

STATUS REPORT

A FEASIBILITY STUDY OF THE USE OF X-RAY EQUIPMENT

FOR SIMULTANEOUS MEASUREMENTS OF OIL AND WATER SATURATIONS

Project BE9, Task 3, Milestone 1

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September 1987

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SUMMARY

The feasibility of using an x-ray technique for simultaneous measurements of oil and water saturations was studied. The experimental procedure selected for this feasibility study was developed by Oak and Ehrlich.¹ The work plan was to determine the feasibility of applying their method using the x-ray equipment (x-ray controller and detection system) currently available at NIPER.

The study revealed that NIPER's x-ray equipment cannot be used for simultaneous measurements of oil and water saturations because of hardware constraints. Adaptation of the equipment to make such measurements possible would require major modifications and expenditures. However, the equipment could possibly be used for the determination of water and oil saturations sequentially.

Further study revealed that, despite extensive efforts in performing the experiments under different conditions, sequential determinations of oil and water saturations were not achievable to the required degree of accuracy. Errors in saturation measurements were greater than 10 percent. We conclude, therefore, that accurate measurements of oil and water sequentially by the x-ray technique are not feasible with the equipment available at NIPER.

INTRODUCTION

The feasibility of adapting the NIPER x-ray apparatus to determine water and oil saturations simultaneously by using the absorption edge of iodine¹ was studied. The advantages of using x-rays for measuring oil and water saturations in porous media are as follows:

1. The x-ray technique can be used to measure the saturations in a core encased in a metal core holder. This procedure permits the study of three-phase relative permeabilities at higher pressures (10,000 psi)

and temperatures (212° F) using aluminum or titanium alloy core holders and heating jackets.

2. The method allows for simultaneous measurement of oil and water saturations, thus avoiding any time lag between the two measurements. The time factor is especially important during unsteady-state or displacement runs because the saturations change continuously.
3. The area of the core scanned for water and oil saturation determinations at a fixed location would be the same since the aperture size (sampling area) is the same.
4. The present limitations on the core size (>1 in. and <1.25 in. diameter) would be eliminated. Any core size larger than 0.5 in. in diameter and less than 3.0 in. in diameter may be used.

THEORY

The absorption edge technique uses the K-absorption edge of elements to resolve the incident x-ray beam into two beams of different wavelengths. Figure 1 shows the absorption edge of different elements. The absorption coefficient of an element is high at wavelengths just shorter than the absorption edge and low at wavelengths longer than the absorption edge. By using an iodine filter and tagging the oil with an iodine component and the water with a bromide, the water and oil saturation can be measured simultaneously. Oil saturation can be obtained by measuring the x-ray absorption at wavelengths just shorter than the absorption edge of iodine. On the contrary, water saturation can be obtained by measuring the x-ray absorption at wavelengths longer than the absorption edge of iodine.

Figure 2 shows the x-ray characteristic curve generated as a function of the tube potential (tungsten target) for the x-ray system used. The wavelengths of interest are from 0.20 to 0.40 Å. From figure 1, it is obvious that some of the elements that exhibit a K-absorption edge around the wavelengths of interest are barium, cesium, and iodine. Thus, by selecting proper operating parameters such as x-ray tube potential, filter type,

concentration, and thickness, it is theoretically possible to measure oil and water saturations simultaneously.

Iodine and bromine were selected as the tag for the oil and water phases, respectively. An iodine filter was used to separate the x-ray beam into two distinct wavelengths. By tagging the oil with iodine solution and the water with bromine solution and measuring the absorption at wavelengths just shorter than the absorption edge of iodine, the oil saturation can be determined. Similarly, the water saturation may be determined by measuring the x-ray absorption at wavelengths higher than the absorption edge of iodine.

