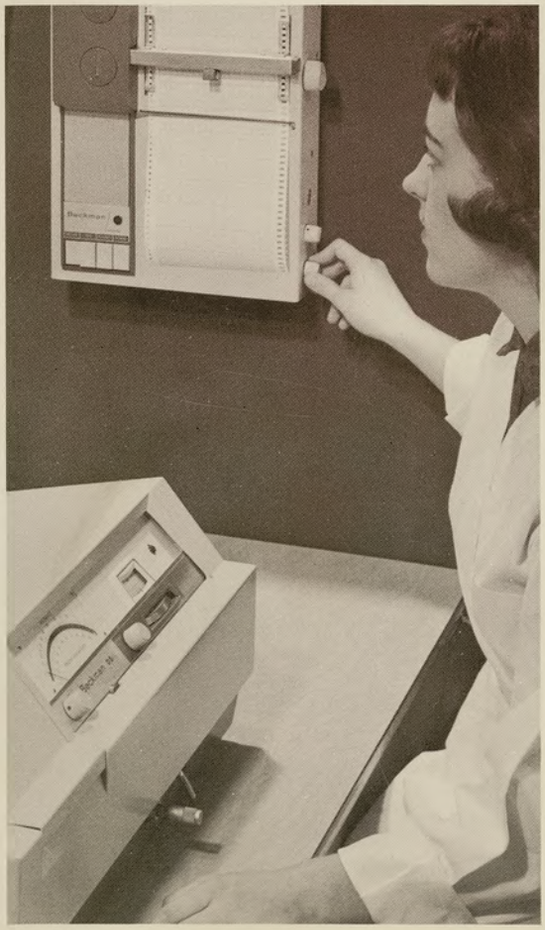
Beckman®

INSTRUCTION MANUAL

MODEL DB SPECTROPHOTOMETER



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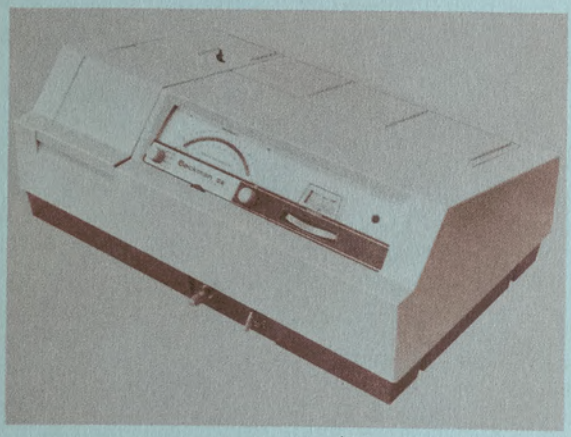
MODEL DB
SPECTROPHOTOMETER
OPERATING INSTRUCTIONS



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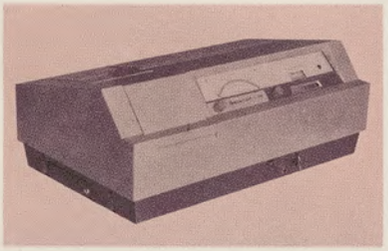
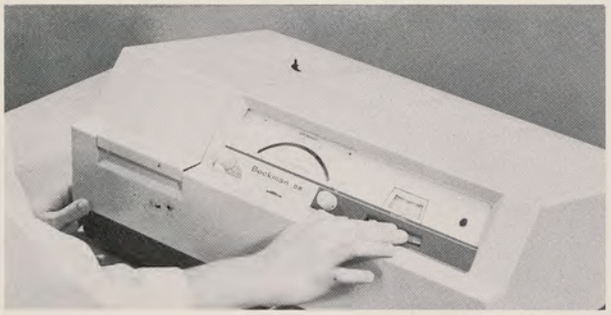
Illustrations

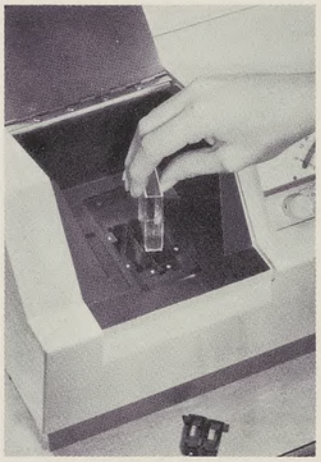
Figure	Title	Page
Front.	Model DB	
Figure 1.	Model DB in Operation	1
Figure 2.	Specifications	2
Figure 3.	Optical Diagram	2
Figure 4.	Front Panel Controls	4
Figure 5.	Micrometer Slit Adjust	5
Figure 6.	Back Panel Controls	6
Figure 7.	Internal Adjustments	8
Figure 8.	Recorder Chart Speed Data	10
Figure 9.	Cell Handling Techniques	16
Figure 10.	Slit System	18
Figure 11.	Vibrating Mirror Assembly	19
Figure 12.	Circuit Diagram, Signal Amplifier	20
Figure 13.	Circuit Diagram, Meter Circuit	20
Figure 14.	Circuit Diagram, Regulator	21
Figure 15.	Circuit Diagram, Flame Operation	23
Figure 16.	Circuit Diagram, Regulated Power Supply	23
Figure 17.	Bandwidth Dispersion Data	27
Figure 18.	Source Light Adjustments	32
Figure 19.	Adjustment Locations	34
Figure 20.	Oscilloscope Patterns	35
Figure 21.	Model DB Circuit Diagram	40

Contents

Reference	Title	Page
SECTION 1	**INTRODUCTION**	
1.1	Optical System	2
1.2	Instrument Location	3
1.3	Installation	3
SECTION 2	**DB OPERATING CONTROLS**	
2.1	Controls on Front Panel	4
2.1.1	Power Control Switch	4
2.1.2	Wavelength Control	5
2.1.3	Slit Program Selector	5
2.1.4	Manual Micrometer Slit Adjust	6
2.1.5	Source Selector	6
2.1.6	100% Adjust	7
2.1.7	Zero Adjust	7
2.1.8	Pilot Light	7
2.2	Controls on Back Panel	7
2.2.1	Dynode Voltage Control	7
2.2.2	Operation Selector	7
2.2.3	Recorder Terminals	7
2.2.4	Recorder Scan Receptacle	7
2.2.5	A.C. Power Cord	7
2.2.6	Fuses	7
2.2.7	Wavelength Drive Accessory Switch	7
2.2.8	Hydrogen Lamp Power Cord	8
2.2.9	Cooling System Ports	8
2.2.10	Tungsten Lamp Power Switch	8
2.2.11	Flame Attachment Ports	8
2.2.12	Burner Lift Accessory Port	8
2.3	Internal Adjustments	8
SECTION 3	**OPERATION**	
3.1	Manual Operation, Double Beam	9
3.2	Per Cent T Recording, Double Beam	11
3.2.1	Reaction Rate Studies	12
3.3	Differential Analysis	12
3.4	Repetitive Scan	13
3.5	Single-Beam Operation	13
SECTION 4	**CELL HANDLING & SOLVENTS**	
4,1	Absorption Cells	15
4.2	Cell-Handling Techniques	15
4.3	Cell Orientation	15
4.4	Cell Care & Cleaning	16
4.5	Solvents	16
4.6	Testing the Solvent for Impurities	16
SECTION 5	**ELECTRONICS**	
5.1	Description of Operation	17
5.2	Monochromator	17
5.3	Slit System	18
5.4	Sample Compartment	18
5.4.1	Light Chopping Assembly	19
5.4.2	Detector Mirror	19
5.5	Photomultiplier	19
5.6	Signal Amplifier	19
5.7	Circuit Operation	20
5.8	Output Signal Switch	20
5.9	Meter Circuit	21
5.10	Regulator	21
5.10.1	Circuit Operation (Reference Signal Absent)	22
5.10.2	Circuit Operation (Reference Signal Present)	22
5.11	Flame (Single Beam) Operation	23
5.12	Power Supply	23
5.13	Regulated Supply	24
5.14	Operation	24
5.15	Vibrating Mirror System	24
5.16	Demodulator Driving Circuit	25
SECTION 6	**SCIENCE OF SPECTROPHOTOMETRY**	
6.1	Transmittance	26
6.1.1	Absorbance	26
6.1.2	Spectral Slit Width	26
6.1.3	Effective Band Width	26
6.1.4	Resolution	27
6.1.5	Stray Light	27
6.2	Band Width	28
6.3	Calculations of Band Width	28
6.4	Absorption	29
6.5	Qualitative Interpretation	29
6.6	Quantitative Interpretation	30
6.7	Beer's Law	30
6.8	Transmittance & Absorbance Measurements	31
SECTION 7	**MAINTENANCE**	
7.1	Replacing Tungsten Source	32
7.1.1	Positioning & Focusing Tungsten Source	32
7.2	Replacing Hydrogen Source	33
7.2.1	Positioning & Focusing Hydrogen Source	33
7.3	Adjusting Manual Slit Zero	34
7.4	Vibrating Mirror Amplitude	34
7.5	Phasing	35
7.6	Reference Amplifier Feedback Potentiometer (Adjustment)	35
7.7	Replacing Photomultiplier	35
7.8	Tube Replacement	36
7.9	Cleaning Mirrors	36
7.10	Sample Compartment Positioning	36
SECTION 8	**ACCESSORIES AND REPLACEMENT PARTS LIST**	37







Section One Introduction

The BECKMAN DB Spectrophotometer is a double-beam/single-beam instrument designed for rapid transmittance and absorbance measurements in the 205 to 770 millimicron spectral range. It provides the technician with a tool for accurate qualitative analysis, quantitative analysis and reaction rate study.

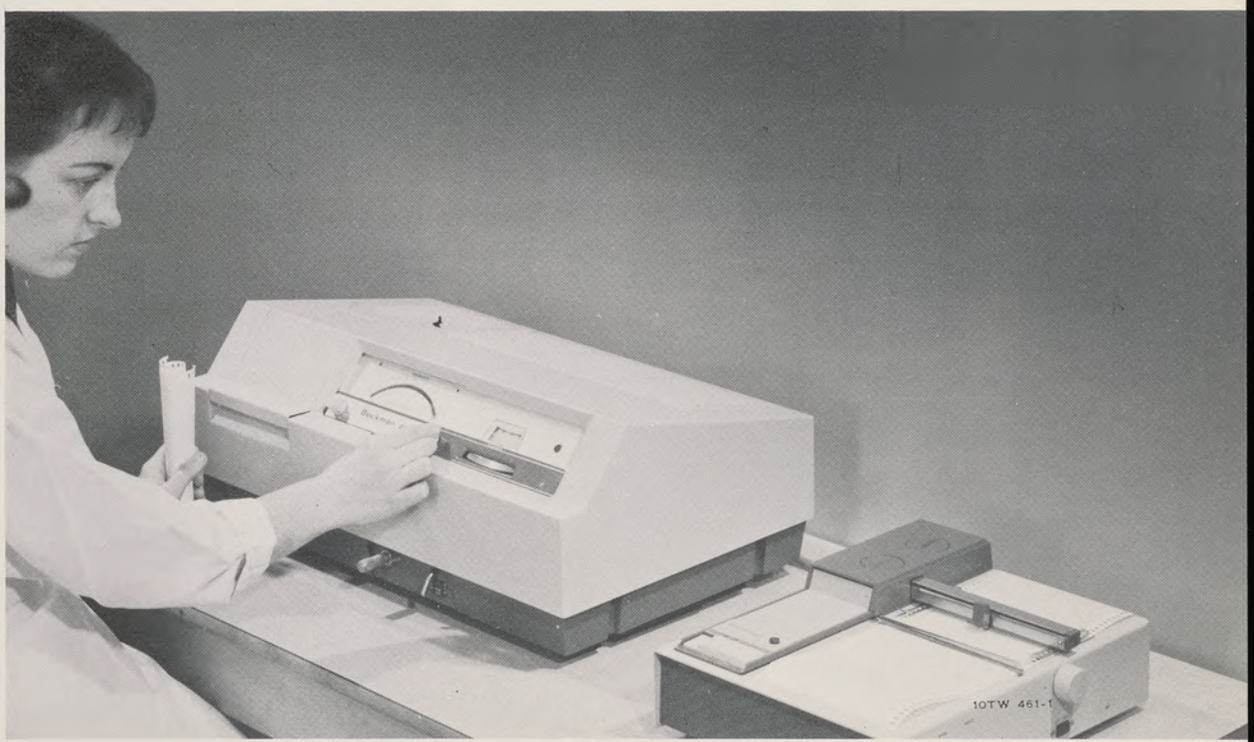
The instrument features a minimum of operating controls and a direct-reading meter, permitting rapid analyses even when operated by relatively inexperienced personnel. Easily installed accessories include Wavelength Drive Units, Flame Attachment, and H_2 Lamp Source.

When used with a Wavelength Drive Unit and a Strip Chart Recorder, the DB records in percent-transmittance and becomes a low cost ratio recording spectrophotometer.

The DB is a single-unit instrument. As shown in the optical diagram (Figure 3), its major components contained in one cabinet are a source, monochromator, beam-switching device, detector, amplifying and regulating circuits and a readout meter.

The DB also provides a means of closely controlling the temperature of the sample compartment for use in those analyses where such control is necessary for the most accurate results. Water or other coolant is circulated through coils molded into the compartment. An accessory kit provides all necessary fittings to attach the DB to a coolant source.

Figure 1. Model DB in Operation



POWER REQUIREMENTS:
1 amp. at 115 volts ± 10 volts, 50/60 cycles

OPTICAL PRINCIPLE:
Double-Beam (vibrating mirror)

PRISM:
33° Fused Silica

WAVELENGTH RANGE:
Ultraviolet (Hydrogen Lamp)
205mμ — 350mμ
Visible (Tungsten Lamp)
325mμ — 770mμ

MONOCHROMATOR:
Littrow Prism

DETECTOR:
1P28 Photomultiplier

RESOLUTION:
Ultraviolet (205-325mμ): Better than 1.2mμ
Visible (325-700mμ): Better than 4.0mμ

ACCURACY:
Wavelength: 220-334mμ: Better than 1.0mμ
335-574mμ: Better than 5.0mμ
575-700mμ: Better than 10.0mμ

LINEARITY:
Photometric: Better than ± 1.0%T
± .01 Abs. units at 0.4 Abs.

