Model '54 Transmission and Reflection Fluorimeter for Determination of Uranium With Adaptation to Field Use

GEOLOGICAL SURVEY BULLETIN 1036-M

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Model '54 Transmission and Reflection Fluorimeter for Determination of Uranium With Adaptation to Field Use

By ERNEST E. PARSHALL *and* LEWIS F. RADER, Jr.

CONTRIBUTIONS TO GEOCHEMISTRY

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MODEL '54 TRANSMISSION AND REFLECTION FLUORIMETER FOR THE DETERMINATION OF URANIUM, WITH ADAPTATION TO FIELD USE

By Ernest E. Parshall and Lewis F. Rader, Jr.

ABSTRACT

The model '54 fluorimeter for the determination of uranium by the fluorimetric method has been designed, built, and used in the U. S. Geological Survey since 1950, and is one of several such instruments developed by the Geological Survey. An adaptation for field use is included in the present description of the instrument. The circuit is a modification of that used in the Beckman DU spectrophotometer and incorporates an electrometer, amplifier, and blue-sensitive phototube as used in the DU instrument because of the extraordinary sensitivity obtained for a phototube of this type. The low-voltage circuit used in the Model '54 fluorimeter results in an unusually broad working range of high sensitivity and stability for the instrument. Changes in temperature have little or no effect on the accuracy of readings made under conditions expected in field work. A detailed description of the instrument and of its use in fields and laboratory is accompanied by photographs, working drawings, and instructions that would make possible its construction in the average machine shop.

INTRODUCTION

Fluorimetric methods for the determination of uranium were developed before commercial fluorimeters were available. Investigators in various laboratories, therefore, generally built their own instruments. Pioneering in the design of fluorimeters were Price and coworkers in 1945 and 1948 (Price, Ferretti, and Schwartz, 1953); Fischer and Pickle (1946); Center (1949); Davey and Florida (1949); Jacobs (1950); Kaufman and others (1950); Zimmerman (1950); Yeaman (1951); Kelley and coworkers (1954); and Galvanek and Morrison (1954).

U. S. Geological Survey chemists have developed numerous fluorimetric instruments, several of which have already been described in publications. Fletcher and May in 1950 and 1954 reported a modification of the Oak Ridge Model R fluorimeter which had been in use since 1948. The first transmission instrument was reported in 1949 and 1954 by Fletcher, May, and Slavin: the Model V transmission fluorimeter in 1950 and 1954 by Fletcher, May, and Anderson. A portable battery-powered field instrument was described in 1950 by May and Fletcher. Several features of the Model V and the field fluorimeters were incorporated into a compact laboratory instrument (the Model VI) by Kinser in 1954.

Reports on this early work, as now available to the public, are listed in the bibliography.

This paper describes another fluorimeter developed in the U. S. Geological Survey. This instrument, the Model '54, has been in use in essentially its present form since 1950. During this time it has been used daily for the determination of uranium in a large variety of materials totaling about 1,000 determinations per month so that the instrument has been thoroughly tested.

Requests from chemists who have received training in the fluorimetric method under the Atomic Energy Commission program and from private companies engaged in reconnaissance for uranium, and related mining activities, show that this instrument is particularly suited to their needs. This report, containing instructions, wiring diagrams, and descriptions of the Model '54 fluorimeter, has been prepared to make such information available to all those interested.

This report concerns work done by the U. S. Geological Survey on behalf of the Division of Raw Materials of the U. S. Atomic Energy Commission.

ACKNOWLEDGMENTS

The advice and assistance of all who made possible the development, construction, testing, and description of this fluorimeter are gratefully acknowledged. G. J. Petretic, formerly of the Geological Survey, was largely responsible for the early development work. C. G. Bay and others of the Geological Survey aided in the design and construction of the instrument in the shops.

DESCRIPTION OF FLUORIMETER

This fluorimeter uses a circuit modified from that of the Beckman DU spectrophotometer: it also uses the three electronic tubes similar to those of the Beckman instrument. The phototube has extraordinary sensitivity for one of this type and requires no cooling arrangement for stable operation that has been found desirable for some types of photomultiplier tubes (Morton, 1955). The blacklight lamp is a 3-watt F 3 RP 12/GL type that operates satisfactorily without cooling. The amplifier unit is so constructed that either a transmission-type search head or a reflection type unit may be used for measurement of fluorescence independently. An adaptation of the instrument for field use with dry-cell batteries also is described. Either the standard laboratory instrument or the field adaptation is

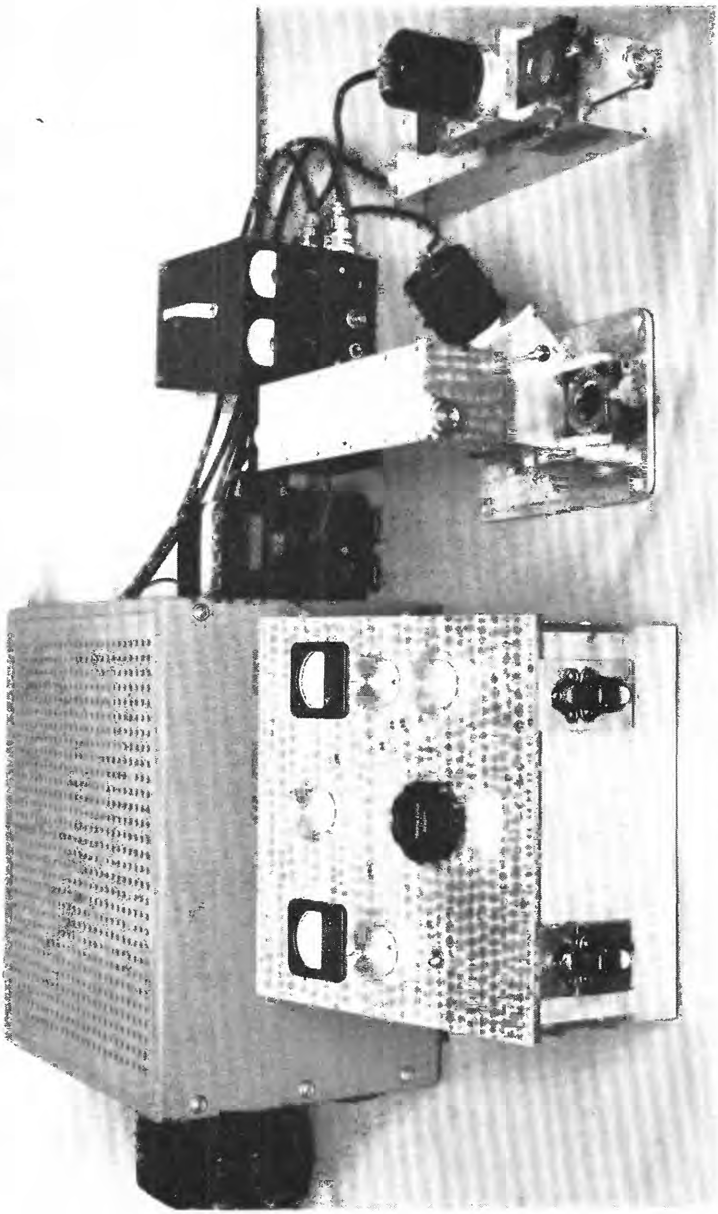

FIGURE 31. Assembled fluorimeter, Model '54.

designed to read a broad range of uranium concentrations in carbonate fluoride flux pads without exceeding the point at which the phototube fails to respond linearly to additional small increments of concentration or at which the phototube fails to recover rapidly from exposure to intense light. Thus it is possible to work in the range of from 2×10^{-9} to 3×10^{-5} grams of uranium in a 2 gram flux wafer, and the use of extremely small samples, large dilutions, or tiny aliquots is generally not necessary.

The assembled instrument as used in the laboratory is shown in figure 31. In the foreground are the amplifier and control cabinet, reflection-type and transmission-type search units, respectively, from left to right. In the background are a commercial power supply designed to power a model DU spectrophotometer, a constant voltage transformer connected ahead of the small power supply in the right background. This power supply is for the two black-light lamps, one on each type fluorimeter search head.

The amplifier and control cabinet is made of ¼-inch sheet aluminum cut and assembled to form a box $10 \times 11\frac{1}{2} \times 8\frac{3}{4}$ inches, sloping to 5½ inches in front.

The amplifier unit may be operated with either a commercial power source, a 6-volt storage battery, or with dry cells as described later for field use. However, this unit also requires six radio C batteries in addition to the power source just mentioned.

ELECTROMETER AND PHOTOTUBE ASSEMBLY

Different views of the phototube assembly and shutter arrangement of the search heads are shown in figures 32 and 33. The housing is made from sheet aluminum. Two sides are ¼ inch thick and two are ⅛ inch thick so that the edges of the thick stock may be milled to light-tight fit. The two ends also are thick stock milled for the same reason. An aperture bored to a diameter of 1.276 inches is cut in one of the thin sides of the housing, 2¼ inches from center to one end of plate, threaded for attachment to the light-filter holder and lamp assembly (36 threads per inch). The overall dimensions of the assembled housing are 10¼ x 2⅞ x 2¾ inches.

All wiring in the phototube housing must be with waxed, cotton-braided, insulated wire because static electricity caused by moving the shutter slide is sufficient to cause serious interference with operation of the instrument if wire covered with synthetic insulation is used.

