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INLINE INSTRUMENTATION:
GAMMA MONITOR, URANIUM COLORIMETER

J. W. Landry

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CHEMICAL TECHNOLOGY DIVISION
Process Design Section

INLINE INSTRUMENTATION: GAMMA MONITOR, URANIUM COLORIMETER

J. W. Landry

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ABSTRACT

Methods of applying gamma scintillation spectrometry and uranium colorimetry based on differential absorption of two wavelengths of light are described which are under development at ORNL for continuous analysis of radiochemical process plant streams. The instruments have features of automatic standardization and background subtraction.

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INTRODUCTION

This report describes a continuous gamma monitor and a continuous uranium-determination colorimeter that can be installed directly in process lines or in continuous recirculating sampling lines. Continuous measurements make possible early detection of process malfunction, and inline analysis decreases the possibility of radiation exposure of analytical and operating personnel and probably decreases costs. The gamma monitor was designed and tested at ORNL. The uranium colorimeter was developed at Savannah River and was tested and modified at ORNL. The work reported here was done by the author and T. S. Mackey of the Chemical Technology Division, R. W. Stelzner of the Analytical Chemistry Division, and M. J. Kelly of the Instrumentation and Controls Division.

GAMMA MONITORING

Three inline gamma monitors have been used in the ORNL pilot plants to record the gamma activity of process streams in counts per minute per milliliter. They can record up to five decades full scale. They record the integrated count rate of gamma activities above 0.05 Mev energy, but can also indicate specific energies for a very rough approximation of specific fission product contaminations.

One such instrument has been operating in the first-cycle organic product stream of the Power Reactor Fuel Processing Pilot Plant (Fig. 1). The scintillation spectrometer head puts out an electric current, which is measured on a recorder and is proportional to incident gamma radiation. The process stream flows up through the Fluorothene tube, the bore of which has been given a 10- μ in. finish to minimize the buildup of background radiation resulting from contamination of the tube by the process stream. An approximate background reading made with the tube drained is manually subtracted from the recorded reading to obtain the activity of the sample. The instrument is standardized by draining the tube and, by means of a solenoid, moving a Cs-137 capsule through a mercury-filled tube to a position in front of the detector. A shield minimizes extraneous radiation. In Fig. 1 the detector is positioned against the tube, which is the position for maximum sensitivity of the instrument. Usually the detector is positioned back from the tube and an absorber and collimator are placed between the detector and tube. A 1/8-in. lead absorber is used to stop beta and soft gamma radiation. The pulse height selector and voltage window of the spectrometer are set for the instrument to integrate the count rate for all gamma radiation of more than 0.05 Mev energy. A removable lead collimator, 1/8 in. i.d. by 2 in. long, is used to decrease background radiation that reaches the detector and to range the instrument. Results obtained with this instrument agree within a factor of 2 with results of sample analysis of the process stream. A scintillometer-spectrometer was selected because of its high sensitivity, gamma energy independence, and ability to discriminate against low-energy gamma radiation. It is not used for spectrometry, however, except for very rough indications. This instrument is similar to ones in successful use at the Hanford plant.

The method of functioning of the monitor is shown in Fig. 2. The detector sees both radiation from the sample of process stream in front of it and the background radiation. This background radiation is from contaminated process equipment and contents, from the contaminated stainless steel pipe connections of the monitor,

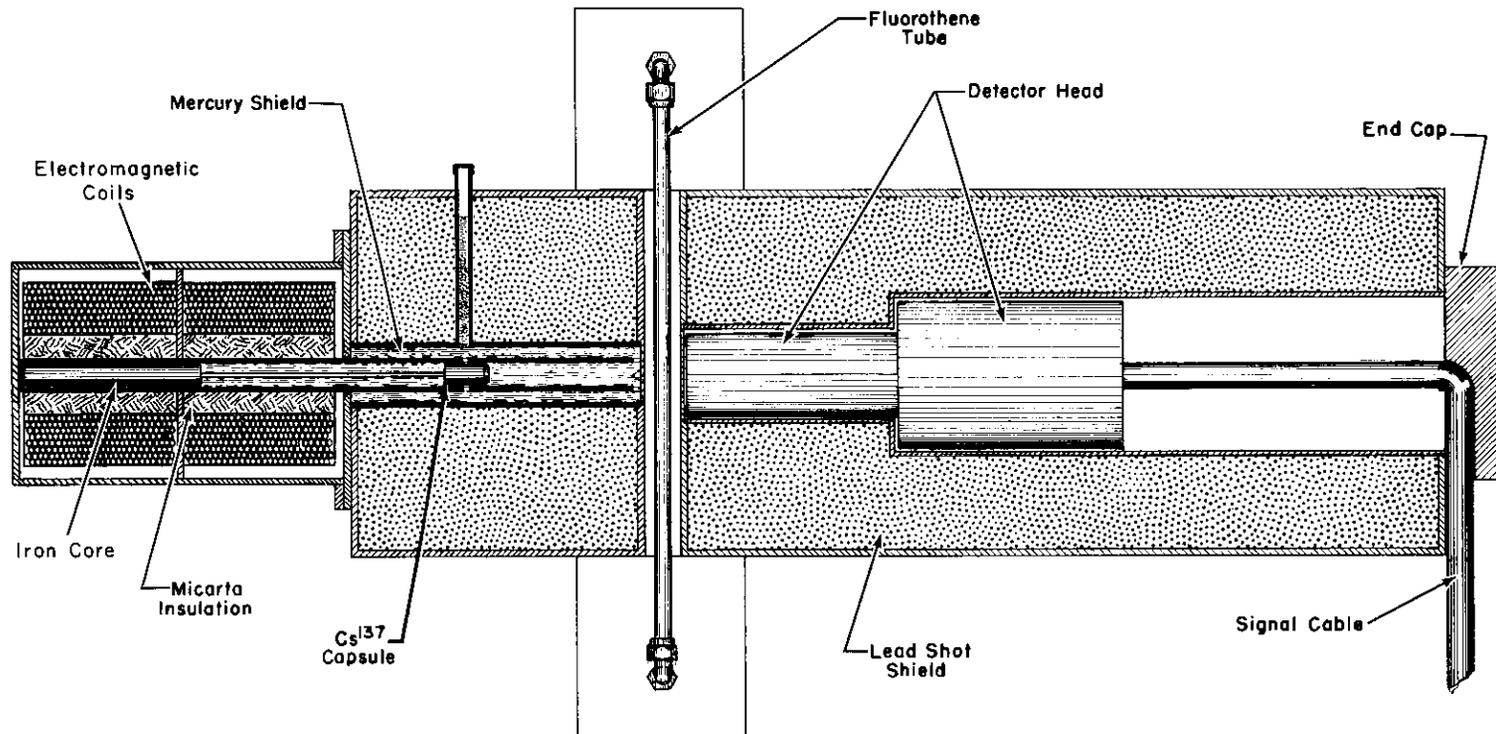


Fig. 1. Inline gamma monitor.

