ni, Zn
Iron	^{109}Cd	Good	Cr, Mn, Co, Ni, Cu
Lead	^{109}Cd	Good	As
Magnesium	^{55}Fe	Fair	Na, Al
Manganese	^{109}Cd	Good	Cr, Fe
Mercury	^{109}Cd	Fair	As, Pb
Molybdenum	^{109}Cd	Good	Nb, U
Nickel	^{109}Cd	Good	Cu, Co, Fe
Niobium	^{109}Cd	Fair	Th, U
Silicon	^{55}Fe and ^{109}Cd	Fair	Al
Sulfur	^{55}Fe and ^{109}Cd	Fair	K
Titanium	^{55}Fe and ^{109}Cd	Good	Ca, V
Tungsten	^{109}Cd	Good	Cu, Zn
Uranium	^{109}Cd	Fair	Mo, Th, Sr, Nb
Vanadium	^{55}Fe and ^{109}Cd	Fair	Ti, Cr, Mn
Zinc	^{109}Cd	Good	Cu, W
Zirconium	^{109}Cd	Good	Th, Nb

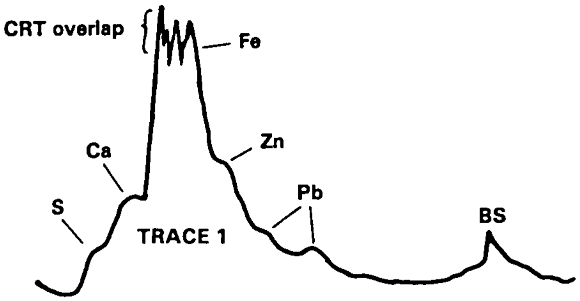
TABLE B-2. - Comparison of minus 200-mesh pulp sample analysis data with PXRFS test results

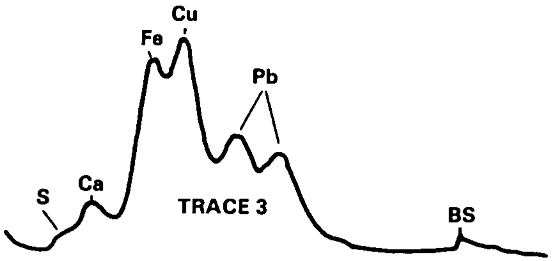
(Ratio value: Total counts divided by backscatter)

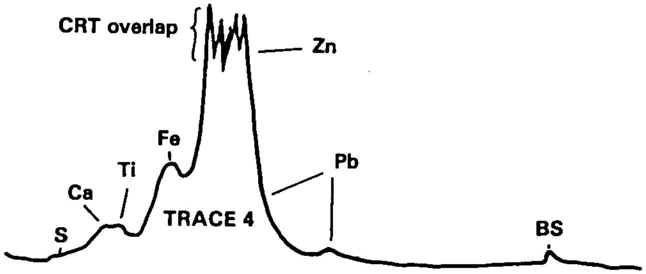
Element	Sample analysis, %	PXRFS results		Comments
		Total counts	Ratio	
GALENA IN IGNEOUS ROCK				
Ca..	NA	87	2.937	ESA; 60-sec ^{109}Cd irradiation. The Fe response interferes with the Zn and Pb X-ray response. See trace 1.
Fe..	NA	1,173	46.000	
Pb..	1.32	51	1.375	
S...	NA	37	1.437	
Zn..	1.95	77	3.500	
SCHEELITE-POWELLITE IN FAULT ZONE				
Ca..	NA	186	0.312	ESA; 300-sec ^{109}Cd irradiation. Fe response interferes with Cu response--Cu appears as small shoulder to right of Fe peak; W plateau to right of Fe peak. See trace 2.
Cu..	NA	81	.375	
Fe..	NA	258	1.062	
Mo..	0.0095	60	.187	
W...	.3200	77	.375	

Element	Sample analysis, %	PXRFS results		Comments
		Total counts	Ratio	
MASSIVE SULFIDE				
Ca..	NA	27	7.375	ESA; 60-sec ^{109}Cd irradiation. Cu response interferes with the Zn response. See trace 3.
Cu..	11.00	153	30.750	
Fe..	NA	133	35.812	
Pb..	14.00	82	18.562	
S...	NA	9	2.812	
Zn..	.11	ND	NAp	
Ca..	NA	77	4.812	ESA; 80-sec ^{109}Cd irradiation. See trace 4.
Cu..	.018	ND	NAp	
Fe..	NA	287	14.125	
Pb..	2.800	89	4.375	
S...	NA	31	1.312	
Ti..	NA	72	4.187	
Zn..	23.500	1,075	52.562	

See notes at end of table.