Identification and location of items in the phototube housing, figures 32 and 33 are: Resistors, R1, R2, R3, R4, and R5, with resistances of 39 ohms, 47 ohms, 1 megohm, 150,000 ohms and 2,000 megohms, respectively; (I) are porcelain insulator mounts, (A) is the electrometer

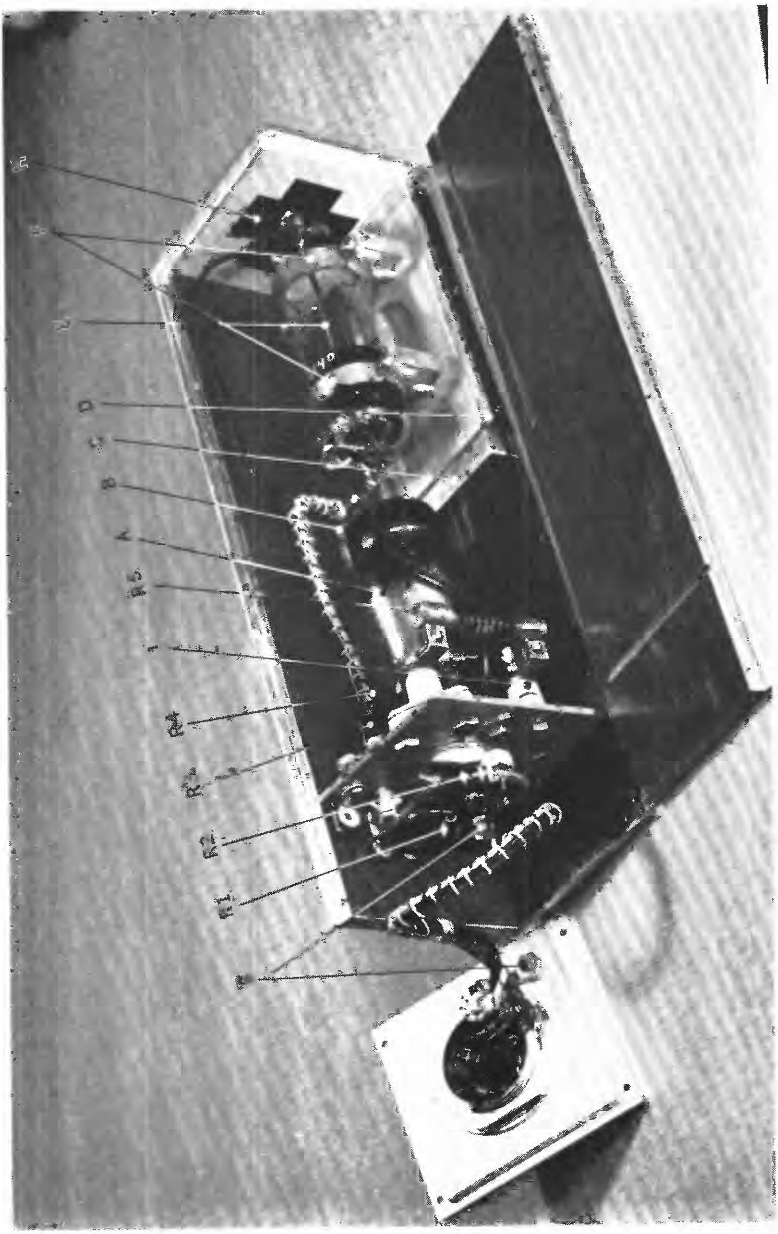

FIGURE 32. Phototube assembly and shutter arrangement.

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FIGURE 33.—Switch and shutter arrangement in search head.

amplifier tube (V2), type 32 for low grid current applications (Sylvania no. 1229, specially selected, Beckman no. 2532, (B) is a dark-current switch (S4) operated by the shutter slide (C).

This dark-current switch is made from one set of contacts from a 12-ampere, type 200-4 Guardian relay contact assembly. Remove one set of contacts from the assembly intact. Also remove the center blade, turn it over so that all connecting lugs are on the same side, and flatten the flute in the end of the blade for attachment of a plexiglass block or shoe that will operate the switch, when finally assembled, whenever the shutter slide is moved. This block is made from ¼ inch thick material cut to clear the end of the lower blade of the switch and carefully drilled and fitted before fastening to the end of the center blade with two round-head number 2-56 thread machine screws. Bend the upper and lower wire lugs away from the center one for more wire clearance. Mount the contact assembly on a piece of plexiglass ½ x 1 x ½ inch as shown (fig. 33). Either drill to bolt through the plexiglass or drill and tap for the number 5-40 thread screws that come with this part. Locate the blades of the assembly on the block with the stack spacer insulating washers flush with the inside edge of the ½-inch dimension of the block in order to gain about ⅛-inch clearance from the side of the case. Also locate the assembly on the block near the right end as shown on figure 33. The block and switch assembly is then fastened to the side of the case with two number 6–32 thread oval-head machine screws ½ inch long. Drill and tap the block for these screws, locating one midway between the two assembly screws and the other spaced ½ inch toward the contact end of the block.

The switch must be positioned so that the shutter has just cleared the light opening before the switch operates. The vertical position is such that the shutter slide raises the center blade to clear the lower contact and to make contact with the upper blade. When the shutter plate closes the light opening, the center blade of the switch, which is connected through resistors R4 and R5 (fig. 32) to the grid of the electrometer tube, is in contact with the lower blade thus grounding the grid circuit through the two resistors so that the dark current may be adjusted to zero on the meter. When the shutter plate is pushed in, light passes to the phototube as the center blade of the switch is raised to clear the lower or grounded blade, and the grid circuit is transferred to the upper blade which is connected to the slide-wire circuit through the range switch. This switch thus prevents operation of the instrument when the opening to the phototube is not completely cleared because no readings can be obtained on the slide-wire dial, and the operator is warned to adjust the position of the shutter plate. Contact tension adjustment of the swich is made by bending. With the light shutter open and switch in raised position, clearance of 0.025

inch should be allowed between contacts. The contacts must be adjusted so that they just make before break in order to avoid severe shock to meter needle when in use.

The shutter plate (*C* figs. 32 and 33) is aluminum, 2⅜ x 3⅜ x ¼ inches, cut so that the width is a free fit between the side of the housing. A brass operating rod for the shutter slide is ⅛ x 3⅝ inches threaded at both ends for attachement to plate and pull knob. Attachment to the shutter plate is made off center so that interference with the opening to the phototube is avoided. The rod length is adjusted to allow about ¼-inch clearance of the opening in the search unit housing when the shutter slide is fully open. The phototube (Beckman 2342) (E) is mounted in plexiglass saddles by means of brass strips (F), bent to the desired radius so that thin cork strips may be placed between the saddles, strips, and the tube at both mounts. The saddles holding the phototube are mounted to an aluminum plate, 2⅝ x 4$\frac{1}{16}$ inches, with a ¼-inch-diameter hole bored to line up with the threaded opening in the housing. This plate is fitted into ⅛-inch-deep slots in both sides of the housing. A clearance of ¼-inch is attained between shutter plate and the bottom of the phototube. One hole is drilled through each side plate of the housing into each slot and tapped for a number 4–40 short Allen setscrew. These screws hold the tube mounting plate in place and allow for adjustment and alinement of the 1¼-inch hole in this plate with that in the tube housing. The plexiglass is drilled and tapped for all screws. Sufficient space between the saddles must be left to allow for minor adjustment of the phototube window to the opening in the housing. The black cross, (H), on end of housing is adhesive rubber insulating tape used to prevent a short circuit.

The electrometer amplifier tube (A, fig. 32) is held in a socket mounted on an aluminum plate, ⅛ x 2½ inches square, recessed into slots in the sides of the housing but loose enough to allow removal or repositioning of plate and tube. The tube is mounted with the plane of the filament in a vertical position. Otherwise the filament may sag and cause failure of the tube. When the search head is turned on its side to be used in a different position than the usual one, the tube mount may be rotated 90 degrees in the housing and the plane of the filament kept vertical. Slots to position the mounting plate are 1$\frac{17}{32}$ inches from center to end of housing plate.

Figure 34 shows the wiring underneath the amplifier cabinet cover and arrangement of parts. The resistors and other parts are further identified in the appendix and text. The circuit wiring diagram is shown in figure 35. At the top left of figure 34 is on-off toggle switch (53). This switch proved unsatisfactory because of variable contact resistance and has been replaced with 90° indexing, four-pole