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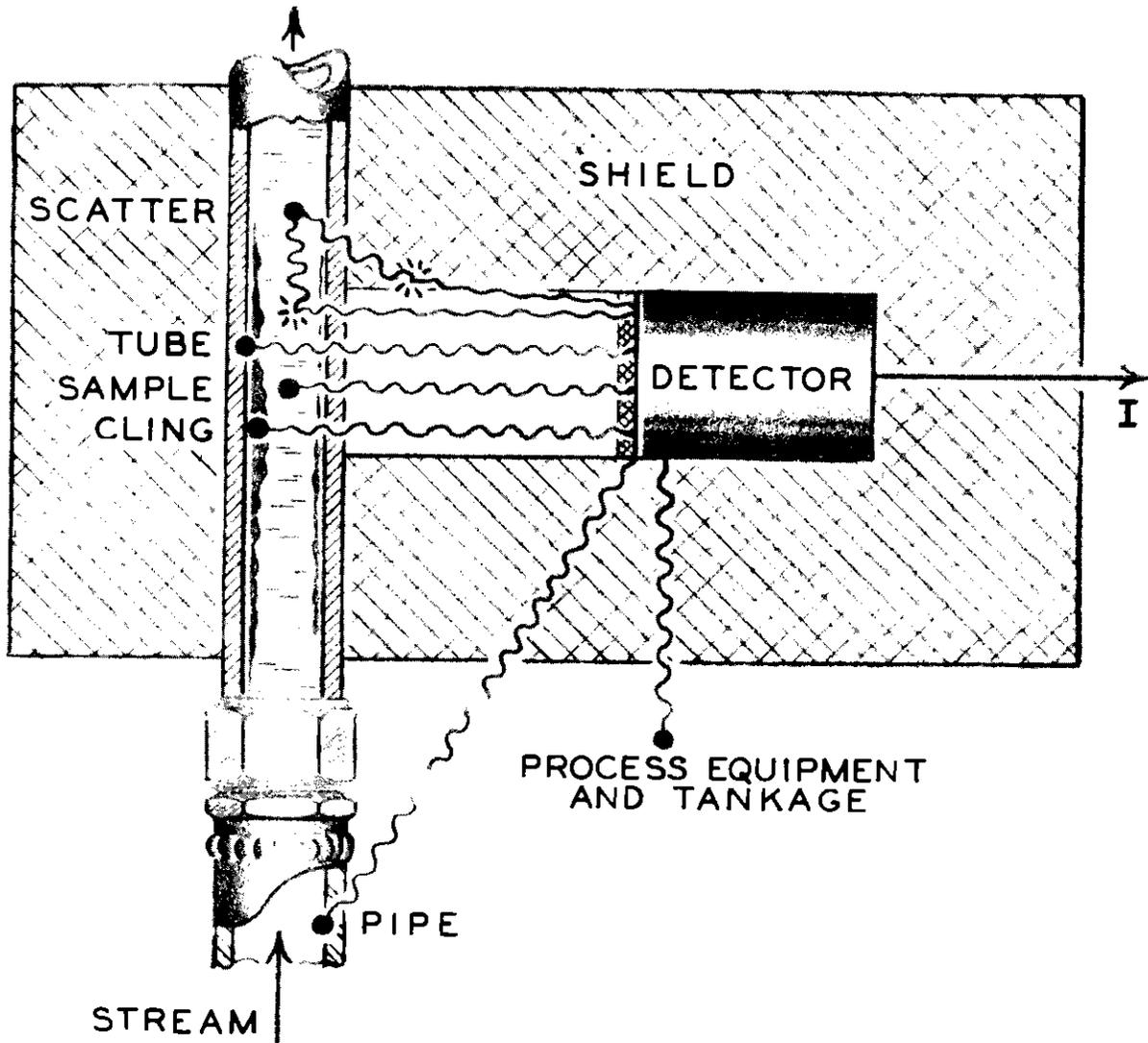


Fig. 2. Method of functioning of inline gamma monitor.

from the contaminated bore of the Fluorothene tube, from the film of liquid that remains in the tube when the tube is drained, and from scatter radiation produced when gamma photons collide with matter.

A major fraction of the scatter radiation originates in the stream above and below the sample because of proximity to the detector. Unfortunately, the stream is drained from the tube in making the background reading so that this scatter radiation is not included in the background reading and subtraction. Scatter radiation consists of degraded photons, which are impossible to distinguish from low-energy gamma emissions such as those from ruthenium. The inability of the instrument to separate this scatter radiation from the sample radiation prevents taking full advantage of the ability of the spectrometer to discriminate.

A gamma spectrogram from this instrument is shown in Fig. 3. A fair indication is obtained of the zirconium and niobium concentration of the stream (a), but the ruthenium peak (b) is obscured in the background (c). It is believed that if the scatter radiation can be separated from the sample radiation, a more significant spectrogram may be obtained. The constituents of background radiation mentioned above are variable.

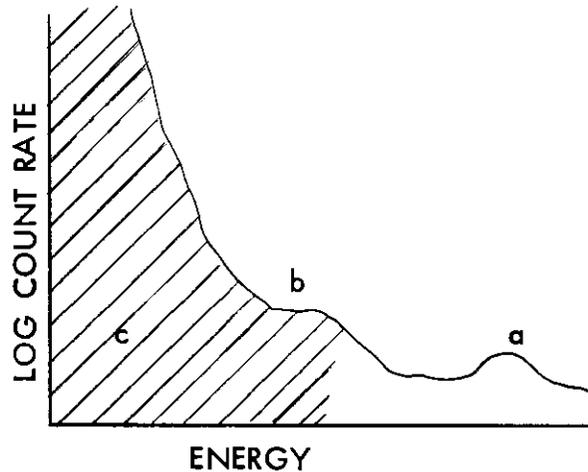


Fig. 3. Gamma spectrogram from inline gamma monitor. (a) Zirconium-niobium peak. (b) Ruthenium peak.. (c) Background.

An example of the variation, with respect to time, of the radiation seen by the gamma detector is shown in Fig. 4. The detector output is an electric current, ΣI , proportional to the incident radiation. It is simple to shield against the detector seeing much radiation from surrounding process equipment. Hence process equipment produces only a small fraction (I equipment) of the detector current. It is difficult to shield against the radiation from the piping (I pipe), which is near the openings in the detector shield. It is impossible to shield against the radiation from the tube (I tube) because shielding would obstruct the radiation from the sample as well. Although the smooth Fluorothene tube becomes contaminated only slightly compared with the stainless steel piping and process equipment, the tube contamination produces a larger fraction of the detector current because it is in the direct view of the detector. The contamination in the process equipment, piping, and tube increases steadily with time. Radioactive process solution accumulating in nearby catch tanks, which are emptied periodically, produces the tankage current (I tankage). A detector current, called "dark current," is produced even when there is no radiation because of the multiplier phototube characteristics of the detector. This dark current (I dark) is variable. The current produced by the radioactivity of the process stream (I sample) varies (top current in Fig. 4). The scatter and cling radiations (I scatter, I cling) also originate from the process stream and therefore vary similarly. The bottom plot of Fig. 4 shows how these detector currents total. The radioactivity of the sample, which corresponds to the height of the shaded band, is determined by making a gross reading of radioactivity and subtracting the background radioactivity reading, which corresponds to the height of the light area below this band. Since the background radioactivity is variable, background readings are made at least daily. A factor is assumed for scatter. The scatter factor requires repeated calibration because it depends on the energy distribution of the radiation, and this varies with time in process streams.

An experimental version of a continuous gamma monitor is shown in Fig. 5. A plant model has not been made, but the principles have been investigated with preliminary models. The process stream flows through, and fills, the tube and bellows. The bellows is surrounded by hydraulic fluid, which is connected by a transmission line to a 1-cycle/sec sinusoid pulser. The bellows is moved from its position on the left as shown in the drawing to the dotted line on the right, thus changing the stream volume by the amount shown by the arrow. The detector current (Fig. 6) changes accordingly. The background currents (a) are for equipment piping, tube, tankage, dark current, cling, and scatter.

The detector current is fed to a condenser and rectifier where only the a-c component passes and is recorded in terms of counts per minute per milliliter. The only radiation thus measured is from the dynamic volume of the stream. This volume is constant and constitutes the sample. All other radiation constitutes background. The background radiation is static over a period of 1 sec and propagates the d-c current. The d-c current can be measured separately for an exact measurement of background radiation. Although background is excluded in the primary record of the monitor, the provision is necessary for following background buildup because an excess can jam the detector.

Periodically the hydraulic pulse is valved so that the bellows is static and the Cs-137 capsule, entering and leaving the counting chamber via a mercury well,