REPEATABILITY:
Wavelength: 220-334mμ: Better than 0.5mμ
335-574mμ: Better than 2.5mμ
575-700mμ: Better than 5.0mμ
Photometric: Better than 0.5%T

SLITS:
Programmed or Manual,
Adjustable (0.01-2.0mm)

STRAY LIGHT:
Less than 0.5% from 205-680mμ

100% LINE VARIATION:
Less than ± 2% from 220-700mμ

ABSORPTION CELLS:
1 mm, 5 mm, 10 mm, 40 mm Rectangular

NET WEIGHT:
60 pounds

SHIPPING WEIGHT:
75 pounds

DIMENSIONS:
23"x 14"x 9"

Figure 2. Specifications

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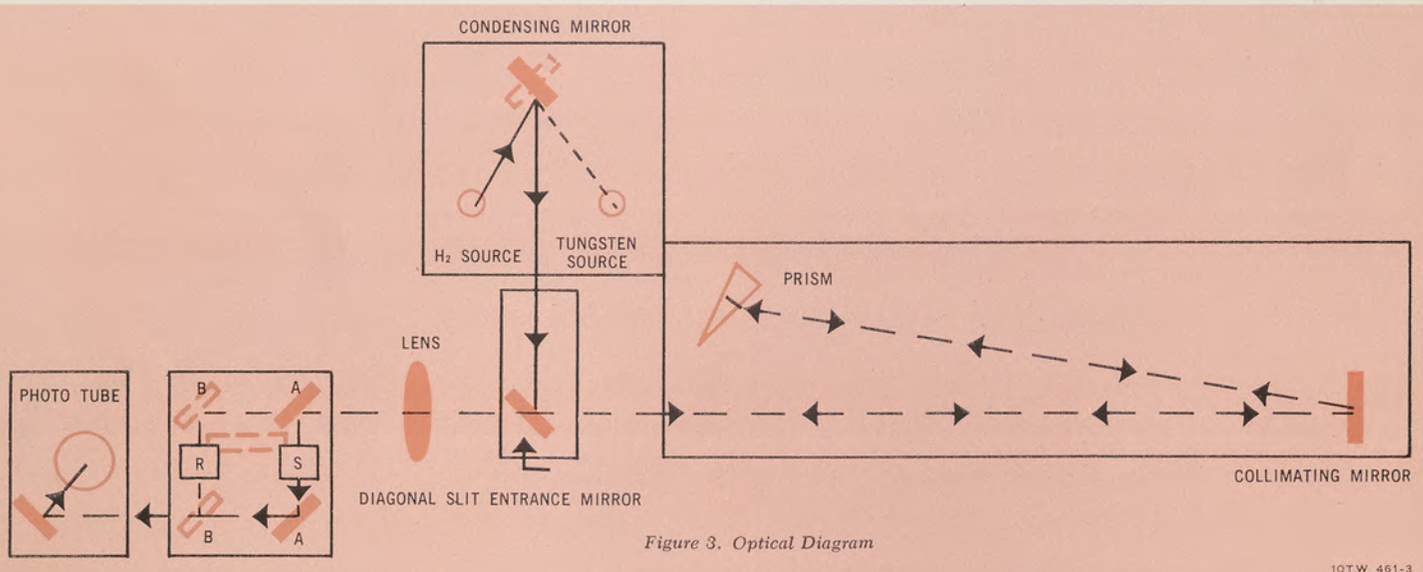

Figure 3. Optical Diagram

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1.1 OPTICAL SYSTEM

As illustrated in Figure 3, radiant energy from the source is focused by the source condensing mirror and directed to the monochromator diagonal mirror. The diagonal mirror directs the energy through the entrance slit to the collimating mirror, where it is reflected to the prism. The DB employs a fused silica prism for dispersion, the far surface of which is aluminized to form a mirror. Thus, the beam passes through the prism twice, undergoing refraction each time. The desired wavelength is selected by rotating the wavelength control, which determines the angular position of the prism in the energy path.

The refracted beam is directed once more to the collimating mirror and the selected wavelength is centered on the exit slit of the monochromator. The narrow band of energy leaving the exit slit is then directed alternately through the sample and reference paths by the beam-switching assembly. Any absorption by the sample reduces the energy in the sample path in comparison to that in the reference path.

The coupled mirrors in the beam-switching device vibrate between position a-a and position b-b, Figure 3, 35 times per second, directing alternate pulses of energy through the sample and reference cells.

With exception of the cells, all DB optical elements are common to both paths. Consequently, any change in source or optical components (other than the reference and sample cells) affects both paths equally and, while total system energy may be altered by such changes, the ratio between the sample and reference paths is not altered.

1.2 INSTRUMENT LOCATION

For maximum stability, the DB should be located in that portion of the laboratory that is least subject to temperature variation.

Place the DB as close as possible to a source of 115-volt, 60 cycle power. The Model DB will also operate satisfactorily on 50 cycle power. However, during 50 cycle operation there will be a proportional increase in scanning time when the instrument is equipped with a wavelength drive accessory. No other utilities are required unless the flame attachment is to be used, in which case a source of fuel and oxygen must be provided.

Instrument performance may be affected by heavy electromagnetic fields, such as those generated by large electric motors and diathermy machines. Any possible disturbances of this nature should be considered in selecting the location for the instrument.

1.3 INSTALLATION

(1) After removal from packing case, remove tape on wavelength dial, sample compartment and source selector.

(2) Inspect all sections for loose components.

(3) Unwrap the louvred covers and place on the instrument.

(4) Place the instrument on a stable, vibration-free table and attach the power cord to a 115-volt, 60 cycle power source.

(5) Turn the power switch (left knob, front panel) to ON position. The indicating light in the upper right-hand corner of the front panel will glow. With the switch in this position, the electronics will stabilize after approximately 2 minutes. Temperature equilibration will require somewhat longer.

Section Two DB Operating Controls

Operating controls on the DB used during normal instrument operation are conveniently located on the front panel. These are shown in Figure 4. Less frequently used controls, connectors and ports are located on the back panel as shown in Figure 6.

2.1 CONTROLS ON FRONT PANEL

The following controls are located on the front panel.

2.1.1 POWER CONTROL SWITCH

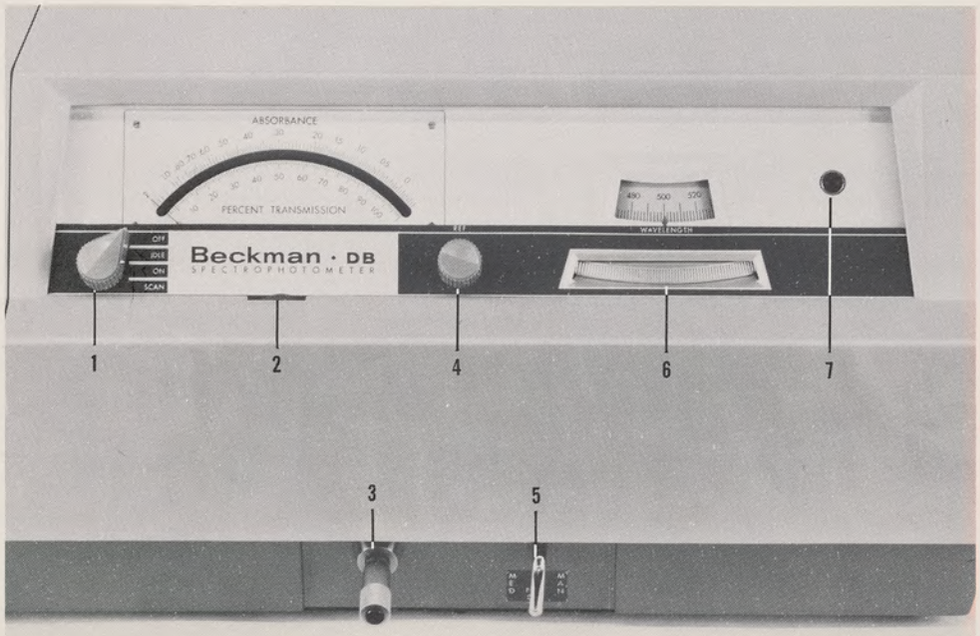
The position of the power control switch determines the following circuit conditions:

OFF - Instrument is inoperative; line voltage is present at fuses F1 and F2, but no line voltage is present in any circuit.

IDLE - Power is supplied to all circuits except the beam-switching circuit. This places the beam-switching unit at rest, while maintaining the operating temperature of the electronics during periods when the instrument is not in use.

ON - Instrument is operative, with power supplied to all circuits. Beam switching operates.

SCAN - Voltage is supplied at the wavelength-scan-motor terminal strip, and the circuit is closed at the recorder scan plug, activating these two accessories if they have been installed.



1. POWER CONTROL SWITCH
2. ZERO ADJUST
3. MANUAL MICROMETER SLIT ADJUST
4. 100% ADJUST
5. SLIT PROGRAM SELECTOR
6. WAVELENGTH CONTROL
7. PILOT LIGHT

Figure 4. Front Panel Controls

10TW 461-4

2.1.2 WAVELENGTH CONTROL

Rotating the wavelength control rotates the wavelength cam, setting the angular position of the prism. This determines the central wavelength of the band of light passing through the exit slit.

The central wavelength is indicated by the indexing pointer at the center of the wavelength window above the wavelength control. The wavelength control also rotates the cams which position the slits.

2.1.3 SLIT PROGRAM SELECTOR

A mechanical, spring-loaded control, the slit program selector allows the operator to select the slit program best suited to the analysis to be made. The settings of the slit program selector indicate the following conditions:

NAR - Designates the narrow slit function and is intended for use with reference materials of high transmittance (approximately 50% and above with respect to air). Pull the control out and turn to lock in the vertical position, with the control arm over NAR on the dial plate.

MED - Designates the medium slit function and is intended for use with reference materials of lower transmittance (below 50% with respect to air). Pull out control and turn to lock in the left position, with the control arm over MED on the dial plate.

MAN - When the control arm is locked over MAN on the dial plate, in the right-hand position, the slits are no longer programmed, and the instrument is set for the use of the manual micrometer slit adjust. In this position, the slit width is controlled by the setting of the manual micrometer.

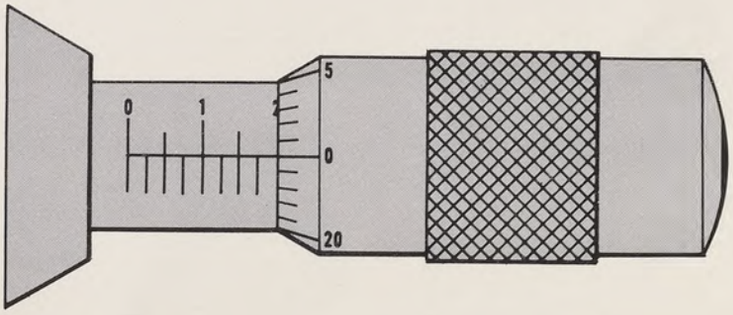


Figure 5. Micrometer Slit Adjust 10TW 461-5

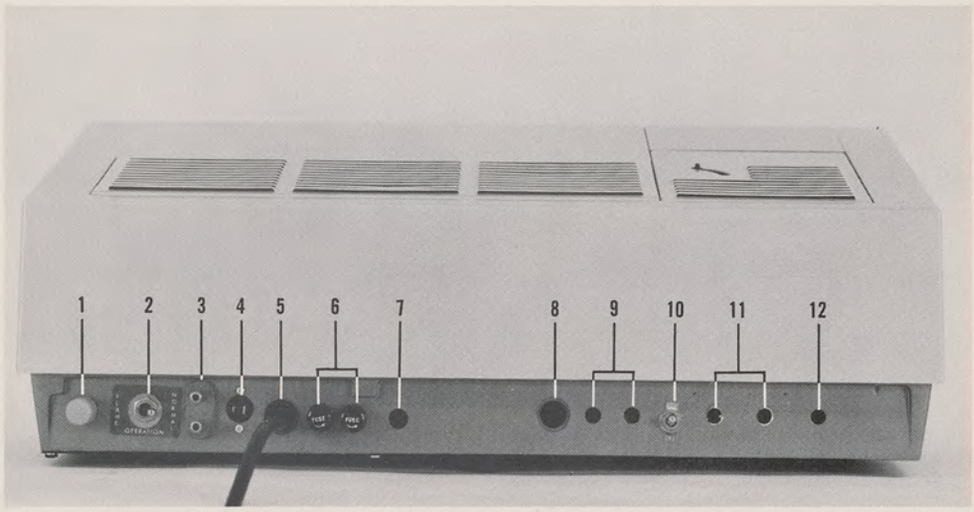
2.1.4 MANUAL MICROMETER SLIT ADJUST (Figure 5)

Turning the thimble of the manual micrometer slit adjust in a counter-clockwise direction causes the slit to open. Each complete turn of the thimble opens the slit 0.25 mm, and eight complete turns open the slit to its maximum width of 2 mm. Complete turns are read on the sleeve scale of the micrometer adjust, which is calibrated in 0.25 mm divisions. See Figure 5.

For the fine adjustments, the radial divisions on the thimble are calibrated to indicate 0.01 mm of slit width, with 25 such divisions making one complete turn of the thimble.

NOTE

Always return the micrometer thimble to zero stop when the slit program selector is returned to either the NAR or MED positions. Otherwise the micrometer may over-ride the action of the slit cams.



1. POWER SWITCH
2. FLAME OPERATION SWITCH
3. RECORDER TERMINALS
4. RECORDER CABLE ACCESSORY PLUG
5. POWER CABLE
6. FUSES

Figure 6. Back Panel Controls

10TW 461-6

2.1.5 SOURCE SELECTOR

The source selector determines the light source. When the source selector is in the VISIBLE (320-800 mμ) position, the source mirror reflects light from the tungsten lamp to the monochromator diagonal entrance mirror. When the source selector is in the UV (200-350 mμ) position, the source mirror reflects light from the accessory hydrogen lamp to the monochromator diagonal entrance mirror.

NOTE

The 1400 Model DB is delivered without accessories. The 1401 Model DB has the Hydrogen Lamp and the 72099 Dual Wavelength Drive (10/40 mμ per min.) as installed features. The Hydrogen Lamp Accessory, Beckman 28190, consists of the hydrogen lamp, lamp plate, cord and mounting

screws. A Hydrogen Lamp Power Supply, 2965, must be ordered separately to provide power for this accessory.

2.1.6 100% ADJUST

The 100% adjust, identified as REF on the front panel, adjusts the amplifier output current to the level required to cause a reading of 100% on the output meter, when sample and reference energy levels are equal.

2.1.7 ZERO ADJUST

The zero adjust allows the meter to be set at zero when no signal is present at the detector. The adjustment is made by aligning the meter needle and zero line, with the instrument ON and an opaque block (supplied in the shipping kit) in the sample beam.

2.1.8 PILOT LIGHT

The pilot light glows, indicating the electronics are on, when the power control switch is in the IDLE , ON or SCAN positions.

2.2 CONTROLS ON BACK PANEL

7. SWITCH FOR WAVELENGTH DRIVE ACCESSORY
8. HYDROGEN POWER CABLE
9. PORTS WATER COOLED SAMPLE COMPARTMENT KIT
10. TUNGSTEN LAMP SWITCH
11. OXYGEN AND FUEL PORTS, FLAME UNIT
12. BURNER LIFT ACCESSORY PORT

The following controls are located on the back of the instrument.

2.2.1 DYNODE VOLTAGE CONTROL

The dynode voltage control adjusts the voltage to the dynodes of the photomultiplier tube which determine gain of the system, and is used only during single-beam and flame operation; i.e., manual control. For double-beam operation, dynode voltage control is automatic.

2.2.2 OPERATION SELECTOR

The operation selector is a two-position switch which changes the instrument from double-beam to single-beam operation. For normal double-beam operation, the switch is set in the NORMAL position. For single-beam and flame operation, the switch is set in FLAME position.

