Chemistry

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GEOLOGICAL SURVEY

THE 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.

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Trace Elements Investigations Report 520

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THE 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

A fluorimeter for the determination of uranium by the fluorimetric method has been designed, built, and used in the U. S. Geological Survey laboratory, Denver, Colo., for the past five years. 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 reliability of readings made under conditions expected for field work. A detailed description and simple instructions are given together with photographs and shop drawings so that construction and use of this instrument may be possible in small laboratories with limited facilities.

INTRODUCTION

The determination of uranium by the fluorimetric method requires use of a suitable instrument to complete the determination unless visual estimation is used. Most laboratories prefer to use instrumentation because of greater precision and convenience. However, commercial development of an instrument, suited particularly to the determination of uranium fluorescence in fused wafers of sodium fluoride or fluoride carbonate flux mixtures, was accomplished only recently.1/ Consequently development work on fluorophotometers or fluorimeters for the determination of uranium has been carried on extensively for about eight years in many laboratories engaged in uranium analyses: Fischer and Pickle (1946); Center (1948); Davey and Florida (1949); Fletcher, May, and Slavin (1954); Fletcher, May, and Anderson (1954); May and Fletcher (1954); Jacobs (1950); Kaufman et al. (1950); Zimmerman (1950); Yeaman (1951); Price et al. (1953); Kinser (1954); and Kelley et al. (1954).

Development of a fluorimeter suited to our needs was started in 1948 and resulted in a satisfactory instrument in 1949. This fluorimeter used a circuit modified from that of the Beckman DU spectrophotometer;2/ it also used the three electronic tubes similar to those of the Beckman instrument. However, the original design has been improved and modified from time to time by substituting an RP-12, 3-w, black-light lamp for the EH-4 lamp, by separating the phototubes from the main amplifier unit for use separately as search heads for both transmission and reflection type units, and by generally improving the range and stability. The basic features of this instrument have been tested through essentially continuous use since 1949.

1/ The Jarrell-Ash Co., 165 Newbury St., Boston 16, Mass.

2/ Trade names and commercial designations have been used to describe many of the components used in construction of the Model '54 fluorimeter. Such designations avoid lengthy descriptions of doubtful clarity, that otherwise would be necessary to identify properly items well known by trade names. This use is not to be construed as an endorsement of any particular product or manufacturer. Undoubtedly equivalent items are made that may be substituted, but no systematic search could reasonably be made for these at present.

An adaptation of the instrument for field use with dry-cell batteries also has been made but only tested in the laboratory and not under field conditions. However, the instrument is versatile, the range of uranium fluorescence may be measured in fused fluoride carbonate flux mixtures, when varied from 0.001 microgram to 0.1 milligram uranium in a 2-g flux wafer, and the sensitivity may be adjusted for a predetermined range required on a particular job, or other adaptations may be made quite simply.

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

The assembled instrument as used in the laboratory is shown in plate 1. The box, A, is 11-1/2 x 14-1/4 x 8-3/4 inches and contains the amplifier unit. Search units, B and C, contain electrometer and phototubes, black-light sources, light filters, and slides to hold both standard uranium glass plates and unknown flux wafers for measurement of uranium fluorescence. Unit B is for reflection-type measurements usually for samples of greatest uranium content, whereas unit C is for transmission-type measurements of wafers con-

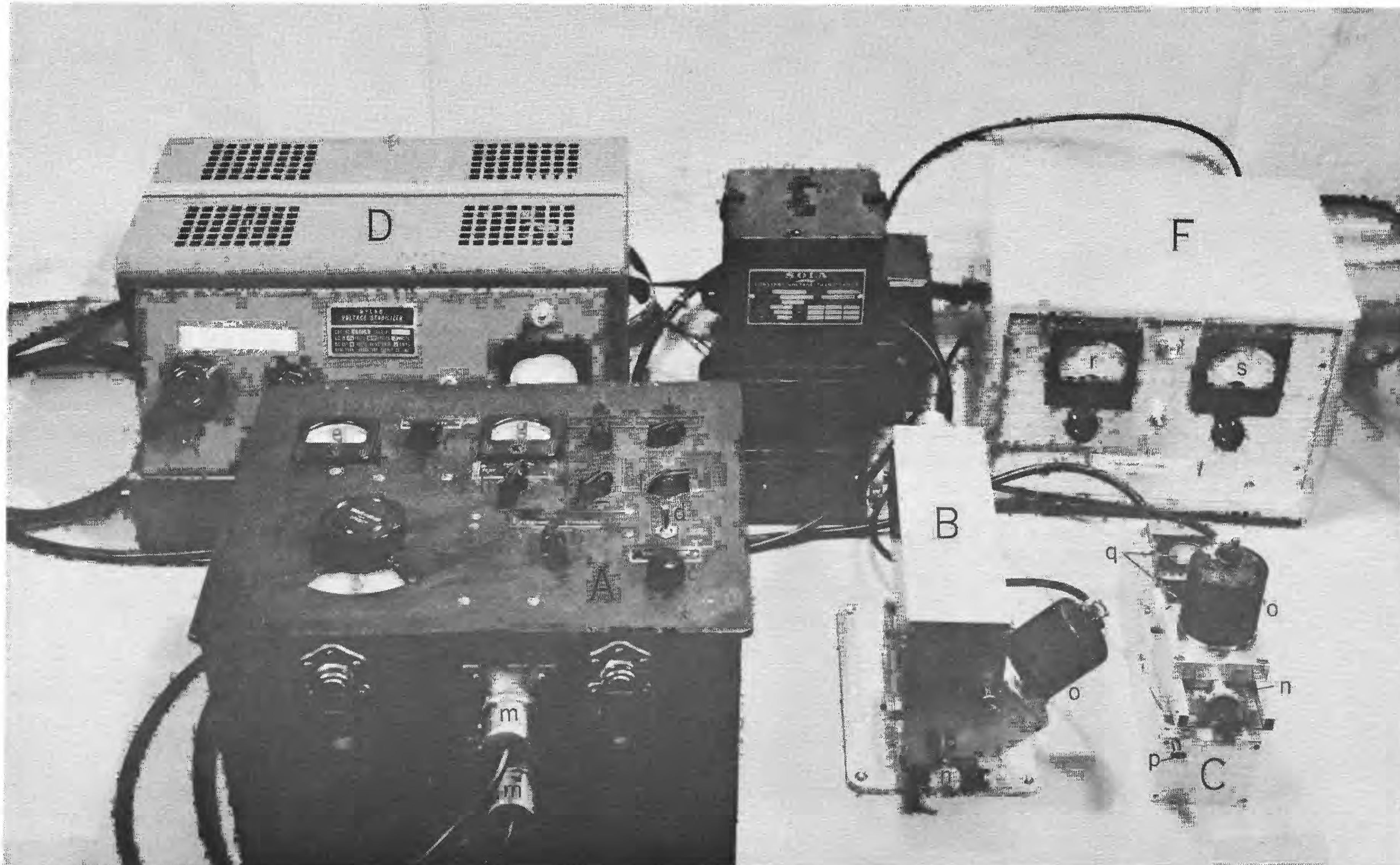

Plate 1.--Assembled fluorimeter, Model '54.

taining very low levels of uranium concentration. D is a commercially available voltage stabilizer and power source. E is a commercially available constant-voltage transformer of 250-va capacity, and F is a double-unit power source used to control electrical current to the black-light lamps used on the search units B and C. The amplifier unit, A, may be operated with either a commercial power source, a 6-v storage battery, or with dry cells as described later for field use. However, the amplifier unit also requires six radio C batteries mounted inside the case regardless of the type of external power-source used.

Plate 1 also shows control knobs mounted on the lid of the amplifier unit, A, as follows: (a) is the slide wire knob and dial, (b) is a selector switch designating use of either the reflection (B) or transmission (C) search units, (c) is for circuit balancing or dark-current adjustment, (d) is an on-off toggle switch, (e) is volt meter, 0-15 v range for testing batteries, (f) is the sensitivity control knob, (g) is a meter having a special scale with a zero in the center of the range for balancing the dark current and indicating the slide wire settings, (h) is a three-position range switch with check, 0.1 and 1.0 positions, (i), (j) and (k) are adjustments for C battery taps so that voltage may be changed as required without making changes inside the cabinet, (l) is a test switch with seven positions for checking voltage of individual batteries, and (m) are amphenol receptacles no. 79-P05F and plugs no. 79-05M (for cables Belden no. 8425) to the two search heads (B) and (C), respectively.

Lettering on items (B) and (C), plate 1, show the following features: (n) is the pull knob for the slide that holds positioned glass standards (q) and the recess for measuring fluorescence of unknown flux wafers, (p) is the pull for the sliding shutter arrangement, (o) is the housing for the 3-w black-light lamps. On F (r) and (s) are 0 to 500 milliammeters in the power circuit for two black-lights on units (B) and (C), respectively.

Electrometer and phototube assembly

Different views of the phototube assembly (B and C, plate 1) and shutter arrangement of the search heads are shown in plates 2 and 3. The housing is made from sheet aluminum. Two sides are 1/4-inch thick and two are 1/8-inch thick so that the edges of the thick stock may be milled to lighttight 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, 2-1/4 inches from center to one end of plate, threaded for attachment to the light filter holder and lamp assembly (36 threads). The overall dimensions of the assembled housing are 10-1/4 x 2-7/8 x 2-3/4 inches.