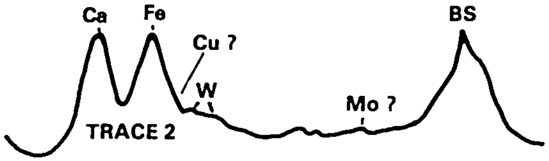
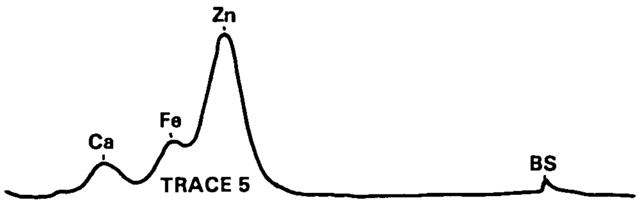
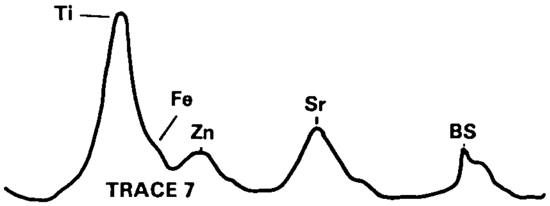


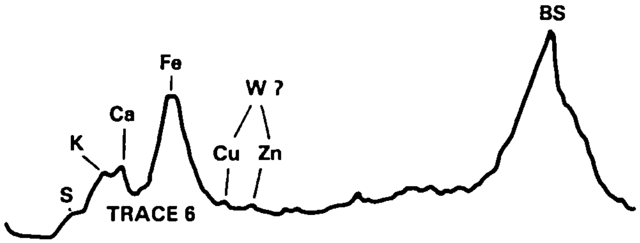
TABLE B-2. - Comparison of minus 200-mesh pulp sample analysis data with PXRFS test results--Continued

Element	Sample analysis, %	PXRFS results		Comments
		Total counts	Ratio	
SULFIDE				
Ca..	NA	132	4.187	ESA; 60-sec ^{109}Cd irradiation. The Pb response is masked by Fe and Zn responses. Fe appears as a shoulder to left of Zn peak. See trace 5.
Fe..	NA	229	6.875	
Pb..	0.4	ND	NAp	
Zn..	NA	551	18.500	
ALTERED RHYOLITE PORPHYRY				
Ca..	NA	32	0.312	AA; 120-sec ^{109}Cd irradiation. Mo detection may indicate only a background value and ratio. Bulge in right side of Fe peak (trace 6) is a distortion caused by the Cu response.
Cu..	0.1000	18	.187	
Fe..	NA	99	.750	
K...	NA	53	.312	
Mo..	.0004	18	.182	
S...	NA	13	.125	
Sc..	NA	46	.312	
W...	NA	28	.125	
Zn..	NA	12	.125	
LIMESTONE				
Ba..	0.0	ND	NAp	ESA; 120-sec ^{109}Cd irradiation. Fe appears to be shoulder on the right side of the Ti peak (trace 7). Ti peak may have intense Ca response contribution rather than just Ti response.
Ca..	7.0	ND	NAp	
Fe..	2.0	41	1.562	
Mg..	2.0	ND	NAp	
Pb..	.3	ND	NAp	
Sr..	.4	63	2.875	
Ti..	.03	172	7.312	
Zn..	.6	45	1.812	
STREAM SEDIMENT FINES				
Ca..	NA	37	0.375	ESA; 90-sec ^{109}Cd irradiation. See trace 8.
Cu..	0.0125	ND	NAp	
F...	.0680	ND	NAp	
Mo..	.0330	47	.187	
S...	NA	18	.125	
W...	.0034	ND	NAp	

See notes at end of table.