This procedure can be illustrated by the following equations. From Beer-Lambert's Law, equations 1 and 2 can be written as follows:

at wavelength 1,

$$\ln \frac{I_1}{I_0} = \sum_{i=O,W,G} - \mu_i^1 S_i \phi l \rho_i \quad (1)$$

at wavelength 2,

$$\ln \frac{I_2}{I_0} = \sum_{i=O,W,G} - \mu_i^2 S_i \phi l \rho_i \quad (2)$$

where I/I_0 = ratio of the x-ray intensity transmitted through the sample to the incident x-ray intensity

μ_i = absorption coefficient of species i

ϕ = porosity of core sample

l = width of core sample

ρ_i = density of species i

S_i = saturation of species i

Assuming that the density and absorption coefficient of gas are small (the x-rays absorbed by gas are negligible), equations 1 and 2 may be written as

$$-\ln \frac{I_1}{I_0} = A S_w + B S_o \quad (3)$$

$$-\ln \frac{I^2}{I_0^2} = D S_w + E S_o \quad (4)$$

$$\text{where } A = (\mu_w^1 \rho_w - \mu_g^1 \rho_g) \phi l$$

$$B = (\mu_o^1 \rho_o - \mu_g^1 \rho_g) \phi l$$

$$D = (\mu_w^2 \rho_w - \mu_g^2 \rho_g) \phi l$$

$$E = (\mu_o^2 \rho_o - \mu_g^2 \rho_g) \phi l$$

Equations 3 and 4 indicate that the water and oil saturation in porous media may be determined by measuring x-ray absorption at two different wavelengths on either side of the iodine absorption edge after selecting the correct window setting and baseline and tagging the oil with iodine and the water with bromine.

EXPERIMENTAL PROCEDURE

To enable simultaneous measurements of x-ray absorption at two wavelengths, the x-ray detection circuit was modified by adding a second detection cable, binary counter, rate meter, pulse height selector, window control, and amplifier.

The detector consists of a scintillation crystal, a photomultiplier, and a preamplifier, which transforms an incident x-ray photon into an electric pulse, the amplitude of which is proportional to the energy of the incident photon. The number of pulses generated is proportional to the number of incident photons. Consequently, in response to incident x-ray photons, the detector generates electrical pulses for which the frequency is proportional to the intensity of the x-rays, and for which the amplitude of each pulse is proportional to the x-ray energy.

The electric pulses so generated are received by the pulse height selectors, each of which is equipped with a baseline discriminator and a window that sets the minimum and the maximum amplitude of pulses to be selected. The settings for both the baseline and the window range from 0 to 100. The frequency of the selected pulses is measured by the linear rate meter.

Since the pulse amplitude is proportional to the incident x-ray photon energy, it is possible to set the baseline and window of each selector in such a way that the selected pulses by each selector correspond to x-rays belonging to each wavelength band. By doing so, the absorption measurements at two wavelengths can be simultaneous and obtained from a single beam.

A series of experiments was performed with filters of various thickness (1/2, 1/4 and 1/16 inch) filled with sodium iodide solution (50, 70, 90 and 100 percent), at various x-ray settings (45 KV-10 mA, 45 KV-30 mA, 50 KV-10 mA, and 50 KV-30 mA). For each of these experimental combinations, cells of various concentrations (5, 10, and 15 percent) and thicknesses (1/16, 1/4, and 1/8 inch) of sodium bromide (simulating brine in core) and idododecane (simulating oil in core) were placed in front of the sodium iodide filter.

A basic program was written to control the x-ray unit and facilitate data acquisition and analysis. Preliminary tests of the second measuring channel indicated that the x-ray intensities were not distributed normally around the center (50 percent window setting). A typical curve for both channels as a function of window settings is shown in figure 3. The manufacturer of the system (Philips Electronics Inc.) was contacted, and a service engineer was dispatched to study the problem. It was found that the first channel (channel A) and the controller were designed to work with the x-ray diffraction equipment and could not support the tungsten tube characteristics without modifications to the APD controller. Thus, simultaneous measurements of oil and water saturations were not possible without major modifications and expenditures.