Identification and location of items in the phototube housing, plates 2 and 3 are: Resistors, R1, R2, R3, R4, and R5, with resistances of 39 ohms, 47 ohms, 1 megohm, 150,000 ohms, and 2000 megohms, respectively; (I) are porcelain insulator mounts, (A) is an electrometer amplifier tube, type 32 for low grid current applications (Sylvania no. 1229, specially selected, or Beckman no. 2532), (B) is a dark-current switch operated by the shutter slide (C).

This dark-current switch is shop-made of phosphor bronze strips 0.020 inch thick by 3/8 inch wide. The upper and lower blades are 2-1/4 inches long, but the center blade is 2-3/4 inches in length (pl. 3). All three blades are mounted on plexiglass block 1/2 inch thick, held in place by two

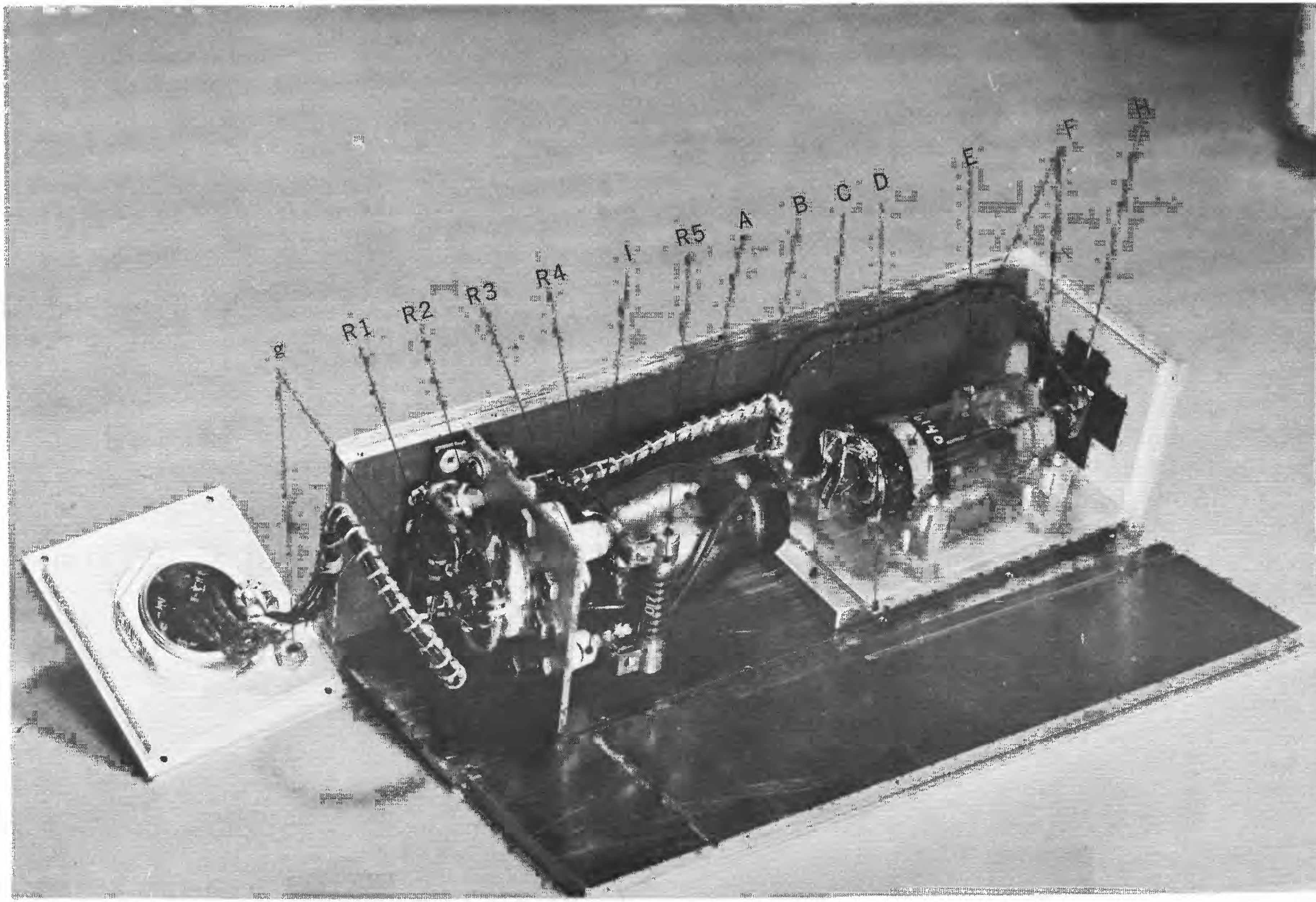

Plate 2.--Phototube assembly and shutter arrangement.

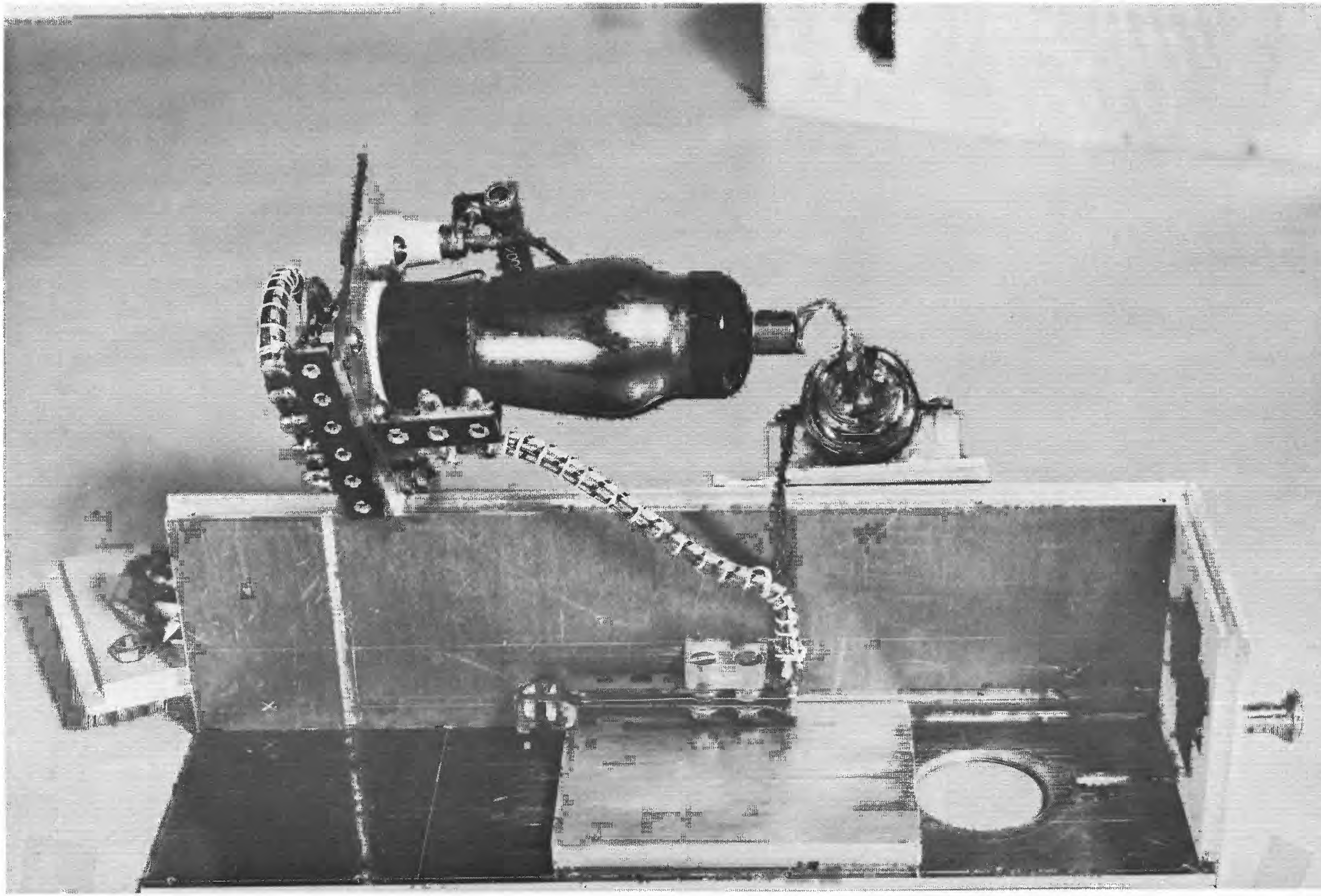

Plate 3.--Switch and shutter arrangement in search head.

oval-head no. 6-32 screws, side plate drilled to clear block and tapped. All blades are fastened to the block with two no. 6-32 machine screws and nuts. The center and lower blades are drilled for extruded fiber washers and are separated with fiber washers of the same diameter as that of the extruded washers. The contact points are of silver, soldered to the bronze strips. The upper blade is drilled to clear the no. 6 screw. All blades are insulated from each other. About 1/8-inch clearance is allowed between switch blades and side plate. Caution must be used in mounting of switch so that contact with the grounded tube coating is avoided during operation.

The end of the long center blade has a piece of plexiglass fastened to it so that the shutter slide will operate the switch. This plexiglass block is recessed to clear the lower blade. 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 make firm 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 (pl. 2) 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.