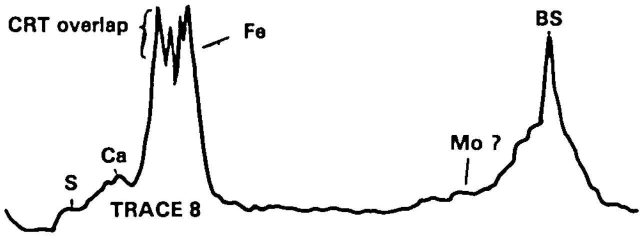
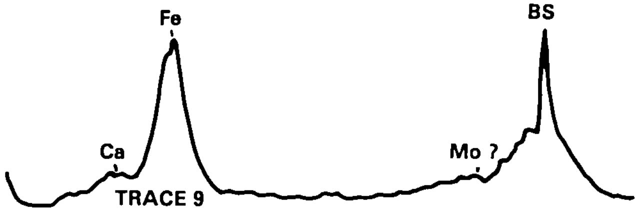


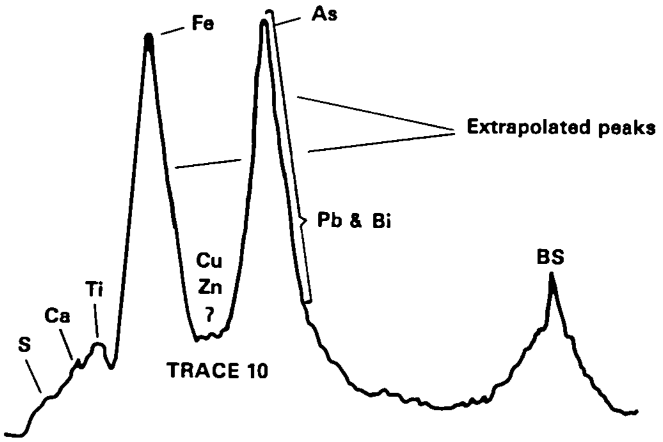
TABLE B-2. - Comparison of minus 200-mesh pulp sample analysis data with PXRFS test results--Continued

Element	Sample analysis, %	PXRFS results: Total counts	PXRFS results: Ratio	Comments
STREAM SEDIMENT FINES--Continued				
Ca..	NA	67	0.312	AA; 60-sec ^{109}Cd irradiation. Mo interference from backscatter peak. See trace 9.
Cu..	0.0025	ND	NAp	
F...	.0520	ND	NAp	
Fe..	NA	313	1.375	
Mo..	.0480	60	.250	
W...	.0042	ND	NAp	
QUARTZ VEIN IN GRANITE				
As..	0.50	286	3.375	ESA; 120-sec ^{109}Cd irradiation. As, Bi, and Pb counts and ratio readings are all very close in value; interference among these three elements (trace 10) makes analysis difficult. Responses between the Fe, Pb, As, and Bi peaks are questionable; however, Cu is known to occur in this sample--Zn possibly in sphalerite as well.
Bi..	.05	274	3.125	
Ca..	.10	49	.625	
Cu..	.05	57	.750	
Fe..	3.00	419	4.562	
Pb..	.10	284	3.500	
S...	NA	25	.250	
Ti..	.02	49	.562	
Zn..	<.10	66	.812	
QUARTZ VEIN IN GRANITE--Continued				
As..	0.50	ND	NAp	ESA; 60-sec ^{55}Fe irradiation. See trace 11.
Bi..	.05	ND	NAp	
Ca..	.10	ND	NAp	
Cu..	.05	ND	NAp	
Fe..	3.00	ND	NAp	
K...	NA	65	0.375	
Pb..	.10	ND	NAp	
Si..	>20.0	18	.062	
Ti..	.02	ND	NAp	

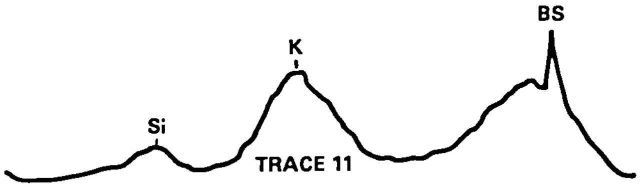
See notes at end of table.



