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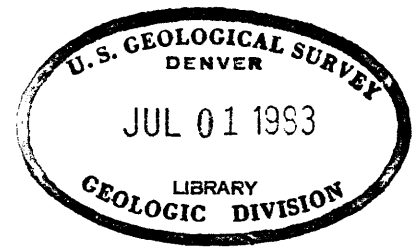
A BATTERY-POWERED FLUORIMETER FOR THE DETERMINATION OF URANIUM

by

Irving May and Mary H. Fletcher

December 1950



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ABSTRACT

A transmission fluorimeter is described which is completely battery-powered and which is suitable for use in the laboratory, in field stations, or in mobile units. The ultraviolet light source is a 3-watt RP-12 lamp. The instrument is sufficiently sensitive for the determination of 0.001 percent uranium in a 0.4-mg sample.

INTRODUCTION

The determination of uranium by measuring the fluorescence of fluoride melts has been used extensively in the past few years as a laboratory procedure. With an appropriate fluorimeter this technique could be developed into a field method for the determination of uranium.

A fluorimeter for field use, either in a mobile laboratory or at a field station, would have to be completely battery-powered. Although there are a number of fluorimeters in general use which employ battery-powered current-amplifying and measuring devices, they all require house current as a source of power for the ultraviolet light source. The fundamental problem in designing a field fluorimeter therefore resolves itself into finding a suitable ultraviolet lamp that can be operated with batteries.

The instrument described in this paper employs an inexpensive 3-watt d.c. lamp as the source of ultraviolet light. The arrangement of the sample support, filters, and shutter is the same as used in the transmission fluorimeter described in a previous paper.1/ The fluorescent light is converted to electrical energy by a battery-powered photomultiplier tube, and the resulting current is read directly with a sensitive but sturdy microammeter.

Acknowledgment

We thank Dr. Francis J. Davis, Division of Health Physics, Oak Ridge National Laboratory, for his recommendations regarding phototubes and meters, and for assembling a prototype current-measuring unit.

DESCRIPTION OF THE INSTRUMENT

The ultraviolet lamp

The ultraviolet light source is a small d.c. RP-12 lamp, manufactured by the General Electric Company for lighting fluorescent instrument dials on aircraft. The lamp is coated on the inner surface with a phosphor having a radiation peak at 3500 A. The RP-12 lamps are available in two wattage ratings, 3 watts,12-16 volts, and 4 watts, 24-28 volts. The lamp has a length of 2 1/3 inches, a maximum diameter of 1 1/2 inches, and a regular double-contact bayonet

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1/ Fletcher, Mary H., and May, Irving, The design of the Model V transmission fluorimeter: U. S. Geol. Survey Trace Elements Investigations Rept. 133 (in preparation).

base with indexed pins.2/ The lamp circuit employed in this fluorimeter is shown in figure 1.

Despite its lower light output, we have adopted the 3-watt lamp for this instrument because of the greater convenience in working with lower-voltage batteries. The light produced by the 3-watt lamp is much less intense than that from 100-watt lamps or larger which normally are employed in fluorimeters. Therefore the 3-watt lamp might seem unsuitable for use in a very sensitive fluorimeter. The over-all sensitivity of the transmission fluorimeter 3,4/ is so great, however, that for normal use in uranium analyses the lamp is raised about 10 inches from the primary filter and the photomultiplier tube is operated at only 450 volts rather than at its maximum voltage. In the field fluorimeter, however, the low light output of the 3-watt lamp is compensated for largely by placing the lamp nearly in contact with the primary filter and operating the photomultiplier tube at 900 volts.