A suitable switch may be made from one set of contacts from a 12 amp, type 200-4, Guardian relay contact assembly, if the materials described above are not readily available. To do this, one set of contacts is removed intact from the rest of the assembly. The center blade is then removed, turned over so that wiring lugs are all on the same side, and reinforced by placing a short contact blade, from a Guardian relay parts kit no. 200-3, on top of the center blade. The terminal lug should be cut from this reinforcing blade and it should be shortened, if the other end touches contact button on center blade. The upper and lower wire lugs are bent away from the center one for more wire clearance. The plexiglass shoe is fastened to the long center blade by two round-head screws (2-56 thread) after carefully drilling and fitting. Contact tension adjustment is made by bending. With the light shutter open and switch in raised position, clearance of 0.025 inch should be allowed between the contacts.

The shutter plate (C, pl. 2 and 3) is aluminum, 2-3/8 x 3-3/8 x 1/4 inches, cut so that the width is a free fit between the sides of the housing. A brass operating rod for the shutter slide is 1/8 x 3-5/8 inches, threaded at both ends for attachment 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 1/4 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, 2-5/8 x 4 x 1/16 inches, fitted into 1/8-inch-deep slots in both sides of the housing. A clearance of 1/4 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 no. 4-40, short, Allen setscrew. These screws hold the tube mounting plate in place and allow for adjustment and alignment of the 1-1/4 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, pl. 2) is held in a socket mounted on an aluminum plate, 2-1/2 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° in the housing and the plane of the filament kept vertical. Slots to position the mounting plate are 1-17/32 inches from center to end of housing plate.

Plate 4 shows the details of wiring underneath the amplifier cabinet lid and arrangement of parts. The resistors and other parts are further identified in the appendix. The circuit wiring diagram is shown in figure 1. Plate 4 also shows the terminal block and leads for connection to 6-v storage battery (when operation on battery is desired), 100 megohm resistor (R-18) for grid on amplifier tube, mounted with porcelain stand-off insulators, and amplifier tube (Beckman 2531). The cabinet is lined with sheet copper

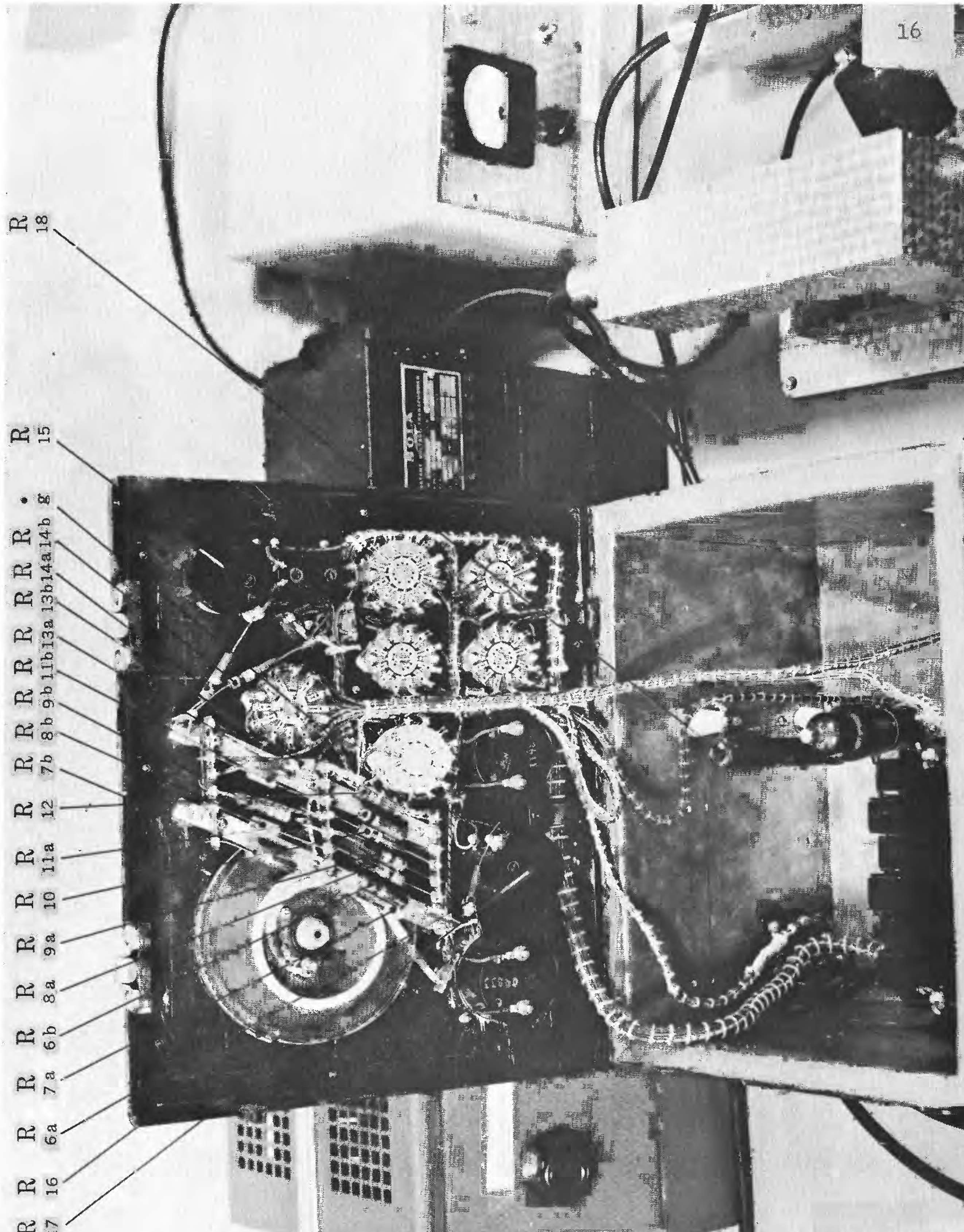

Plate 4.--Location of wiring and parts in amplifier cabinet.

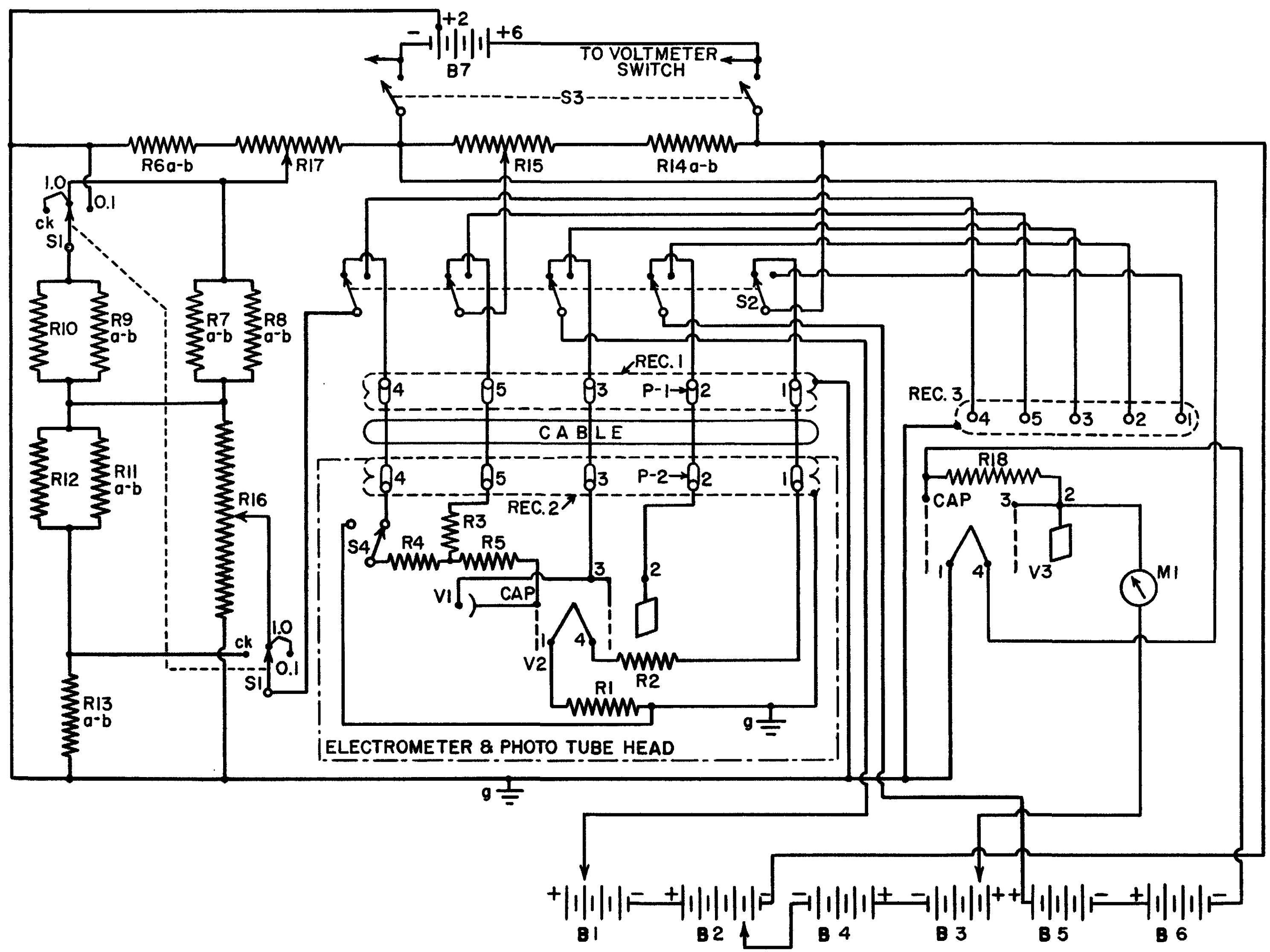

Figure 1. WIRING DIAGRAM OF MODEL '54 FLUORIMETER

PARSHALL. 8-9-54

for shielding, except the lid which is made from 1/4-inch sheet aluminum. Pointed setscrews in the lid (not shown, pl. 4) contact the copper sheet to obtain maximum shielding of the circuit.