However, we decided to study the possibilities of determining water and the oil saturations sequentially using channel B. The use of only one channel would not allow for simultaneous measurements of both oil and water but should facilitate sequential measurements. The time lag involved would be the time needed for an additional reading (10 to 20 seconds).

Several sodium iodide concentrations, cell thicknesses, and x-ray tube potentials were tried. Results show that the x-ray beam cannot be differentiated into two distinct monochromatic beams, and the x-ray intensity transmitted through the filter is not strong enough to obtain accurate saturation measurements throughout the core. The use of varying tag concentrations in the brine and oil were also investigated.

Figure 4 shows the x-ray intensity through an iodine filter 0.75 in. thick and containing 90 percent NaI solution at an x-ray potential of 45 KV and 10 mA. This figure was generated by measuring the x-ray intensities at window widths of 1 unit increments. The K-absorption edge of iodine is evident around a window width of 52 percent. The resolution of the two peaks is limited. Various filter concentrations and thicknesses were tried to improve the resolution, but the improvement was not significant. This may be due to the detector having a gaussian distribution about a mean height proportional to the photon energy. Because of this overlap in the two peaks, the calibration may be nonlinear. Thus, by selecting window widths (48 to 51 and 53 to 55 percent) and measuring the x-ray intensities, oil and water saturations can be calculated using equations 3 and 4.

Using this technique (solving equations 3 and 4), the concentrations of sodium bromide and idododecane were calculated and compared with experimental values (known concentration in cells) while varying the window setting and baseline. The difference between calculated concentrations of iodine and bromine and experimental value were greater than 10 percent. Hence for the system studied, using the hardware available at NIPER, accurate measurements of oil and water concentrations sequentially by the x-ray technique is not feasible.

The use of a sodium iodide filter to resolve the x-ray beam into two monochromatic beams reduces the intensity of the x-ray by a factor of 100. Thus the signal to noise level increases. The noise level which is due to statistical fluctuation in the detector signal is the principal limitation on the sensitivity of the system. To increase the sensitivity of measurement, one must increase the total number of counts. Increasing the number of counts requires increasing the intensity of fluorescent radiation and the detector efficiency.

CONCLUSIONS AND RECOMMENDATIONS

- Simultaneous measurement of oil and water saturations is not possible without major modifications to available x-ray equipment. Consequently, the feasibility of measuring water and oil saturations sequentially was studied.
- The difference between calculated concentrations of iodine and bromine and the experimental values were greater than 10 percent. Hence, for the system studied, using the hardware present at NIPER, accurately measuring oil and water saturations sequentially by the x-ray technique is not feasible.
- The intensity can be increased by increasing the primary beam intensity, using a large current constant potential x-ray machine. A more practical and less expensive (\$15,000.00) solution is using a lanthanum target in the x-ray tube instead of tungsten. As shown in figure 5, lanthanum produces two high intensity fluorescent beams which have an energy spread of only 0.4 KeV, on either side of the iodine K-absorption edge. Thus, the lanthanum tube would not only allow for the measurement of oil and water saturations in a three-phase system but would also substantially increase the accuracy of measuring iodine concentration, which allows the oil saturation to be determined in a two-phase system.
- The detector efficiency can be increased either by using a detector with a very short resolving time, high resolution at low energy levels, or by using a current integrating detector. The lanthanum target option is the most promising.

REFERENCE

1. Oak, M. J. and R. Ehrlich. A New X-ray Absorption Method for Measurement of Three-phase Relative Permeability. Pres. at the Soc. Petrol. Eng. 60th Ann. Tech. Conf. and Exhibition. Las Vegas, NV, Sept. 22-25, 1985. SPE paper 14420.