2.2.3 RECORDER TERMINALS

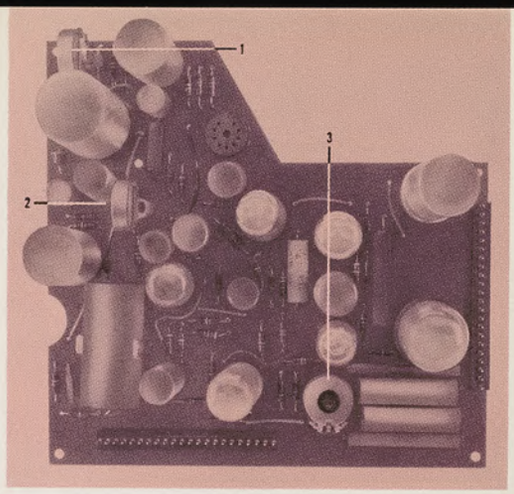
The input cable to the recorder is connected to the recorder terminals. The top terminal is positive, the bottom terminal negative or ground.

2.2.4 RECORDER SCAN RECEPTACLE

The external recorder cable, supplied with a Wavelength Drive Motor Accessory, is plugged into the recorder scan receptacle on the DB, and also into the recorder. This cable is used only with the Beckman 5-Inch Strip Chart Recorder.

2.2.5 A.C. POWER CORD

Three prong power cord plug may be inserted in 2-conductor receptacle by using 16988 adaptor provided. Connect pigtail lead of adaptor to ground.



CHOPPER AMPLITUDE CONTROL R49 1.
DEMODULATOR PHASING CONTROL R64 2.
DYNODE FEEDBACK CONTROL R17 3.

Figure 7. Internal Adjustments

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2.2.6 FUSES

Fuses F1 and F2, (rated at 1 amp Slo-Blo) are located in fuse holders.

2.2.7 WAVELENGTH DRIVE SPEED SELECTOR

When a Dual Wavelength Drive Accessory has been installed in the instrument, the speed selector switch is located in this pre-drilled hole.

2.2.8 HYDROGEN LAMP POWER CORD

The hydrogen lamp power cord supplies power to the Hydrogen Lamp Accessory, and is supplied with the accessory.

2.2.9 COOLING SYSTEM PORTS

These access ports accommodate fittings necessary to connect the liquid cooling jacket of the sample compartment to a source of coolant.

2.2.10 TUNGSTEN LAMP POWER SWITCH

The tungsten lamp power switch turns the tungsten lamp on and off.

2.2.11 FLAME ATTACHMENT PORTS

These are access ports for fuel and oxygen lines and flame adjusting mechanism, to be used with the Flame Attachment.

2.2.12 BURNER LIFT ACCESSORY PORT

When a Flame Attachment Accessory has been installed in the instrument, a lever inserted in this port is used to adjust the focus.

2.3 INTERNAL ADJUSTMENTS (Figure 7)

Adjustment of the internal potentiometers of the DB is seldom required. See Section 7. These internal potentiometers are:

(1) Chopper amplitude control (R49), which adjusts the amplitude of the vibrating mirror system.

(2) Demodulator phasing control (R64), which adjusts the switching position of the Clare relay.

(3) Dynode feedback control (R17), which adjusts the feedback within the dynode high voltage supply.

Section Three Operation

The DB Spectrophotometer is designed for meter readout or for operation with a Wavelength Drive Motor Accessory and a recorder.

Transmission or absorbance of the sample compared to the reference can be read directly on the meter. With the Wavelength Drive Accessory installed and a strip chart recorder connected to the instrument, the DB becomes a percent-T recording spectrophotometer.

3.1 MANUAL OPERATION, DOUBLE-BEAM

(1) With the power control switch in ON position, allow the instrument to warm up to temperature equilibrium; good operation may be had after 2 minutes; maximum stability is reached after 1 hour.

(2) With the sample beam blocked, adjust meter to zero, using the zero adjust control.

(3) If a run is to be made in the ULTRAVIOLET range:

Verify that the hydrogen lamp is installed and the lamp power supply is connected and operative.

Fire the hydrogen lamp in accordance with its power supply instructions.

Move the source selector to the U.V. position.

(4) If a run is to be made in the VISIBLE range:

Move the source selector to the VISIBLE position. Turn the tungsten lamp power switch to the ON position.

(5) Select the slit program to be used in the following manner:

Check the transmission of the solvent by placing a sample of the solvent in a cell, inserting the cell into the sample beam compartment (marked S), and comparing its transmission against an empty air path in the reference compartment (marked R).

If solvent transmission is 50% or above, set the slit program selector at the NAR position. This position provides the narrowest programmed slit operation, and therefore the best programmed resolution.

If the solvent transmission is less than 50%, set the slit program selector at the MED position. This position provides the widest programmed slit operation and therefore greater energy for low signal level.

If the manual slits are to be used, set the slit program selector in the MAN position. Next, check the manual slit zero in the following manner: Set the micrometer slit adjust on the zero stop position and note the background level on the meter. Now turn the micrometer adjust in a counterclockwise direction until the meter

reading begins to increase. The point at which this increase begins is the manual slit zero position.

Dial desired slit width on the micrometer adjust.

(Note that the micrometer adjust must be returned to its zero stop position before using the slit program selector on the NAR or MED positions.)

(6) Set the instrument at the desired wavelength by turning the Wavelength Control.

When recording with fixed slits (in MAN position) check 100% line over the region of interest. Rotate wavelength dial through this region and see that meter reads 100%, and the noise level is acceptable. If not, widen the slits.

(7) Fill both sample and reference cells with the solvent to be used, following the cell handling recommendations in paragraph 4.1. Install the cells in the instrument, each in its proper compartment. Re-establish a meter reading of 100% transmission, if necessary, by adjustment of the REF control.

NOTE

The solvent should be tested for impurities in accordance with the procedure described in paragraph 4.5.

(8) Discard solvent in sample path cell and refill with sample-solvent mixture. Note that the cell should be replaced in the instrument in the same position used in reading 100% T. See cell handling recommendations, paragraph 4.2.

(9) Close sample compartment cover and read transmission or absorbance on the appropriate scale of the meter.

Figure 8. Recorder Chart Speed Data

MODEL DB WAVELENGTH DRIVE MOTOR	Range 200 to 350 $m\mu$ RECORDER CHART SPEEDS (in/min)			Range 320 to 800 $m\mu$ RECORDER CHART SPEEDS (in/min)		
	½"	1"	2"	½"	1"	2"
	LENGTH OF CHART (INCHES)			LENGTH OF CHART (INCHES)		
5 $m\mu$/min	15.0	30.0	60.0	48.0	96.0	192.0
25 $m\mu$/min	3.0	6.0	12.0	9.6	19.2	38.4
10 $m\mu$/min	7.5	15.0	30.0	24.0	48.0	96.0
40 $m\mu$/min	1.875	3.75	7.5	6.0	12.0	24.0
20 $m\mu$/min	3.75	7.5	15.0	12.0	24.0	48.0
80 $m\mu$/min	0.94	1.875	3.75	3.0	6.0	12.0

10TW 461-8

3.2 PERCENT-T RECORDING, DOUBLE-BEAM

(1) With the power control switch in ON position, allow the instrument to warm up to temperature equilibrium. Two minutes is sufficient for good operation. Maximum stability is reached after 1 hour.

(2) The DB is supplied for use with 100 mv recorders. Two resistors are supplied with the instrument so that it may be adapted to the input requirements of either a 50 mv (500 ohm resistor) or a 10 mv (56 ohm resistor) recorder. If required, connect the appropriate resistor across the recorder terminals and connect the strip chart recorder to the recorder terminals.

(3) Insert the opaque block in the sample beam and adjust the recorder to zero, using the zero adjust on the recorder.

(4) If a run is to be made in the ULTRAVIOLET range, repeat steps 3, 5, 6, 7, 8 and 9 of paragraph 3.1 before proceeding farther.

(5) If a run is to be made in the VISIBLE range, repeat steps 4, 5, 6, 7, 8 and 9 of paragraph 3.1 before proceeding farther.

(6) To prepare for scan:

Select a recorder speed which offers the best chart presentation at the DB scanning speed. Chart interpretation is easier if the relationship between the scanning progress through the spectrum and the chart-paper advance is such that each vertical line on the chart denotes a readily-determined wavelength. For example, if a 20-millimicron-per-minute wavelength drive motor is used, and a recorder speed of two inches per minute is available, the two speeds result in a chart presentation in which each one-inch abscissa (vertical) chart line indicates a ten-millimicron interval. Thus, from any known starting wavelength, determination of the wavelength at subsequent points on the chart is a simple measurement. See data in Figure 8.

At the beginning of the run make a note on the chart paper of the starting wavelength, slit setting and speed.

Then, set the Wavelength Control to the longer wavelength end of the section to be scanned.

(7) With sample and reference cells installed and filled only with solvent, turn the power control switch to SCAN . The Beckman 5-Inch Strip Chart Recorder will automatically start the chart drive when the scanning operation begins. For all other recorders, simultaneously turn the power control switch to SCAN and the recorder chart drive to ON .

(8) In order to calibrate for cell difference, run a 100% line over the area to be scanned. This line establishes a reference for corrections on the recorded %T curve.

(9) After the 100% line has been run, remove the sample cell, rinse, and fill with sample-solvent mixture. It is important that the same cell be used and that it be reinserted in the sample compartment in the same position it occupied during the 100% line run in step 8.

(10) Return the chart to the starting point, with the pen position coinciding with the start of the previously-traced 100% line.

(11) Repeat scan.

(12) The recorder pen will now indicate the transmittance of the sample as a percentage of reference transmission (100%) over the range of wavelengths being scanned.

NOTE

The DB will scan continuously from long to short wavelengths and then repeat after passing over the back side of the wavelength cam. For available scanning speeds, see Accessories Section.

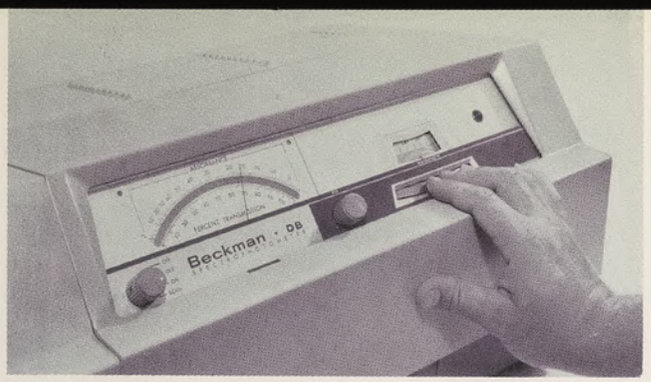
3.2.1 REACTION RATE STUDIES

When the DB is operated as a percent-T recording spectrophotometer, sample reaction or precipitation over a period of time may be recorded by setting the instrument at an appropriate wavelength, inserting the sample and turning on the recorder. The set-up for this operation is identical to that outlined in paragraph 3.2, except that in step 7 the power control switch is turned to the ON position rather than the SCAN position, and the recorder energized.

3.3 DIFFERENTIAL ANALYSIS

In a double-beam system, the meter (or recorder pen) indicates the ratio of the transmittance of the sample to the transmittance of the reference. Therefore, the same spectrophotometer that records the ratio between reference and sample transmittance can also be employed to reveal the difference between similar but not identical samples. Since the DB indicates 100%T whenever the sample and reference transmittance is identical, the instrument meter will read 100% or the recorder pen will trace a straight line at the 100% level on the chart, EXCEPT AT THOSE POINTS OF DIFFERENCE BETWEEN SAMPLE AND REFERENCE. These differences become immediately apparent as deviations from the 100% line or reading. Thus, by using a known material, the extent to which a slightly dissimilar material departs from these characteristics can be measured easily and precisely.

The following factors must be taken into account when the DB is employed for differential analysis:



The solvent should exhibit no deep absorbance within the area of the run.

Cells of matched pathlength should be used in order to confine all differences in sample and reference paths to the differences between the sample and reference materials.

If the reference absorbs more energy than the sample, the pen will go above the 100%T line. Unless it can be determined beforehand that the stronger absorber will be in the sample path, the REF control should be turned counterclockwise to bring the 100%T line down to the 80%T level. If the pen still rises above the 100%T line, contract the scale by further adjustment of the REF control, or by interchange of the cells.

NOTE

For highly absorbant differential runs, run the sample with air as a reference. Do not rely upon differential data in regions exhibiting less than 50%T. In such cases, use sample solutions of lower concentration.

3.4 REPETITIVE SCAN

If it is necessary to study a time reaction of a sample over a specific band of wavelengths, follow the procedure described in paragraph 3.2, and allow the instrument to repeat the scan as many times as required. The operator may then observe the scan in the wavelength region of interest.

3.5 SINGLE-BEAM OPERATION

(1) With the power control in the ON position, allow the instrument to warm up to temperature equilibrium; two minutes is sufficient for good operation. Maximum stability is reached after 1 hour.

(2) Turn operation selector to FLAME position.

(3) Turn slit program selector to MAN position.

(4) Block reference beam by inserting the opaque block in the reference channel.

(5) Check manual slit zero and dial desired slit width on the manual micrometer slit adjust.

(6) Set the desired wavelength by turning the wavelength control.

NOTE

Check the source selector to determine that the proper source has been selected for the region.

(7) Block the sample beam with the opaque block and check zero on the meter. If necessary, set the meter at zero with the zero adjust.

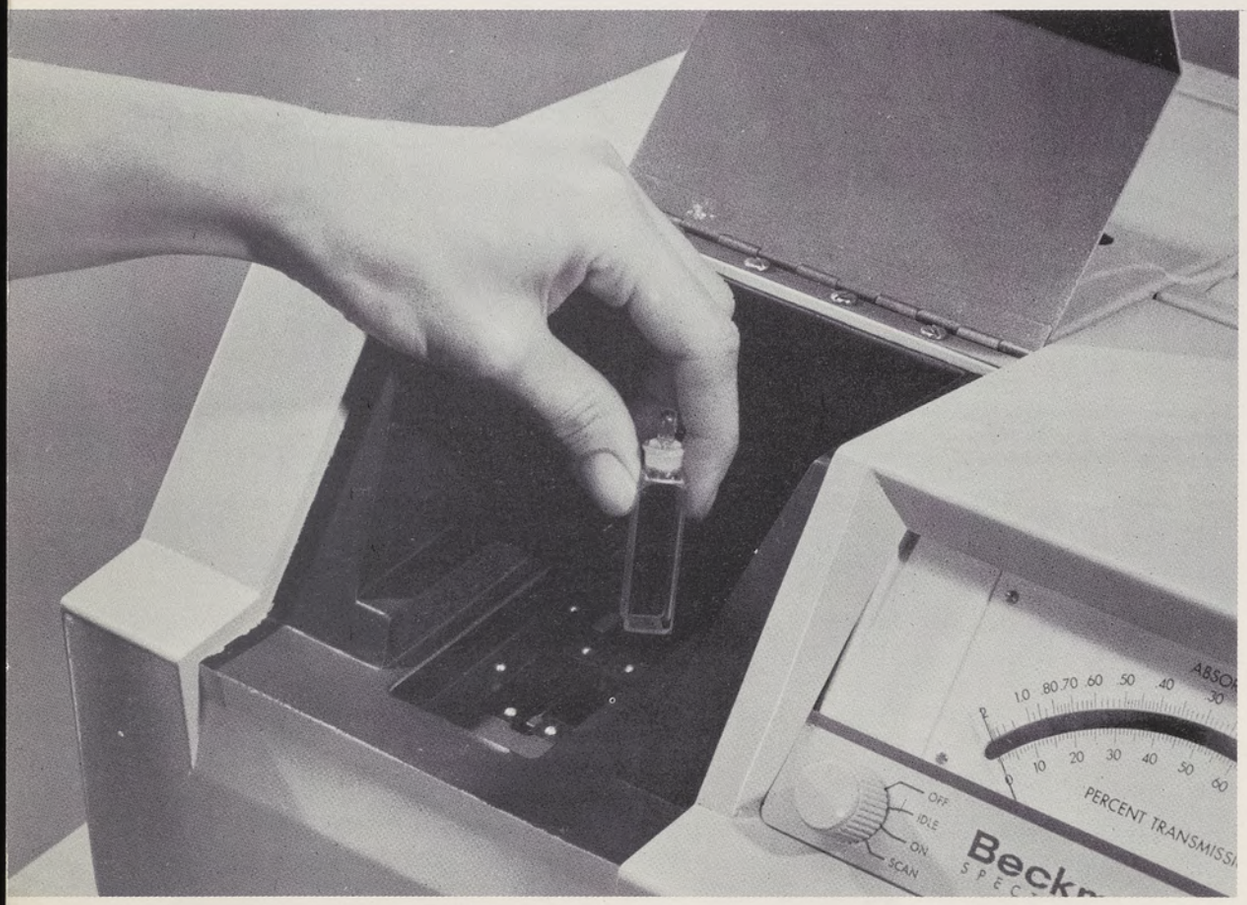
(8) Remove the opaque block in the sample beam and insert cell containing only solvent.