The slide-wire dial should be purchased complete with knob, shaft and stationary contact. (See appendix.) The slide wire is mounted on the 1/4-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 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 bore of the knob. As this diameter is slightly more than half an 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 head bolt 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 "0" end of the resistance wire. Some of this detail cannot be seen in plate 4 because certain parts are hidden by the resistor assembly. 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 5/16-inch, standard, fine thread, hexagonal nut. Shielded wire 17-1/2 inches long is satisfactory for leads (Belden no. 8431, phonograph pick-up arm cable). The shield is connected to the "0" end of the resistance wire, the other end of wire to the two-lug terminal strip.

The resistor mounting (pl. 4) consists of two metal strips bent to clear the slide-wire assembly. Three 8-lug terminal strips (Cinch-Jones no. 56 C) are mounted on these strips. The top of each lug is removed to provide an open slot to hold the resistors and permit changes or adjustment if necessary.

Plate 5 shows one view of the mount for six 7-1/2 v C-batteries. Another view is shown in plate 6. This mount is built of plastic, wired on the underneath side to an arrangement of contact springs that give the proper connections to the battery binding posts. These springs must be long enough to make contact with the large diameter base of the battery terminal posts and strong enough to keep tension on the contacts. The diameter of the springs is sufficient to allow entry of the binding posts when the nuts are removed. The batteries are mounted upside down in the mount with battery number 1 in the left position of holder (pl. 6).

Plate 5 also shows the assembled housing used for transmission measurements but with the black-light lamp holder removed from the top and the

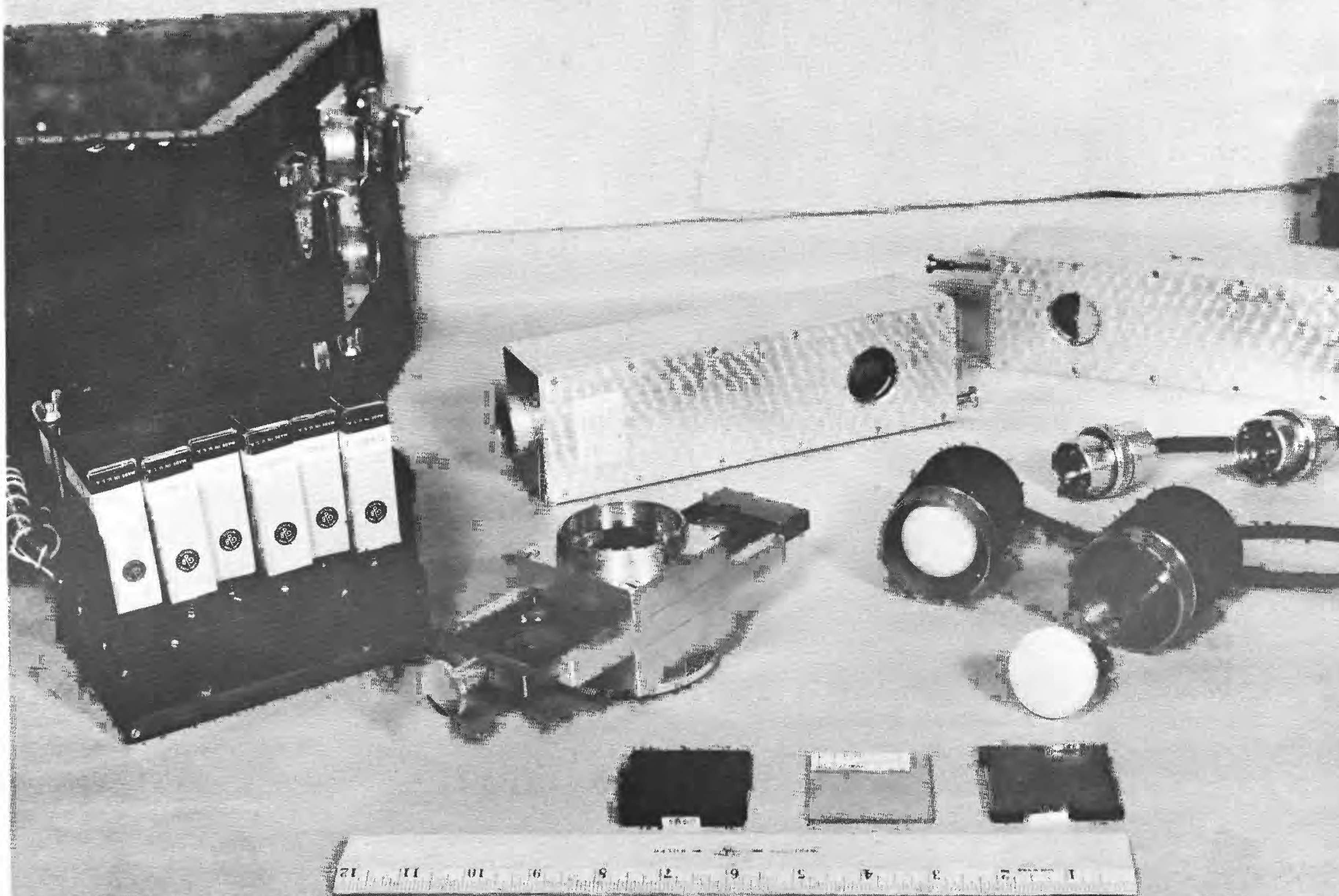

Plate 5.--Transmission assembly, RP-12 ultraviolet lamps and housings, light filters, and mount for 7 1/2-v C batteries.

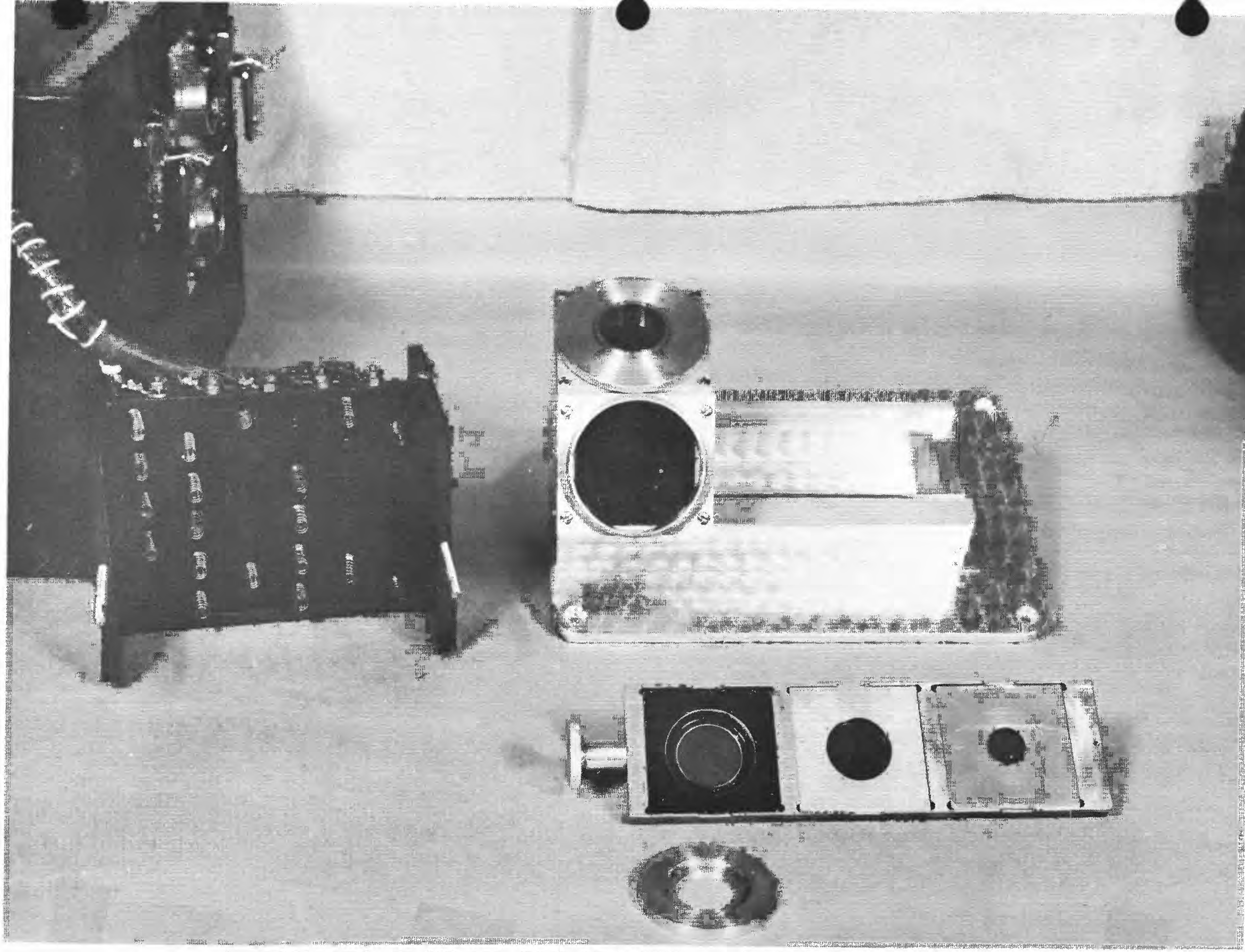

Plate 6.--Reflection assembly, slide holding position for flux wafer and two masked glass standards, and detail of mount for C batteries.

search-head removed from the bottom. Shop drawings for construction of this unit are given in figure 2. 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-in. square light filter (Corning 5840) and the black-light lamp with holder. Below the slide are mounted Corning light filters nos. 3486 and 9780, also 2-in. polished squares, and a threaded fitting for attachment to the search-head. A light-tight fit is obtained by machining the parts and lapping them together for close fit.