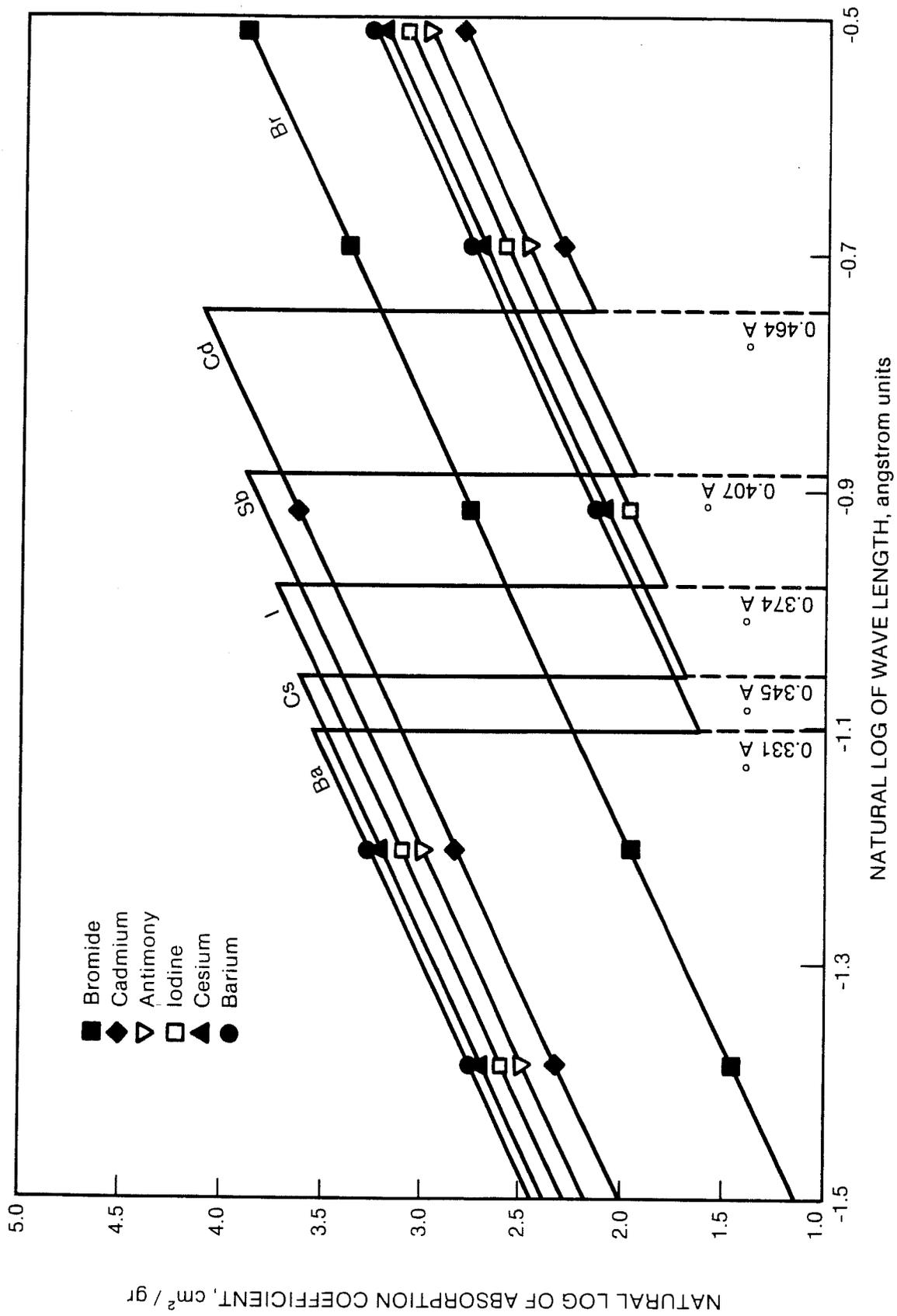


FIGURE 1. - Mass absorption of major components of tracers and filters.