TRACE 9



TRACE 10



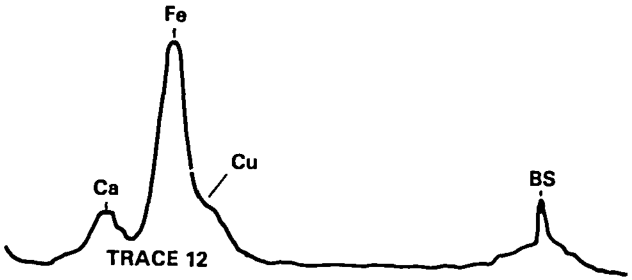
TRACE 11

TABLE B-2. - Comparison of minus 200-mesh pulp sample analysis data with PXRFS test results--Continued

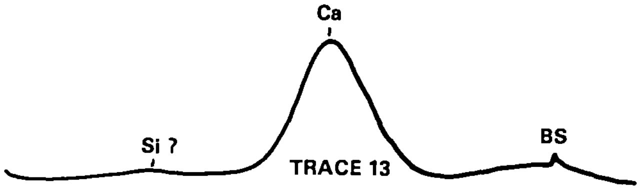
Element	Sample analysis, %	PXRFS results Total counts	PXRFS results Ratio	Comments
ANDESITE				
Ca..	10.00	159	1.125	ESA; 180-sec ^{109}Cd irradiation. Fe peak (trace 12) is distorted by the Cu response that shows up as a shoulder to the right of the Fe peak.
Cu..	2.00	202	1.312	
Fe..	7.00	612	6.312	
Mg..	1.00	ND	NAp	
Pb..	.09	ND	NAp	
Rb..	NA	27	.187	
Ti..	.20	ND	NAp	
Zn..	.01	ND	NAp	
Ca..	10.00	145	2.687	ESA; 60-sec ^{55}Fe irradiation. See trace 13.
Cu..	2.00	ND	NAp	
Fe..	7.00	ND	NAp	
Mg..	1.00	ND	NAp	
Pb..	.09	ND	NAp	
Si..	10.00	8	.062	
Ti..	.20	ND	NAp	
Zn..	.01	ND	NAp	
TALC AND CHLORITE, ALTERED DIKE				
Ca..	[1]0.57	45	0.437	ESA; 60-sec ^{109}Cd irradiation.
Fe..	[2]12.20	297	3.875	
S...	NA	16	1.870	

Element	Sample analysis, %	PXRFS results Total counts	PXRFS results Ratio	Comments
SPHALERITE IN SCHIST				
Ca..	NA	140	0.550	ESA; 60-sec ^{109}Cd irradiation.
Fe..	NA	440	22.750	
Pb..	0.006	ND	NAp	
Zn..	32.500	1,512	81.875	
MOLYBDENITE IN BULL QUARTZ				
Ca..	NA	35	0.187	ESA; 60-sec ^{109}Cd irradiation.
Fe..	NA	40	.187	
Mo..	0.29	103	.562	
PHOSPHATE ROCK				
Ca..	26.3000	187	1.500	ESA; 180-sec ^{109}Cd irradiation. Cr, Ti, and V responses are masked by the much stronger Ca response. Much longer analysis time would be required to confirm the presence of Zn and Nb.
Cr..	.0860	ND	NAp	
Cu..	.0085	21	.187	
Fe..	.2500	36	.250	
Nb..	NA	26	.187	
S...	NA	19	.187	
Sr..	.0990	41	.312	
Ti..	.0800	ND	NAp	
V...	.0290	ND	NAp	
Zn..	NA	31	.187	

See notes and footnotes at end of table.



TRACE 12



TRACE 13

TABLE B-2. - Comparison of minus 200-mesh pulp sample analysis data with PXRFS test results--Continued