(9) Adjust the dynode voltage control on the back panel until the meter reads 100%.

(10) Remove cell containing the solvent, discard the solvent, and fill the cell with solvent-sample mixture.

(11) Place the cell in the sample beam compartment in the same position as in Step 8. Read the transmittance or absorbance on the appropriate scale of the meter.

(12) Repeat steps 5, 6, 8, 9 and 10 for all wavelengths of interest.



Section Four Cell Handling and Solvents

Extreme flexibility of Model DB application is guaranteed by the wide assortment of liquid and gas cells available.

4.1 ABSORPTION CELLS

TYPE OF CELL	PATHLENGTH IN MILLIMETERS	DESCRIPTION	WAVELENGTH	TO ORDER SPECIFY BECKMAN NO.
LIQUID	1	Silica Rectangular with Polyethylene Cover	205-770	46028*
LIQUID	5	Silica Rectangular with Polyethylene Cover	205-770	46003**
LIQUID	10	Silica Rectangular with Polyethylene Cover	205-770	46005
LIQUID	10	Silica Rectangular with Ground-Glass Stopper	205-770	46007
LIQUID	40	Silica Rectangular	205-770	46029***
SPACER	9	Silica Spacer used to reduce light path of all 10 mm Rectangular Cells to 1 mm.		

* Requires #72085 Cell Adapter
** Requires #40861 Cell Adapter
*** Requires #73206 Stainless Steel Cover

4.2 CELL-HANDLING TECHNIQUES (Figure 9)

Do not touch optical surfaces of any absorption cell. Be particularly careful to ensure that portions of the optical surfaces that will be in the light path are kept clean. Natural oils or perspiration on the hands will form smudges on the optical surfaces, causing localized absorption or reflection of light.

Be certain that the inside and outside of cells are dry before inserting them in the Model DB. Do not fill cells while holding them over the instrument.

4.3 CELL ORIENTATION

A small Beckman trademark decal appears on one of the non-optical surfaces of each rectangular cell. This decal may be used to standardize orientation of the cell in the holder by making sure that the trademark faces the same direction each time the cell is inserted. For cells that do not bear a trademark decal, a special mark should be made on the cell body for this purpose.

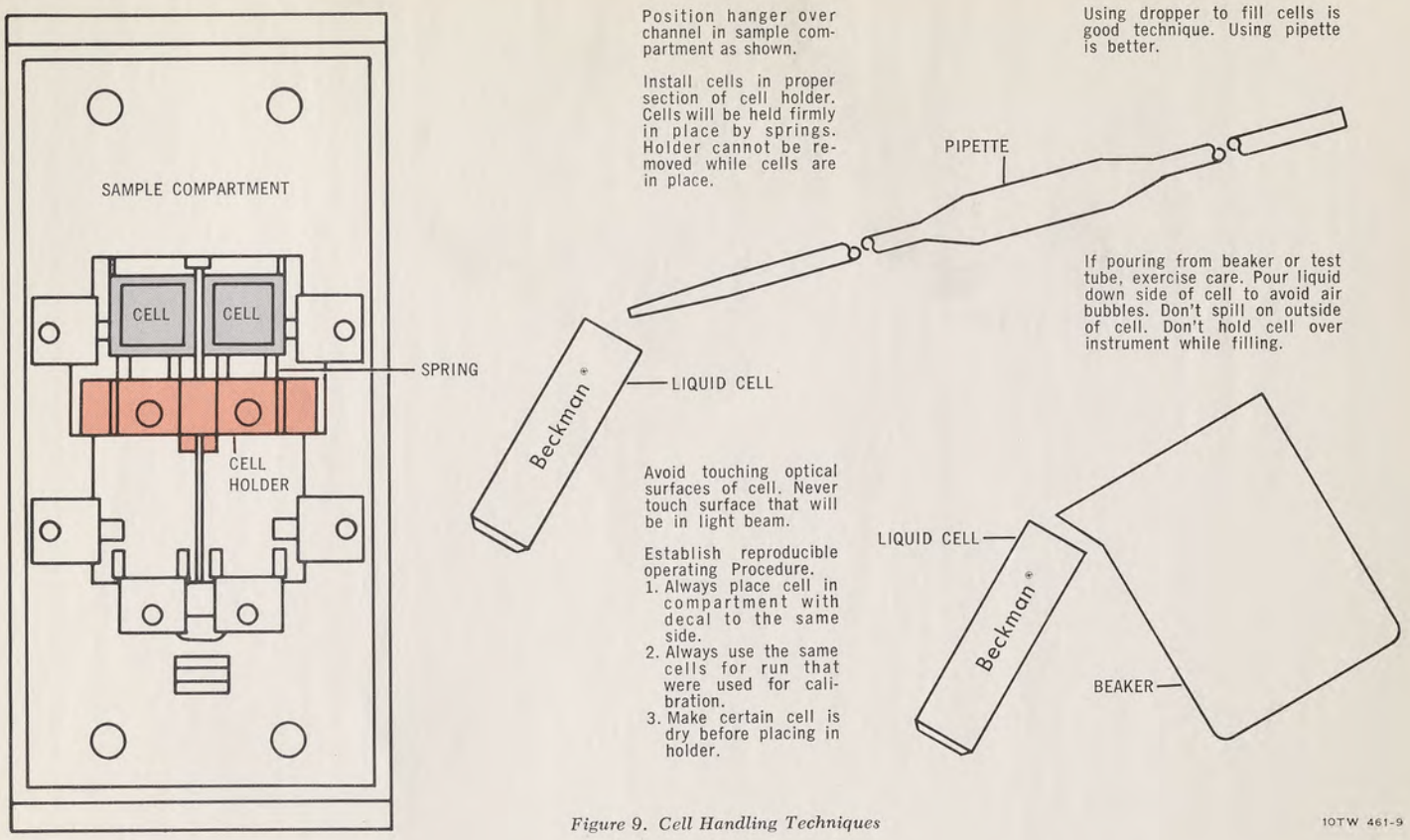


Figure 9. Cell Handling Techniques

4.4 CELL CARE AND CLEANING

When an absorption cell is positioned in the light path, it becomes an integral part of the instrument's optical system and must be treated with the same care given other optical components. Scratched or contaminated cell windows reflect or absorb light, resulting in inaccurate measurements.

Unless contamination is severe, cells may be cleaned by rinsing them thoroughly in distilled water or in solvent in which the sample is soluble.

If rinsing in distilled water or solvent fails to remove contaminants, cells may be cleaned with mild sulphuric acid or mild sulphonic detergents. Avoid using hot concentrated acids or alkalies or other cleaning agents which may etch the polished optical surfaces. Store cells in the box in which they were supplied.

4.5 SOLVENTS

Liquids used as solvents in spectrophotometric work must be chemically inactive to the material being investigated and transparent over the spectral range of the investigation. The chart in Section 8 lists the most widely used solvents for both the visible and the ultraviolet range.

4.6 TESTING THE SOLVENT FOR IMPURITIES

Every solvent must be tested for impurities before use. Most solvents will produce a flat, straight-line recording or steady, single-value meter reading when a 10 millimeter cell of pure solvent is scanned in the sample compartment against an air path in the reference compartment of the DB. Peaks (other than cut-off peaks) indicate impurities requiring purification or discard of the solvent.

Section Five Electronics

In order to describe the components and circuitry of the Model DB Spectrophotometer, the instrument as a whole is discussed in this section, then separated into its components and discussed in detail.

5.1 DESCRIPTION OF OPERATION

All DB optical elements are common to both the sample and the reference path. Any change in the source or in any mirror or lens will cause an equal change in both sample and reference paths. Therefore, while any change will affect system energy, it will not affect the relationship between the sample and the reference output.

Light from either the tungsten lamp or the hydrogen lamp is selected by the source mirror and directed into the monochromator by the diagonal mirror. The light leaves the monochromator as a narrow band of wavelengths whose intensity and spectral width has been determined by the slit width and whose position in the spectrum has been determined by the angular position of the prism.

The light is then alternated through the sample and reference compartments at a 35-cycle rate by a vibrating mirror light-chopping system.

The photomultiplier tube transforms the alternating sample-reference light pulses into current pulses. The amplifier raises the level of the photomultiplier output to 85 volts and passes the alternating voltage pulses to the output-signal switch. The dynode voltage of this tube is regulated so that its output will maintain the reference pulse level at 85 volts at the amplifier output.

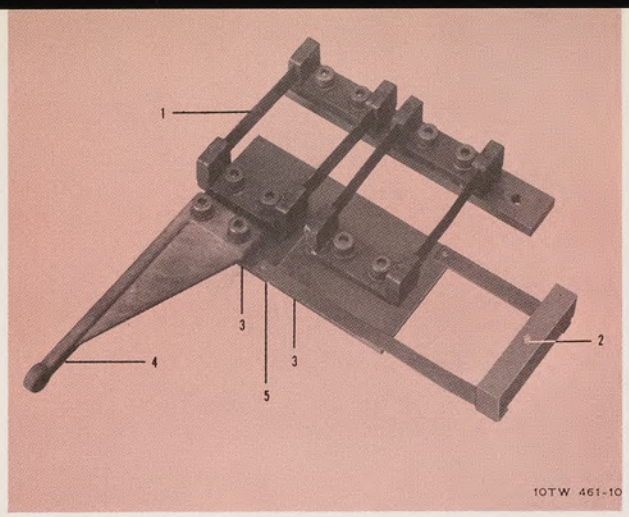
The output signal switch is synchronized with the light chopper. Its function is to separate the sample and reference pulses. The sample pulses are sent on to the meter circuit, where their amplitude is indicated on the meter. The reference pulses are sent to the Automatic Reference-Standardizing circuit.

The Automatic Reference-Standardizing circuit determines the difference between the amplitude of the reference pulses and the 85 volt (100%) reference level. If there is such a difference-voltage, it is applied to effect the change in photomultiplier output necessary to return the reference signal to a level of 85 volts. A change in dynode voltage will also change the amplitude of the sample pulses proportionally.

The power supply provides all filament, bias and plate voltage, as well as regulated high voltage for the photomultiplier tube and power for the tungsten lamp.

5.2 MONOCHROMATOR

Light from either the tungsten, hydrogen or mercury lamp is directed by the source mirror to the diagonal entrance mirror. The diagonal entrance mirror directs the light through the entrance slit and into the monochromator, to the collimating mirror. There it is collimated and reflected to the prism, where it undergoes refraction. The back



1. FLEXIBLE REEDS
2. PIVOT
3. SLIT JAWS
4. SLIT FOLLOWER ARM
5. SLIT

Figure 10. Slit System

surface of the prism is aluminized and reflects the refracted light back through the prism, again refracting it as it emerges from the prism. The desired wavelength of light is selected by the Wavelength Control which adjusts the angular position of the prism. The spectrum is directed back to the collimator which, as determined by the position of the prism, centers the chosen wavelength on exit slit of the monochromator.

5.3 SLIT SYSTEM (Figure 10)

The slits are mounted on a pair of reed parallelograms. Opening motion applied to the slit follower arm through linkage to the concave defining jaw is also applied to the convex defining jaw through the slit links and pivot arm. Initial tension in the reeds provides the required closing force when motion is reversed.

The exit slit is positioned directly above the entrance slit, and a single set of slit jaws determines the width of both. Thus, the entrance and exit slit widths of the Model DB are always equal. The two slit jaws are mounted on flexible reeds and are linked together so that bilateral motion is assured.

Either of two slit cams mounted coaxially with the wavelength cam will program the slits to compensate for non-uniformity of the sources and detector as a function of wavelength. Energy will be effected as a square function of the total slit width. The two cams differ by the square root of 2 in slit opening or a factor of 2 in energy. The Slit Program Selector positions the cam follower on the smaller of the two cams for the NAR slit function and on the larger cam for the MED slit function. In the MAN position, the Slit Program Selector removes the follower from both cams and allows adjustment of the slit with the Manual Micrometer Slit Adjust. The micrometer permits manual adjustment of the slits from the closed position to two millimeters maximum.

The selected wavelengths pass through the exit slit, the exit lens, and into the sample compartment.

5.4 SAMPLE COMPARTMENT

The sample compartment encloses the light chopping assembly and the sample and reference cells. Light of the selected wavelength enters this assembly, passes alternately through the sample and reference, and is focused on the photomultiplier-detector by the detector mirror.

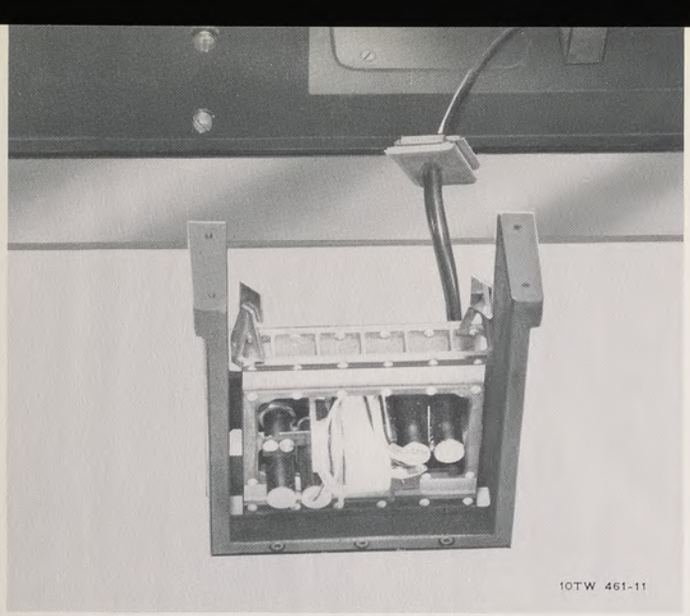


Figure 11. Vibrating Mirror Assembly

A partition fits into the cell compartments to secure the 10 millimeter cells. Springs are built into the back and sides of cell compartments to secure 40 millimeter cells.