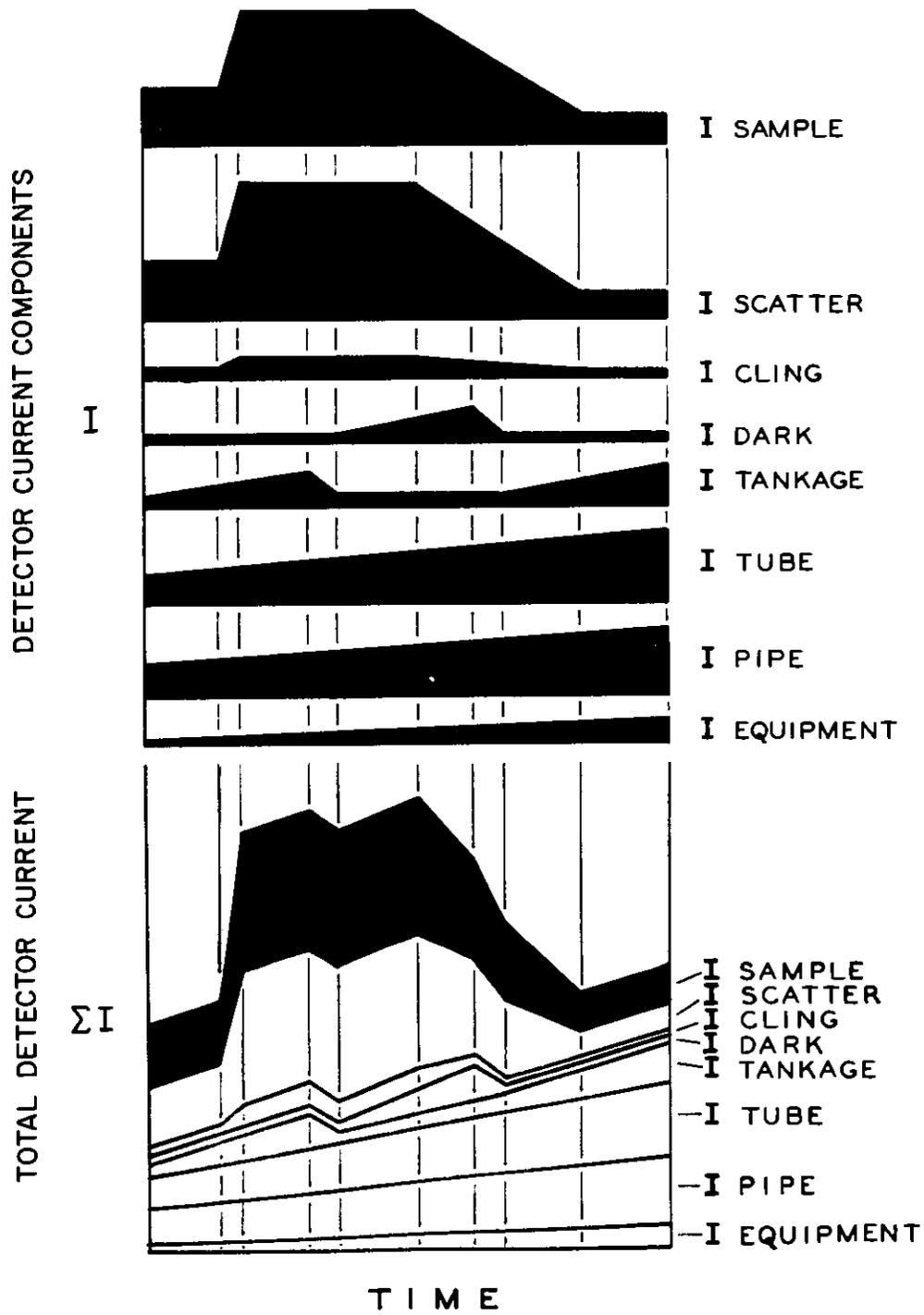


Fig. 4. Detector current with respect to time from inline gamma monitor.

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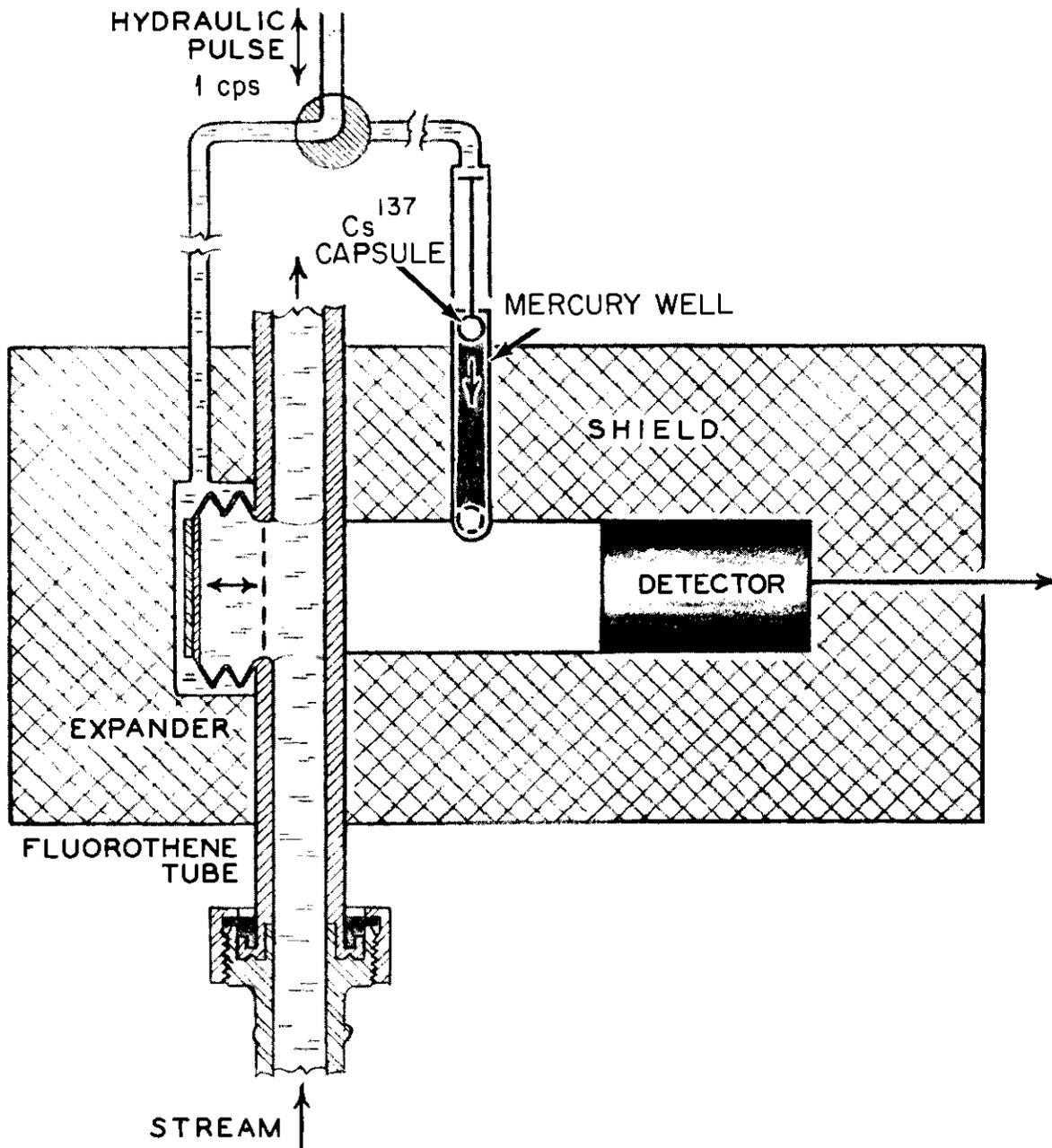


Fig. 5. Experimental inline gamma monitor.

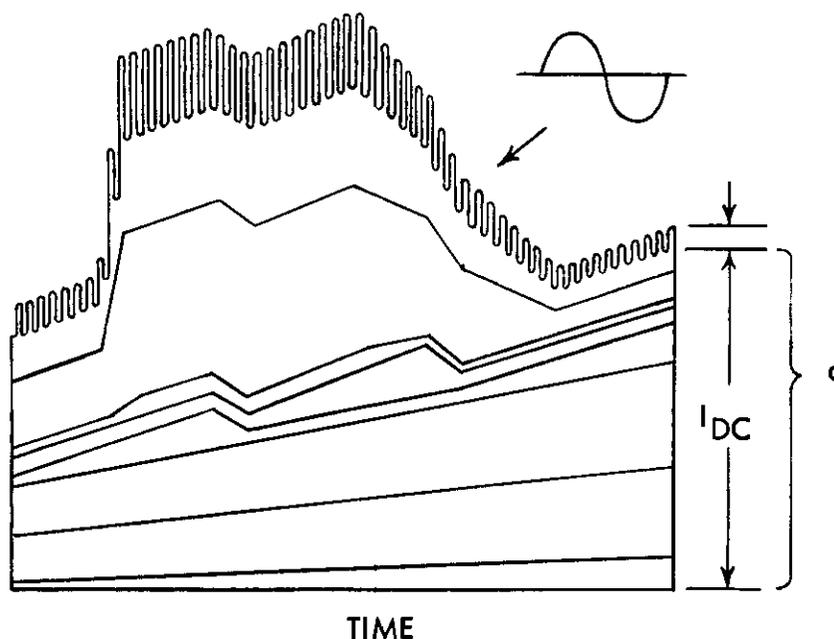


Fig. 6. Detector current from experimental inline gamma monitor, 60 cycles per minute.

pulses radiation to the detector. The resulting a-c current, a background-free standardization measurement, is fed to an amplifier. The output voltage of the amplifier is proportional to the count rate of the standard, either directly or logarithmically depending on the amplifier, and is fed to a Brown recorder which has been modified to be a ratio recorder. The voltage from measuring the standard is applied to the slidewire so that the measured count rate of the standard reads 100% scale on the recorder. The standard is selected to have approximately the same count rate as the flowsheet stream so that standardization is effected under approximately the same conditions as stream measurement.