The lamp generates very little heat; even when it is close to the filter and covered with a reflecting dome, there is no necessity for

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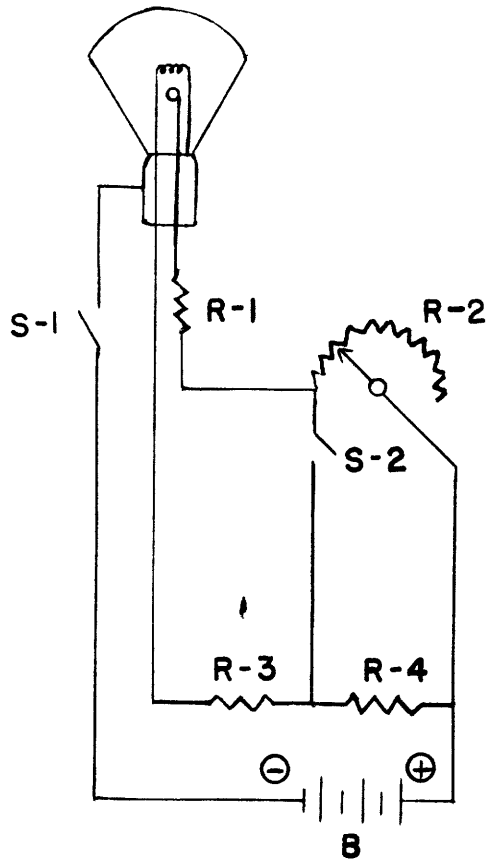
2/ Sockets for these lamps are manufactured by the Cole-Hersee Co., 20 Old Colony Ave., Boston, Mass., and the H. A. Douglas Manufacturing Co., Bronson, Mich.

3/ Fletcher, Mary H., and May, Irving, The design of the Model V transmission fluorimeter: U. S. Geol. Survey Trace Elements Investigations Rept. 133 (in preparation).

4/ Fletcher, Mary H., May, Irving, and Slavin, Morris, A transmission fluorimeter for use in the fluorimetric method of analysis for uranium: U. S. Geol. Survey Trace Elements Investigations Rept. 104, 1949.



S-1 On-off switch
S-2 Starting switch, normal position open
R-1 Resistor, 16 ohms
R-2 Variable resistor, 25 ohms
R-3 Resistor, 6 ohms
R-4 Resistor, 24 ohms
B Battery, 12-16 volts

Figure 1.– Lamp circuit

cooling with a fan. The light output of the lamp becomes reasonably stable less than five minutes after lighting.

Power supply for the lamp

We have investigated two types of batteries to supply current for the lamp: mercury dry-cell batteries for portable instruments, and lead storage batteries for use in a laboratory, field station, or mobile unit.

The recently developed mercury batteries, manufactured by P. R. Mallory and Co., Inc., provide several times more current per unit volume than do conventional dry cells. These batteries consist of 14 type-4RF cells in series, providing a starting voltage under load of 16 volts.

Performance tests of the batteries were made in which 500 milliamperes were drawn in the lamp circuit. Under continuous drain, the battery delivered this current for 90 minutes. After an overnight rest, an additional 50 minutes of service was obtained. Two such batteries, connected in parallel, provided 5 hours of service at the same current drain, followed by 1 1/4 hours' service after resting. This current drain represents an appreciable overload on the batteries and results in poorer performance compared to the rated capacity of 3000 ma. hours.

A storage battery is the most convenient power supply for the lamp where ease of portability is unimportant. The battery used consists of seven Willard No. 25-2 radio cells in series. The cells have transparent non-spillable polystyrene cases with built-in indicator

floats. The dimensions of the unit cell are 2 5/16 by 2 1/2 by 6 inches so that a battery of seven cells is only 18 inches long. The capacity of these cells is 26.5 ampere hours which is therefore equivalent to about 50 hours of current supply for the lamp per battery charge.

A voltage regulator is not required inasmuch as the storage battery provides a steadier current than that generally obtained with voltage-regulated house current.

We have assembled a convenient charging unit for the storage batteries consisting of a Powerstat, a Fansteel selenium rectifier stack (output of 18 volts), and an ammeter. Convenient control of the charging rate is obtained by varying the output voltage of the Powerstat. A pin has been mounted on the face of the Powerstat to limit its output so that the rectifier cannot be overloaded.

The current flowing through the lamp circuit can be changed by varying resistance R-2 (fig. 1). Variation of the resistance produces a change in the light output of the lamp. This adjustment is employed for standardizing the over-all sensitivity of the instrument. (Larger changes in sensitivity may be made by changing the voltage on the photomultiplier tube as described below.)