5.4.1 LIGHT CHOPPING ASSEMBLY (Figure 11)

The light-chopper or beam-switching assembly consists of a pair of parallel mirrors rigidly mounted to a self-resonating reed system that moves with simple harmonic motion consecutively from sample to reference position.

5.4.2 DETECTOR MIRROR

The detector mirror produces a reduced prism image on the photo-sensitive cathode of the detector.

5.5 PHOTOMULTIPLIER (1P28)

The alternating pulses of light, falling on the photocathode of the 1P28 photomultiplier tube, cause electrons to be emitted from the surface. The electrons are attracted to the first dynode by its more positive voltage. Striking the first dynode, each of the electrons causes secondary electrons to be emitted which are, in turn, attracted to dynode Number 2 by its higher positive potential. At dynode Number 2, and at each of the nine dynodes of increasingly positive potential, each impinging electron causes additional secondary electrons to be emitted, until, at the anode of the tube, a current gain of 100,000 can be effected.

The dynode voltage, varying inversely to the energy of the reference path, is divided equally among the dynodes.

5.6 SIGNAL AMPLIFIER (Figure 12)

The DB signal amplifier consists of a pentode amplifier stage and a cathode follower output stage. V7 is operated in a region where its open loop gain is greater than 800. It employs four feedback loops that establish the operating point and stabilize the gain. Plate feedback, C10 and R34, effectively increase the plate load impedance of V7 thus increasing the open loop gain of V7; this feedback is always less than unity by virtue of degeneration in the cathode follower. Screen feedbacks R37, R38 and C12 provide compensation for tube differences. The screen voltage is automatically established regardless of the trans-

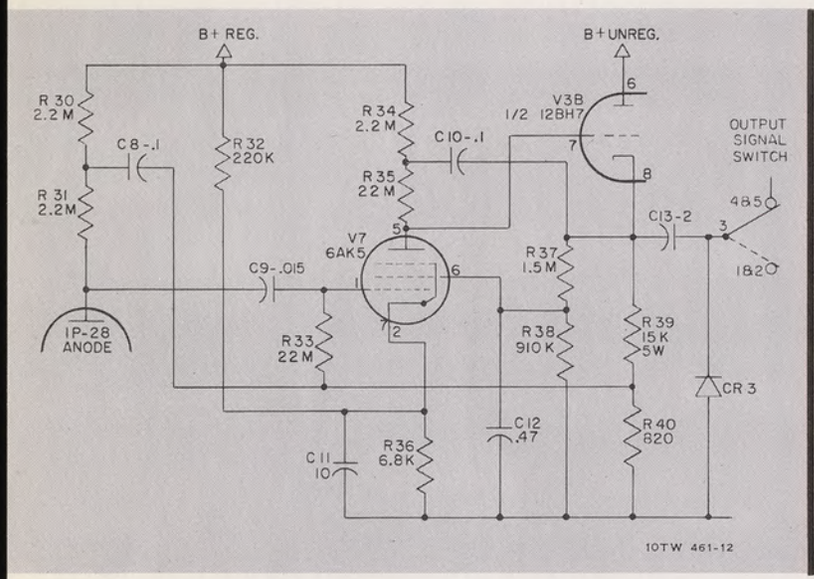


Figure 12. Circuit Diagram, Signal Amplifier

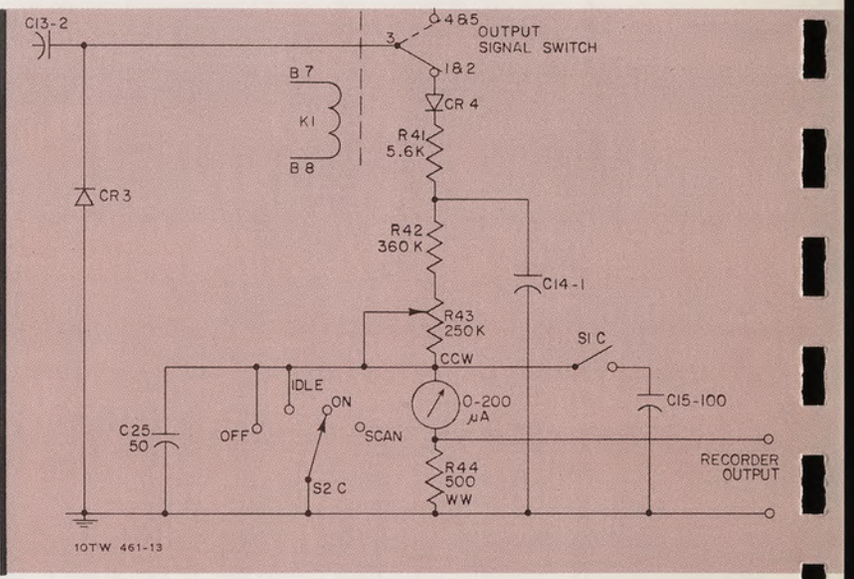


Figure 13. Circuit Diagram, Meter Circuit

conductance of V7. C8 and R30 prevent the amplifier from becoming "blocked" when the photomultiplier is overloaded with unchopped radiation. R39, R40 and R33 stabilize the closed loop gain to approximately 20.

5.7 CIRCUIT OPERATION

Electrons collected on the anode of the photomultiplier produce a negative going voltage on passing through R31. This voltage is applied to the grid of V7 through C9 decreasing the current through V7. The plate of V7 and grid of V3B go in a positive direction when the current is reduced in R35. The cathode of V3B follows the grid. This voltage is divided by R39 and R40 and the divided portion is applied to the grid of V7 through R33 to stabilize the gain of the amplifier to approximately 20. C13 couples the amplifier to the regulator or to the meter circuit depending on the position of the signal switch. CR3 clamps the cathode voltage on C13 when the cathode of V3B is in the negative portion of the cycle.

5.8 OUTPUT SIGNAL SWITCH (Figure 13)

The Output Signal Switch is synchronized with the light-chopping circuit so that it switches the reference component of the amplifier output to the automatic Reference-Standardizing Circuit and switches the sample components of the amplifier output to the meter circuit.

The Output Signal Switch is a magnetic switch with mercury-wetted contacts, hermetically sealed in a hydrogen atmosphere. The switch forms the armature of its activating coil and both are enclosed in a steel envelope. Capillary action draws the mercury from a reservoir in the bottom of the glass capsule to wet the switch surfaces.

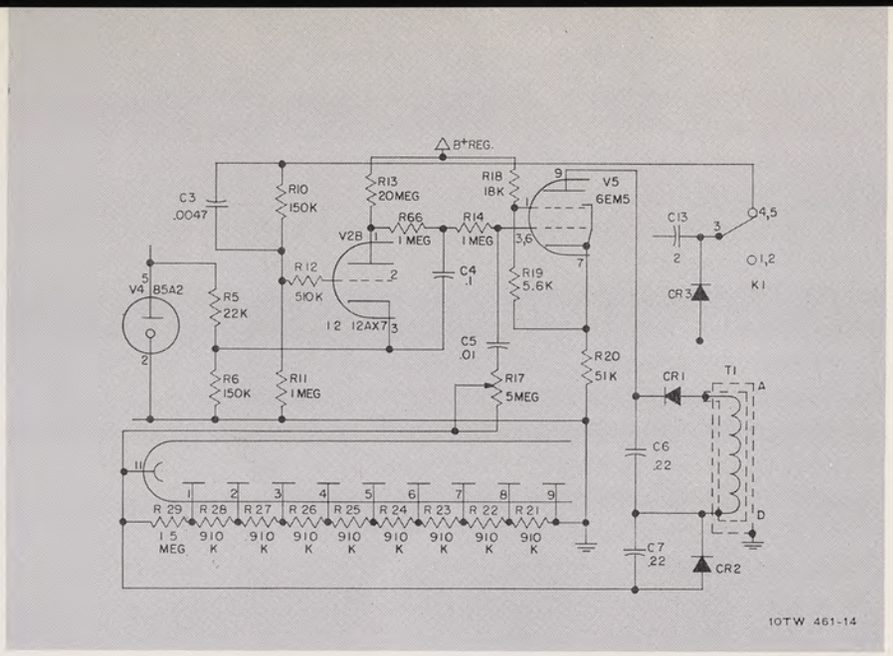


Figure 14. Circuit Diagram, Regulator

5.9 METER CIRCUIT (Figure 13)

When the cathode of V3B is at a minimum signal this voltage is clamped on C13 by diode CR3. When the cathode of V3B is at its maximum positive voltage the clamped voltage on C13 plus the residual voltage on C14 must equal this maximum voltage. If not, CR4 will conduct, resetting the voltage on C14. CR4 permits peak reading of the sample signal. R41 filters the noise on charging C14. C14 maintains the current through the meter during reference and clamp portion of the cycle. R42 and R43 (100% adjust) limit the current through the meter. R43 is adjusted for 100%T when the transmittance of the reference and sample beams are equal.

Meter current through R44 serves as a voltage source for an external recorder. C25 is a filter condenser to filter current pulses through the meter. Switch S-2C, a section on the power and control switch, shunts the meter during the off and idle positions of control. C15 is an additional filter condenser added when S-1C, a section of the operation switch, is in flame position.

5.10 REGULATOR (Figure 14)

The DB measures the per cent T relationship of the sample and reference signal at an 85 volt level. This is accomplished by comparing the amplitude of the reference signal to a fixed voltage from the Power Supply. The difference is amplified and used to bias the control tube of the dynode voltage regulator. The control tube (6EM5) and photomultiplier divider share the voltage of a floating power supply. The relative voltages appearing across these elements is determined by the bias of control tube.

When the reference signal appears on C13 it is switched to the dividing network C3, R10 and R11. R12 couples the divided portion to the grid of V2B where it is compared with the reference voltage appearing on the cathode of V2B. R5 and R6 divide the voltage of V4 in the same ratio as the reference signal divider, R10 and R11, so the reference signal will be regulated at an 85 volt level within the bias of V2B. The 85 volt level was selected so the bias stability of V2B and contact potential of CR3 would be small compared to the linearity and stability of the instrument. If the divided portion of the reference signal is equal to the reference

voltage of the cathode, V2B will conduct, resetting the voltage of C4 through integrating resistor R66. The cathode voltage on V2B plus the voltage on C4 is coupled to the grid of V5 through R14. The voltage divider R18, R19 and R20 establish the operating point of V5. The cathode voltage on V2B plus the voltage on C4 minus the voltage across R20 is the bias voltage on V5. The photomultiplier divider R21 through R29 is the load resistor for V5. Thus the floating power supply is shared by V5 and the dynode resistors R21 through R29. If the bias of V5 is decreased by an increase in the voltage on C4, the current through V5 will increase; this increased current flowing through R21 through R29 will increase the voltage across the dynode resistors. The photocathode will be negative because of the location of the ground point in the plate circuit of V5.

CR1, C6, C7 and CR2 with the shielded winding of T1, is a half wave voltage-doubling power supply for the control stage V5. The double Faraday shield around the winding of T1 is to prevent capacity coupling of 60-cycle and line transients to the high voltage supply.

R14, C5 and R17 is a feedback network that serves two purposes: Transients originating in the high voltage supply are coupled to the grid of V5, their effect on the dynode voltage is reduced by the gain of V5; also any change in the voltage on C4 is degenerated by feedback through C5 and R17. The effective capacity of C5 is increased by the gain of V5 by its position in the circuit. Thus the time constant of C5 and R14 is approximately equal to the sustaining time constant of R13 and C4. The time constant R13 plus R66 and C4 maintain the voltage on the grid of V5 during the absence of the reference signal.

5.10.1 CIRCUIT OPERATION (REFERENCE SIGNAL ABSENT)

When there is no reference signal across R10 and R11 the grid of V2B is connected to ground through R11. The cathode is at the divider voltage of R5 and R6, therefore V2B is cut off . The voltage on C4 slowly increases by current through R13 and R66. This change in voltage is applied to the grid of V5 where it increases the current through V5. The increased current through the dynode resistors R21 through R29 increases the voltage across the feedback network R17, C5 and R14. When R17 is properly adjusted the feedback voltage will compensate for the change of voltage on C4 with minimum change in dynode voltage.

5.10.2 CIRCUIT OPERATION (REFERENCE SIGNAL PRESENT)

When the reference signal first appears across the divider R10 and R11, it will be large enough to cause V2B to conduct. This in turn resets the voltage on C4 by discharging C4 through R66. The reduced voltage of C4 applied to the grid of V5 reduces the current through V5. The reduced voltage on the dynode resistors of the photomultiplier reduces the current gain of the photomultiplier thus the amplitude of the reference pulse is decreased. This action continues until the amplitude of the reference pulse is again equal to 85 volts. At this time C4 is "reset". V2B is near "cut off" and the circuit is ready to "coast" during another sample pulse.

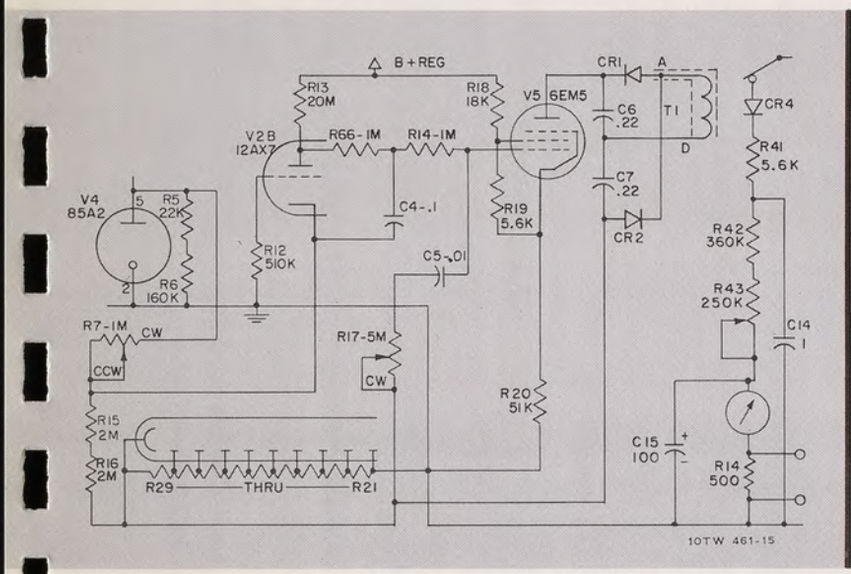


Figure 15. Circuit Diagram, Flame Operation

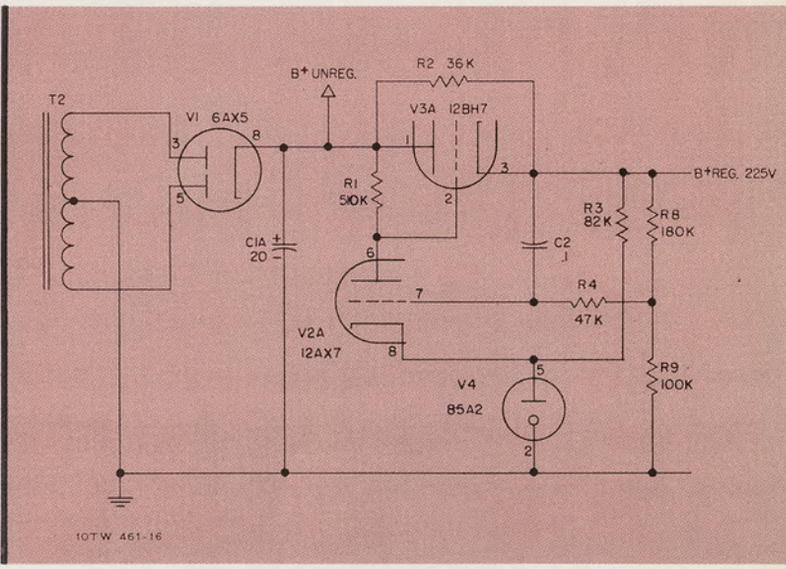


Figure 16. Circuit Diagram, Regulated Power Supply

5.11 FLAME (SINGLE BEAM) OPERATION (Figure 15)

In single beam or flame operation, all components beyond V2B operate in the same manner as in double beam or reference control operation. When the operation switch is in flame position, the following circuit changes are made: The grid of V2B is connected to ground through R12; the cathode of V2B is connected to the junction R7 and R15. C15 is connected across the meter circuit for additional filtering. With the grid of V2B returned to ground through R12 and the cathode connected to 85 volts through R7, V2B will remain cut off until C4 is charged through R13 and R66 to the point where the conduction of V5 increases the voltage across R21 through R29.