Element	Sample analysis, %	PXRFS results		Comments
		Total counts	Ratio	
ALTERED RHYOLITE PORPHYRY				
Ca..	NA	38	0.375	AA; 60-sec ^{109}Cd irradiation.
Cu..	0.0800	ND	NAp	
Fe..	NA	104	1.000	
Mo..	.0006	ND	NAp	
ANDESITE				
Ca..	NA	47	0.375	AA; 60-sec ^{109}Cd irradiation.
Cu..	0.0100	ND	NAp	
Fe..	NA	121	1.062	
Mo..	.0035	ND	NAp	
ARKOSIC SANDSTONE				
Ca..	NA	44	0.500	AA; 60-sec ^{109}Cd irradiation.
Cu..	0.0625	ND	NAp	
Fe..	NA	169	1.275	
Mo..	.0012	ND	NAp	
ARGILLITE				
Ca..	NA	49	0.625	AA; 60-sec ^{109}Cd irradiation.
Cu..	0.0075	ND	NAp	
Fe..	NA	145	1.312	
Mo..	.0002	ND	NAp	
LIMESTONE				
Al..	>4.0	ND	NAp	ESA; 60-sec ^{109}Cd irradiation.
Ca..	>10.0	125	2.437	
Fe..	3.0	47	.687	
K...	<.06	179	2.687	
Mg..	.6	ND	NAp	
Ti..	.2	ND	NAp	
STREAM SEDIMENT FINES				
Ca..	NA	85	0.750	ESA; 60-sec ^{109}Cd irradiation.
Cu..	0.0125	ND	NAp	
F...	.0380	ND	NAp	
Fe..	NA	1,957	2.687	
Mo..	.0450	ND	NAp	
S...	NA	35	.437	
W...	.0135	ND	NAp	
Ca..	NA	49	.187	AA; 60-sec ^{109}Cd irradiation.
Cu..	.0005	ND	NAp	
F...	.0260	ND	NAp	
Fe..	NA	34	.125	
Mo..	.0004	ND	NAp	
W...	.0010	ND	NAp	

Element	Sample analysis, %	PXRFS results		Comments
		Total counts	Ratio	
QUARTZ VEINS AND STRINGERS IN GRANITE				
Ca..	NA	77	0.250	AA; 120-sec ^{109}Cd irradiation.
Cu..	0.0010	ND	NAp	
Fe..	NA	129	.562	
Mo..	.0028	ND	NAp	
Zn..	.0050	ND	NAp	
Ca..	NA	66	.187	AA; 60-sec ^{109}Cd irradiation.
Cu..	.0150	ND	NAp	
Fe..	NA	90	.312	
Mo..	.0070	ND	NAp	
Zn..	.0005	ND	NAp	
Ca..	NA	73	.187	Do.
Cu..	.8010	ND	NAp	
Fe..	NA	69	.187	
Mo..	.0055	ND	NAp	
W...	.0007	ND	NAp	
Ca..	NA	58	.250	AA; 90-sec ^{109}Cd irradiation.
Cu..	.0005	20	.062	
Fe..	NA	50	.187	
Mo..	<.0002	ND	NAp	
W...	<.0002	ND	NAp	
GRANITE				
Al..	4.000	ND	NAp	ESA; 90-sec ^{109}Cd irradiation.
Au..	NA	20	0.062	
Ca..	.500	69	.250	
Cb..	[3].010	ND	NAp	
Cu..	.002	ND	NAp	
Fe..	1.000	102	.312	
Ni..	.002	ND	NAp	
Pb..	.010	ND	NAp	
Rb..	NA	65	.125	
Ti..	.030	ND	NAp	
W...	NA	46	.125	

See notes and footnotes at end of table.

TABLE B-2. - Comparison of minus 200-mesh pulp sample analysis data with PXRFS test results--Continued