The voltage from measuring the process stream is found, by the recorder, on the slidewire so that the record is in terms of counts per minute per milliliter referred to the standard. The voltage is divided, by a manually adjustable resistor, so that the process stream reading is positioned on the recorder scale and chart with space to deviate in either direction as desired. This adjustment is provided also so that the reading can be made to register exactly with the chart calibrations when there is variation in supplies of chart paper. When necessary, the instrument can be ranged with the adjustable resistor until the standard can be changed.

A logarithmic amplifier is preferred in pilot plant operations because it extends the range of the recorder, but the standardization method will apply for a linear amplifier as well. Standardization is made on a periodic basis which is adequate, although the instrument could be standardized continuously by pulsing the sample and standard simultaneously but at different frequencies. The two measurements would be separated on the basis of frequency.

The experimental version of the gamma monitor may have three advantages over existing plant models:

1. It provides automatic and continuous background subtraction.
2. It subtracts the background which results from scatter radiation.
3. It requires no draining or rinsing operation for background measurement and therefore eliminates the valve and bypass arrangements.

This instrument is primarily a monitor of gross gamma activity. However, with its improved background control, it might provide significant gamma spectrograms of high-activity process streams such as the first-cycle product streams. A laboratory model of this instrument operated well when set to measure integrated detector current (i.e. gross gamma activity) and did not discriminate on other than streams of very high activity. Its dynamic response to decreased count rates was incompatible with 1-cycle/sec sinusoidal presentation of the radiation. When the 1-cycle/sec sinusoid pulser was replaced with a 2-cycle/min square wave pulser, the instrument provided a good spectrogram of a low-activity source. In a plant model of this revised spectrometer, automatic standardization and background subtraction could be provided by replacing the condenser and rectifier with four condensers and a switch. In this arrangement the detector current is fed to an amplifier whose output is a voltage proportional to the count rate. The voltage can be stored on a condenser.

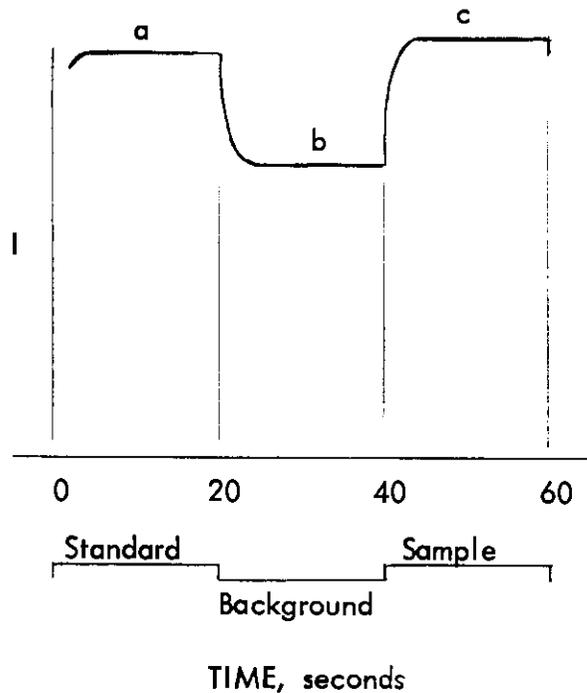


Fig. 7. Detector current from experimental inline gamma monitor, 1 cycle per minute.

Figure 7 shows a 1-min square wave cycle for the inline spectrometer shown in Fig. 5. With the bellows contracted the standard is positioned before the detector and counted for 15 sec (a), 5 sec being allowed always for preliminary equilibrium. The count rate is stored as voltage on a condenser. Then the standard is withdrawn for 20 sec to make a 15-sec background count (b), which is stored on each of two condensers. For the final 15 sec the bellows is expanded and the sample count is made (c) and stored on the remaining condenser. The two condensers with the background count are switched with the other two condensers to make the background subtractions. The difference voltages are fed to a modified Brown recorder as described previously. A standardized background-free reading in counts per minute per milliliter is recorded.

Figure 8 shows the gamma monitor circuit diagram. When the switch positions 1 are closed, the detector is measuring standard plus background and the recorder is recording the stream gamma activity. When the switches are closed in positions 2, the detector is measuring background and the recorder is zeroing. When positions 3 are closed, the detector is measuring sample plus background and the recorder is standardizing. The background subtraction removes all detector current except what originates from the sample radiation. This automatically takes care of the zero adjustment in the detector and preamplifier. Zero adjustments in the amplifier and recorder are made with a counter-potential adjustment, which is driven by the recorder while the amplifier is at zero input. The proportionality adjustment for the detector, preamplifier, amplifier, and recorder is made by the standardization. The spectrometer window setting is switched to a specific gamma energy band during the standardization operation. The activity of the process stream is recorded in counts per minute per milliliter.

URANIUM COLORIMETRY

Figure 9 shows an inline uranium colorimeter developed by the Savannah River Laboratory inline instrumentation group and, at their request, tested in the ORNL Metal Recovery Plant for one year. Their instrument is described in DP-229. It continuously measured uranium concentrations in the range 45-55 g/liter in a flowing aqueous stream with an accuracy within ± 1 g/liter based on analysis of samples of the stream that were drawn at the point indicated on the figure. This instrument can be adjusted to measure uranium concentration between 1 and 350 g/liter. The instrument is regarded, after the year of operation, as a good plant instrument.

The SRL inline colorimeter makes use of the yellow color of uranyl nitrate. Most of the current chemical processes for recovering uranium use nitrate systems in which the uranium is present as the uranyl ion. Uranyl solutions viewed in white light appear increasingly yellow, with increasing uranyl concentration, due to the property of uranyl to transmit yellow light and absorb blue light. Absorption is maximum at a wavelength of 416 millimicrons. The instrument contains a light source, filter, and glass-windowed cell in which the light can be passed through the process stream (Fig. 10). The filter, which makes a monochromatic source of light for the wavelength that is strongly absorbed by uranyl, is an interference filter or a grating replica. These provide a narrow passband. Since only the 416-millimicron wavelength is needed for uranyl measurement, the narrow passband eliminates other wavelengths and thus minimizes possible interference from other colored ions in the process stream.

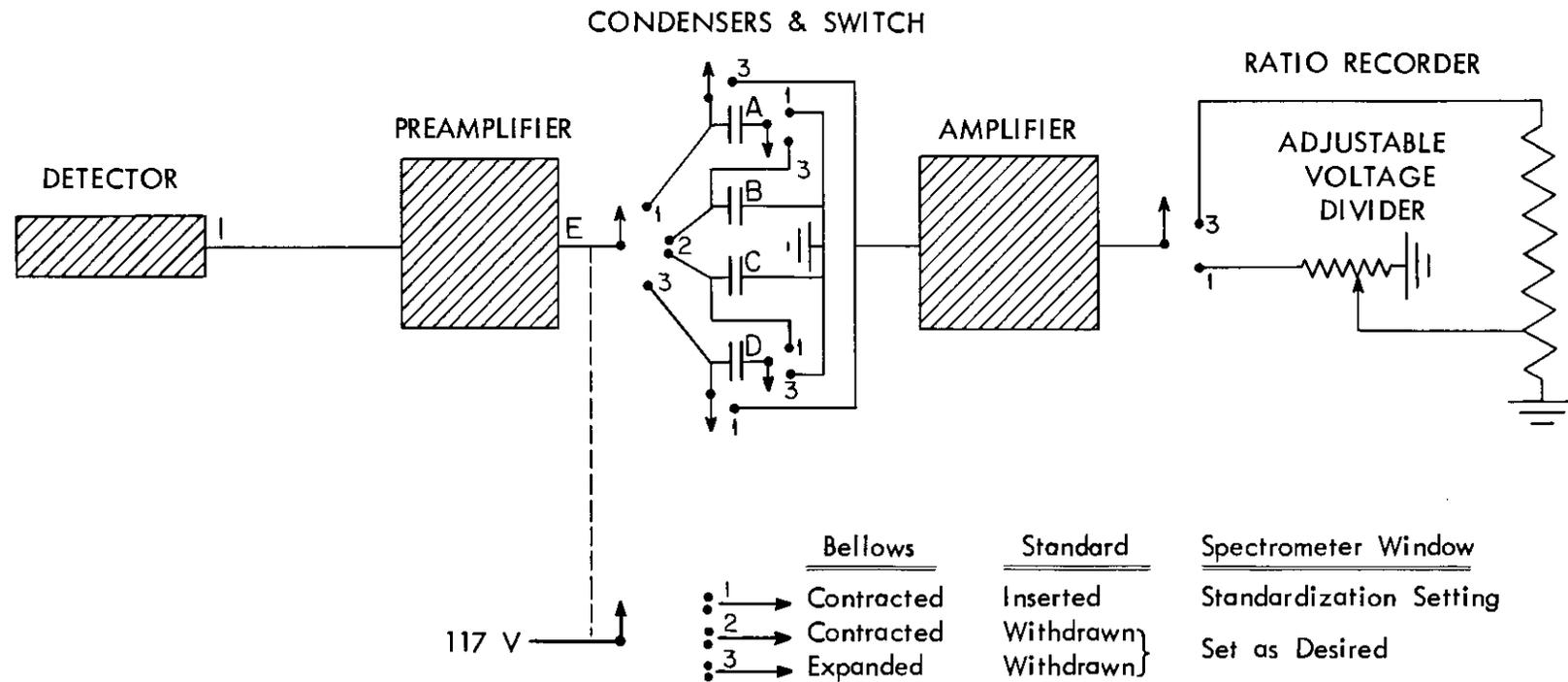


Fig. 8. Circuit diagram for experimental inline gamma monitor, 1 cycle per min. Switch is three-position, clock driven. Condensers: A - Standard + background; B - Background; D - Stream + background.