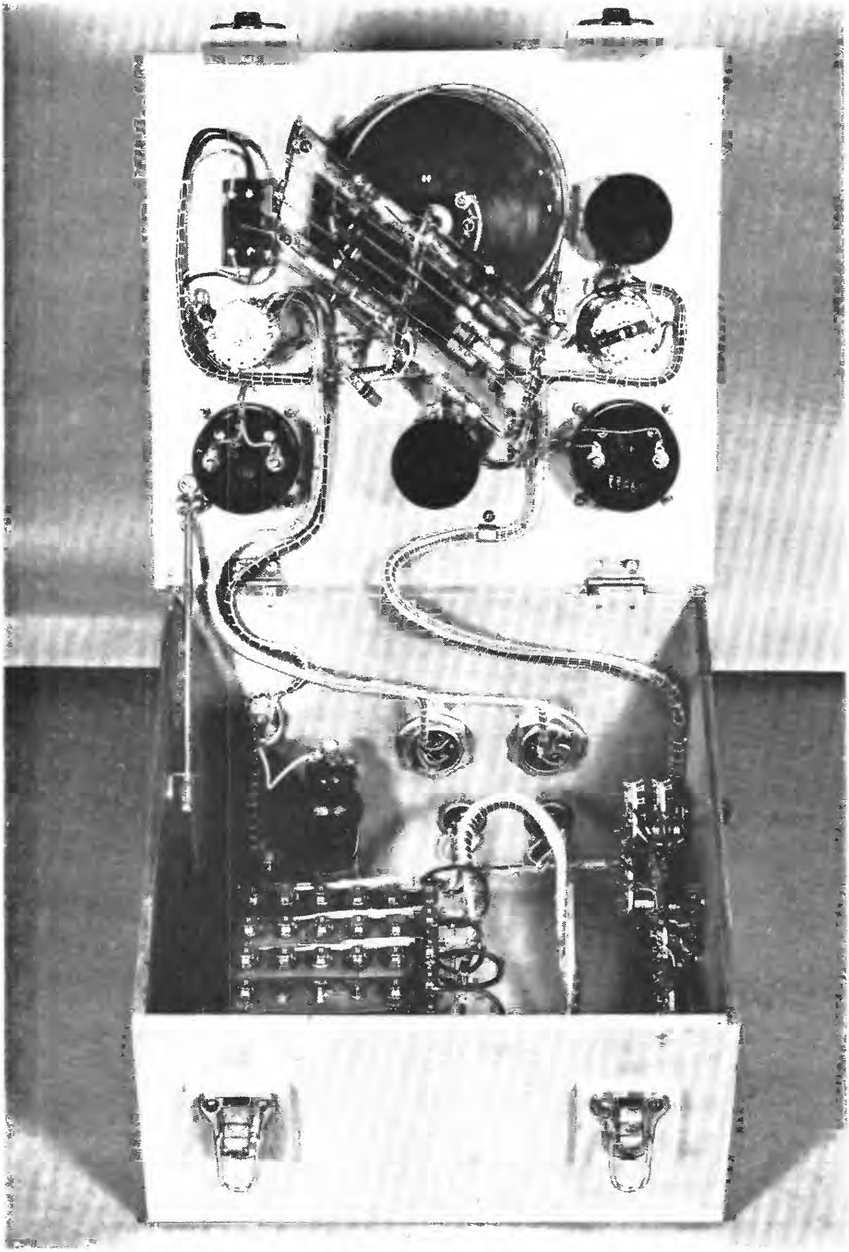

FIGURE 34.—Location of wiring and parts in amplifier cabinet.

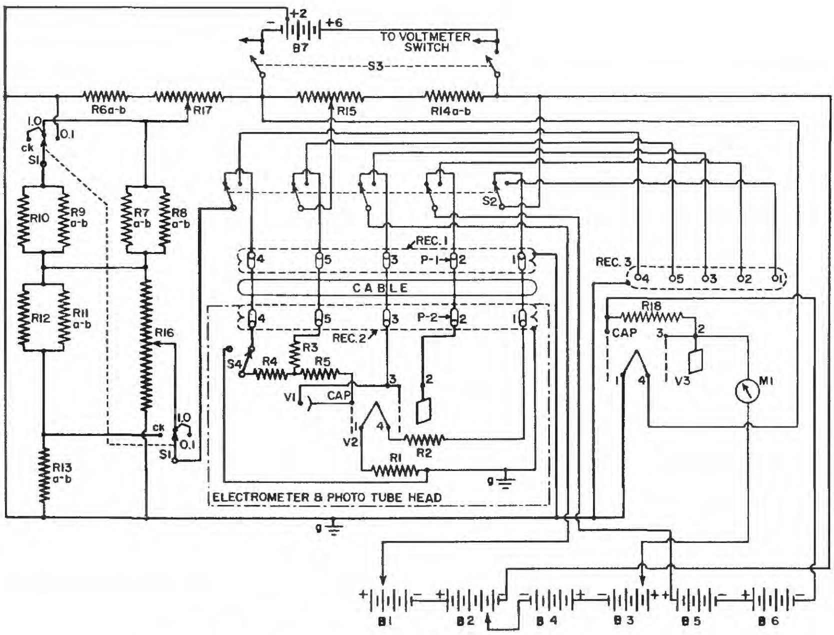

FIGURE 35.—Wiring diagram of Model '54 fluorimeter.

rotary ceramic switch with two poles wired in parallel to minimize contact resistance. At top center is slide wire (R16), and at top right is dark-current helipot (R15). Ceramic switches: left center is selector "reflection-transmission" (S2), right center is range switch (S1). At lower left is battery-test voltmeter, 0–15 volt direct current, lower center is helipot (R17) referred to as sensitivity control, lower right is meter with zero-center scale (M1). Parts visible in the cabinet are: Left rear, 100-megohm resistor (R18) mounted on standoff insulators. The resistor shown is an IRC type MVP number 5304. However, the small resistor (R18) listed on page 249 is desirable. The tube is (V3) Beckman amplifier, number 2531. The two receptacles held in place with hexagonal nuts are REC 1 and 3, figure 35. The two small receptacles are P4, figure 36, at the left, and one similar to P4 except that it is a four-wire type to prevent improper connections and replaces the cable connector, figure 36, for 6-volt storage battery connection. Four of the C batteries are shown. These batteries are held in the case by a bakelite box $4\frac{3}{8}$ x $5\frac{9}{16}$ x 2 inches, ID, cut from $\frac{1}{4}$-inch material. This box is fastened to the floor of the cabinet with two flathead 6–32 machine screws cut to exact length so that they do not protrude. The C batteries are positioned with number 1 nearest the tube and number 6 nearest the front of cabinet when connected according to diagram, figure 37. On the right side of the

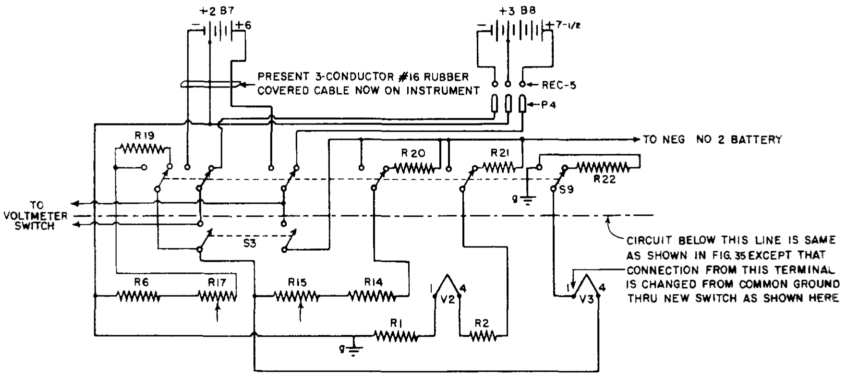

FIGURE 36.—Modification of wiring diagram for operation on dry cell battery.

cabinet (fig. 34) are switches S5, S6, S7, and S8 (fig. 37) and S9, R19, R20, R21 and R22 (fig. 36). Shafts of all switches except S9 are cut short and slotted for screwdriver adjustment. S9 has a knob attached.

The C batteries are held together in the bakelite box by a sheet of plexiglass that hooks under a spring arrangement not shown. This sheet is 5¾ x 4¾ x 1/16 inches. Drill 6 rows of six holes each in the plexiglass (fig. 38). Locate the first row of holes 3/8 inch from the edge that will be positioned in front of cabinet. Space the other rows 1 inch apart. Across the other dimension start the first row ½ inch from left side and space ¾ inch apart. All holes are 19/64-inch diameter except the last row at the right, figures 34 and 38, and these are drilled to clear a number 6 machine screw. Terminal nuts are required on all posts of batteries 1, 2, and 3 but only on the end terminals of batteries 4, 5, and 6. Therefore prepare connections to these terminals by inserting a number 0 brass grommet and plain washer, with the washer down, and crimp in place. Crimp only enough to hold and prevent rotation. Solder connecting wires to the edge of the flange so that terminal nuts are cleared. The 6-volt terminal of batteries 4, 5, and 6 is not connected but needs to be fastened for support when batteries are handled as a unit. Brass machine screws and thumb nuts (6–32 x ½) are used in the small holes along the right side of the plexiglass as terminals to connect the 7½-volt pigtail leads of the C batteries. The wires from these terminals to the amplifier are soldered to lugs that are held in place by hexagonal nuts.

The slide-wire dial (fig. 34) should be purchased complete with knob, shaft and stationary contact. (See list, p. 249.) The slide wire is mounted on the ¼-inch-thick panel through a brass bushing. This bushing is made from 9/16-inch brass stock, cut to 0.760+ inch long, drilled for a running-fit of the knob shaft. One end is turned to 0.500

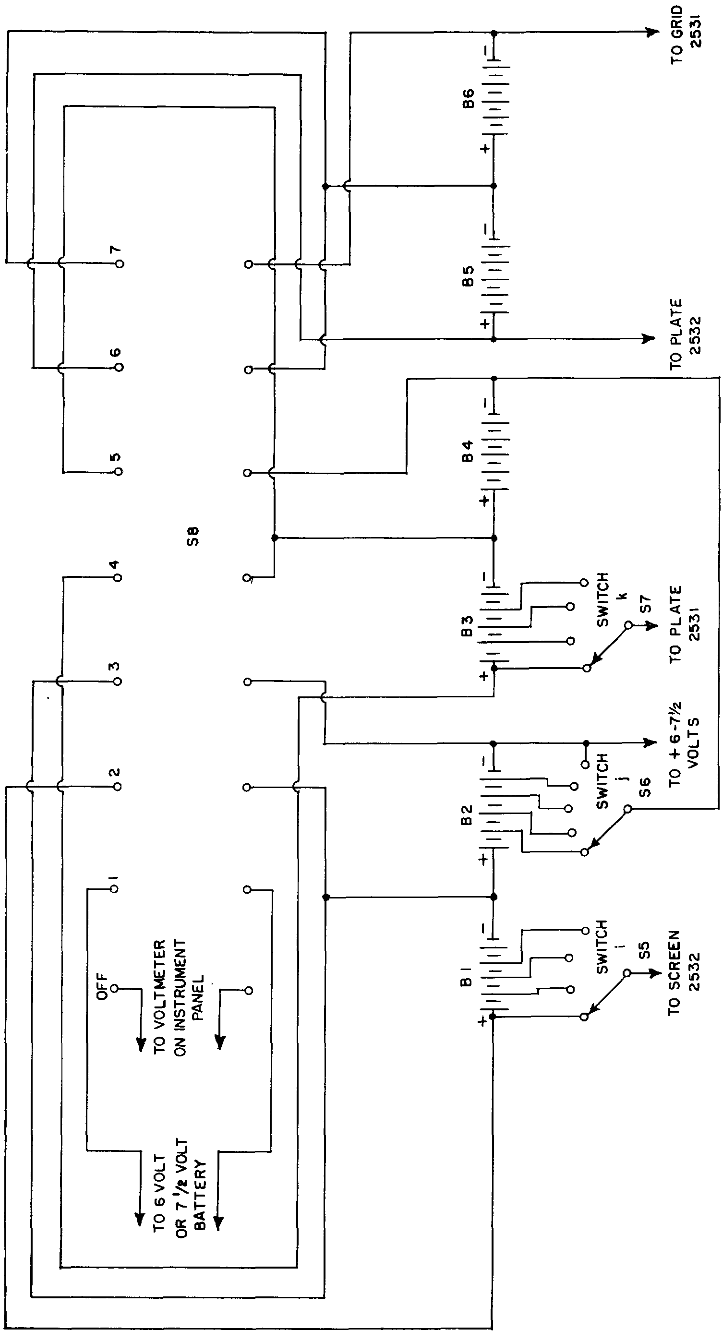

FIGURE 37. Wiring diagram of battery test circuit.