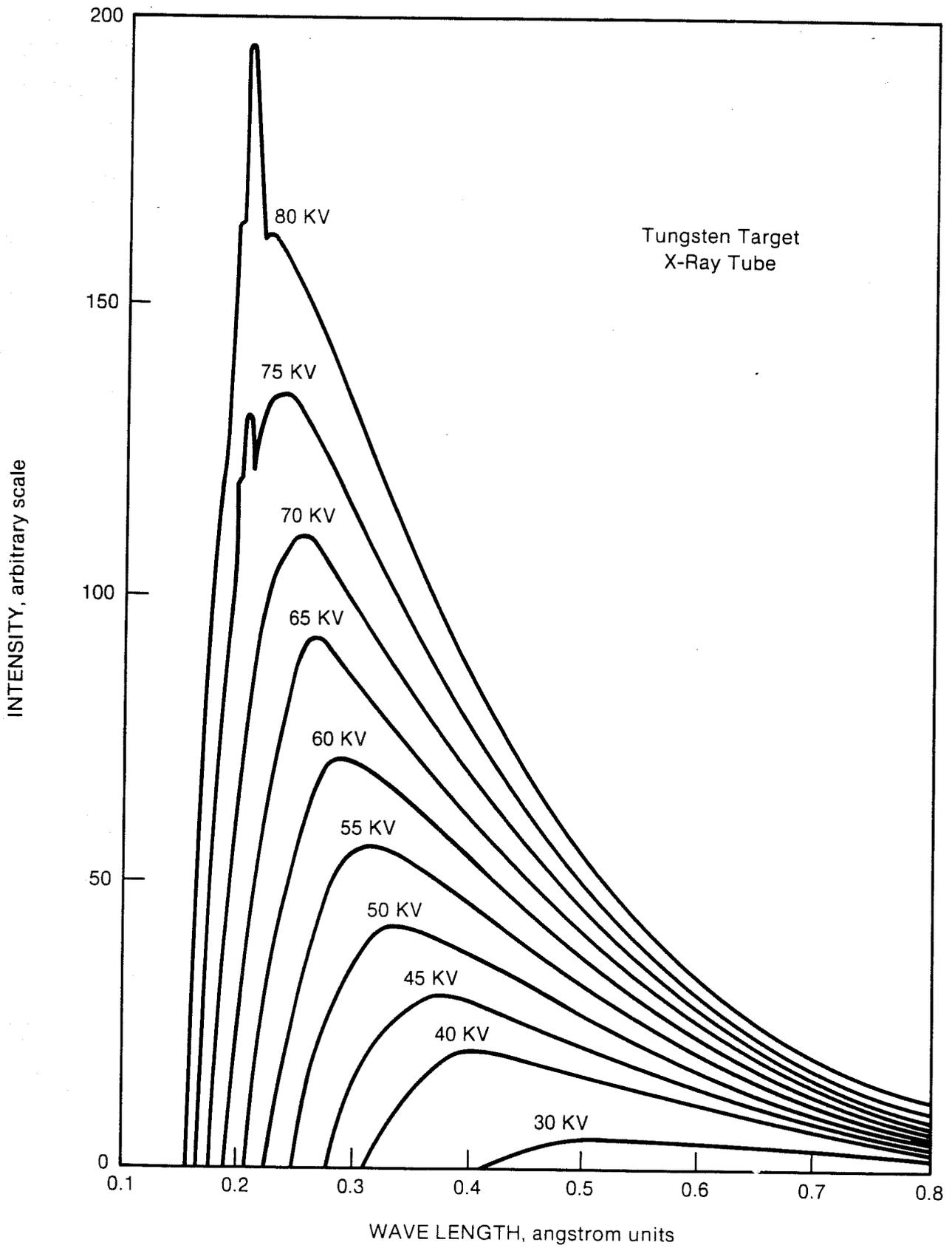


FIGURE 2. - X-ray spectra at different voltages.