The dynode voltage is applied to the divider R16, R15 and R7. When this current is sufficient to produce 85 volts across R7, the cathode voltage will approach the grid voltage and V2B will conduct. V2B will continue to regulate the dynode voltage at this level until R7 is changed. If R7 is decreased, the dynode voltage will be increased, and conversely, if R7 is increased, the dynode voltage will be regulated at a lower level.

5.12 POWER SUPPLY

The power supply consists of a full wave rectifier condenser input filter with conventional series regulator for regulated DC of 225 volts. Transformer T2 includes two 6-volt windings so the filaments may be referenced to a DC voltage near their cathode potential and a 5.5-volt winding with full wave rectifier and condenser input to filter the voltage to the tungsten source. Switch 2 has five sections for the following functions: Section 2A connects 115 volts a.c. to transformers T1 and T2 in the last three of four positions. Section 2B connects 115 volts a.c. to the tie point TP1 in the fourth or scan position of switch 2 for the wavelength drive motor. Section 2C connects a short across the meter in the first and second (off and idle)

positions of switch 2. Section 2D connects B+ unregulated to the cathode followers V8B and V9 in the third and fourth (on and scan) positions of V2. Section 2E closes the circuit to scan receptacle P5 in the fourth or scan position.

5.13 REGULATED SUPPLY (Figure 16)

The 800-volt center tapped winding of T2 supplies 400 volts RMS to each plate of the full wave rectifier V1. The d.c. voltage appearing on the cathode of V1 is filtered by C1A and supplies unregulated d.c. voltage to the cathode followers V2B, V8B, V9 and filament reference divider R45 and R46. V3A is a series regulator shunted by R2, to increase its current capacity, and controlled by V2A through load resistor R1.

V2A is a voltage amplifier with its cathode fixed at 85 volts by V.R. tube V4. R8 and R9 form a divider for the grid of V2A and determine regulating point of B+. C2 and R4 increase the high frequency feedback and thus reduce the ripple in regulated B+.

5.14 OPERATION

As the regulated voltage appearing at the junction of dividers R8 and R9 increases toward 85 volts the current through V2A increases. This increased current through R1 lowers the plate voltage of V2A as well as increasing the bias on series regulator V3A.

The increased bias of V3A increases the voltage drop across the tube correcting for the change in B+ regulated. The circuit operates in the same manner for ripple and transients with the exception of the regulating divider. Ripple and transients are divided by C2, R4, and R9, therefore the feedback is a function of the impedance which in turn is a function of the frequency of the transient. The regulator will maintain B+ regulated to better than 1%.

5.15 VIBRATING MIRROR SYSTEM

The vibrating mirror system is a four bar linkage with pivots of gold-plated steel reeds. A silicon-steel armature is attached to one of the side members. The armature passes through a coil form which has polarizing magnets molded in. When a current passes through the coil, the flux induced in the armature bar reacts with the polarizing magnets and displaces the mirror system.

A vane, attached to the linkage, interrupts a light beam illuminating a cadmium selenide cell, producing a signal in phase with the vibrating mirror system. This signal is amplified and coupled to the cathode follower with the driving coil in the cathode circuit. Thus, the mirror system is driven at resonance with simple harmonic motion.

The amplitude is limited by another vane which uncovers a cadmium selenide cell, illuminating it at full amplitude. This signal is coupled by means of a bridge circuit to decrease the driving signal, regulating the amplitude.

5.15.1 CIRCUIT DESCRIPTION

A photosensitive cadmium selenide cell (CD1) is illuminated by a neon bulb (I2). An opaque vane attached to one leg of the linkage is positioned between the neon bulb and sensitive area of CD1, interrupting half of the light when the linkage is at rest. Moving the linkage in one direction increases the illumination, decreasing the resistance of CD1. R51 and CD1 form a voltage divider across an electrical bridge with legs R49 and a thermister on one side and CD2 and R52 bypassed by C18 on the other. When the resistance of CD1 decreases, the voltage at the junction of CD1 and R51 decreases. This voltage is coupled to the grid of V8A by C17 and R53, decreasing the grid voltage.

Decreasing the grid voltage decreases the voltage drop across plate load resistor R55. This positive going voltage is shifted in phase by R56 and C21, then coupled to the grid of cathode follower V9 by C23, grid limiter R26 and grid return R60. The cathode of V9 follows the grid, increasing the current through the driving coil K2. Condenser C1B blocks the direct current through the coil, preventing overheating. Resistor R63 is a biasing resistor in series with resistor R58 to form a d.c. return. When the linkage reaches an extreme position, the signal is reversed and the mirror system is driven in the opposite direction. When the system reaches full amplitude, a vane uncovers CD2 illuminated by neon bulb I3 in one extreme position. Thus CD2 acts as a rectifier, increasing the voltage drop across R52, filtered by C18. Raising the voltage at the junction of R52 and CD2 decreases the voltage across CD1 and R51, decreasing the driving signal.

Potentiometer R49 serves as an amplitude control by setting the proper voltage across the bridge. A thermistor compensates for the temperature coefficient of CD2.

5.16 DEMODULATOR DRIVING CIRCUIT

Mercury relay K1 is driven by the same signal that drives the bridge, switching the sample and reference signals in phase with the vibrating mirror system.

The signal at the plate of V8A is shifted in phase by potentiometer R64 and C20. This signal is again shifted in phase by R57 and C22, then coupled to the grid of cathode follower V8B by C24 and grid return R61. The coil K1 is driven by the cathode with C1C blocking the d.c. R65 is a biasing resistor in series with R59, forming a d.c. return.

The switching point is adjusted by potentiometer R64, while interchanging connections B-B will provide 180° phase change.

Section Six The Science of Spectrophotometry

Absorption spectrophotometry is based on the observation and comparison of absorption spectra. The absorption spectrum -- a curve showing the amount of radiant energy absorbed at each wavelength -- is a characteristic property of many chemical compounds, i.e., each such chemical compound has its own absorption spectrum. Basically, the spectrophotometer provides absorption data to assist the chemist in qualitative and quantitative analysis. Before considering spectrophotometric analysis in more detail, here are a few commonly encountered terms.

6.1 TRANSMITTANCE

Transmittance (T) is the ratio of the radiant energy transmitted by the sample (P) to the energy incident upon the sample (P_o). Both radiant energies must be obtained at the same wavelength, with the same spectral slit width.

$$T = \frac{P}{P_o} \qquad \text{or} \quad \%T = \frac{P(100)}{P_o}$$

Transmittance is usually given in per cent.

Since it is seldom possible to measure these radiant energies directly because of the presence of a sample cell, it is customary to consider the transmittance of the sample as the ratio of the light transmitted by the cell and the sample to the light transmitted by some arbitrary standard. In other words, the sample is compared to a standard. This standard is often the cell filled with the liquid in which the sample is to be dissolved. The transmittance of this standard, of course, is defined as 100 per cent. Therefore, when the transmittance of a sample is given, it is necessary to specify the standard with which the sample was compared.

6.1.1 ABSORBANCE

Absorbance (A) is the negative logarithm to the base 10 of the transmittance: $A = -\log T = \log 1/T$ (T is expressed as a decimal fraction, not in per cent.) As with transmittance measurements, the standard with which the sample was compared should always be included with the absorbance data. The absorbance of the standard is defined as zero.

6.1.2 SPECTRAL SLIT WIDTH

Spectral Slit Width is the total range of wavelengths emerging from the exit slit, neglecting stray light and spherical aberrations. Spectral slit width is important to the spectroscopist because it indicates which wavelengths are involved in a given transmittance measurement. Spectral slit width = 2 x dispersion in $m\mu$/mm x slit width in mm.

6.1.3 EFFECTIVE BAND WIDTH

Effective Band Width refers to the span of wavelengths emerging from the exit slit of the monochromator, each of which contributes at least half as much energy as does the wavelength with the greatest energy. See Figure 17A. Effective band width is expressed in millimicrons.

6.1.4 RESOLUTION

Resolution is the ability of the instrument to distinguish between two peaks closely spaced in wavelength.

6.1.5 STRAY LIGHT

Stray Light is extraneous light which reaches the phototube at wavelengths which do not correspond to the Wavelength Scale setting.

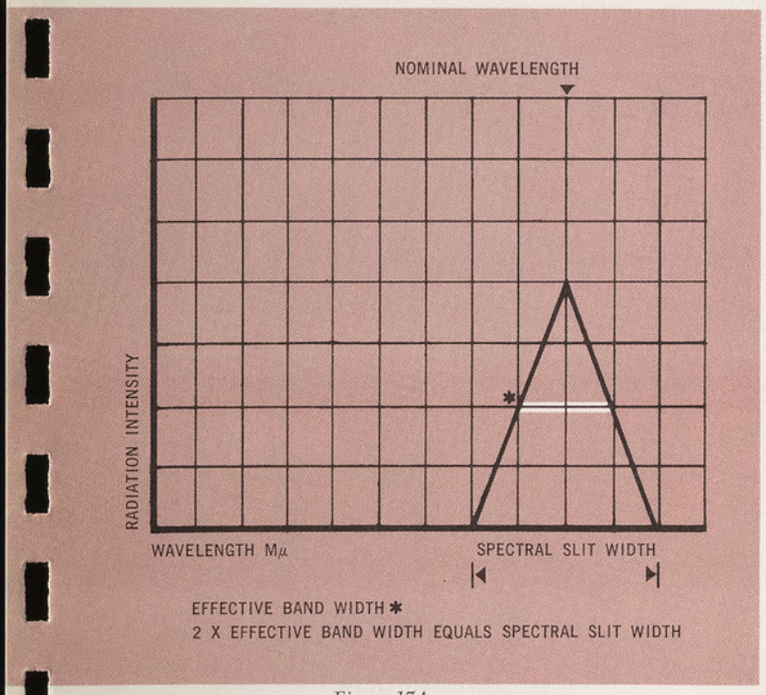
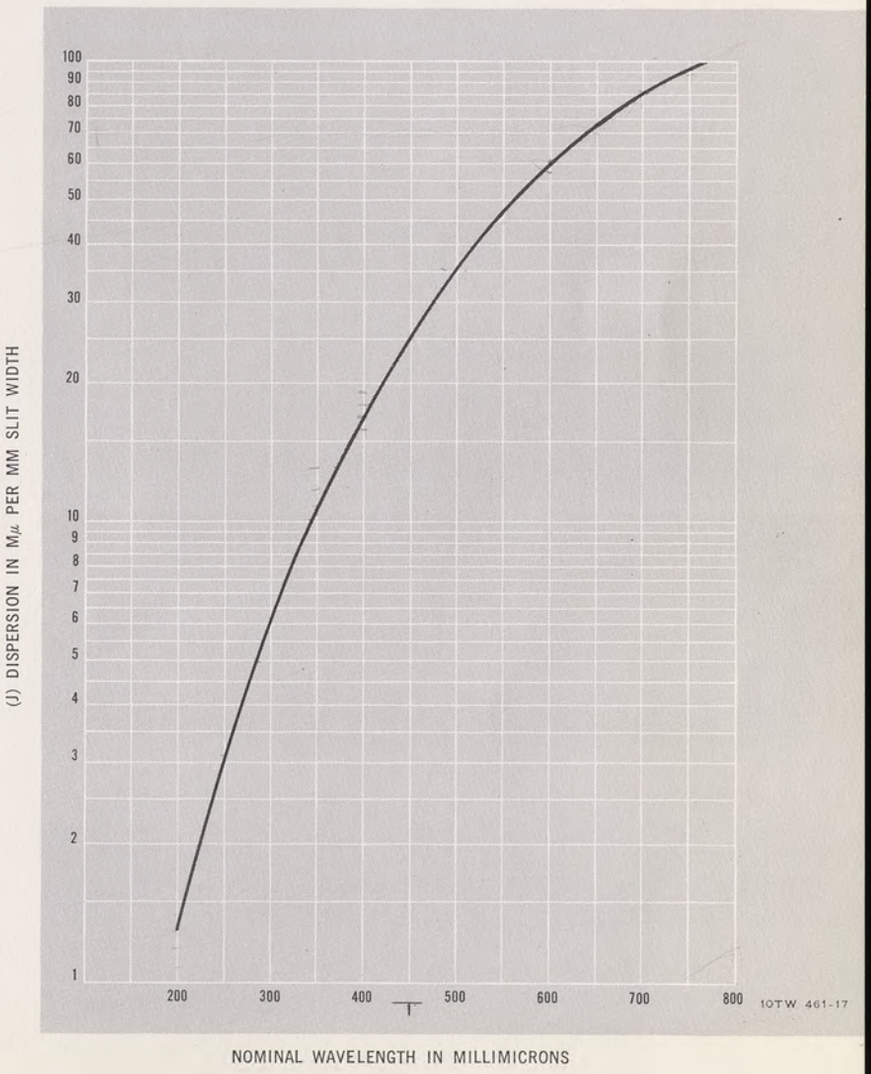


Figure 17A

Figure 17. Bandwidth Dispersion Data



6.2 BAND WIDTH

The light emerging from a monochromator with a continuum source does not consist of a single wavelength but of a group of wavelengths. If the light emerging from the exit slit were examined in detail and its intensity plotted as a function of wavelength, a curve would result similar to that in Figure 17A. The nominal wavelength is that wavelength which corresponds to the peak of the curve. The curve in Figure 17A is an idealized one that would be obtained with a perfect optical system. In practice, unavoidable aberrations result in the effective widening of the slit image, and thus the triangle shown should be slightly rounded at the top and bottom.