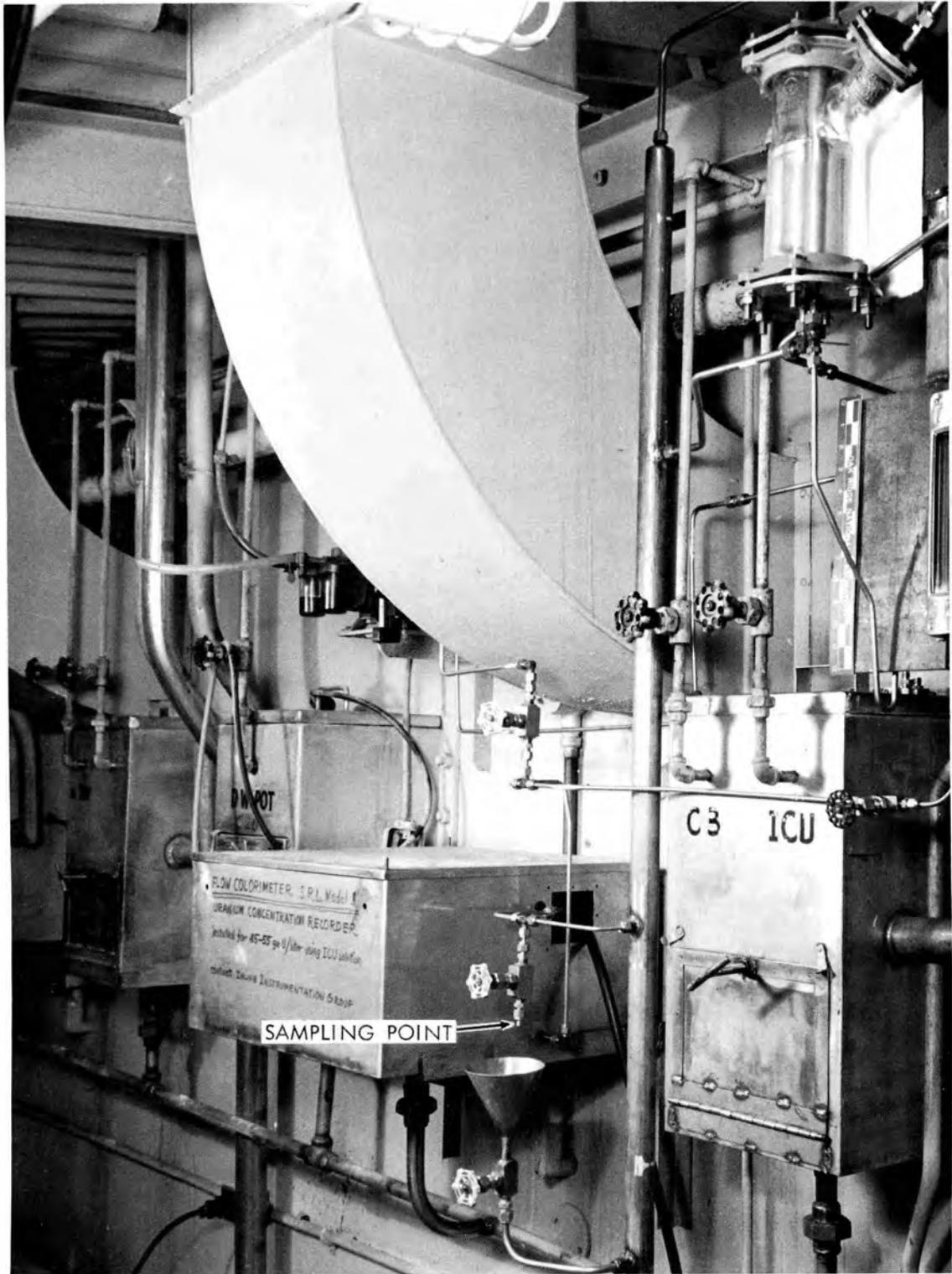


Fig. 9. SRL inline colorimeter.

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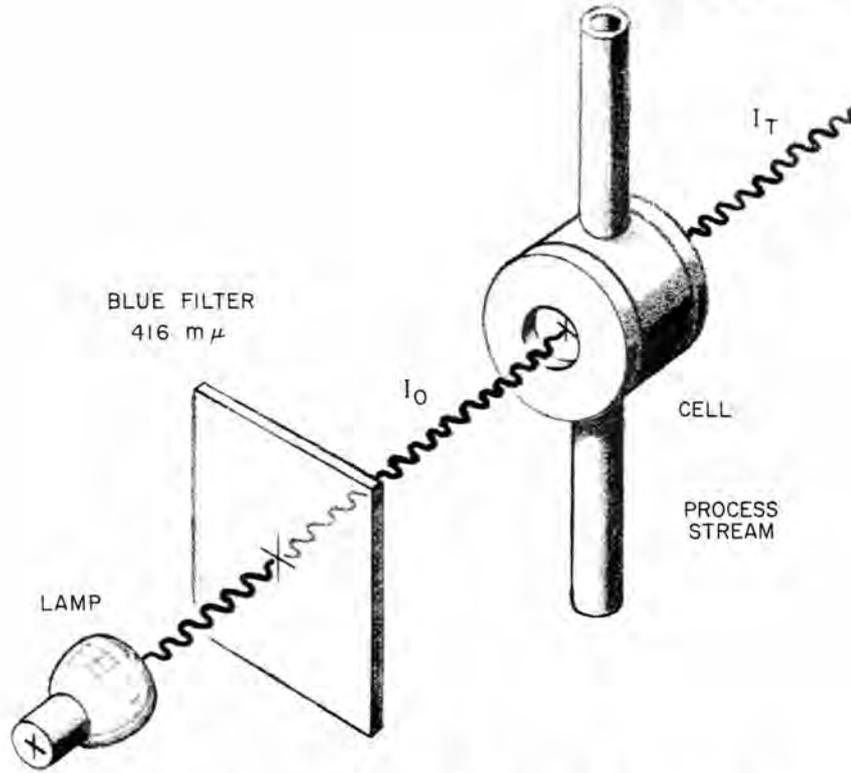


Fig. 10. Principle of SRL inline colorimeter.

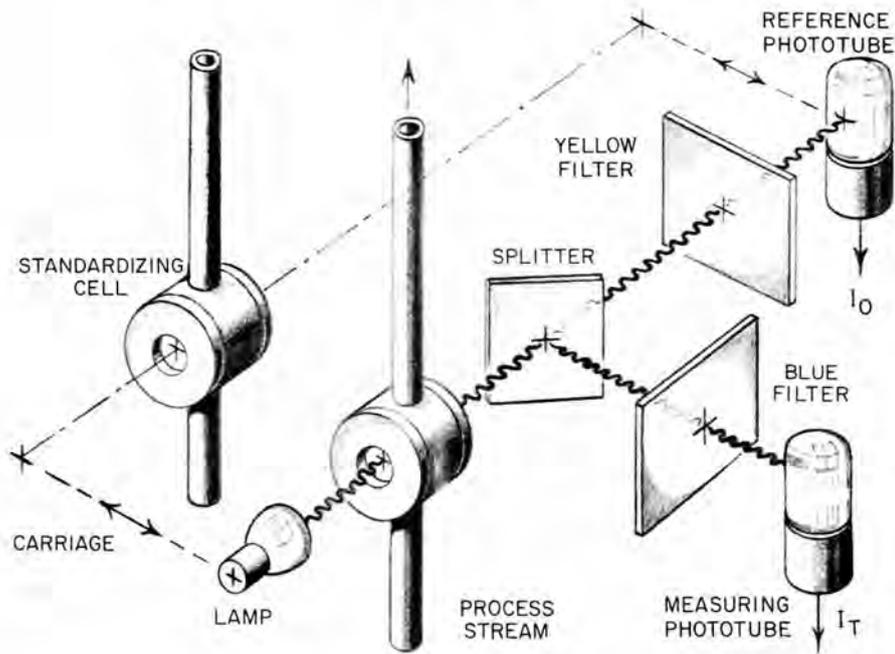


Fig. 11. Sketch of SRL inline colorimeter.