Plate 6 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 more uniform light distribution and intensity could be achieved than was possible with a round hole of limited diameter. 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. 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 in figure 3.

Plate 7 and plate 1, F, show the power supply for the two black-light lamps when the instrument is operated on 110-v a-c house current. The wiring diagram and list of parts required to assemble this unit are given in figure 4.

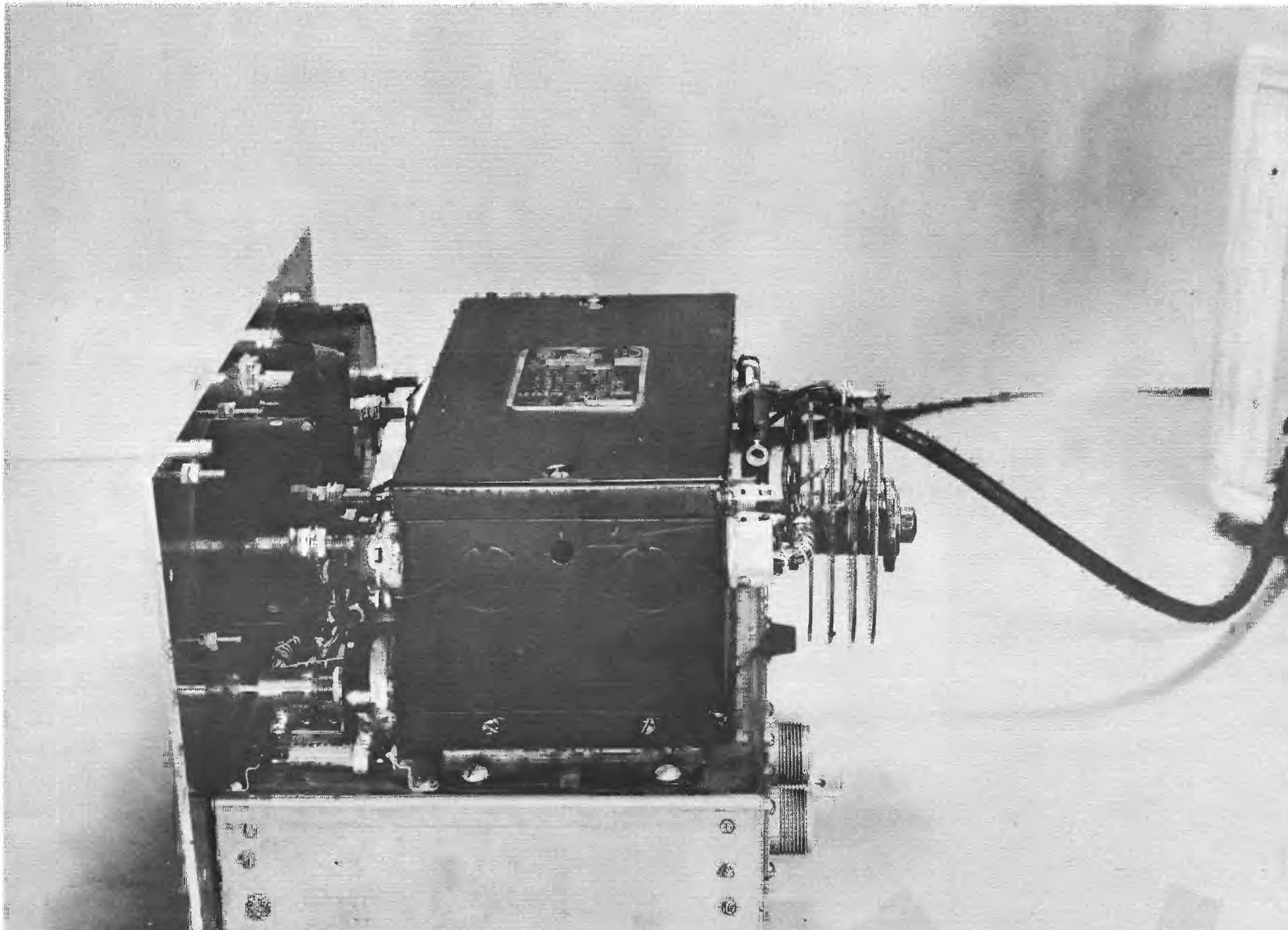

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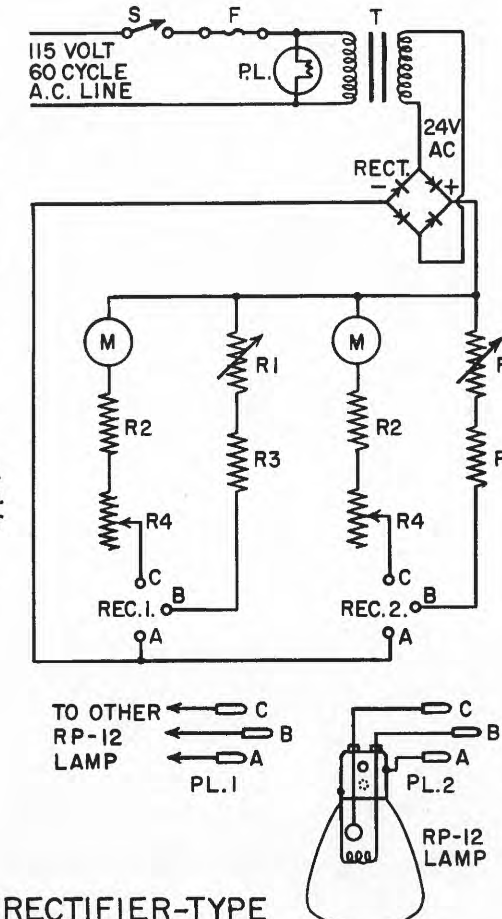
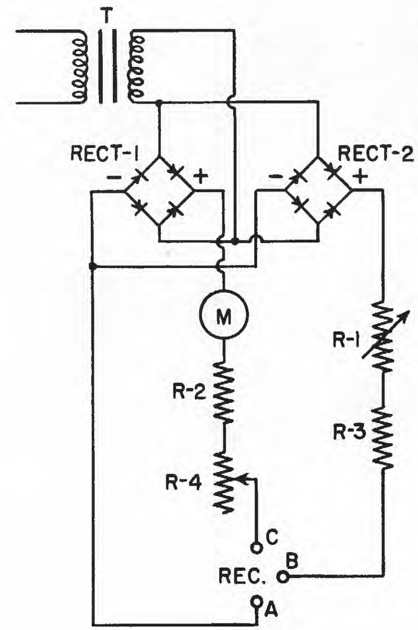
Plate 7.--Ultraviolet lamp power source operated on house circuit.



S- S.P.S.T. WALSCO #S-330 TOGGLE SWITCH.
F- 1 AMP. 3 AG FUSE & CHASSIS MOUNTING.
P.L.- NE51 PILOT LITE WITH RESISTOR SOCKET.
T- JEFFERSON SIGNALLING TRANSFORMER. 50 V.A. 115 V.A.C. PRI.- SECONDARY TAPPED TO 24 VOLTS MAX. AT 2 AMPS.
RECT.-FEDERAL CAT. #2103 OR #2104. 26 VOLT A.C. 20 VOLT D.C. 3 OR 6 AMP.
M- 0/500 MILLIAMMETERS. 2 REQ'D.
R1- 75 Ω 10 WATT ADJUSTABLE RESISTORS. 2 REQ'D.
R2- 30 Ω 10 WATT RESISTORS. 2 REQ'D.
R3- 25 Ω 10 WATT RESISTORS. 2 REQ'D.
R4- 40 Ω 4 WATT VARIABLE CLAROSTAT CAT. #10-40. 2 REQ'D.
REC. 1-REC. 2 - POLARIZED RECEPTACLES.
PL.1-PL.2- PLUGS TO MATCH REC. 1 & 2.
RP-12 FLUORESCENT LAMP.

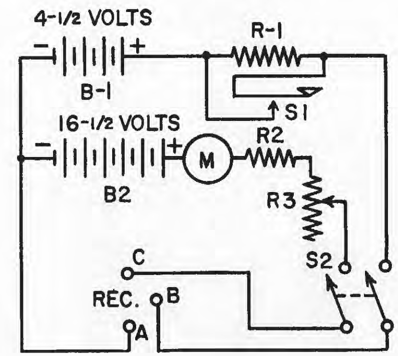
SINGLE TRANSFORMER AND RECTIFIER-TYPE POWER SUPPLY FOR OPERATION OF TWO RP-12 LAMPS



T- G.E.CO. CAT. #RT-1 TRANSFORMER 115/24 VOLT, 60 CYCLE, 35 V.A. REMOTE CONTROL TYPE.
RECT-1 & RECT-2. FEDERAL SELENIUM RECTIFIERS, CAT. #1017. 25 VOLT A.C. INPUT, 600 Ma. D.C. OUTPUT.