In the Model DB the dispersion of the Fused Silica -- its separation of light into its component wavelengths -- is nonlinear with wavelength. For example, two spectral lines whose wavelengths differ by 10 millimicrons in the blue region, are separated (in the dispersed beam) by much greater physical distance than a corresponding pair of spectral lines in the red region.

Conversely, therefore, any given slit width will pass a much wider band of wavelengths in the red range than in the blue.

In order to achieve true per cent transmittance readings, two peaks must be completely resolved. The apparent per cent transmittance will depend upon the slit width and will include such influences as the shape and slope of the transmittance curve within the wave band being transmitted and the variation of the transmittance of the optical system with respect to wavelength. It is usually easy to determine if these effects are significant by increasing or decreasing the slit width by a factor of two or more; a change in apparent transmittance then indicates that these effects are pertinent. Whenever very reliable values must be obtained, the narrowest possible slit width should be used, and the above check involving change of slit width should be made.

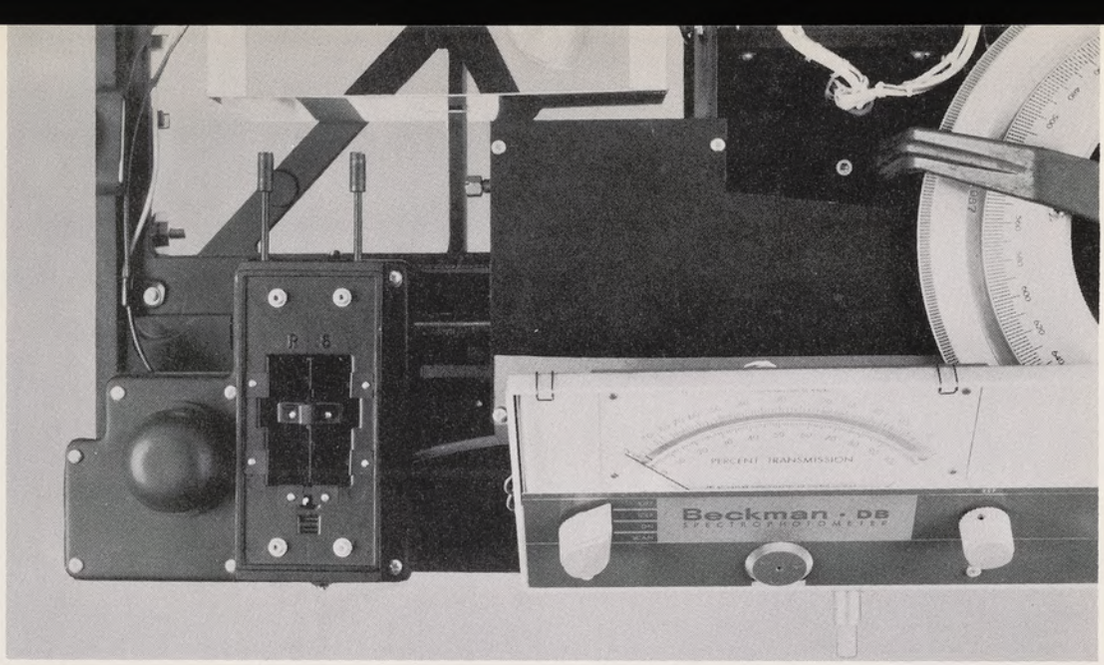
6.3 CALCULATION OF BAND WIDTH

To calculate band widths, it is necessary to know the dispersion of the instrument in $m\mu$ per millimeter of slit width at the wavelength being used, the actual slit width being used, and the effective widening of the image by diffraction effects in the optical system. The effective band width is proportional to the width of both the entrance and exit slits. Since in the DB the slit widths are equal, the effective band width is the linear dispersion times the width of the exit slit plus diffraction effect.

The effective band width, due to strictly geometrical considerations, is $\Delta\lambda_S = JS$.

Where: J = dispersion in $m\mu$/mm
S = width of slit in mm.

This formula for effective band width disregards the effects of slit diffraction.



In the wavelength range of the DB these diffraction effects are very small compared to the slit widths necessary due to energy limitations. As an example, diffraction effects should be considered only when the geometrical slit width approaches the slit width caused by diffraction effects. These slit widths are approximately figured by the formula $S_d = 10\lambda$. This is true of a monochromator with an f10 system. Considering one wavelength, 800 mμ, where the diffraction effect will be the greatest, the formula gives a slit width of .008 mm. This slit width is less than the .01 mm minimum slit width that is recommended for use on the DB. It is also much less than is practical due to energy available in this region.

6.4 ABSORPTION

All substances transmit some portions of the electromagnetic spectrum and absorb others. The color of red glass, for example, results from the fact that it absorbs light of short wavelengths, the green and the blue, and transmits light of long wavelengths, the red and some yellow light. The concentration of the solution and its thickness will further affect the absorbance. If the absorbance is plotted against wavelength the spectral absorbance curve of the sample is obtained.

Just as each component has its own absorbance spectrum, a mixture of two or more components will yield an absorbance spectrum containing characteristics of all the components. The absorbance at each wavelength is the sum of the absorbances of the components of the mixture at that wavelength; the contribution of each component is proportional to the concentration and to its absorptivity at that wavelength. Absorptivity is the ratio of the absorbance to the product of concentration and path length.

6.5 QUALITATIVE INTERPRETATION

Problems involving the identification of organic components can be solved by spectrophotometric techniques through use of the matching fingerprint method. If the spectroscopist has an extensive library of absorbance curves for known compounds, positive identification of the unknown is established in a very short time if the unknown spectrum matches any of the known spectra. If the unknown spectrum does not match any of the knowns, the negative data is still important because it restricts the field of possibilities.

6.6 QUANTITATIVE INTERPRETATION

Usually in quantitative work the constituents of the sample are known and it is desired to determine the concentration of one or more of them. If, however, it is necessary to decide what components are present, this information may be obtained from the history of the sample, by ordinary qualitative chemical analysis, or by comparing the spectrum of the mixture with the spectra of various pure samples as discussed above.

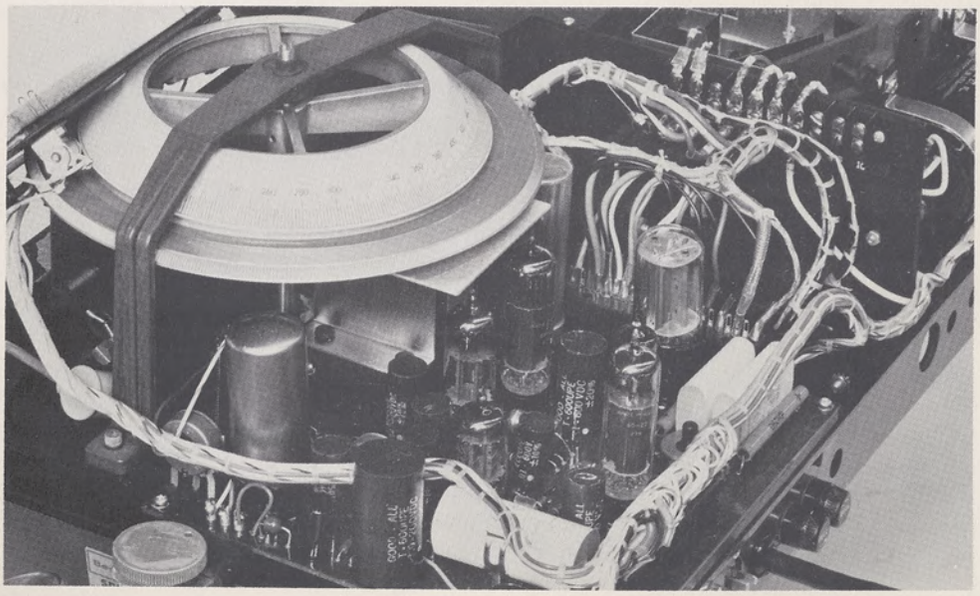
6.7 BEER'S LAW

Quantitative spectrophotometry is based upon the fact that the absorbance of an absorbing material is dependent upon its concentration. If the absorbance is directly proportional to the concentration, the system is said to obey Beer's Law. The absorbance of a sample is also directly proportional to the length of the light path within the sample. (In much practical spectrophotometric work, the cell length is a constant, and therefore, it cancels out in Beer's Law calculations). Mathematically:

$$A = abc$$

where A is the absorbance, a is the absorptivity (unit area per unit mass), b is the length of the light path, and c is the concentration (mass per unit volume).

Assuming that Beer's Law is obeyed strictly, it would be very simple to obtain the concentration of an unknown by simply comparing the absorbance of a known sample. Concentration of the unknown could then be readily obtained by simple arithmetic; i.e., by proportion.



In practice, however, slight deviations from Beer's Law are noted. In making quantitative measurements, it may be necessary, therefore, to plot the absorbance of a series of standards (solutions containing known concentrations of the absorbing material) against their concentrations. In these graphs, concentration is given linearly on one axis and absorbance is given linearly on the other axis. Usually a slight curved line passing through the origin is obtained. When the absorbance of an unknown has then been obtained, its concentration may be easily read from the graph.

The applicability of Beer's Law depends on adherence to certain requirements. The samples must be compared with a 100 per cent reference in which the concentration of the absorbing substance is zero; the absorbing substance must not participate in an equilibrium in the solution which would be shifted by change in concentration; essentially monochromatic light must be made at wavelengths where the absorbance does not change appreciably with wavelength; i.e., a horizontal portion of the absorbance curve.

The absorbance of a mixture at a particular wavelength is the sum of the absorbances of all the components of the mixture.

6.8 TRANSMITTANCE AND ABSORBANCE MEASUREMENTS

Transmittance or absorbance measurements are always made by comparing a sample to a standard (or reference). As the standard would obviously have a transmittance of 100 per cent compared with itself, it may be called the 100 per cent reference. It is possible to define various standards, but usually the standard is a blank , i.e., a solution identical in composition with the sample except that the absorbing material being measured is absent. In making the spectrophotometric measurement, the operator sets the instrument at 100 per cent by using the 100 per cent reference. The sample is then placed in the light path and its transmittance or absorbance is read.

The 100 per cent reference contains none of the absorbing substance. In the Beer's Law graph discussed above, therefore, the 100 per cent reference will lie at zero on the concentration axis. In this instance, the 100 per cent reference may be considered to be one of the series of standards used in preparing the graph.

Often, quantitative techniques are developed where the standard is a known concentration of the sample. Use of such a standard allows greater measuring accuracy and precision of measurement, particularly where the samples are stable and where strong absorption bands must be used for measurement.

Section Seven Maintenance

7.1 REPLACING TUNGSTEN SOURCE

The tungsten source in the DB is operated at reduced voltage (5.5 volts at 115-volt line) for longer life. When the source burns out always replace with BII Part No. 2305; G.E. Part No. 2331.

(1) Remove power cord from 115-volt outlet.

(2) Remove source and electronic louvres.

(3) Loosen two screws at each end of die cast cover, lift and tilt forward to remove.

NOTE

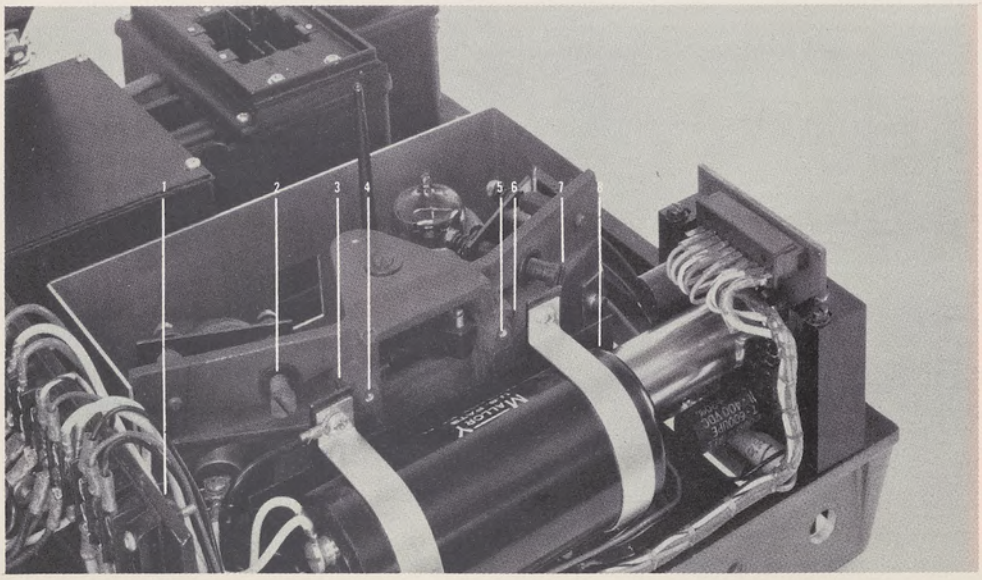
All adjustments with the cover removed must be made by qualified electronic personnel.

(4) Wrap the tungsten bulb with a cloth, twist counterclockwise until detents are loose and remove.

(5) Wrap the replacement bulb with a cloth, orient detent slots to fit the projecting pins of the socket, press firmly in place and twist clockwise until the detents lock.

7.1.1 POSITIONING AND FOCUSING TUNGSTEN SOURCE (See Figure 18)

With the cover removed and the operation switch in flame position (wavelength at 500 mμ) make the following adjustments, each time reducing gain or slit width until the meter reads mid-scale.



1. TUNGSTEN SOURCE VERTICAL ADJUST
2. TUNGSTEN SOURCE FOCUS ADJUST
3. HYDROGEN SOURCE MIRROR ADJUST
4. 4-40 LOCK SCREW
5. 4-40 LOCK SCREW
6. TUNGSTEN SOURCE MIRROR ADJUST
7. HYDROGEN SOURCE FOCUS ADJUST
8. HYDROGEN SOURCE VERTICAL ADJUST

Figure 18. Source Light Adjustments

10TW 461-18

(1) Adjust the source vertical screw to center the beam vertically on the entrance port just in front of the slit diagonal mirror.

(2) Loosen the 4-40 lock screw and adjust the tungsten source mirror screw to center the beam horizontally on the entrance slit aperture.

(3) Adjust the source horizontal screw (focus adjustment) for maximum energy, maintaining horizontal centering as in Step 2. The focus adjustment is not independent.

Tighten the 4-40 lock screw on the Horizontal Adjustment.

7.2 REPLACING HYDROGEN SOURCE (Part No. 8333)

(1) Remove power cord from 115-volt outlet.

(2) Remove source and louvered covers.

(3) Loosen two screws at each end of the die-cast cover, lift and tilt forward to remove.

(4) Loosen three lugs adjacent to the hydrogen source (note color position for replacement).

(5) Loosen two clamp screws, rotate the source window to open area of clamps and lift out.

(6) Insert new source, rotate the source window between the clamps until in faces the source mirror. Center vertically and tighten the clamps.

(7) Replace the lugs on the terminal strip, noting color code.

7.2.1 POSITIONING AND FOCUSING HYDROGEN SOURCE (See Figure 18)

Plug the hydrogen lamp cord into the hydrogen source power supply and fire according to instructions.

NOTE

Protective glasses must be worn when adjusting the hydrogen source, since ultraviolet radiation is harmful to the eyes.