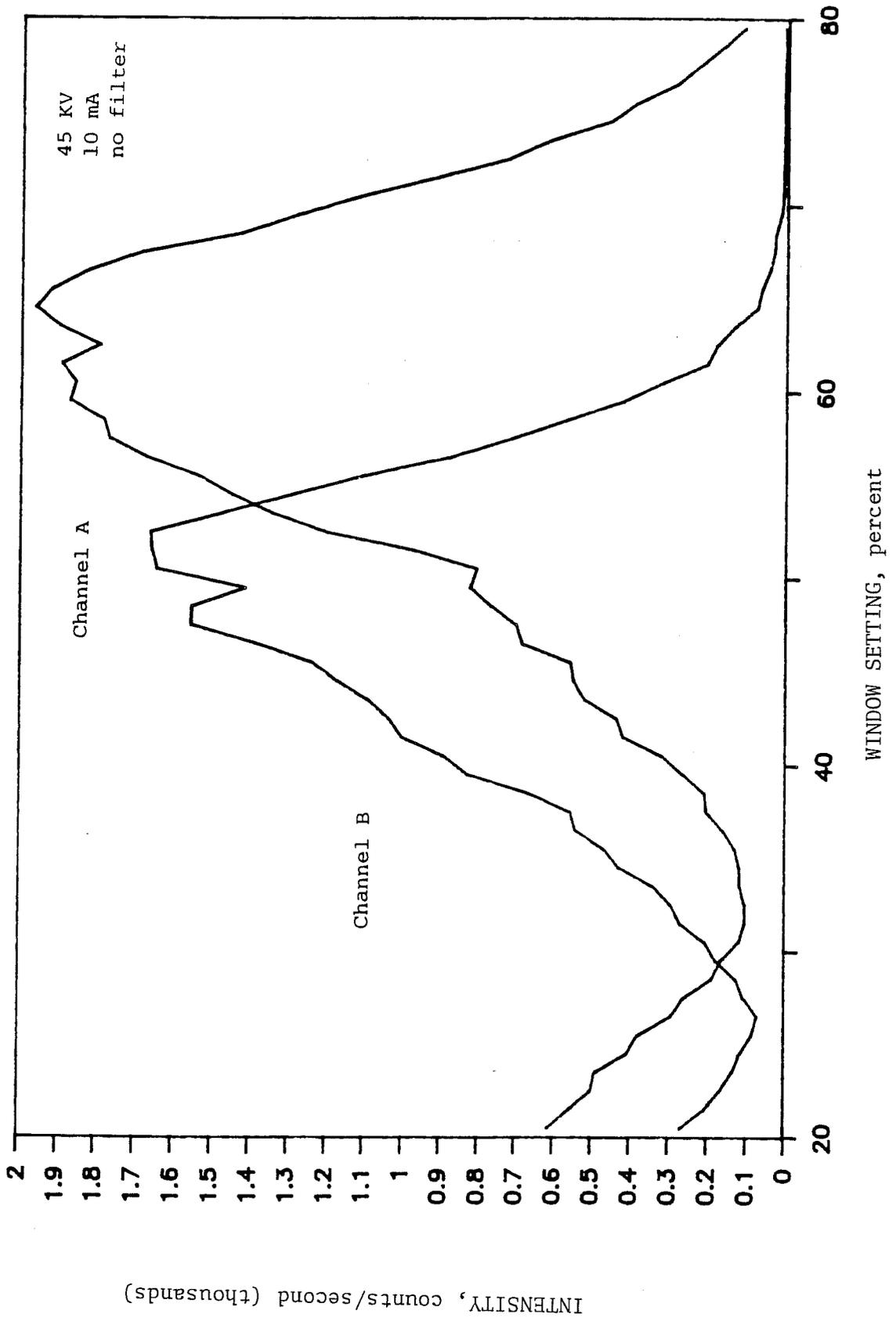


FIGURE 3. - Typical pulse height distribution curve for channels A and B.