For monochromatic light the relation between the intensities of the incident and transmitted light is:

$$I_T = I_0 e^{-\mu CL} \quad (\text{Beer's law}) \quad (1)$$

where I_T is the intensity of the transmitted light, I_0 is the intensity of the incident light, μ is a constant depending on the ion species, C is the ion concentration, and L , the path-length of the light through the solution, is constant for a given system. The concentration is therefore equal to a calibration constant, K , times the logarithm of the ratio of intensities of incident blue light to transmitted blue light:

$$\text{Conc} = K \ln(I_0/I_T) = K(\ln I_0 - \ln I_T) \quad (2)$$

Practical limitations in applying Beer's law to inline colorimetry are darkening of the lamp, dust on the filter, dust and condensate on the exterior of the cell windows, deposits from the process stream on the interior of the cell windows, radiation-darkened filter and cell windows, reflection by the surfaces of the cell windows, suspensoids in the process stream, dust on the phototube used to measure the intensity of the transmitted light, and variable response of the phototube. These extraneous effects result in a sizable and variable reduction in the incident blue light and in the measured, transmitted blue light; this reduction has no relation to the uranium concentration of the process stream.

To properly determine their ratio the incident, as well as the transmitted, blue light must be measured. The incident blue light is measured in a way that circumvents the practical limitations of Beer's law. In effect this is done by measuring the intensity of the transmitted blue light with the darkened bulb, the dusty filter and cell windows, the cloudy process stream, the reflections, and everything else included in the light path except the uranium. This measurement is made on the process stream by using yellow light whose intensity at the source is proportional to that of the blue light and to which these extraneous effects are opaque but to which uranyl is transparent. These extraneous effects have been found to be gray effects; they will decrease the intensity of light but will not affect the proportion of blue and yellow light.

If the blue filter is alternated with a yellow filter, the intensity of the light transmitted by the blue filter will be equal to the intensity of the light transmitted by the yellow filter multiplied by a factor. The factor is practically constant and is the product of two ratios: the ratio of blue to yellow light in the lamp output and the ratio of the filter efficiencies. The uranyl ion is transparent to yellow light of 543 millimicrons wavelength. The intensity of the yellow light transmitted by the process stream is the same whether or not uranyl is present. This intensity, when multiplied by the factor previously defined, would be the intensity of the blue light transmitted by the process stream in the absence of uranyl ion. Thus, for inline colorimetry, I_0 in eqs. 1 and 2 is the measured intensity of the transmitted blue light for zero uranium concentration in the process stream and the extraneous, or gray, effects are avoided. Since the phototube current is directly proportional to light intensity, I_0 and I_T stand for phototube currents as well as light intensities in eqs. 1 and 2.

Figure 11 shows how Beer's law is applied in the SRL inline colorimeter. Light transmitted through the process stream is split equally by a semigilded mirror and directed to two filters. The intensities of the blue (416 millimicrons wavelength) and yellow (543 millimicrons wavelength) transmitted light are measured by two phototubes, and the phototube currents are fed to two linear amplifiers. The lamp, splitter, filters, and phototubes are mounted on a movable carriage. Periodically the carriage is moved manually to a standardizing cell, which is filled with a static sample of the process stream made up to standard flowsheet concentration.

With the carriage in the standardizing position the output voltages of the two amplifiers are adjusted to be equal, the condition for which the recorder prints the uranium concentration of the standardizing solution. With the carriage positioned on the product stream any difference in uranium concentration between the product stream and the standardizing solution results in a differential voltage between the amplifiers. This voltage is recorded in terms of grams of uranium per liter. A strong source of light is used so that the dark current effects from the phototubes are negligible. Dark current is about 0.05 microamp compared with a reading current of about 20 microamp. Since the instrument is standardized at mid-range, the nonlinearity effects on its accuracy are not serious over the range 40-50 g/liter, the range in which it was operated in the Metal Recovery Plant.

A single-channel dual-beam inline colorimeter was designed by the ORNL Inline Instrumentation Group. The basic principle (Fig. 12) was investigated with a model and found usable. The yellow and blue filters are alternated in a wheel which rotates at 3600 rpm. The output current of the phototube alternates accordingly.

The output current of the phototube is a square wave function (Fig. 13). When the yellow filter is transmitting, the phototube current is I_0 . When the blue filter is transmitting, the phototube current is I_T . The phototube current is fed to a logarithmic amplifier whose output is a voltage proportional to the logarithm of the phototube current:

$$E = B \ln I$$

where B is the proportionality factor. The amplifier voltage also will be a square wave function. When the yellow filter is transmitting the voltage is proportional to the logarithm of I_0 . When the blue filter is transmitting, the voltage is proportional to the logarithm of I_T . The amplifier voltage can be fed to a suitable condenser and rectifier circuit and the a-c voltage component can be separated and measured. The measured voltage is equal to half the difference in these voltages:

$$B \ln I_0 - B \ln I_T = 2 E_{ac} \quad (3)$$

Combining eqs. 2 and 3 shows that the concentration is equal to the measured voltage times a constant:

$$\text{Conc} = K \frac{2}{B} E_{ac} = K' E_{ac}$$

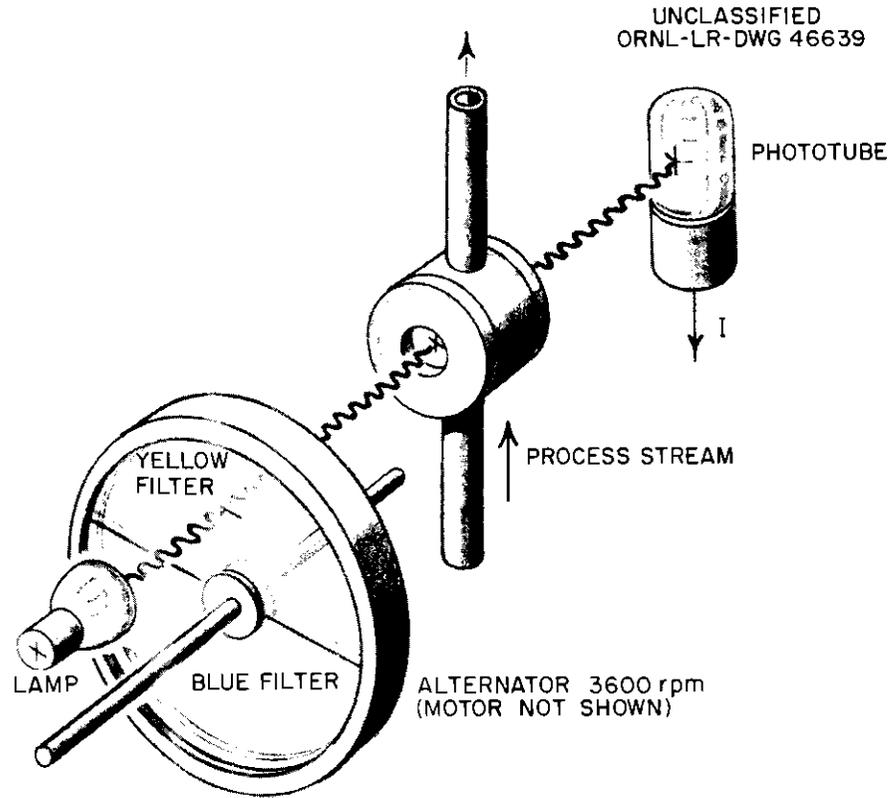


Fig. 12. Sketch of experimental single-channel dual-beam inline colorimeter.