Sample-support, filter, and shutter assembly

The arrangement of the sample support, filters, and shutter is of the transmission type previously described.5/ The ultraviolet-lamp

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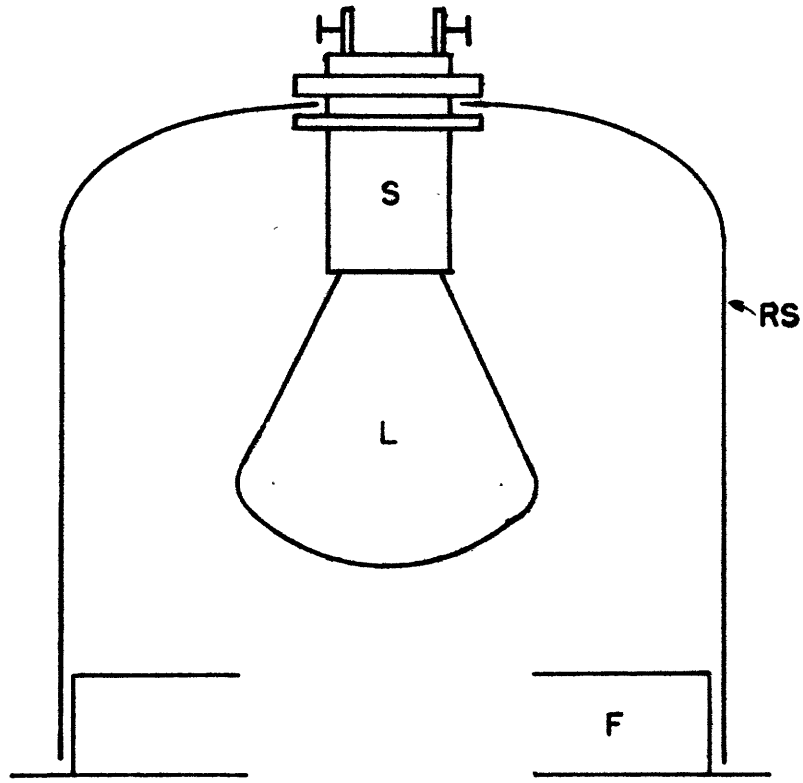
5/ Fletcher, Mary H., and May, Irving, The design of the Model V transmission fluorimeter: U. S. Geol. Survey Trace Elements Investigations Rept. 133 (in preparation).

support seats directly on the unit (fig. 2). The assembly is so constructed that it may be used interchangeably with either the RCA-5819 or the RCA-1P-21 photomultiplier tube. The primary filter is the Corning No. 5874 (2-inch-square, polished thickness); the secondary filters consist of one each of Corning Nos. 3486 and 9780 (2-inch-square) filters.

Photomultiplier tube and circuit

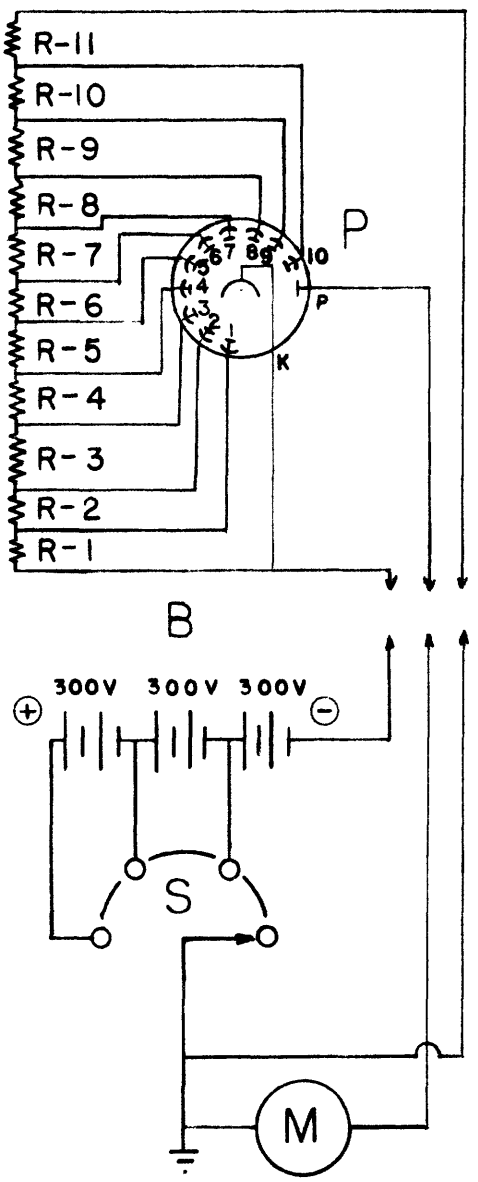
The RCA-5819 photomultiplier tube is being employed in the instument. The power source for the photomultiplier tube consists of three 300-volt Eveready No. 493 Mini-Max batteries in series. The power circuit for the tube is shown in figure 3. With a total voltage of 900 volts the amplification given by the tube is about 200,000 times. The amplification may be doubled, as desired, by increasing the voltage to 1000 volts. Switch S (fig. 3) provides for a step-wise application of voltage to the photomultiplier tube. Thus the tube may be operated over a considerable range of sensitivity. When reading the fluorescence during the analysis of a sample of unknown composition, it is desirable first to take readings at the lowest voltage and then at progressively higher voltages, if necessary, in order to avoid damage to the photomultiplier tube. Average anode currents of more than 750 microamperes will damage the tube. For maximum stability, the anode current should not exceed 100 microamperes.