ALL OTHER PARTS SAME AS TWO LAMP POWER SUPPLY.

LOW-COST POWER SUPPLY FOR OPERATION OF ONE LAMP



B1- 3 #6-1-1/2 VOLT DRY CELLS
B2- 11 #6-1-1/2 VOLT DRY CELLS OR 2-6 VOLT HOTSHOT BATTERIES AND 3 #6-1-1/2 VOLT DRY CELLS.
R1-R2. 10 Ω 10 WATT RESISTORS.
S1- WALSCO CAT. #S-340 2-CIRCUIT MOMENTARY CONTACT SWITCH, CONNECTED NORMALLY OPEN.
S2- WALSCO CAT. #S-332 D.P.S.T. TOGGLE SWITCH.
R3- *(SAME AS R4 IN TWO LAMP UNIT.)*
M- *(SEE TWO LAMP UNIT.)*
REC.- " " " "

OPERATION OF RP-12 LAMP ON DRY CELL POWER

Figure 4. WIRING DIAGRAM AND PARTS

PARSHALL. 8-2-54

A power source to operate only one lamp has been described by Kinser (1954). A power source to operate one lamp has also been built and used extensively for the instrument described here. The circuit diagram and parts for this unit are also shown in figure 4. This small, compact power unit has proved quite satisfactory for operation of one black-light lamp and costs less to build than other models.

The power source to operate two lamps is housed in a cabinet 11 x 7-1/8 x 9 inches (inside dimensions) with an aluminum panel on the front to hold the meters, switch, pilot light, and knobs on the variable resistors (pl. 1). All the parts are mounted on a chassis with wiring underneath where possible (pl. 7). The rectifier and two, 30-ohm, 10-w resistors are mounted on the rear of the chassis to insure adequate ventilation (pl. 7). One end of resistors are connected directly to rectifier, the other to separate terminals on tie-point strip mounted on rear of transformer. Adjustable resistors, 75 ohm, 10-w, are mounted, one on each end of chassis, toward panel, accessible for adjustment. The variable resistors, 40-ohm, 4-w, are located one below each meter and are mounted on chassis directly in front of transformer. These are connected through shaft extensions to knobs on front panel. Resistors, 25-ohm, 10-w, are mounted in front-center of chassis between meters and are supported on tie-point straps. The two output receptacles (amphenol A. N. type connectors) are mounted on rear of chassis. This power unit is fused for protection, the cap on the extractor-type fuse holder being visible on plate 7. The low-voltage wires are carried through the low-voltage end of transformer case.

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 (Amphenol, series 91, microphone connector). This additional connection is necessary even though the amplifier unit will operate on either a commercial power source, or on a 6-v automobile battery connected to the terminal plug shown in plate 4, because effective field use requires operation on 7-1/2-v 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 change-over switch on the amplifier case to make possible a quick change from power to battery operation. A suitable switch for this purpose is Centralab no. 2517 or equivalent. This additional switch must be placed in the circuit ahead of the one used to select the proper search-head. (3) Addition of three resistors to the circuit as shown on figure 3. These resistors are 133, 30 and 16 ohms, respectively, but the values are not critical and 130, 27 and 15 ohm resistors would be satisfactory. It is important that the additional resistor on tube V3 (fig. 5) 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 of either five no. 6, 1-1/2-v, dry-cell batteries, or plug-in type, Radio A, Eveready no. 741, connected in series to produce 7-1/2 v. The intermediate tap is

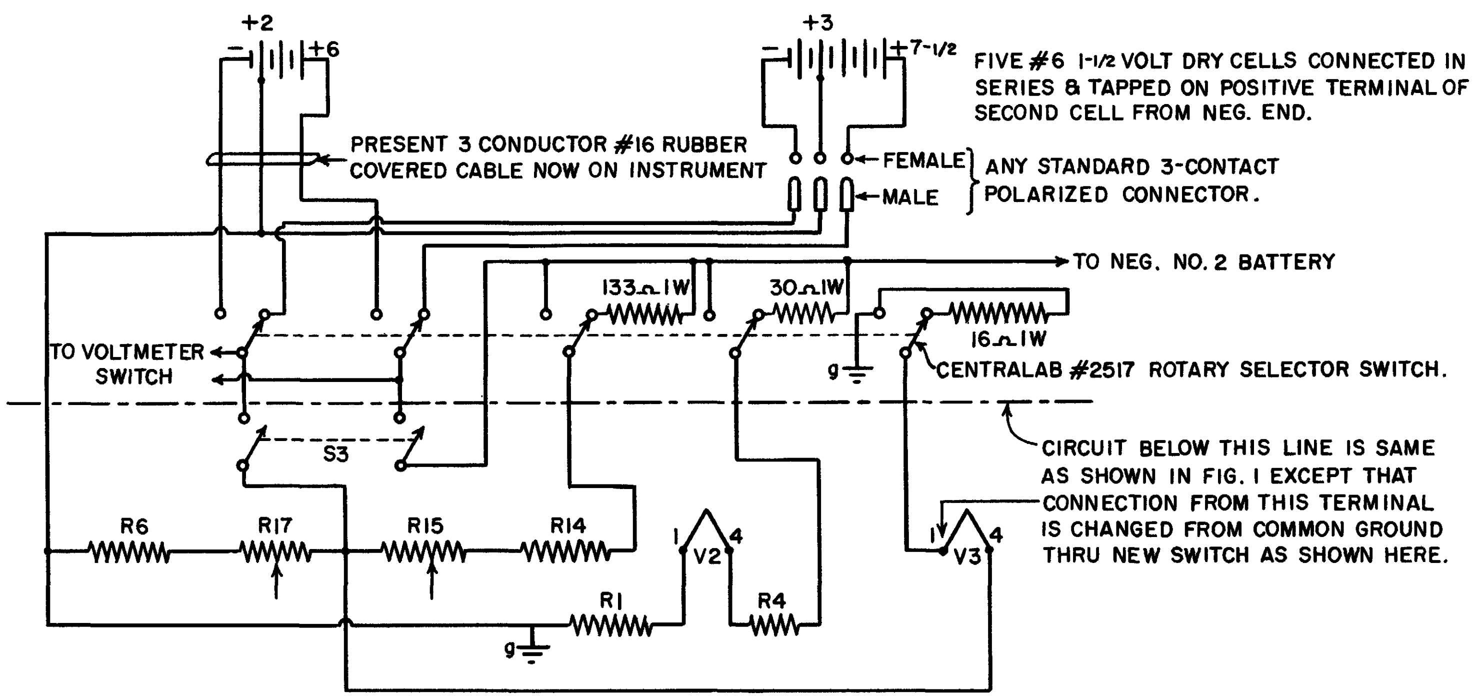


Figure 5. MODIFICATION OF WIRING DIAGRAM FOR OPERATION ON DRY CELL BATTERY

connected to the positive of the no. 2 cell to obtain 3 v. The drain on each of the battery taps is as follows: negative, 145 ma maximum; positive 3 v, 49 ma minimum, 116 ma maximum; positive 7-1/2 v, 34 ma constant. Thus it seems reasonable to expect intermittent operation of the instrument about four 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 advised for at least two reasons, namely, this kind of battery is generally available in small town shops at reasonable cost, and operation of the instrument is more stable and reliable with dry cells than with a 6-v 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 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 transmission search units interchangeably. The carrying case for this power supply has two compartments. Three no. 6, 1-1/2-v, dry cells are one unit to supply 4-1/2 v to the filament. This unit is wired so that only the correct connection may be made because more than 4-1/2 v would destroy the lamp filament. The other compartment is built to hold eleven no. 6, 1-1/2 v, dry cells to supply 16-1/2 v. 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 5. The switch

(S-1, fig. 4) 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 2, fig. 4) gives just enough voltage to start the lamp. The use of the two sets of batteries was selected in order to keep the drain in each set low and insure longer life. The filament current is about 170 ma and the glow circuit requires about 200 ma 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-w, black-light 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 v but will withstand 4-1/2 v momentarily to start. A minimum of 150 ma and a maximum of 200 ma operating current is required for the filament circuit. The glow circuit operates at 7 to 9 v but requires 16 v minimum to start. The minimum operating current in this circuit is 200 ma with a maximum not to exceed 500 ma. 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 flow 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-w 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 d-c voltage amplifier. Operation of the amplifier depends on a voltage drop across the 2000 megohm resistor in the grid circuit of the no. 2532 electrometer tube. A potential is maintained across the elements of the phototube at all times by C batteries nos. 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 nos. 5 and 6 also changes the grid voltage with a resultant larger change in the plate current of tube no. 2531, which is read on the zero-center meter. Power from C batteries nos. 3 and 4 serves this part of the circuit. When the circuit is adjusted for constant current through the meter and tube no. 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 no. 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 no. 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" and "check" circuits govern and maintain proper potential ratios of this voltage.

The "check" circuit (fig. 1) is calibrated by changing resistors R 13 a and b. When wired as shown, it works only on the 1.0 range for a calibrated setting of 100 percent transmission on the dial.