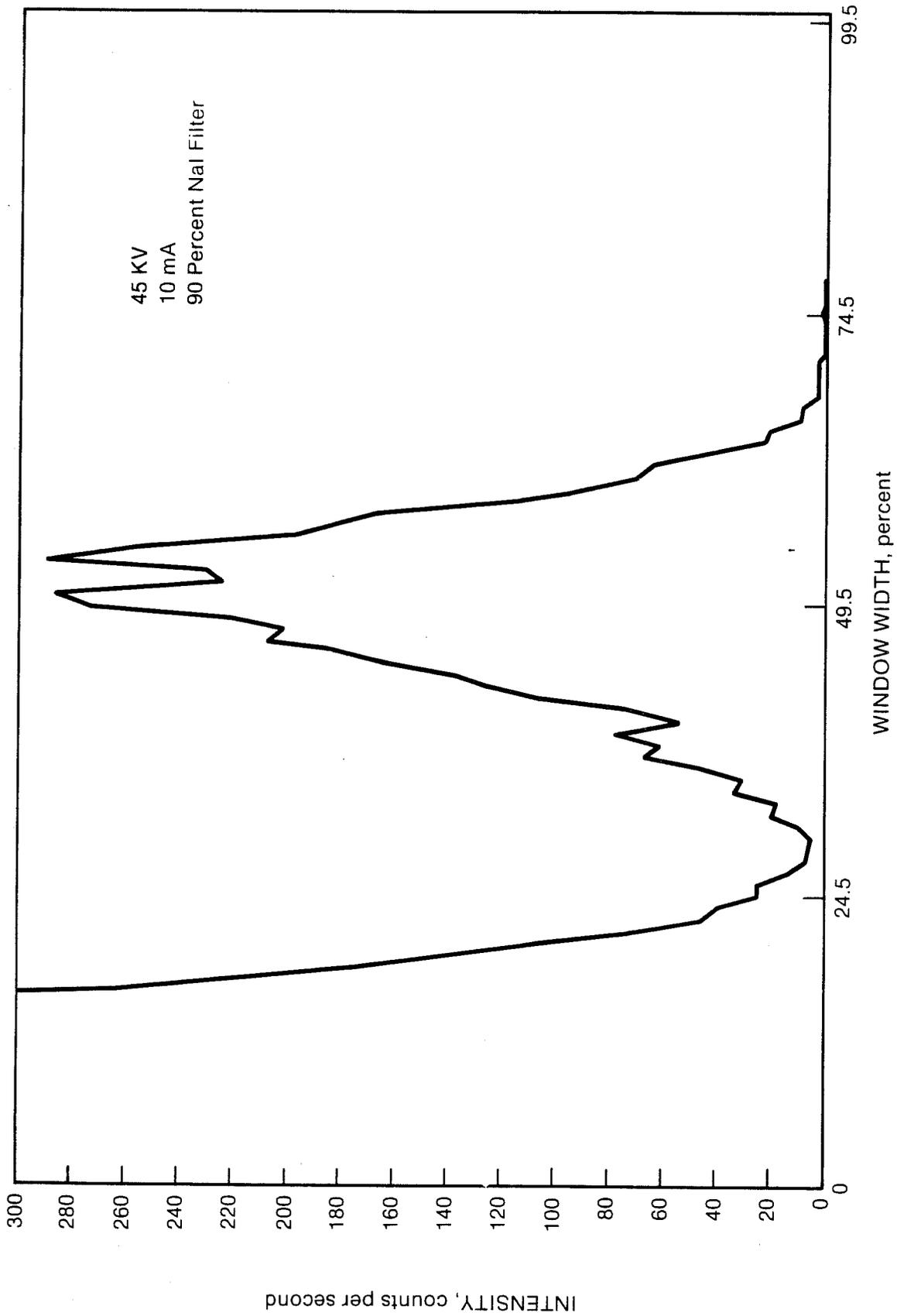


FIGURE 4. - X-ray spectra measured through a scintillation detector and a pulse height selector.