inch in diameter for a length of 11/32 inch and threaded at 32 threads per inch. The other end also is machined down just to clear the inside of the knob. As this diameter is slightly more than 1/2 inch, a shoulder is thus obtained to stop the bushing when screwed into the threaded hole in the panel. When completed the bushing will be slightly longer than required, but it is carefully shortened to match the length of shaft in knob. The knob and shaft must be free-turning when the slide-wire hub is tightened against the shoulder at the end of the thread on the knob shaft. The flat eccentric headbolt and locknut on the slide wire must also have proper clearance on the under side of the panel. This spacing is also controlled by the length of the panel bushing. A fillister-head screw (no. 4–40) is screwed into the underside of panel to strike the eccentric stop on the slide-wire dial. This screw determines the zero position of the transmission scale of the slide wire and must be located so that the zero is lined up with the index line of the window.

The stationary contact is so mounted that when the zero scale setting is correct, the contact is just at the "O" end of the resistance wire. The leads to the terminals on the slide wire must be flexible, coil and uncoil as dial is rotated, and long enough to prevent binding. A support for these leads is provided by a circular piece of 1/8-inch plexiglass slightly smaller than the slide-wire drum. This support is separated from the drum with spacer washers and fastened to the dial-knob shaft with a standard 5/16-inch fine-thread, hexagonal nut. Shielded phonograph pick-up arm cable 17½ inches long is satisfactory for leads. The shield is connected to the "O" end of the resistance wire, the other end of wire to the two-lug terminal strip.

The resistors shown underneath the cover of the amplifier unit, figure 34, are listed and identified on page 249 and also designated by number, figure 35. This arrangement required multiple resistors and parallel connections that may be replaced or simplified as follows: Replace R6a and b with a single 25- or 27-ohm 1-watt resistor; R7a and b and R8a and b may be combined in one 100-ohm 1-watt resistor; likewise combine R9a and b and R10 in a single 10-ohm 1-watt resistor; replace the combination R11a and b, R12, and R13a and b with one 1000-ohm 1-watt resistor; also replace R14a and b with a single 150- or 180-ohm 1-watt resistor. Calibration of the 0.1 to 1.0 range may be accomplished by changing either the 10- or the 100-ohm resistor or possibly both. This should cause either a greater or smaller change in the ratio of 0.1 to 1.0 and indicate whether more or less resistance is required for the correction. Usually an assortment of 10- and 100-ohm resistors varying within the 10 percent tolerance of standard resistors will enable one to find a particular resistor that will produce the correct and desired value, provided the resistance of each is

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measured to select one either higher or lower in resistance, as indicated by the above test, to make the ratio correct.

The assembled cartridge for holding desiccant is shown, on figure 38, in position in the phototube housing and removed for a better view. This cartridge is made from a 4-inch piece of ½-inch thin-wall conduit, drilled through on both sides, 90 degrees apart, to obtain 20 openings. Solder a brass disk 1/16 inch thick and ¾ inch in diameter to one end of the conduit and a screw cap, which is removable, to the other so that the desiccant may be changed quickly. A suitable cap may be made from an Amphenol series 91 plug by cutting the large-diameter shell away from the threaded end, leaving 1/16 inch of the large diameter on the threaded part for soldering to the conduit. The screw cap is made by soldering a disk in the beveled end of the small knurled nut from the plug. A series 91 Amphenol chassis receptacle is used to hold the cartridge in the phototube housing. Remove the insert by turning off the crimped part of the shell but without removing the flange. The locating notch in the threaded shell is closed by soldering a piece of metal in it. This piece is then mounted in a hole bored in the corner of the front plate of housing. Locate this hole so that the flange of the receptacle just clears the inner side and top of the phototube housing. The round threaded nut holds this piece in the end plate. The large knurled ring from the plug clamps the cartridge in the receptacle. A liner of filter paper is cut to fit the assembled cartridge. This liner prevents dust from the desiccant from falling inside the phototube housing.

Figure 38 also shows the assembled housing used for transmission measurements but with the black-light lamp holder removed from the top and the search head removed from the bottom. Shop drawings for construction of this unit are given on plate 3A. The assembly is essentially a track, milled to hold a slide that carries three recessed square openings for the flux-pad holder and two glass standards. Mounted above the slide is a 2-inch-square light filter (Corning 5840) and the black-light lamp with holder. Below the slide are mounted Corning light filters 3486 and 9780, also 2-inch polished squares, and a threaded fitting for attachment to the search head. A lighttight fit is obtained by machining the parts and lapping them together for close fit.

Figure 39 shows a side view of the assembly used for reflection measurements but with the slide, search head, and black-light lamp housing removed. Machine work and fitting of parts on the reflection assembly are more difficult than with the rest of the instrument. The unit must be lighttight and distances for light passage in the assembly should be as short as possible. A square opening is specified in this unit, rather than a round one, because tests showed that a

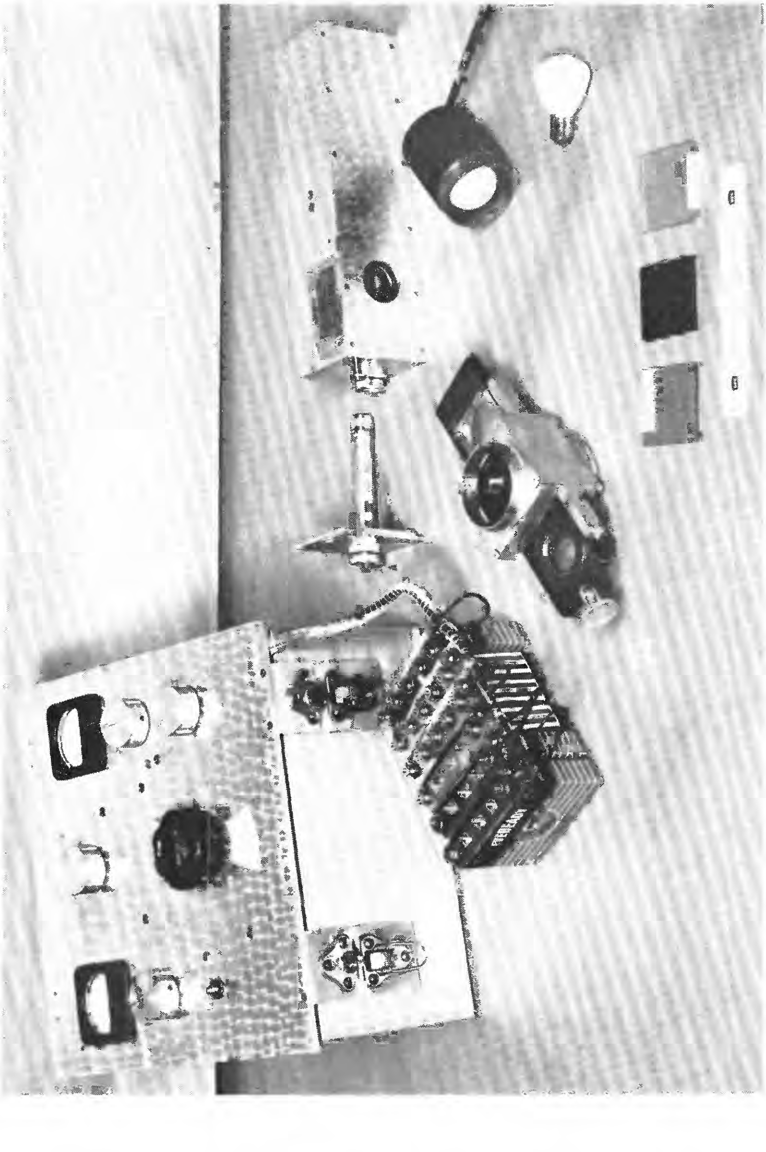

FIGURE 38.—Transmission assembly, RP-12 ultraviolet lamps and housing, light filters, and mount for 7½-volt C batteries.