ADJUSTMENT OF ASSEMBLED INSTRUMENT

Arrange all parts and make all connections. Connector rings on cables must be tight. Use a 6-v storage battery rather than a commercial power supply during the test and adjustment period. Be sure the instrument toggle-switch is off (pl. 1, A, d). Check proper battery connections and condition of C batteries with battery test voltmeter (pl. 1, A, j), 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 (pl. 1, A, c) 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 (i, j, k, pl. 1), one position at a time beginning with (i) that is connected to battery no. 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 (i), 7-1/2 v positive tap of battery 1; switch (j), 7-1/2 v negative tap of battery 2; and switch (k), 7-1/2 v positive tap of battery 3. The wiring diagram of battery test circuit is shown in figure 6.

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.

Now check the low range of the transmission unit. Prepare several flux buttons of known uranium content. We use the shallow, 7-ml 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-g 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 mg 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 ma and the slide-wire dial to 90 percent transmission. Open the shutter on the search head by pushing

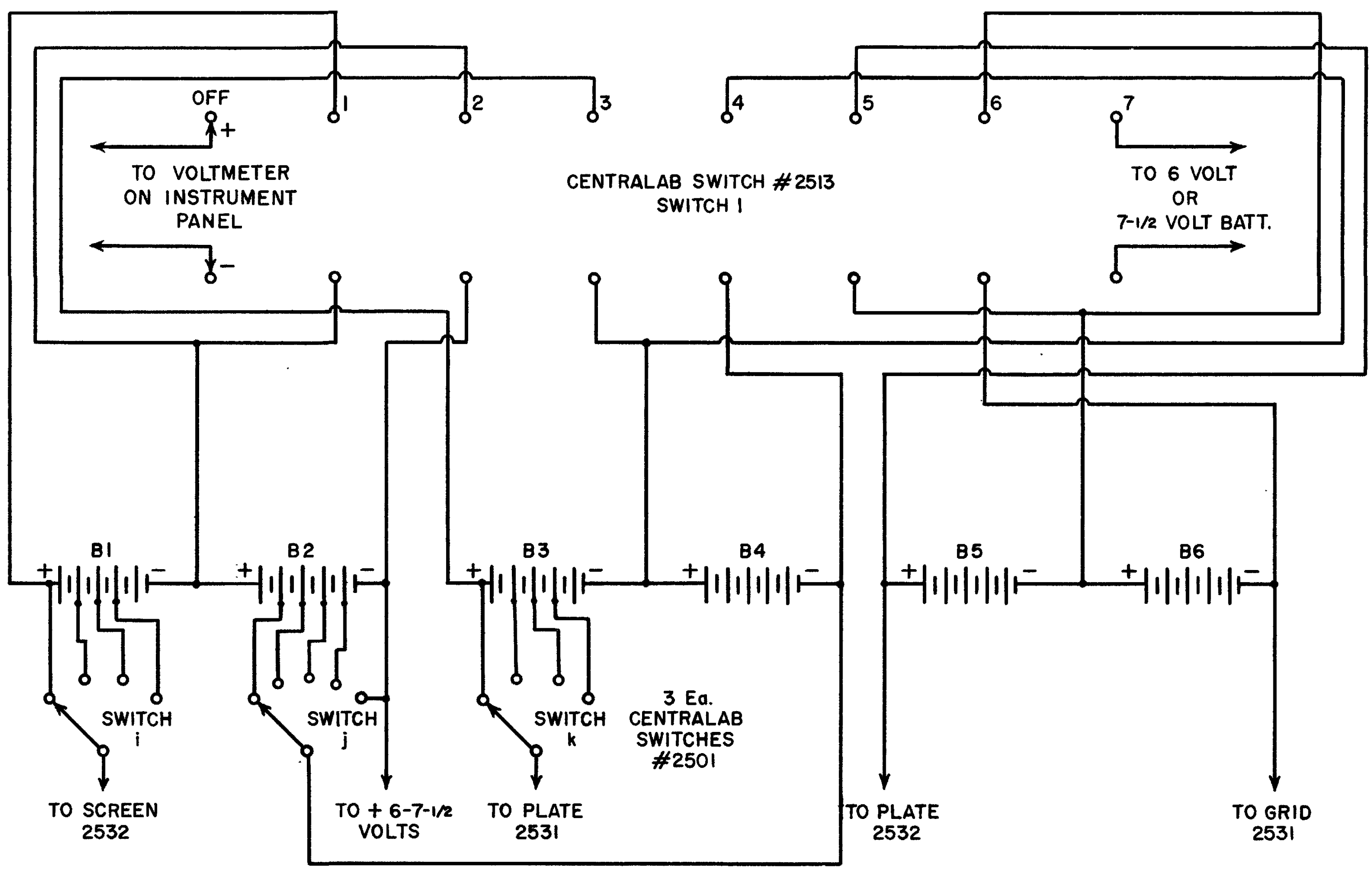


Figure 6. WIRING DIAGRAM OF BATTERY TEST CIRCUIT

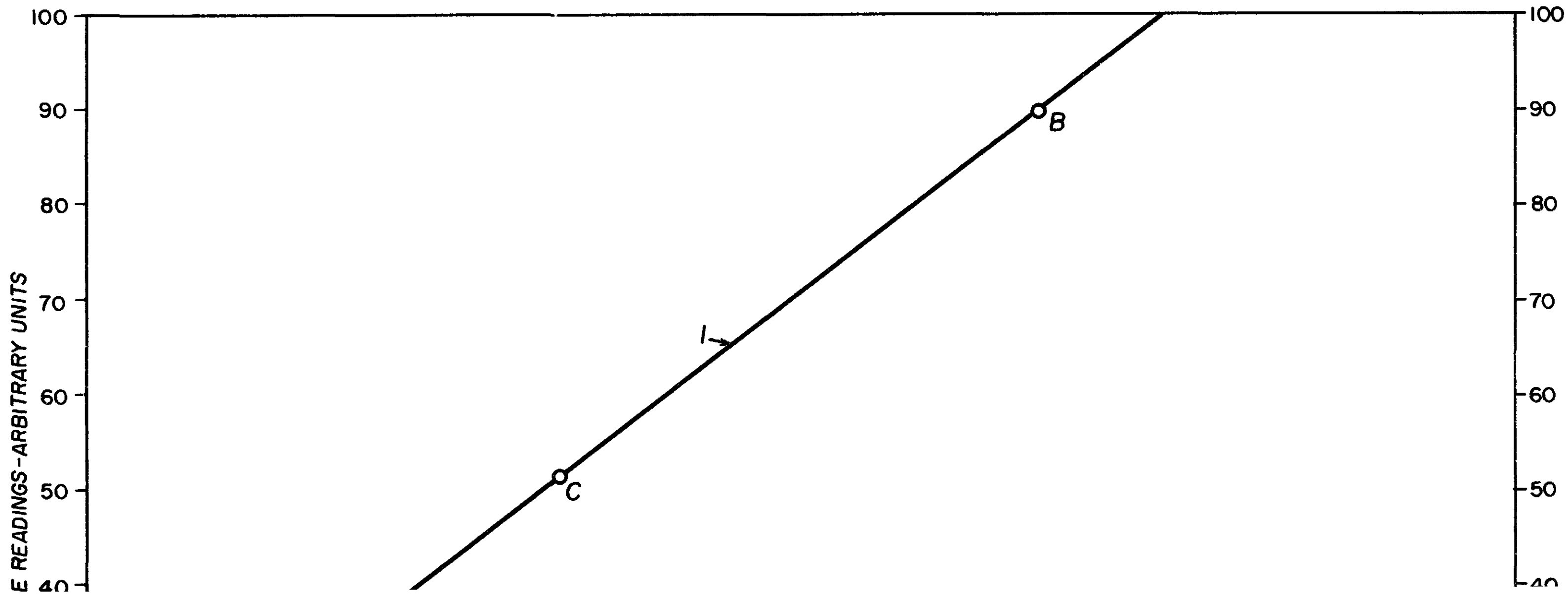
PARSHALL. 8-4-54

rod clear in. Rotate the selectivity control knob (pl. 1, A, f) 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. Repeat this test with the range switch set to 1.0 instead of to 0.1 if necessary.

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 one 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 mg of uranium and blanks. Switch to the transmission unit and set the range for 0.1. Place a 0.0001-mg 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 ma. All buttons should be read when the light intensity is exactly the same. Obtain readings on all standard buttons and graph the machine readings vs. 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 7. 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 fluorescent value by covering the film with a copper disk, approximately 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 copper mask it is convenient to start with a 3/32 inch hole in the center and the disk scribed with concentric rings about 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 and search unit 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 opened up with a rat-tail file until the desired reading is obtained.


E READINGS-ARBITRARY UNITS
100
90
80
70
60
50
40
B
C
I

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 fluorescent value of the permanent standard on the 0.1 range. Change the range switch to 1.0. If the ratio of the 0.1 to 1.0 switch is correct, the slide-wire dial reading should be 10. If the hand on the center meter does not read zero with the slide-wire reading 10, an adjustment of resistors is indicated. Either increase or decrease the values of R 9 a, b (fig. 1 and pl. 4) 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 experimentally determined. 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 mg uranium, on the 0.1 scale, then the 1.0 scale should be very nearly 0.001 mg uranium for a slide-wire setting of 100 percent transmission. However, for exact work working 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. Our 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.