Element	Sample analysis, %	PXRFS results		Comments
		Total counts	Ratio	
GRANITE--Continued				
Al..	4.000	ND	NAp	ESA; 60-sec ^{55}Fe irradiation.
Ca..	.500	ND	NAp	
Cb..	[3].010	ND	NAp	
Cu..	.002	ND	NAp	
Fe..	1.000	ND	NAp	
K...	NA	46	0.375	
Ni..	.002	ND	NAp	
Pb..	.010	ND	NAp	
Si..	>20.000	11	.062	
Ti..	.030	ND	NAp	
Al..	8.00	ND	NAp	ESA; 180-sec ^{109}Cd irradiation. The Sr response may actually be a Y response--they are adjacent to one another in periodic table and have similar emission energies.
Ca..	[1].70	119	.375	
Cb..	[3].02	ND	NAp	
Fe..	4.00	362	1.312	
Pb..	.02	ND	NAp	
Sr..	NA	53	.250	
Y..	.01	ND	NAp	
Al..	8.00	ND	NAp	ESA analysis; 60-sec ^{55}Fe irradiation.
Ca..	[1].70	ND	NAp	
Cb..	[3].02	ND	NAp	
Fe..	4.00	ND	NAp	
K...	NA	75	.562	
Pb..	.02	ND	NAp	
Si..	>20.00	12	.062	
Y...	.01	ND	NAp	
QUARTZ VEIN IN DIORITE				
As..	0.070	ND	NAp	ESA; 60-sec ^{109}Cd irradiation. As, Co, Cr, Ti, V, and Zn responses all masked by strong Cu and Fe responses.
Ca..	NA	85	3.625	
Co..	.020	ND	NAp	
Cr..	.008	ND	NAp	
Cu..	7.000	961	42.000	
Fe..	6.000	587	24.812	
Ti..	.300	ND	NAp	
V...	.070	ND	NAp	
Zn..	.030	ND	NAp	
QUARTZ VEIN IN DIORITE--Continued				
As..	0.070	ND	NAp	ESA; 60-sec ^{55}Fe irradiation.
Co..	.020	ND	NAp	
Cr..	.008	ND	NAp	
Cu..	7.000	ND	NAp	
Fe..	6.000	ND	NAp	
K...	<.600	12	0.062	
S...	NA	27	.125	
Ti..	.300	ND	NAp	
V...	.070	ND	NAp	
Zn..	.030	ND	NAp	
QUARTZ VEIN IN GRANITE				
As..	2.0	349	0.562	ESA; 120-sec ^{109}Cd irradiation.
Bi..	.2	ND	NAp	
Cu..	.2	ND	NAp	
Fe..	7.0	417	10.187	
Pb..	>1.0	328	.562	
Zn..	.2	ND	NAp	
As..	2.0	ND	NAp	ESA; 60-sec ^{55}Fe irradiation.
Bi..	.2	ND	NAp	
Cu..	.2	ND	NAp	
Fe..	7.0	ND	NAp	
K...	NA	55	.312	
Pb..	1.0	ND	NAp	
Si..	>20.0	10	.062	
Zn..	.2	ND	NAp	
As..	1.0	883	53.500	AA; 45-sec ^{109}Cd irradiation.
Ca..	.4	ND	NAp	
Cu..	.8	ND	NAp	
Fe..	>10.0	387	25.875	
Pb..	>1.0	ND	NAp	
Zn..	.7	ND	NAp	

AA Original analysis by atomic absorption. BS Backscatter. ESA Original analysis by mass spectrometer. NA Not analyzed. NAp Not applicable. ND Not detected or no reading.

[1] Analyzed as CaO. [2] Analyzed as Fe_2O_3. [3] Analyzed as Cb_2O_5.

TABLE B-3. - Comparison of USGS sample standards[1] with PXRFS test results

(Ratio value: Total counts divided by backscatter)

Element	Sample analysis, %	PXRFS test results[2]		Element	Sample analysis, %	PXRFS test results[2]	
		Total counts	Ratio			Total counts	Ratio
Copper...	0.01	21	0.125	Rubidium.	0.010	6	0.062
	.01	25	.125		.021	16	.062
	.05	30	.125		.046	18	.187
	.10	25	.187		.100	31	.250
	.20	54	.312		.215	73	.625
	.50	92	.625		.464	116	1.187
	1.00	130	1.000				
	2.00	301	2.312	Strontium	.001	10	0
	5.00	945	9.062		.010	13	0
					.021	12	.062
Iron.....	.046	18	.062		.046	13	.125
	.100	20	.125		.100	16	.250
	.220	17	.125		.215	83	.625
	.460	37	.250		1.000	267	2.812
	1.000	52	.437				
	2.150	104	1.000	Zinc.....	.01	27	.125
	4.640	288	2.375		.02	22	.125
	10.000	781	8.250		.05	24	.125
					.10	26	.187
Lead.....	.01	10	.062		.20	62	.375
	.02	14	.062		.50	111	.750
	.05	16	.125		1.00	182	1.437
	.10	30	.125		2.00	419	3.125
	.20	28	.187		5.00	1,371	14.750
	.50	54	.437				
	1.00	89	.750				
	2.00	163	1.625				
	5.00	348	4.687				
Manganese	.010	376	3.812				
	.022	384	3.875				
	.046	406	4.250				
	.100	404	3.937				
	.215	361	4.187				
	.464	444	4.375				
	1.000	434	5.750				

[1] These element standards have a granitic matrix.