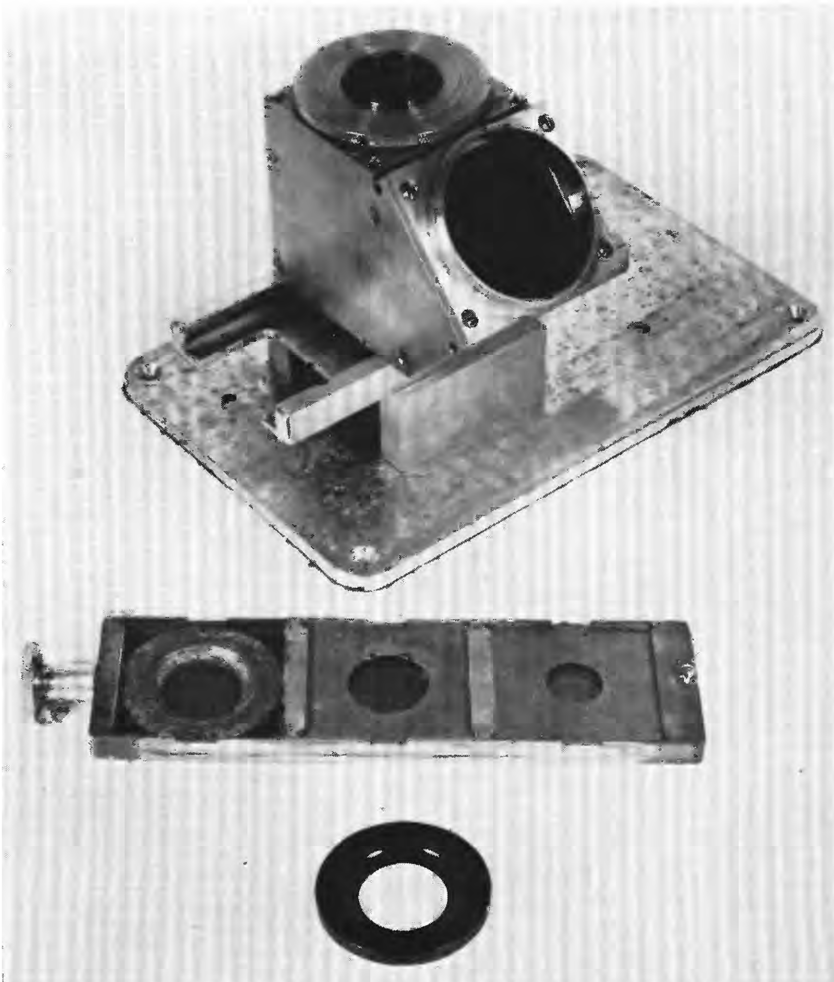

FIGURE 39.—Reflection assembly, slide holding position for flux wafer, and two masked glass standards.

more uniform light distribution and intensity could be achieved than was possible with a round hole. Construction of this unit may be simplified by having a casting made of the part above the button slide and machining to proper dimensions. Light filters of the same size and characteristics as those used in the transmission unit also are used in this unit except that primary filter 5860 gives better light control than 5840 in this instance. The angle of reflection is 45° with the black-light lamp mounted on one side and the search head on top of the unit. Shop drawings of this unit are shown on plate 3*B*.

Construction of the reflection unit (see *B*, separation drawing, pl. 3) may be simplified by eliminating parts marked Ha, Hb, Hc, and

Hd, and substituting part F shown on plate 3A. However, the opening in part F (1⅞-inch hole) need not be cut, but the overall width of part F must be the same as the base, part E, plate 3*B*, in order to obtain a fit. The track for part G, plate 3*B*, is milled so that the openings, G2 (part G, pl. 3*B*) will locate properly in the opening in the bottom of part E, plate 3*B*. The assembly is mounted on base J, plate 3*B*, with spacers 1 inch long.

Figure 40 shows some construction detail for the power supply for the two black-light lamps when the instrument is operated on 110-volt alternating current. The housing for this unit is a commercially available miniature amplifier foundation cabinet. The wiring diagram is that shown in figure 41 for the low-cost power supply except that the transformer (T2) feeds a duplicate rectifier and resistor circuit as shown for a second lamp (fig. 41). The two-lamp circuit (fig. 41) may be used if desired but will cost twice as much. The meter (M) and resistor (R23) shown face down in the housing are paired one set for each lamp. Near the edge of the chassis, left foreground, figure 40, is shown a 75-ohm adjustable resistor in series with a 25-ohm 10-watt fixed resistor. This is duplicated on the other side of the transformer but not visible on the illustration. Adjustment of the fila-

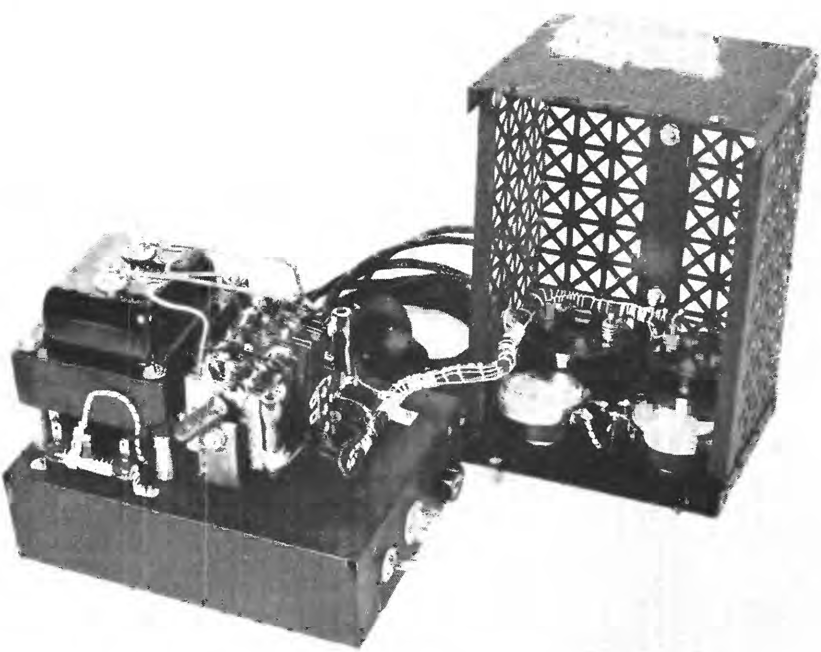

FIGURE 40.—Ultraviolet lamp power source operated on house circuit.

ment voltage is made by removing cover of housing and changing position of slide contacts on the 75-ohm adjustable resistors. If the variable resistor (R23) called for (fig. 41) is used, probably it will be necessary to rearrange parts or use a larger box. Place all resistors and rectifiers above the chassis for better ventilation. Figure 41 shows the switch (S12) fuse, and pilot light ahead of T1 in the circuit. Use the same arrangement in this unit. Receptacles are not visible in figure 40, but are shown in figure 31. If only one black-light lamp is to be operated, the small compact unit given in figure 41 is quite satisfactory and inexpensive.

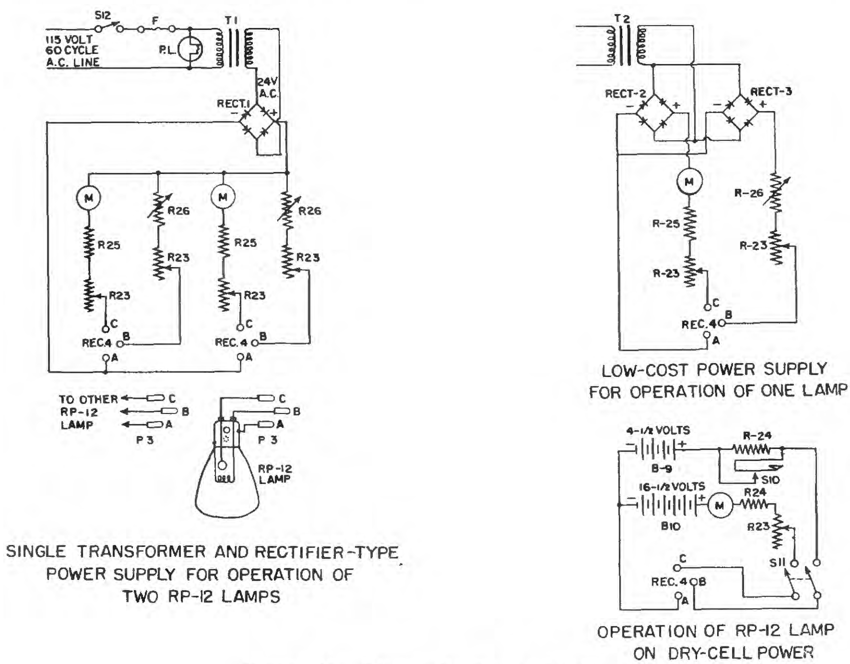

FIGURE 41.—Wiring diagram and parts.

ADAPTATION TO FIELD USE

The basic instrument is the same for either laboratory or field use. However, field use requires a battery power source not only for the amplifier unit but also for the operation of the black-light lamps. The following minor additions or changes are also required: (1) addition of a polarized receptacle or self-locating type plug in the amplifier case for connection with the battery power source. This additional connection is necessary even though the amplifier unit will operate on either a commercial power source or on a 6-volt automobile battery connected to the terminal plug shown in figure 34, because effective field use requires operation on 7½-volt dry-cell batteries, as will be explained subsequently. Hot leads from batteries should not be connected to exposed pins because of possible short circuits with metal objects during transportation for field use. (2) An additional changeover switch on the amplifier case to make possible a quick change from power to battery operation. A suitable switch for this purpose is S9 described in the parts list (p. 250). This additional switch must be placed in the circuit ahead of the one used to select the proper search head. (3) Addition of four resistors to the circuit as shown on figure 36. These resistors are of 133, 30, 16, and 56 ohms, respectively, but the values are not critical and 130-, 27-, 15-, and 56-ohm resistors would be satisfactory. It is important that the additional resistor of tube V3 (fig. 36) be placed in the circuit between the ground and the filament in order to avoid a biasing effect on the tube due to a negative return.