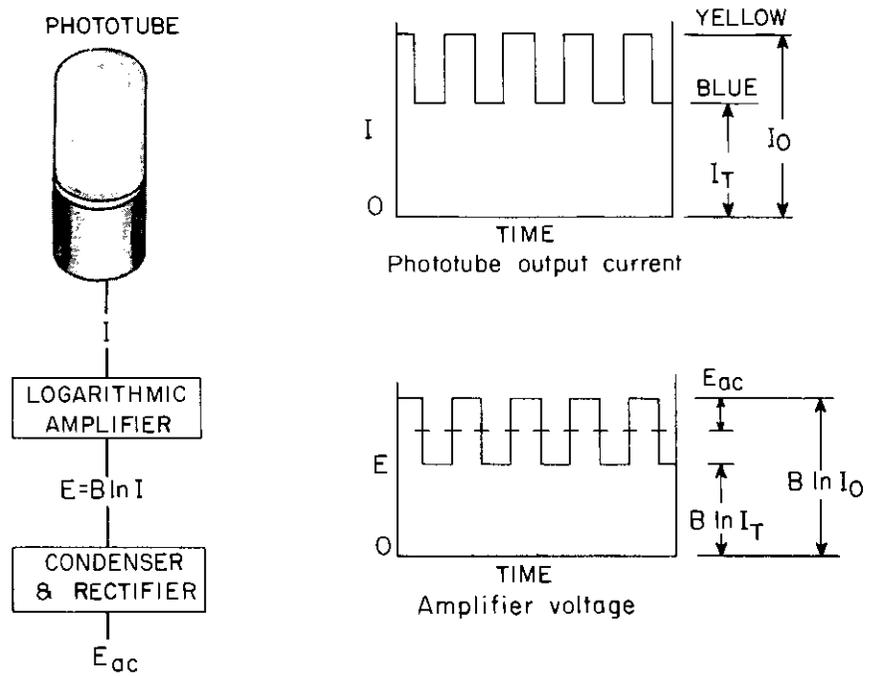


Fig. 13. Readout of experimental single-channel dual-beam inline colorimeter.

This constant is determined once when the instrument is new (Fig. 10) by filling the cell with a standardizing solution.

Advantages of this instrument over the first described one are: it uses a single phototube and amplifier, thereby eliminating imbalance between two channels of phototubes and amplifiers. Frequent calibration is thus probably no longer necessary and the movable carriage and second cell probably can be eliminated. Various components also become unnecessary. The logarithmic response of the instrument follows Beer's law and a linear chart can be used over any range. The a-c amplifier is more stable than the d-c amplifiers.

In another version of the colorimeter (Fig. 14) the lamp, filters, and phototube can be moved from the cell containing the process stream to the cell containing a standardizing solution. This allows the instrument to be checked against a standard of known concentration, thereby providing operator confidence. It also makes the instrument capable of calibrating itself at any time. This feature would be useful in case variable characteristics of the bulb, filter, cell window, and phototube changed the ratio of yellow and blue lights. This might occur, for instance, in the presence of high level radiation fields. To avoid temperature difference effects between the stream cell and the standardizing cell the standardizing cell might be jacketed with the solution leaving the stream cell.

In this colorimeter arrangement (Fig. 14) the concentration of the stream is taken as the reading of the stream times a calibration factor:

$$\text{Conc of stream} = \text{reading of stream} \times \frac{\text{conc of standard}}{\text{reading of standard}}$$

By Beer's law,

$$\begin{aligned} \text{Conc of stream} &= \frac{K \ln \frac{I_0 \text{ stream}}{I_T \text{ stream}}}{K \ln \frac{I_0 \text{ standard}}{I_T \text{ standard}}} \text{ conc of standard} \\ &= \frac{(\ln I_0 - \ln I_T) \text{ stream}}{(\ln I_0 - \ln I_T) \text{ standard}} \text{ conc of standard} \end{aligned} \quad (4)$$

The phototube current is handled the same as explained in Fig. 13 so that again the voltage

$$2 E_{ac} = B \ln I_0 - B \ln I_T \quad (5)$$

Combining eqs. 4 and 5,

$$\text{Conc of stream} = \frac{E_{ac} \text{ stream}}{E_{ac} \text{ standard}} \text{ conc of standard}$$

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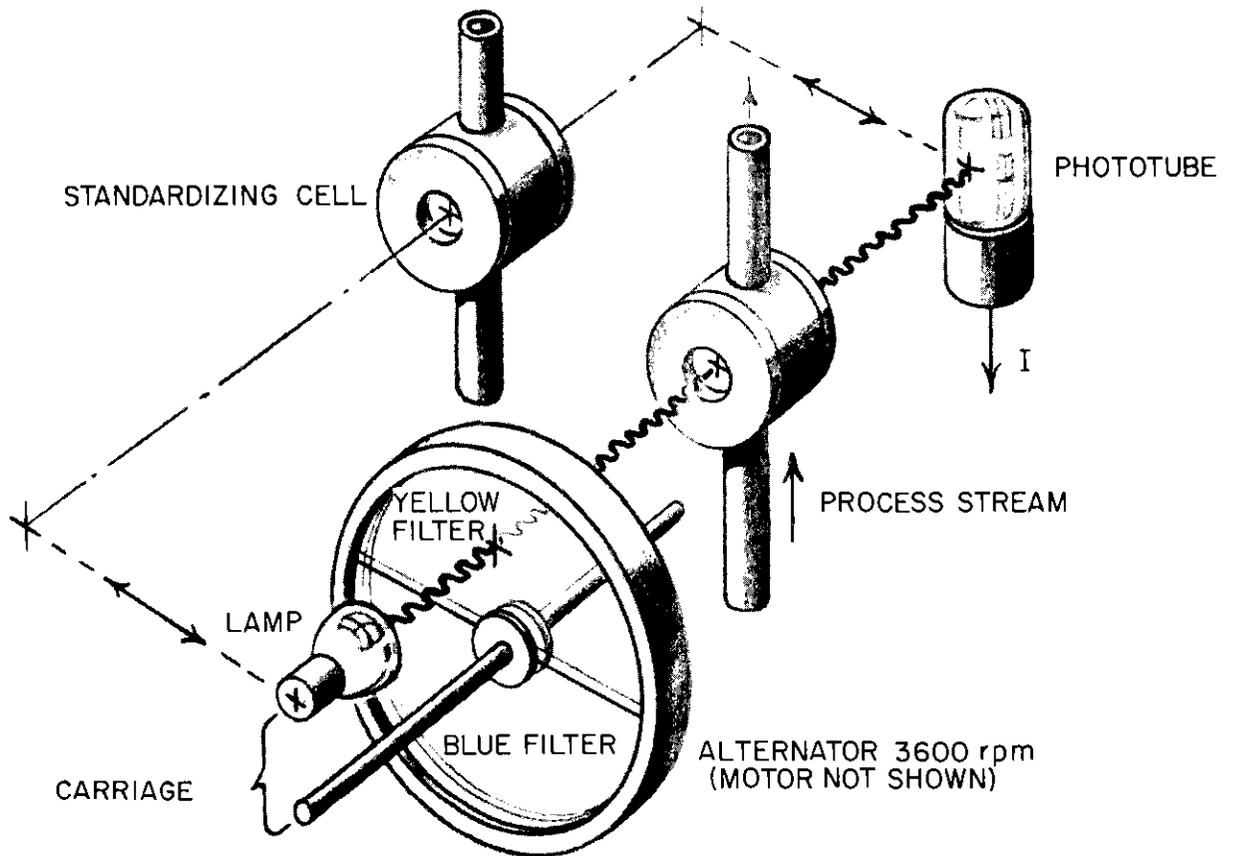


Fig. 14. Sketch of experimental dual-channel dual-beam inline colorimeter.

The voltage from measuring the standard is applied to the slidewire of a Brown recorder which has been modified to be a ratio recorder. The reading of the standard is thus 100% scale on the recorder. The standard is selected to have approximately the same concentration as the flowsheet stream so that standardization is effected under approximately the same conditions as stream measurement.

The voltage from measuring the process stream is found, by the recorder, on the slidewire so that the record is in terms of grams per liter referred to the standard and is linear. The voltage is divided, by a manually adjustable resistor, so that the process stream reading is positioned on the recorder scale and chart with space to deviate in either direction as desired. This adjustment is provided also so that the reading can be made to register exactly with the chart calibrations when there is variation in supplies of chart paper. When necessary, the instrument can be ranged with the adjustable resistor until the standard can be changed.

Radiation degradation products, which sometimes build up in the organic solvent in solvent extraction equipment, absorb blue light and interfere with the use of a colorimeter for measuring uranium in process solvent streams. A three-cell colorimeter was designed (Fig. 15) in which the solvent passes through cells before and after it extracts the uranium. The yellow color of the inlet solvent is measured as equivalent uranium concentration which is subtracted from the apparent uranium concentration measured in the product stream. Since the amount of degradation products produced in a single pass through an extractor is small, this method should give the true uranium concentration regardless of large accumulations of degradation products, corrosion products, and other interferences. The concentrations are measured as voltages and are subtracted using a circuit of condensers. The third cell contains solution for standardization.