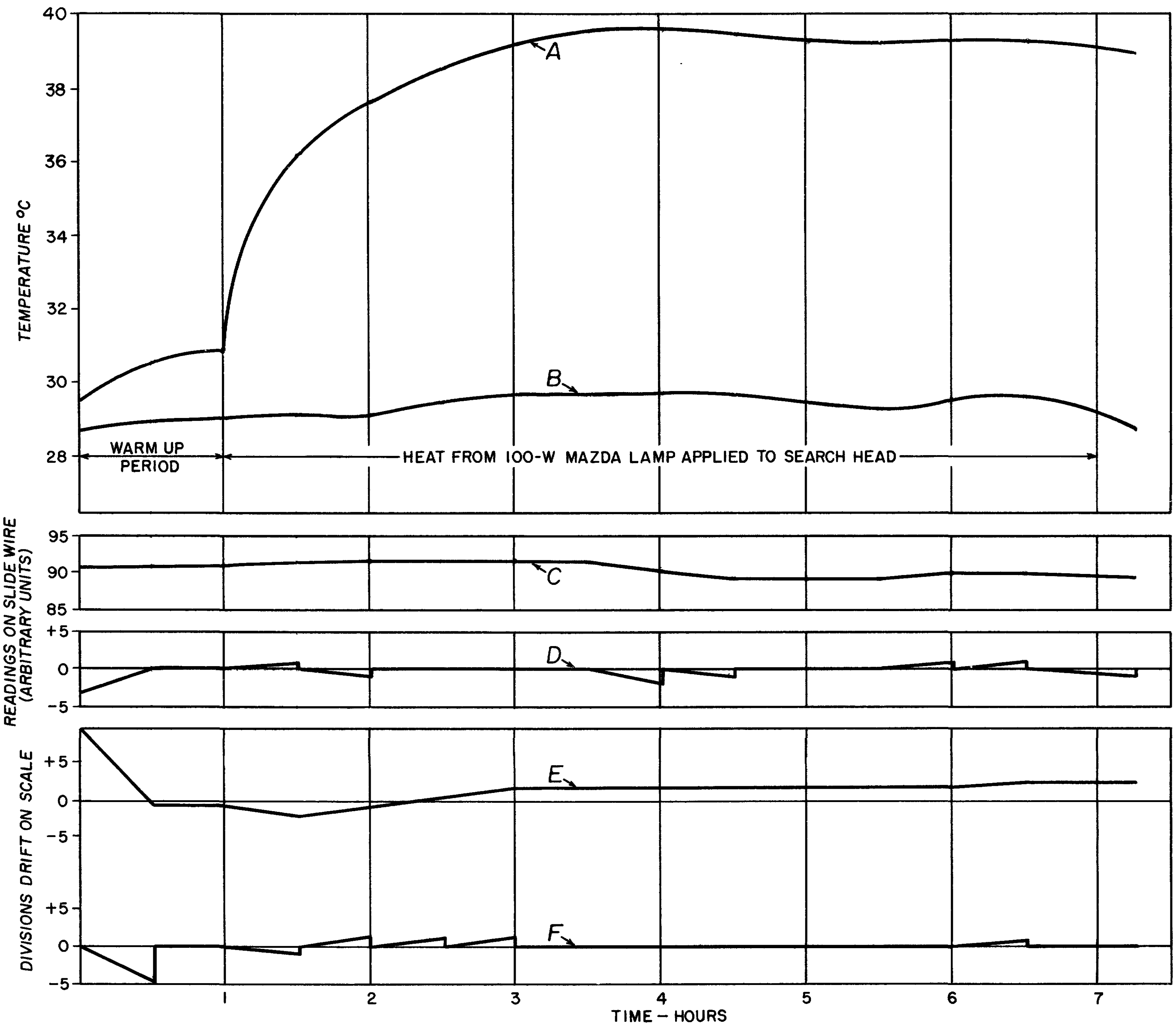
The calibration of the reflection unit may be made so that the working range of uranium fluorescence extends the range of the transmission unit. However, we prefer to set the high-value glass standard to read 100 percent transmission on the 1.0 scale of the reflection unit for the maximum amount of uranium usually expected in routine work. Then the 0.1 scale extends the working range down to lower values. Such an arrangement results in an overlap of the reflection and transmission ranges but this is highly desirable. 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 filters, 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 mμ 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 Denver, Colo. 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 ore-grade materials, may 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 v maximum with most of the circuit operating at 6 v 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, such as Drierite, in the amplifier unit and phototube housing when the instrument is used in areas of high relative humidity will aid stability of measurements made under such conditions.

Figure 7 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 mg uranium, respectively. This curve is similar to the range that will be obtained if the instrument is calibrated as described. Also shown in figure 7 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 mg uranium. Thus the top range of the low scale on the 0.1 setting may be varied from 0.000075 to 0.000025 mg uranium by changing only the setting of the sensitivity control knob (figure 7). 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 8 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).



LEGEND

A- TEMPERATURE °C INSIDE SEARCH HEAD, AS HEAT WAS APPLIED.
B- ROOM TEMPERATURE °C.
C- VARIATION OF SLIDE WIRE DIAL READINGS OF GLASS STANDARD AS TUBE WAS HEATED.
D- DIVISIONS DRIFT OF POINTER ON ZERO-CENTER METER WHEN READING GLASS STANDARD.
E- DARK CURRENT DRIFT DURING HEATING PERIOD (SPECIAL SCALE ON DARK CURRENT KNOB TO ENABLE MEASUREMENT).
F- DIVISIONS DRIFT OF POINTER ON ZERO-CENTER METER WHEN SET ON DARK CURRENT.

FIG. 8- EFFECT OF TEMPERATURE CHANGE (28 to 40°C) ON STABILITY OF PHOTOTUBE AND REPRODUCIBILITY OF INSTRUMENT READINGS

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APPENDIX

Partial list of parts for Model '54 fluorimeter

Code on figure 1	Shown on plate	Item, type, and source
B1-6	5	Radio C batteries, 7-1/2 v, Eveready no. 773, General no. 208. RCA not suitable.
B7	--	Battery, 6 v storage, automobile type, 120 amp-hr capacity or larger. Not needed if commercial-type power source is used.
--	--	Cable, 5 conductor, shielded. Belden no. 8425. Require 2 lengths of 5-feet each.
g	--	Common ground.
M 1	1 (g)	Milliammeter, Beckman DU, zero-center (range unknown).
P1, P2	5	Plugs, Amphenol no. 79-05M, heavy-duty radio connectors.
Rec 1,2,3	5	Receptacles, Amphenol no. 79-P05F, heavy-duty radio connectors.
R 1	2	Resistors, 39 ohms, 1 w.
R 2	2	Resistors, 47 ohms, 1 w.
R 3	2	Resistors, 1 megohm, 1/2 w.
R 4	2	Resistors, 150,000 ohms, 1/2 w.
R 5	2	Resistors, 2000 megohms (Beckman).
R 6a	4	Resistors, 2.7 ohms, 1 w.
R 6b	4	Resistors, 22 ohms, 1 w.

Code on figure 1	Shown on plate	Item, type, and source
R 7a	4	Resistors, 4.7 ohms, 1 w.
R 7b	4	Resistors, 120 ohms, 1 w.
R 8a	4	Resistors, 27 ohms, 1 w.
R 8b	4	Resistors, 470 ohms, 1 w.
R 9a	4	Resistors, 27 ohms, 1 w.
R 9b	4	Resistors, 4.7 ohms, 1 w.
R 10	4	Resistors, 15 ohms, 1 w.
R 11a	4	Resistors, 39 ohms, 1 w.
R 11b	4	Resistors, 560 ohms, 1 w.
R 12	4	Resistors, 150 ohms, 1 w.
R 13a	4	Resistors, 820 ohms, 1 w.
R 13b	4	Resistors, 120 ohms, 1 w.
R 14a	4	Resistors, 68 ohms, 1 w.
R 14b	4	Resistors, 100 ohms, 1 w.
R 15	4	Helipot, 300 ohms, 3 revolution, Beckman model C dark-current adjustment.
R 16	4	Slide-wire disk, 100 ohms, Beckman DU (percent transmission).
R 17	4	Helipot, 200 ohms, 10 revolution, Beckman model A. Sensitivity control.
R 18	4	Grid to plate resistor, 100 megohms, Beckman.
S 1	1 (h)	Switch, check-range selector, Centralab no. 2505.
S 2	1 (b)	Switch, head selector, Centralab no. 2571.
S 3	1 (d)	Switch, toggle, battery on-off, DPST, 12 amp, 125 v, GC no. 1350, Insuline Co. no. 1281.
S 4	3	Switch, dark current, in phototube head.
V 1	2 (E)	Phototube, Beckman no. 2342-1, blue sensitive, 220-625 $m\mu$.

Code on figure 1	Shown on plate	Item, type, and source
V 2	2 (A)	Electrometer tube, Beckman no. 2532, Sylvania no. 1229.
V 3	4	Amplifier tube, Beckman no. 2531, Sylvania no. 1229.

Jobbers of Beckman equipment should be able to supply resistors R 3, R 15, R 16, R 17, R 18; meter m 1; and tubes V 1, V 2, and V 3. The slide wire (R 16) should be ordered complete with knob, shaft, slide-wire drum, and the stationary contact assembly.