Field operation of the amplifier circuit is accomplished by use either of five no. 6, 1½-volt dry-cell batteries, or of plug-in type Radio A heavy-duty batteries, connected in series to produce 7½ volts. The intermediate tap is connected to the positive of the no. 2 cell to obtain 3 volts. The drain on each of the battery taps is as follows: negative, 145 milliamperes maximum; positive 3 volt, 49 milliampere minimum, 116 milliampere maximum; positive 7½ volt, 34 milliampere constant. Thus it seems reasonable to expect intermittent operation of the instrument about 4 hours a day for 30 days as a minimum before it would be necessary to change batteries. Operation of the instrument on dry-cell batteries is advisable for at least two reasons, namely, this kind of battery is generally available in smalltown shops at reasonable cost and operation of the instrument is more stable and reliable with dry cells than with a 6-volt automobile battery because such a battery could not be recharged conveniently in the field. This would result in supply voltage drops and variations in voltage with changing conditions that could not be compensated for, particularly if a trickle-charger was not available. Dry-cell operation avoids such troubles during the life of the batteries because the slow drop in voltage with use is compensated for when dark-current and sensitivity adjustments are made during use of the instrument. The instrument shown and described in this paper has the field adaption features built in at time of construction.

The battery power supply for field use has been designed to power only one black-light lamp. However, the same lamp may be used on either the reflection or the transmission search unit interchangeably. The carrying case for this power supply has two compartments. Three no. 6 1½-volt dry cells make up one unit to supply 4½ volts to the filament. This unit is wired so that only the correct connection may be made because more than 4½ volts would destroy the lamp filament.

The other compartment is built to hold eleven no. 6, $1\frac{1}{2}$ volt dry cells to supply $16\frac{1}{2}$ volts. This is about the minimum voltage required to start and operate the black-light lamp, but it has proved satisfactory. The wiring diagram for the battery power supply is shown in figure 41. The switch (S-10, fig. 41) is for starting the lamp, by raising the filament voltage momentarily to start conduction. This switch is closed for only a few seconds until the lamp starts. The battery (B 9, fig. 41) gives just enough voltage to start the lamp. The two sets of batteries are used in order to keep the drain in each set low and insure longer life. The filament current is about 170 milliamperes and the glow circuit requires about 200 milliamperes for satisfactory light output. Batteries arranged in two units as described should permit operation 4 hours a day for at least 30 days.

DISCUSSION OF BLACK-LIGHT LAMP

The small, 3-watt black-lamp used with this fluorimeter was first used in 1950 by May and Fletcher (1954) and also has been used by Kinser (1954). It is listed in the 1949 General Electric lamp catalog as F3 RP 12/360 BL. This lamp operates on very little power, generates but little heat, and produces an unusual amount of radiation in the wavelength region of 3650 A. The light output is extremely constant. Continuous operation 24 hours a day for a 3-month test period in the laboratory failed to show significant change. The operating characteristics of this lamp have not been available and so certain tests have been made as a guide to the use of this lamp. The filament operates on 4 volts but will withstand $4\frac{1}{2}$ volts momentarily to start. Because of variations in filament resistance in different lamps it is necessary to use a 40-ohm potentiometer-type variable resistor (R23) or other adjustable type resistor in the circuit. When starting a lamp for the first time or if lamps are changed it will be necessary to connect a high resistance voltmeter (20,000 ohms/volt) across the common and filament terminals of the lamp socket. Use either the 6- or 10-volt range and adjust resistor 23 for 4 volts filament circuit. This should start the lamp but after about 5 minutes the voltage must be readjusted to exactly 4 volts when the normal operating current of 200 to 250 milliamperes in the glow circuit has been set. Both filament voltage and glow circuit current should be adjusted at the same time because a large change in either circuit will result in some change in the other. The glow circuit operates at 7 to 9 volts but requires 16 volts minimum to start. The minimum operating current in this circuit is 200 milliamperes but 250 milliamperes is recommended for normal operation. The filament of the lamp causes ionization that in turn causes a current flow through the glow circuit and excitation of the fluorescent coating on the inside of the bulb. The current in the filament circuit is limited by a dropping resistor. At the instant of starting the glow circuit has infinite resistance resulting in the full voltage across the elements of the lamp. Thus the filament may be considered the cathode and the glow element the anode. When the filament heats up, conduction between the cathode and anode occurs which causes a lowering of the internal resistance and starts current flow. The current flow is controlled as conduction increases by suitable current-limiting resistors in order to prevent damage to the lamp. The intensity of the lamp for use on the fluorimeter is controlled by a 40-ohm variable resistor placed in the glow circuit. A potentiometer for this purpose has the advantage of having no "off" position. The RP–12, 3-lamp has a double contact index base. One bayonet locking-pin is opposite the other, but staggered in distance from the base to prevent operation except in one position. Sockets for these lamps are available from Cole-Hersee Co., Old Colony Ave., Boston, Mass., and the H. A. Douglass Manufacturing Co., Bronson, Mich. A usable socket for these lamps may be made from a double-contact bayonet-socket with threaded metal outer shell, by cutting out a new pin socket in one side to match the lamp-base pins.

OPERATION OF AMPLIFIER CIRCUIT

This instrument is a null-point type with provision for adjusting and balancing the entire circuit—both dark-light and reading positions— each time before measurements are made. It is substantially a direct-current voltage amplifier. Operation of the amplifier depends on a voltage drop across the 2000-megohm resistor in the grid circuit of the 2532 electrometer tube. A potential is maintained across the elements of the phototube at all times by C batteries 1 and 2. Phototube conduction is entirely dependent on the amount of light striking the tube. Therefore, in total darkness no current flow occurs in this circuit. Internal and external leakage, dust, moisture, fumes, and careless handling must be guarded against. All tubes are carefully selected, matched for characteristic performance, and aged at the factory so that replacements, when necessary, very nearly match the performance of original tubes of the same type. The electrometer tube is quite sensitive to very small grid voltages or variations in applied voltage. Any change in grid voltage varies the current in the plate circuit. Thus a change in current flow from C batteries 5 and 6 also changes the grid voltage with a resultant larger change in the plate current of tube 2531, which is read on the zero-center meter. Power from C batteries 3 and 4 serves this part of the circuit.

When the circuit is adjusted for constant current through the meter and tube 2531 with the meter hand on zero, any variation in the circuit will cause deflection of the meter hand. Thus, light striking the phototube may be measured. Adjustment of the dark-current potentiometer so that the pointer on the zero-center meter is at zero causes a potential drop across the 2000-megohm resistor sufficient to draw current through the amplifier tube 2532, which is grounded through the 150,000-ohm and 2000-megohm resistors, until a balance is reached that holds the zero-center meter on zero. Then when the switch is thrown to operating position the 2000-megohm resistor is no longer grounded but is connected to the variable voltage slide wire. Light striking the phototube now causes a current through the grid of tube 2532 with resultant deflection of the meter hand. The slide wire is connected in the circuit so that a voltage equal to that caused by light on the phototube is passed to the grid of the other tube, but in the opposite direction, and the circuit may again be balanced. Readings on the slide-wire dial are relative values of this cancelling voltage. The sensitivity-control potentiometer selects the value of the cancelling voltage available, whereas the resistors in the "range" circuits govern and maintain proper potential ratios of this voltage.

INSTRUCTIONS FOR ADJUSTMENT OF INSTRUMENT

Arrange all parts and make all connections. Connector rings on all cable connectors must be tight. Use a 6-volt storage battery rather than a commercial power supply during the test and adjustment period. Be sure the instrument on-off switch is off. Check proper battery connections and condition of C batteries with battery test voltmeter, if this feature was included during construction. Turn the instrument switch on, set the range switch to 0.1 position, turn the selector switch to position for operation of transmission head, and close the opening to the phototube by pulling shutter slide knob all the way out. After a period of about 10 seconds the hand on the zero-center meter will travel from left to right across the scale but will probably return to the left end of the scale. Turn the dark-current adjustment knob (R15) through the entire range (3 revolutions). The meter hand should respond at some point during rotation of knob. If not, then set the dark-current knob at about center of rotation range and change battery taps on C batteries one at a time by turning switches (S5, S6, S7, fig. 37) one position at a time beginning with S5 that is connected to battery number 1. Normally only one position change on switch is required to shift the meter hand to the opposite end of scale. The switch-tap combination that permits the meter hand to zero on the scale with the dark-current knob about the center of its rotation range is correct. The initial setting of switches should be as follows: Switch S5, 7½ volt positive tap of battery 1; switch S6, 7½ volt negative tap of battery 2; and switch S7, 7½ volt positive tap of battery 3. The wiring diagram of battery test circuit is shown in figure 37.

To prevent fluorescent particles from the flux buttons and other foreign matter from falling through the opening onto the light-filter glass of the transmission unit, it is desirable to place a 2-inch square of glass in the bottom of the slide opening that holds the metal flux-button retainer. Eastman 2-inch transparency picture-slide mounts are quite satisfactory as they require no cutting and have little or no fluorescence under light of the wavelength used in this instrument.

Check the low range of the transmission unit. Prepare several flux buttons of known uranium content. The writers use the shallow 7-milliliter platinum dish described by Grimaldi, May, and Fletcher (1952) to prepare flux buttons, although other sizes may be used, provided the button holder is modified. The size of the button from a 2-gram charge prepared in these dishes is 1.390 inches diameter and 0.036 inch thick. Standard flux buttons made to contain 0.000075 and 0.0001 milligram of uranium are suitable for this check test. Place one of the standard buttons in the instrument. Set the range switch to 0.1, adjust the dark current to zero center of meter, set the selector switch to transmission head, adjust the lamp current to 250 milliamperes and the slide-wire dial to 90 percent transmission. Open the shutter on the search head by pushing rod entirely in. Rotate the selectivity control knob (R17) until the meter hand can be set at zero. In case the meter hand cannot be centered by rotating the selectivity control knob, set this knob at one extreme end of the range, and start turning the slide-wire dial toward the lower end of the scale. If this does not allow a centering of the meter hand, then repeat the operation, but with the selectivity control knob set at the other extreme of its range. Disregard a tendency of the meter to zero, as the slide-wire reading nears zero on the scale, because this part of the scale is essentially the dark current. If necessary, repeat this test with the range switch set to 1.0 instead of to 0.1.