The lamp, filters, and phototube are mounted on a carriage which revolves so that the locus of the axis of the lamp, filters, cell, and phototube is a cone. This arrangement minimizes movement of the phototube and its connections. It also simplifies the mechanics because rotation of the carriage, as it revolves, is prevented with a simple coupling.

A three-cell inline colorimeter was built by the ORNL Inline Instrumentation Group for installation in the first cycle product (organic) stream in the ORNL Metal Recovery Plant (Fig. 16). The instrument uses the dual-beam principle that was proved in the operation of the SRL colorimeter in the Metal Recovery Plant. It differs from the SRL colorimeter in that it includes the third channel, for subtracting solvent color, and the carriage is motorized for remote operation.

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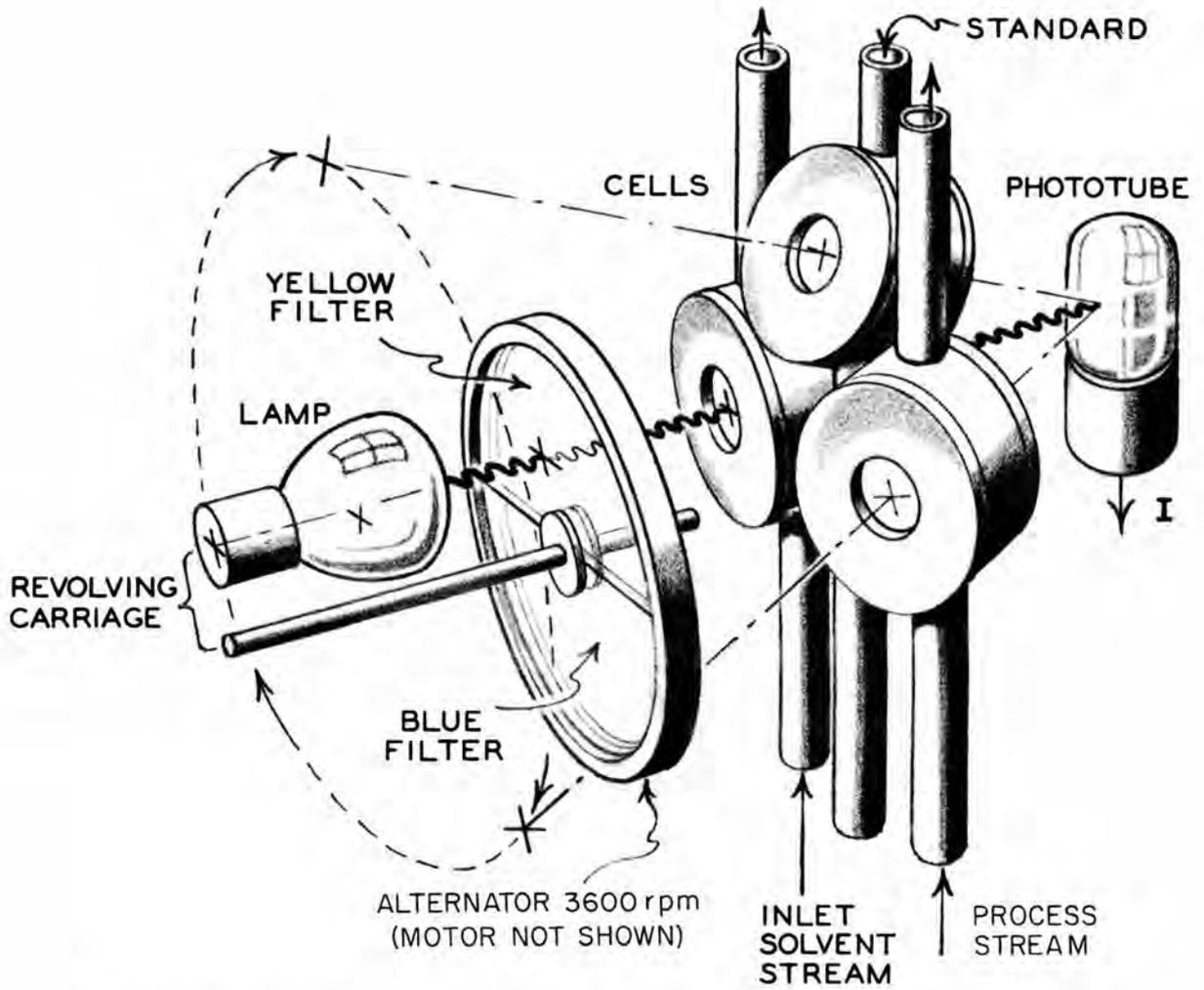
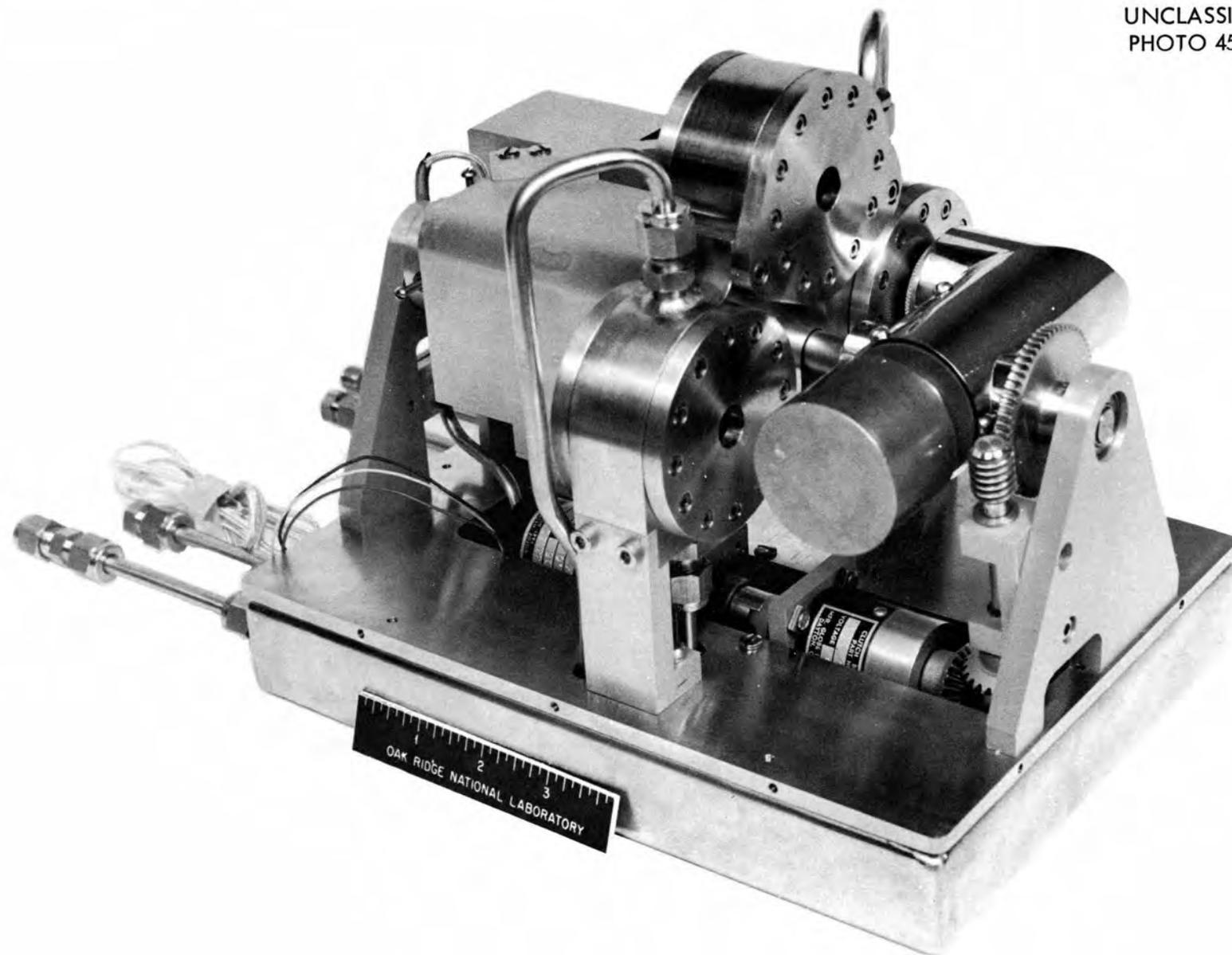


Fig. 15. Sketch of experimental triple-channel dual-beam inline colorimeter.



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Fig. 16. ORNL inline colorimeter for uranium in first cycle organic product stream.

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