As an alternative arrangement the RCA-1P-21 photomultiplier tube may be used in place of the 5819 tube. The smaller size of the 1P-21 tube is advantageous where maximum portability is desirable.



RS Reflecting shield
L Lamp
S Lamp socket
F Primary filter holder

Figure 2.—Reflecting support for lamp



P RCA photomultiplier tube 5819
M Microammeter
S Selector switch
B Eveready Micromax batteries, 300 volts each
R-1 to R-11 Resistors, 1 megohm, 1/2 watt

Figure 3-Power source for photomultiplier tube

Microammeter

The currents generated by the photomultiplier tube are read directly with a microammeter. The meter in use until recently was a Rawson type 507C,6/ giving full-scale readings of 4, 40, 400, and 4000 microamperes. Recently we replaced this with a Rawson 507C meter having full-scale readings of 5, 15, 50, and 150 microamperes. The scales on this new meter were chosen to give a convenient gradation of sensitivities within the useful range of the photomultiplier-tube output.

PERFORMANCE OF THE INSTRUMENT

The over-all performance of the instrument is characterized by good stability over a very wide range of sensitivity. At the sensitivities normally employed here, the instrument is standardized by adjusting the reading obtained with no sample in position and with the shutter open, to full-scale deflection on the most sensitive scale of the microammeter (4 microamperes).7/ This adjustment is made by varying the current supply of the ultraviolet lamp with resistance R-2 (fig. 1).

A standard curve for the instrument is shown in figure 4. The fluoride melts were prepared by techniques which have been described elsewhere 7,8/ and the melts were fused in specially designed platinum

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6/ Rawson Electrical Instrument Co., Cambridge, Mass.

7/ Fletcher, Mary H., May, Irving, and Slavin, Morris, A transmission fluorimeter for use in the fluorimetric method of analysis for uranium: U. S. Geol. Survey Trace Elements Investigations Rept. 104, 1949.

8/ Fletcher, Mary H., U. S. Geol. Survey Trace Elements Investigations Rept. 130 (in preparation).