Correction of wiring on the sensitivity control or the dark-current control or both, by reversing the connections, may be necessary in case the direction of rotation of these controls is not the same as that of the slide wire. The lower or bottom terminal of the helipot is the moving contact. *Do not change this connection.* When controls are correctly wired more light on the phototube deflects the hand on the zero-center meter to the left. Clockwise rotation of the dials will again center the meter hand.

CALIBRATION AND ADJUSTMENT OF RANGE

Turn on instrument, power source, and lamps. Allow a warm-up period of about 1 hour before use. Prepare sets of standard flux buttons of known uranium content and blanks. Three or more buttons for each uranium value are desirable, for example, 0.000025, 0.00005, 0.000075, 0.0001 milligram of uranium and blanks. Switch to the transmission unit and set the range for 0.1. Place a 0.0001-milligram button in position. Adjust dark current to zero on dial with shutter on search head closed (pull knob full out). Turn the slide wire to read about 90 on scale. Open the shutter by pushing knob all the way in. Zero the center meter again by rotating sensitivity control knob. If this cannot be done, rotate slide-wire knob until the meter hand centers. In case the button contains too much uranium to be read on the 0.1 range, switch to the 1.0 range and obtain a reading from the slide-wire dial. The black-light lamp current may be adjusted to give either more or less light intensity as desired, but it should not be operated at less than 200 milliamperes. All buttons should be read when the light intensity is exactly the same. Obtain readings on all standard buttons and graph the machine readings against the uranium content. This should establish the low range of the instrument. A plot of data similar to that obtained by following the preceding instructions is shown in figure 42. From this data a permanent reference standard may be prepared. Acetate film 0.005 inch thick with matte surface on only one side is quite satisfactory for preparation of a low-value permanent standard. A disk of this material, cut to fit the button holder, and masked to attain the desired fluorescence value by covering the film with a copper disk, approximately $\frac{1}{16}$-inch thick, bored to remove a portion of the center, has been very stable with little or no change in fluorescence detectable over a period of 9 months. When preparing the cooper mask it is convenient to start with a $\frac{3}{32}$-inch hole in the center and the disk scribed with concentric rings about $\frac{1}{16}$-inch apart. Place the standard flux button of desired value that is to be the upper limit of range for a particular scale setting in one slide opening. The masked standard is placed in the adjacent slide position so that they may be read alternately. The hole in the mask is then enlarged with a rat-tail file until the desired reading is obtained. Filing of the hole must be kept centered by following the scribed rings as a guide. When completed the film may be fastened permanently to the bottom edges of the mask with transparent tape. One side of the film will fluoresce differently from the other, therefore the standard must be positioned the same for all readings.

When the low range of the instrument has been established and permanent standard prepared for the 0.1 setting of the transmission unit, the range of the 1.0 scale may be established as follows: Balance the dark current and set the slide wire to read 100 percent transmission for the fluorescence value of the permanent standard on the 0.1 range. Change the range switch to 1.0. If the ratio of the 0.1 switch is correct, the slide-wire dial reading should be 10. If the hand on the center

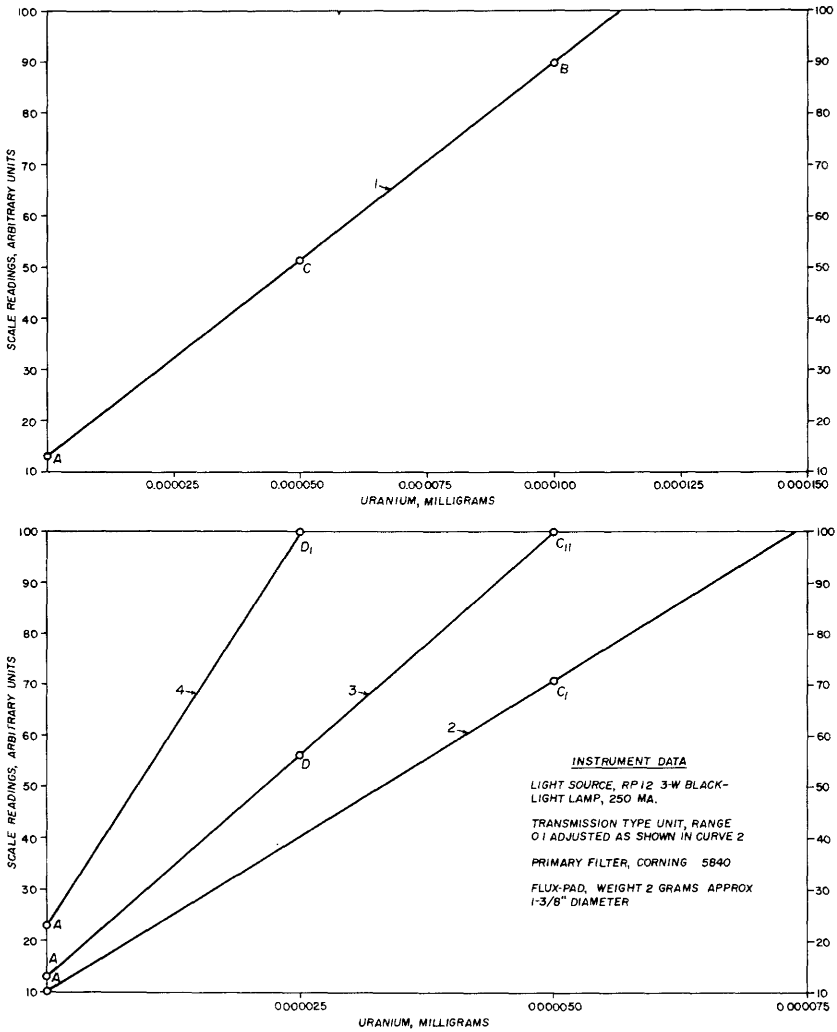

FIGURE 42.—Change of range and sensitivity of low scale available within range of sensitivity control without other changes in instrument.

meter does not read zero with the slide-wire reading 10, an adjustment of resistors is indicated. Either increase or decrease the values of R9 a, b (figs. 34, 35) by 10 ohms to determine whether an increase or decrease in resistance is required. The exact resistance value required to give the desired ratio must be determined experimentally. Repeat the above test until the proper ratio is attained within the limit of error of reading the dial. Be sure machine has warmed up and dark-current balance is correct and stable when range ratio is 10 to 1.0. If the permanent standard was calibrated to read 100 percent transmission, equivalent to 0.0001 milligram uranium on the 0.1 scale, then the

1.0 scale should be very nearly 0.001 milligram uranium for a slide-wire setting of 100 percent transmission. However, for precise work empirical curves should be prepared using flux buttons of known uranium content because of errors inherent in the method and possible instrument error.

The reflection unit is independent of the transmission unit and must be calibrated separately. A fluorescent glass standard of greater brilliance than the standard used in the transmission unit may be prepared from a suitably masked 2-inch square Corning glass no. 3750. The range of fluorescence may be adjusted over rather wide limits as desired. However, the range of the sensitivity control knob, black-light intensity, and characteristics of tubes in the instrument limit and control the working range. It may be desirable to obtain several tubes of each number, particularly no. 2532, so that the search-head assemblies may be tested for range and performance. The writers' experience indicates that changing from the search head of the transmission unit to the search head of the reflection unit requires no change in the rest of the instrument, such as changing battery taps, etc. If a particular tube combination requires such changes in operation, then the faulty tube must be located and replaced.

The calibration of the reflection unit may be made so that the working range of uranium fluorescence extends the range of the transmission unit. However, the writers prefer to set the high-value glass standard to read 100 percent transmission on the 1.0 scale of the reflection unit for the maximum amount of uranium usually expected in routine work. Then the 0.1 scale extends the working range down to lower values. Such an arrangement results in a highly desirable overlap of the reflection and transmission ranges. Setting the reflection unit to read a definite high value on the 1.0 scale also permits one to control the slope of the high standard curve and results in greater sensitivity and accuracy for determinations in the high range. In case the fluorescent glass standard, when masked, is still too brilliant for a reasonable calibration, the intensity may be reduced by placing a wire mesh over the light filter or by changing the primary filter. The following Corning filters may be used: nos. 5840, 5874, and 5860 that pass 55, 50, and 21 percent of black light of 365 millimicron wavelength, respectively.

DISCUSSION

The fluorimeter described here has a sensitivity equal to or better than that of several fluorimeters of different design that have been tested under working conditions at the Denver, Colo., laboratories of the Survey. The working range for concentrations of uranium in the fused-flux wafers is unusually advantageous for laboratories analyzing different types and grades of samples. Analysis of samples containing traces, as well as samples of ore-grade materials, can be accomplished without resorting to unusual sample size, large dilutions, or great variation in aliquot size. The low-voltage circuit of this instrument, about 18 volts maximum with most of the circuit operating at 6 volts or less, tends to minimize leakage caused by moisture, dust, and fumes, with resultant stability and reliability of operation under adverse conditions. Provision for use of a desiccant, in the amplifier unit and phototube housing when the instrument is used under conditions of high relative humidity will aid stability of measurements made under such conditions.