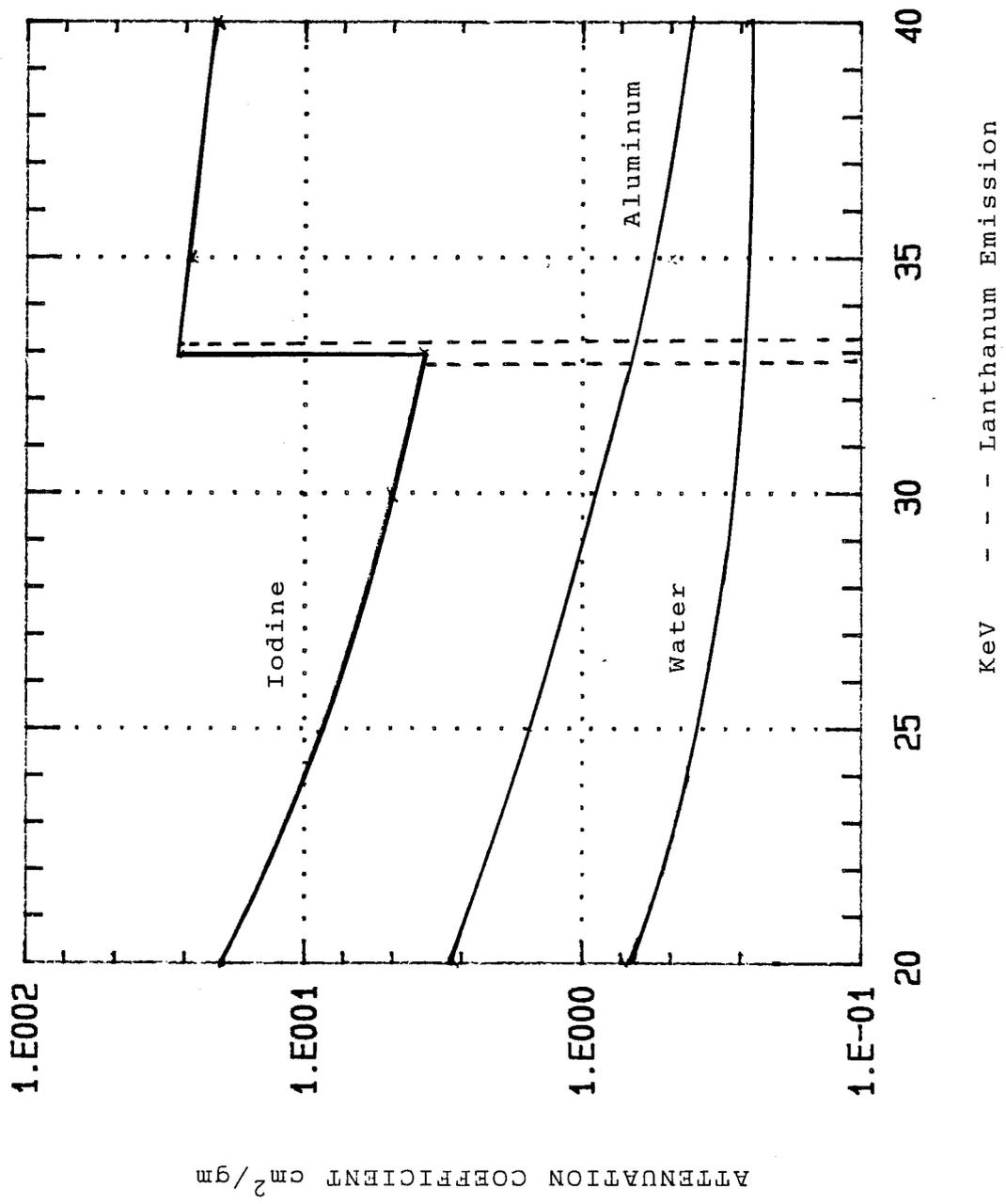


FIGURE 5. - Emission lines of iodine, gerium, and Lanthanum.