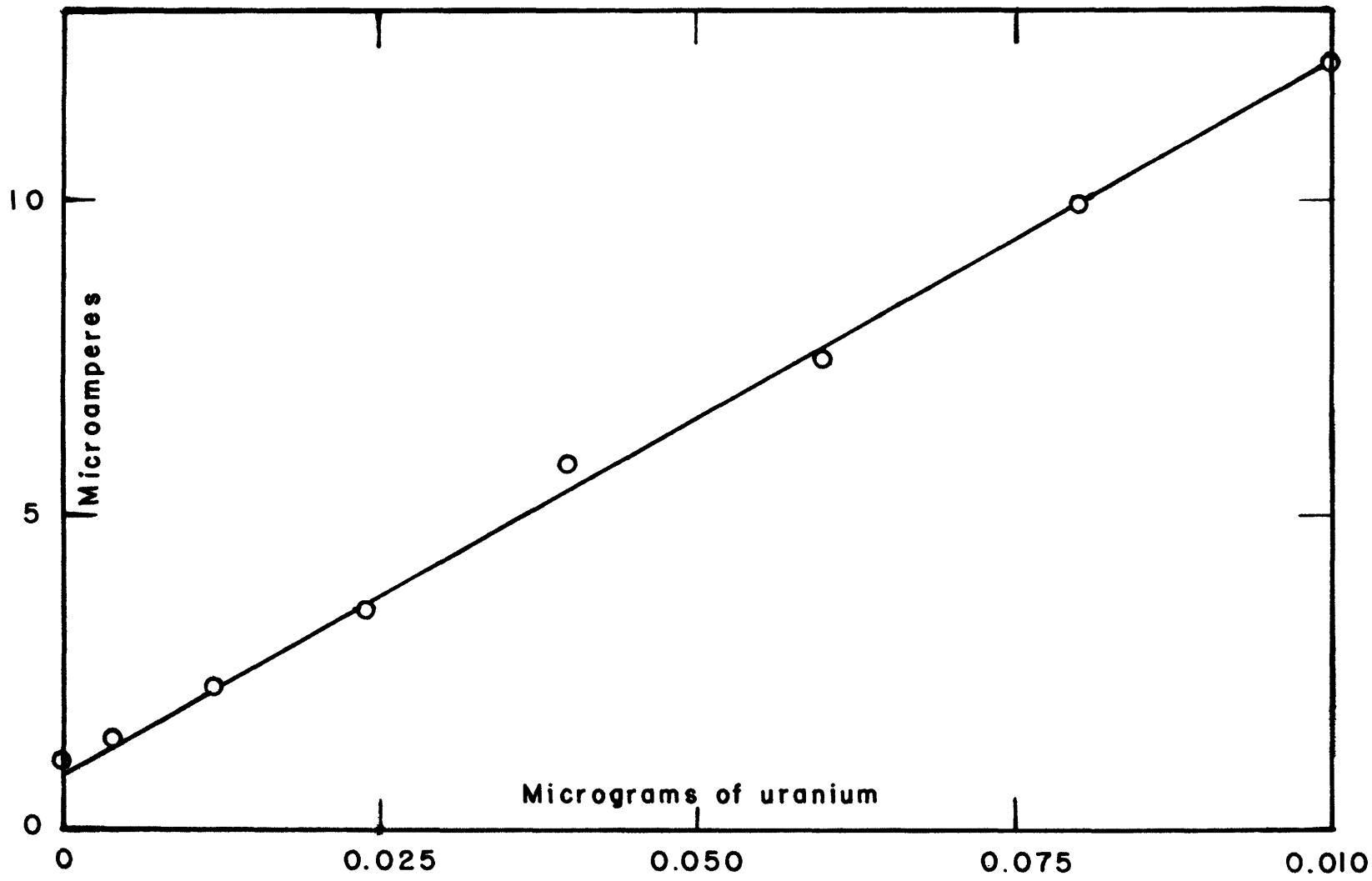

Figure 4 – Standard curve

dishes (fig. 5). The ultraviolet lamp was powered by the storage-battery unit and the photomultiplier-tube voltage was 900 volts. The sensitivity is approximately 1 microampere for 0.01 microgram of uranium. Inasmuch as the meter can be read to the nearest 0.01 microampere on the 4-microampere scale, the sensitivity is adequate for this range of uranium concentrations.

Table 1 shows the precision with which fluorescence measurements may be made with the fluorimeter. The fluorescence of melts prepared in the routine analysis of rocks for uranium was measured with the fluorimeter. Each melt was measured four times, and was rotated 90° between successive measurements. The over-all variance of the measurements is 0.028.