Figure 42 shows a calibration curve for the transmission unit of the Model '54, set to read on the 0.1 range. Points B and C, curve 1, show readings of 90 and 51.5 on the slide-wire dial for uranium values of 0.0001 and 0.00005 milligram uranium, respectively. This curve is similar to the range that will be obtained if the instrument is calibrated as described. Also shown in figure 42 are curves 2, 3, and 4 that demonstrate different settings of sensitivity made possible within the range of the sensitivity control knob. No changes in the instrument are required except rotation of the sensitivity control to a different part of its range. Point C_1, curve 2, reads 71 for the 0.00005 milligram uranium button, but this same button may be read as 100 (C_{11}, curve 3) by merely adjusting the sensitivity control knob. Likewise points D, curve 3, and D_1, curve 4, show different settings that may be assigned a flux button containing 0.000025 milligram uranium.

Thus the top range of the low scale on the 0.1 setting may be varied from 0.000075 to 0.000025 milligram uranium by changing only the setting of the sensitivity control knob (figure 42). This change in sensitivity also changes the blank readings from 10 to 23 but affords a good working curve of almost any desired slope for accurate work. This illustration does not cover the full range of settings possible on the range of the sensitivity control adjustment but does demonstrate the versatility of the instrument.

Figure 43 shows the stability of the instrument and the phototube when the temperature inside the phototube housing was raised by heating the exterior to cause a temperature change in the range of 28° to 40° C (82.4° to 104.0°F). Normal changes in temperature have little or no effect on the precision of uranium determinations made with this instrument.

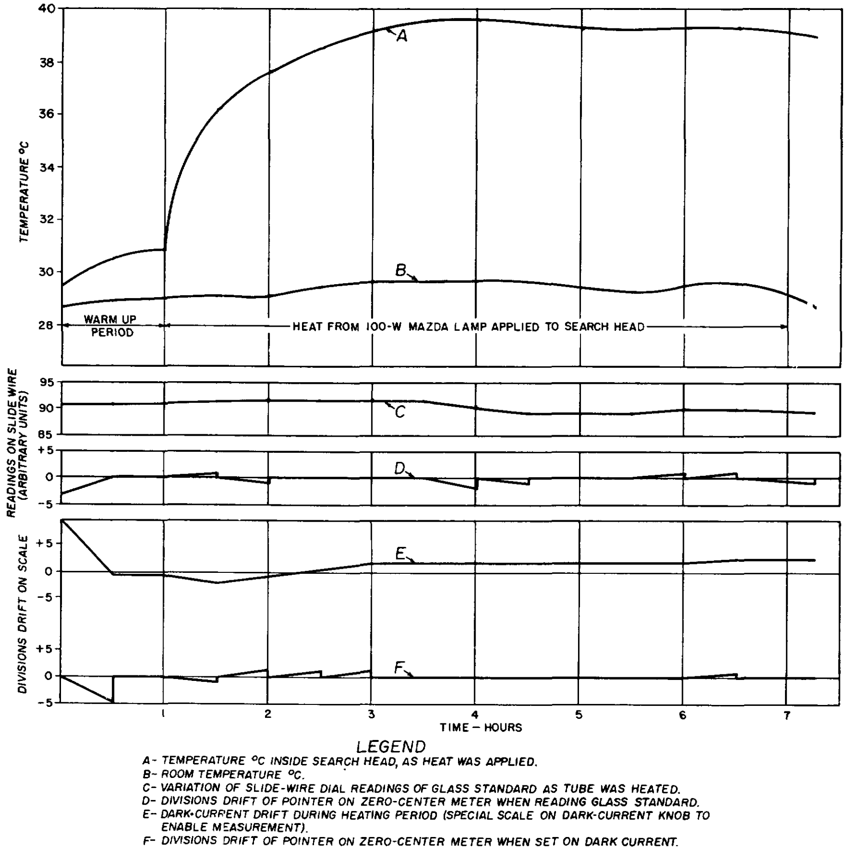

FIGURE 43.—Effect of temperature change (28° to 40° C.) on stability of phototube and reproducibility of instrument readings.

LIST OF PARTS FOR MODEL '54 FLUORIMETER

Code	*Figure no.*	*Item and description*
B1–6	35, 38	Batteries, radio C, 7½ volt.
B7	36	Battery, 6 volt, storage, automobile type, 120 ampere-hour capacity or larger. Not needed if commercial-type power source is used.
B8	36	Batteries, five 1½ volt, radio plug-in type.
B9	41	Batteries, three no. 6, 1½ volt, dry cells.
B10	41	Batteries, eleven no. 6, 1½ volt, dry cells.
g	35	Common ground.
M1	31 (g), 35	Milliammeter, Beckman DU, zero-center (range unknown).
P1, P2	38, 35	Plugs, Amphenol no. 79–05 M, heavy-duty radio connectors.

Code	Figure no.	Item and description
P3	41	Plugs, polarized, to fit receptacle 4.
P4	36	Plugs, polarized, to fit receptacle 5.
Rec 1, 2, 3	38, 35	Receptacles, Amphenol no. 79–P05F, heavy-duty radio connectors.
Rec 4	41	Receptacles, three wire standard type to fit polarized plugs.
Rec 5	36	Do.
R1	32, 35	Resistor, 39 ohms, 1 watt.
R2	32, 35	Resistor, 47 ohms, 1 watt.
R3	32, 35	Resistor, 1 megohm, ½ watt.
R4	32, 35	Resistor, 150,000 ohms, ½ watt.
R5	32, 35	Resistor, 2,000 megohms (Beckman).
R6a	34, 35	Resistor, 2.7 ohms, 1 watt.
R6b	34, 35	Resistor, 22 ohms, 1 watt.
R7a	34, 35	Resistor, 4.7 ohms, 1 watt.
R7b	34, 35	Resistor, 120 ohms, 1 watt.
R8a	34, 35	Resistor, 27 ohms, 1 watt.
R8b	34, 35	Resistor, 470 ohms, 1 watt.
R9a	34, 35	Resistor, 27 ohms, 1 watt.
R9b	34, 35	Resistor, 4.7 ohms, 1 watt.
R10	34, 35	Resistor, 15 ohms, 1 watt.
R11a	34, 35	Resistor, 39 ohms, 1 watt.
R11b	34, 35	Resistor, 560 ohms, 1 watt.
R12	34, 35	Resistor, 150 ohms, 1 watt.
R13a	34, 35	Resistor, 820 ohms, 1 watt.
R13b	34, 35	Resistor, 120 ohms, 1 watt.
R14a	34, 35	Resistor, 68 ohms, 1 watt.
R14b	34, 35	Resistor, 100 ohms, 1 watt.
R15	34, 35	Helipot, 300 ohms, 3 revolution, Beckman model C, dark-current adjustment.
R16	34, 35	Slide-wire disk, 100 ohms, Beckman DU (percent transmission).
R17	34, 35	Helipot, 200 ohms, 10 revolution, Beckman model A (sensitivity control).
R18	34, 35	Grid to plate resistor, 100 megohms, Beckman.
R19	36	Resistors, 56 ohms, 1 watt.
R20	36	Resistors, 133 ohms, 1 watt.
R21	36	Resistors, 30 ohms, 1 watt.
R22	36	Resistors, 16 ohms, 1 watt.
R23	41	Resistors, 40 ohms, 4 watt. Variable Clarostat.
R24	41	Resistors, 10 ohms, 10 watt.
R25	41	Resistors, 40 ohms, 10 watt.
R26	41	Resistors, 75 ohms, 10 watt, adjustable, set at 60 ohms.
S1	31 (h), 35	Switch, range selector, rotary cermaic, two circuit, two position.
S2	31 (b), 35	Switch, head selector, rotary ceramic six circuit, two position.
S3	31 (d), 35	Switch, battery on-off, rotary ceramic, four circuit, two position.
S4	33, 35	Switch, dark current, in phototube head.
S5	31 (i), 37	Switch, tap, battery no. 1, rotary ceramic, one circuit, four position.

Code	*Figure no.*	*Item and description*
S6	31 (j), 37	Switch, tap, battery no. 2, rotary ceramic, one circuit, five position.
S7	31 (k), 37	Switch, tap, battery no. 3, rotary ceramic, one circuit, four position.
S8	31 (l), 37	Switch, battery test, rotary ceramic, two circuit, eight position.
S9	36	Switch, battery change-over, 6–7½ volt, rotary ceramic, six circuit, two position.
S10	41	Switch, lamp start, 1 ampere, 125 volt, two circuit, momentary contact, push to close circuit.
		Cable, 5-conductor, shielded, rubber covered.
S11	41	Switch, on-off, DPST toggle, 3 ampere, 125 volt, bat handle.
S12	41	Switch, on-off, SPST toggle, 3 ampere, 125 volt, bat handle.
V1	32 (E), 35	Phototube, Beckman no. 2342–1, blue sensitive, 220–625 mμ.
V2	32 (A), 35	Electrometer tube, Beckman no. 2532, Sylvania no. 1229.
V3	34, 35	Amplifier tube, Beckman no. 253, Sylvania no. 1229.
F	41	Fuse, 1 ampere, 3 AG, and chassis mounting.
P. L	41	Pilot light, NE51, with resistor socket.
T1	41	Transformer, 115 volt, 60 cycle, primary; 50 va, 24 volt, secondary.
T2	41	Transformer, 115 volt, 60 cycle, primary, 35 va, 24 volt, secondary remote-control type.
Rect 1	41	Rectifier, full wave bridge, 25 volt a-c, 20 volt d-c, 3-ampere, dry-plate type.
Rect 2, 3	41	Rectifiers, full wave bridge, 25 volt a-c, 20 volt d-c, 600-milliampere, dry-plate type.
M	41	Milliammeters, d-c, 0/500.
Lamp	41	Lamp, 3 watt, 12–16 volt, d-c, fluorescent black-light, indexing base, RP–12 bulb.

Specify 2-inch polished light filters by color specification number, otherwise a molded, somewhat thicker, glass will be obtained.

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