[2] 120-sec ^{109}Cd irradiation.

TABLE B-4. - PXRFS (^{109}Cd) analysis of selected Bureau of Mines mineral display specimens

Sample and composition (9)	Elements detected by PXRFS	Counts	Element to backscatter ratio	Comments
Carnotite in sandstone; $K_2(UO_2)_2(VO_4)_2 \cdot nH_2O$.	K	77	1.687	30-sec irradiation.
	Ca	66	1.437	
	Cu	45	1.375	
	U	213	4.750	
Cinnabar and antimony[1]...	Hg	123	2.312	60-sec irradiation. Sb not detectable with this instrument.
	S	39	.250	
	Ca	51	.812	
	Fe	23	.437	
Cupro-tungstate; CuO and WO_3.[1]	Cu	265	9.375	60-sec irradiation. W response is masked by intense Cu response combined with Ni and Co responses.
	Fe	170	5.625	
	Co	185	6.312	
	Ni	282	9.187	
	Pb	535	17.312	
	Ca	50	1.875	
Erythrite; 37.5% CoO, 38.4% As_2O_5, 24.1% H_2O.	Co	189	(2)	30-sec irradiation. Overflow indicates a ratio is not possible because backscatter is nonexistent.
	As	184	(2)	
Ferberite; 23.7% FeO and 76.3% WO_3.	Fe	94	(2)	Do.
	W	175	(2)	
Galena; 86.6% Pb and 13.4% S.	Pb	580	56.562	60-sec irradiation.
	S	42	3.500	
	Ca	35	4.500	
	Fe	62	5.625	
Garnierite; $(Ni,Mg)SiO_3 \cdot nH_2O$.	K	38	3.375	30-sec irradiation.
	Ni	710	57.000	
Gold telluride[1]..........	Ca	115	3.437	60-sec irradiation. Double peak could be from Hg or Pb response as well as Au.
	Fe	24	.875	
	Au	138	4.500	
Niccolite; 43.9% Ni and 56.1% As.	Ni	156	26.000	30-sec irradiation.
	As	84	12.562	
	Ca	60	8.000	
Rhodonite; 54.1% MnO and 45.9% SiO_2.	Mn	671	31.437	45-sec irradiation.
Scheelite in granite; 19.4% CaO and 80.6% WO_3.	Ca	93	2.187	60-sec irradiation.
	W	84	1.500	
	Fe	54	.812	
	Rb	4	.250	
Scheelite and pyrite; 19.4% CaO, 80.6% WO_3, 46.6% Fe, 53.4% S.	Ca	48	1.062	Do.
	W	78	1.375	
	Fe	120	2.312	
Uranium in sedimentary rock.[1]	U	106	.875	Do.
	Ca	42	.312	
	Fe	123	1.062	

[1] Exact composition percent not known. [2] Overflow.

APPENDIX C.--NUCLEAR REGULATORY COMMISSION (NRC) LICENSING INFORMATION

All manmade radioactive materials are strictly controlled by the NRC, and it has issued comprehensive regulations for controlling and licensing radioactive isotope sources (called "byproducts" because of their origin in a nuclear reactor).

Briefly, the NRC regulations require the following relative to the PXRFS:

1. The licensing of any company or individual (including Government agencies) that uses isotopes of the strength required in portable X-ray fluorescence spectrometers.
2. The isotopes be under the control of a trained and qualified person at all times.
3. The material be kept reasonably secure against theft or loss by accident.
4. Safety precautions be adequate to protect the user and others in the vicinity from radiation.
5. An authorized individual user be present and directly supervise the use of the spectrometer at any temporary job site.
6. User qualifications include, as a minimum, the completion of the instrument manufacturer's training course.
7. Training by companies is permissible, but the NRC must approve the training course.
8. If multiple users are listed on the license, a radiation protection officer must be named.

"A Guide for Preparation of Byproduct Material Application for the Use of Sealed Sources in Portable and Semiportable Gauging Devices" can be obtained by writing: Materials Licensing Branch, Division of Fuel Cycle and Material Safety, Nuclear Regulatory Commission, Washington, D.C. 20555.

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