Table 1.--Precision of fluorescence measurements

Sample	Scale	Readings (microamperes)	Mean of readings	Variance
1	40	30.3, 30.4, 30.6, 30.7	30.5	0.033
2	40	22.8, 22.8, 22.8, 22.9	22.8	0.0033
3	40	31.4, 31.6, 31.6, 31.8	31.6	0.027
4	40	29.5, 29.5, 30.0, 29.4	29.6	0.073
5	40	21.0, 21.4, 21.0, 21.1	21.1	0.037
6	40	24.7, 24.7, 24.8, 24.6	24.7	0.0067
7	4	1.18, 1.18, 1.17, 1.20	1.18	0.017
				0.028 Over-all variance

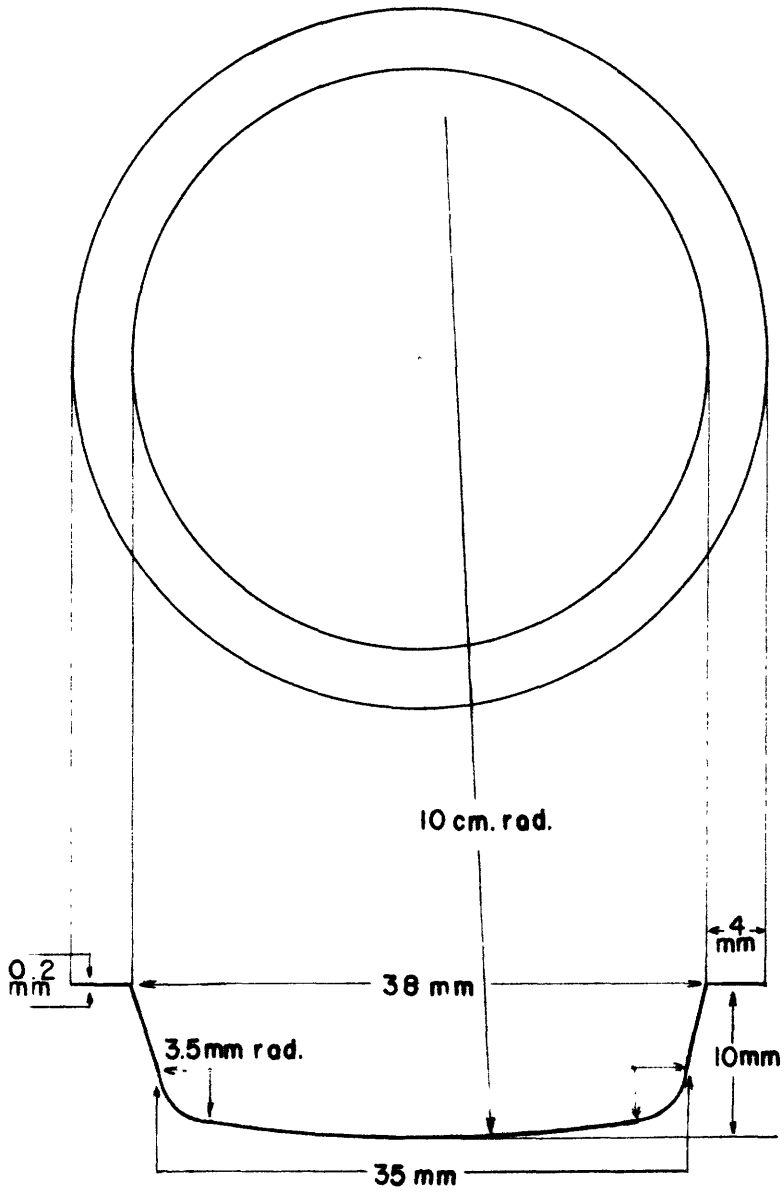

Figure 5-Platinum dish used for fusions

CONCLUSION

A new fluorimeter has been designed and constructed for the measurement of the fluorescence of fluoride melts for the determination of uranium. The fluorimeter is powered completely by batteries and is therefore suitable for use in places where house current is not available, such as field stations and mobile units. The instrument is also very useful as a laboratory fluorimeter when the greater sensitivity of the Model V fluorimeter is not required. Its advantages are: a steady light output without the need for a voltage regulator, and a cool operating lamp not requiring forced ventilation. The instrument is sufficiently sensitive for the determination of 0.001 percent uranium in a 0.4-mg sample. Additional sensitivity is possible with increased voltage on the photomultiplier tube.

Several of these fluorimeters are now being built. Some of them will be assembled so that they may be used conveniently as field instruments. The others will be employed in our laboratories for the routine determination of uranium.