With the cover removed and the operation switch in flame position, wavelength at 300 mμ, proceed as follows, reducing gain or slit width until the meter reads midscale after each step.

(1) Adjust the source vertical screw to center the beam vertically on the entrance port just in front of the slit diagonal mirror.

(2) Loosen the 4-40 lock screw and adjust the hydrogen source mirror screw to center the beam horizontally on the entrance slit aperture.

(3) Adjust the source horizontal screw (focus adjust) for maximum energy. Since the focus adjust is not independent, maintain horizontal centering as in Step 2.

(4) Tighten the 4-40 lock screw on the Horizontal Adjustment.

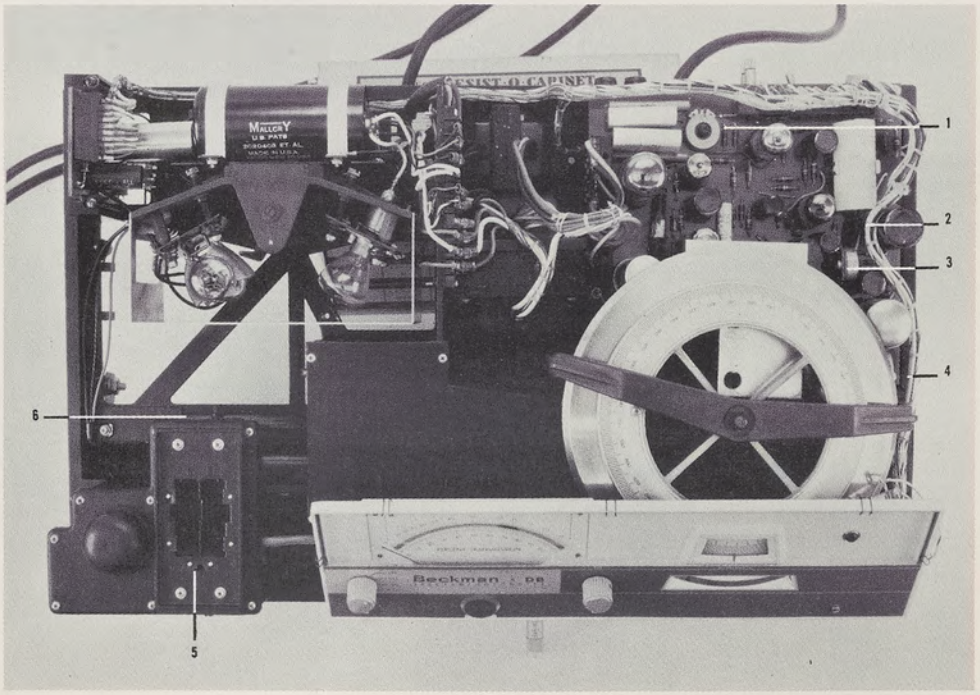
7.3 ADJUSTING MANUAL SLIT ZERO

The DB must be in flame operation, slits in manual position, wavelength at 500mμ, tungsten source and gain maximum. Note the meter displacement with the tungsten source off and micrometer slit adjust in maximum clockwise position. Turn the tungsten source on, set the micrometer slit adjust on zero, loosen the setscrew at the bottom of the cone-shaped thimble and slide the micrometer slit adjust in or out until the meter reads five divisions higher than it did with the source off and slit adjust in maximum clockwise position. Tighten the setscrew maintaining the meter reading.

7.4 VIBRATING MIRROR AMPLITUDE

The top of the vibrating mirror structure may be seen through the inspection port in the front of the sample compartment if it is illuminated by a flashlight through the beam exit port of the sample compartment. See Figure 19.

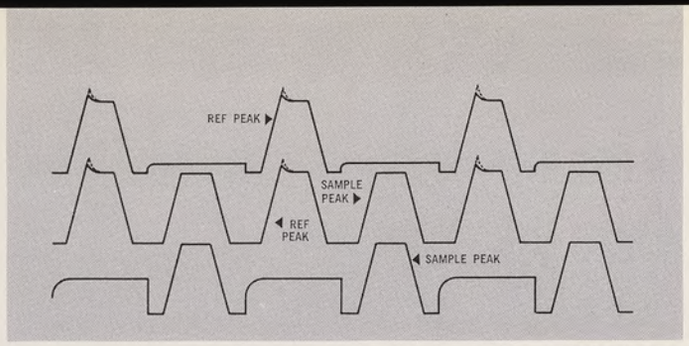
Since the vibrating mirror system is moving with simple harmonic motion it momentarily stops at the extremes of its travel and the gold-plated reeds appear to stand still in these positions. The mirrors are moving with the correct amplitude when the reeds appear as one. If two slightly displaced reeds are seen, adjust (R49) amplitude control (see figure 19) until the reeds appear as one.



1. R17 — AMPLIFIER FEEDBACK CONTROL POTENTIOMETER
2. JUNCTION OF C13 AND CR3
3. R64 — PHASING CONTROL POTENTIOMETER
4. R49 — AMPLITUDE CONTROL POTENTIOMETER
5. INSPECTION PORT
6. REAR OF SAMPLE COMPARTMENT

Figure 19. Adjustment Locations

10TW 461-19



10TW 461-20

Figure 20. Oscilloscope Patterns (Scope on A.C.)

7.5 PHASING

The phasing control potentiometer (R64) adjusts the switching position of the Clare relay. See Figure 19. The Clare relay must switch during the blank portion of the cycle. There are two methods for determining correct setting.

Method 1 (without oscilloscope):

With the DB operating normally in double-beam, block the sample beam with the opaque block. Rotate the phasing control slowly first in one direction until the meter begins to move off zero; note angular position of the control. Then rotate in the other direction until the meter begins to move off zero. Return the control to a position midway between these two positions. This is the correct setting for the phasing control.

Method 2 (with oscilloscope):

Attach a scope between the junction of R10 and C3 and B-(bottom pin of recorder terminal). Set oscilloscope attenuator for 100 volts and sweep rate of approximately 18 cycles per second with internal triggering. Two reference pulses will be displayed on the screen. Switching "spikes" will be seen on both sides of the reference pulses. The phasing control is adjusted properly when the reference pulse is centered between these switching "spikes". See Figure 20. (Ref Pulse After Switching)

7.6 REFERENCE AMPLIFIER FEEDBACK POTENTIOMETER ADJUSTMENT (R17, Figure 19)

The reference amplifier feedback is properly adjusted when the instrument leaves the factory and should not require adjustment unless it is accidentally moved. To display the reference pulse, connect an oscilloscope in the same manner as Method 2 of Phasing. Rotate the potentiometer in both directions and note appearance of a spike on the top leading edge of the reference pulse. R17 is properly adjusted when this spike is just discernable.

7.7 REPLACING PHOTOMULTIPLIER

(1) Remove power cord from 115-volt receptacle.

(2) Remove the top cover and bottom panel.

(3) Remove the rubber cap located at the left of the sample compartment.

(4) Support the top of the photomultiplier with one hand and push on the centering pin of the octal socket with a pencil or suitable rod.

(5) Replace with BII Part No. 11754.

(6) Replace rubber cap and bottom panel.

(7) Operate the DB in single-beam and adjust the horizontal and vertical detector mirror screws for maximum output, lock in position.

7.8 TUBE REPLACEMENT

All tubes used in the DB are standard tubes and checks made on a transconductance checker are indicative with the exception of V7(6AK5). V7 can be tested in operation by checking the open loop gain of the amplifier.

7.9 CLEANING MIRRORS

If the dust cannot be blown from the mirrors with dry nitrogen, proceed as follows:

(1) Mix a rich suds of a suitable detergent, such as Dreft (never use soap).

(2) Soak a ball of fresh surgical cotton in the suds, handle the cotton with tweezers only. Body oils are difficult to remove from mirrors.

(3) Draw the saturated cotton over the mirror. Do not touch the mirror with the tweezers.

(4) Rinse immediately with distilled water.

(5) Rinse immediately with clean ethyl alcohol.

Do not let the mirror start to dry between rinses.

NOTE

Do not remove mirrors for cleaning. Special fixtures are required for aligning. The only mirrors exposed to dust are the source mirror and entrance slit diagonal. These may be cleaned by placing a tissue at the base of the mirror and using fine-tipped squeeze bottles for rinsing. Always remove power cord from socket before cleaning any mirrors.

7.10 SAMPLE COMPARTMENT POSITIONING

The sample compartment supports the sample and reference cells as well as blocking the beam in between the sample and reference pulses. This compartment is properly positioned at the factory and should not be removed unless an oscilloscope is available for positioning. To position the sample compartment, connect an oscilloscope between the junction of C13, CR3 and B- (lower terminal of recorder output). See Figure 19. Set attenuator for 100 volts and sweep rate at approximately 70 cycles per second, internal trigger, folding the reference pulse over the sample pulse. With the DB operating normally in double beam, adjust the rear of the sample compartment to superimpose the sample and reference pulses as received on the scope. Position the front of the sample compartment to center the sample and reference beams on the exit ports. Note: Widen the slits and set the wavelength at approximately 500 mμ. Place a small rectangular card in each of the beams and visually determine the centering. Remove the cards and recheck beam superposition. See Figure 20 for oscilloscope pattern.

Section Eight Accessories and Replacement Parts List

A number of accessories are available for use with the Model DB. These accessories are designed to provide the instrument with greater flexibility and scope of operation.

8.1 SINGLE-SPEED WAVELENGTH DRIVE ACCESSORY

Beckman No. 28180

This unit adapts the 1400 Model DB for use with a recorder at a standard scanning rate of 50 mμ/min, providing per cent T readings. The unit, delivered ready for installation, includes instructions and a 49890 Recorder Cable Accessory for use with the Beckman 5-Inch Strip-Chart Recorder.

8.2 DUAL-SPEED WAVELENGTH DRIVE ACCESSORY

Beckman No. 74475	5/25 mμ/min speeds
Beckman No. 72099	10/40 mμ/min speeds
Beckman No. 73272	20/80 mμ/min speeds

These accessories provide flexibility of operation by permitting a choice of two scanning speeds. Speed selection is made by means of a switch on the back panel of the instrument. Each of these accessories is delivered with installation instructions and a Recorder Cable Accessory.

8.3 FLAME PHOTOMETRY ATTACHMENT

Beckman No. 28260 (supplied without burner)

The Flame Photometry Attachment provides rapid qualitative and quantitative determinations of more than 30 metallic elements. The attachment is provided with a regulation unit, bracket assembly, exhaust chimney, fuel and oxygen hoses, and a package of 12 five-ml beakers. An Oxygen-Hydrogen or Oxygen-Acetylene Atomizer-Burner Assembly is required but not supplied.

8.3.1 FLAME ATTACHMENT BURNERS

To be ordered separately when the 28260 Flame Photometry Attachment is ordered.

4020 Atomizer-Burner, Oxygen-Hydrogen or Air-Hydrogen, medium bore.
4030 Atomizer-Burner, Oxygen-Acetylene, medium bore.
4040 Atomizer-Burner, Oxygen-Acetylene, small bore.
4050 Atomizer-Burner, Oxygen-Hydrogen, or Air-Hydrogen, small bore.
4060 Atomizer-Burner, Oxygen-Hydrogen or Air-Hydrogen, large bore.
4090 Atomizer-Burner, Oxygen-Acetylene, large bore.

8.4 ULTRAVIOLET ACCESSORY

Beckman No. 28190

Extends the wavelength range of the Model DB to 205 mμ. The accessory

includes an 8333 Hydrogen Lamp (30 watts), a mounting plate assembly and all necessary hardware. Requires, but does not include, a Hydrogen Lamp Power Supply.

8.5 HYDROGEN LAMP (UV) POWER SUPPLY

Beckman No. 2965

Includes single housing with necessary electronics for 115-volt, 60-cycle a.c. line operation of the Hydrogen Lamp. The Ultraviolet Accessory, including the Hydrogen Lamp, is not included.

8.6 REPLACEMENT PARTS LIST

DESCRIPTION	BECKMAN NO.	DESCRIPTION	BECKMAN NO.
Fuse - 1 amp - F1 - F2	148	Prism - Assembly	28026
Switch - SW3	9889	Mirror Assembly - Diagonal	28132
Tube - 12AX7 - V2	15093	Mirror Assembly - Receiver	49807
Tube - 85A2 - V4	20820	Adjust. Assembly - Manual Slit	49822
Tube - 12BH7 - V8, V3	23742	Plug - Hole	49854
Tube - 6AX5 - V1	24327	Diode Rectifier - CR1, CR2	79502
Potentiometer - 250K - R43	24747	Diode Rectifier - CR5, CR6	79483
Transformer - Photo - T1	28040	Diode Rectifier - CR3, CR4	79296
Transformer - Power - T2	49874	Capacitor - 10,000 MFD	79484
Switch - SW1	54045	Coil Assembly	49842
Knob	79284	Mirror - Chopper	49865
Compartment Assembly - Sample	28128	Plug - Photocell Holder	49882
Holder Assembly - Cell	28129	Photocell Assembly	49880
Circuit Board Assembly - Large	49886	Lamp Assembly	49879
Board Assembly - Small	49891	Potentiometer - R17 - 5 Meg.	79506
Switch Assembly - SW2	49900	Potentiometer - R49 - 100K	79507
Tube - 6AK5 - V7	63162	Potentiometer	810073
Knob	79285	Potentiometer - R64 - 250K	79426
Tube - 6EM5 - V5	79513	Plug Adapter	16988
Relay - Chopper	79516	Screw - Compensating	74315
Tube - 6973 - V9	99617	Gasket - Sample Compartment	49827
Lamp	99931	Bezel Assembly	28148
Lamp - Tungsten	2305	Spring - Extension	28151
Socket - Lamp	22746	Chopper Assembly	49885
Mirror - Source	28042	Vane	49833
Panel - Glass	28104	Vane	49834
Lamp Assembly - Pilot	28172	Cable - Chopper	49881
Gasket - Panel	28177	Capacitor - C1	79514
Knob - Meter Zero	28142	Photomultiplier 1P28 (Selected)	11754
Mirror - Collimator	28021		

SOLVENTS FOR VISIBLE AND UV SPECTROSCOPY

SOLVENT	DB λ RANGE IN MILLIMICRONS	COMMENTS
Water, distilled	200-800	Limited to water-soluble samples. Use freshly-boiled water to avoid air bubbles.
Ethyl Alcohol	210-800	Very valuable; dissolves the great majority of organic compounds. Use good absolute alcohol, not dried by azeotropic distillation (which leaves traces of benzene).
Isopropyl Alcohol	210-800	Same properties as Ethyl Alcohol. Traces of water and acetone impurities.
Ether	220-800	Very useful; rapid evaporation dictates use in sealed, one-piece cells.
Cyclohexane n-Hexane n-Heptane Iso-Octane n-Pentane	210-800	Saturated hydrocarbons; excellent when pure. Pour through chromatographic column filled with silica gel to remove traces of benzene.
Acetonitrile N, N-Dimethylformamide	200-800 270-800	Widely used in UV work.
Chloroform	245-800	Dissolves materials not readily soluble in alcohol; contains a minimum 1% alcohol as stabilizer.
Carbon Tetrachloride	265-800	

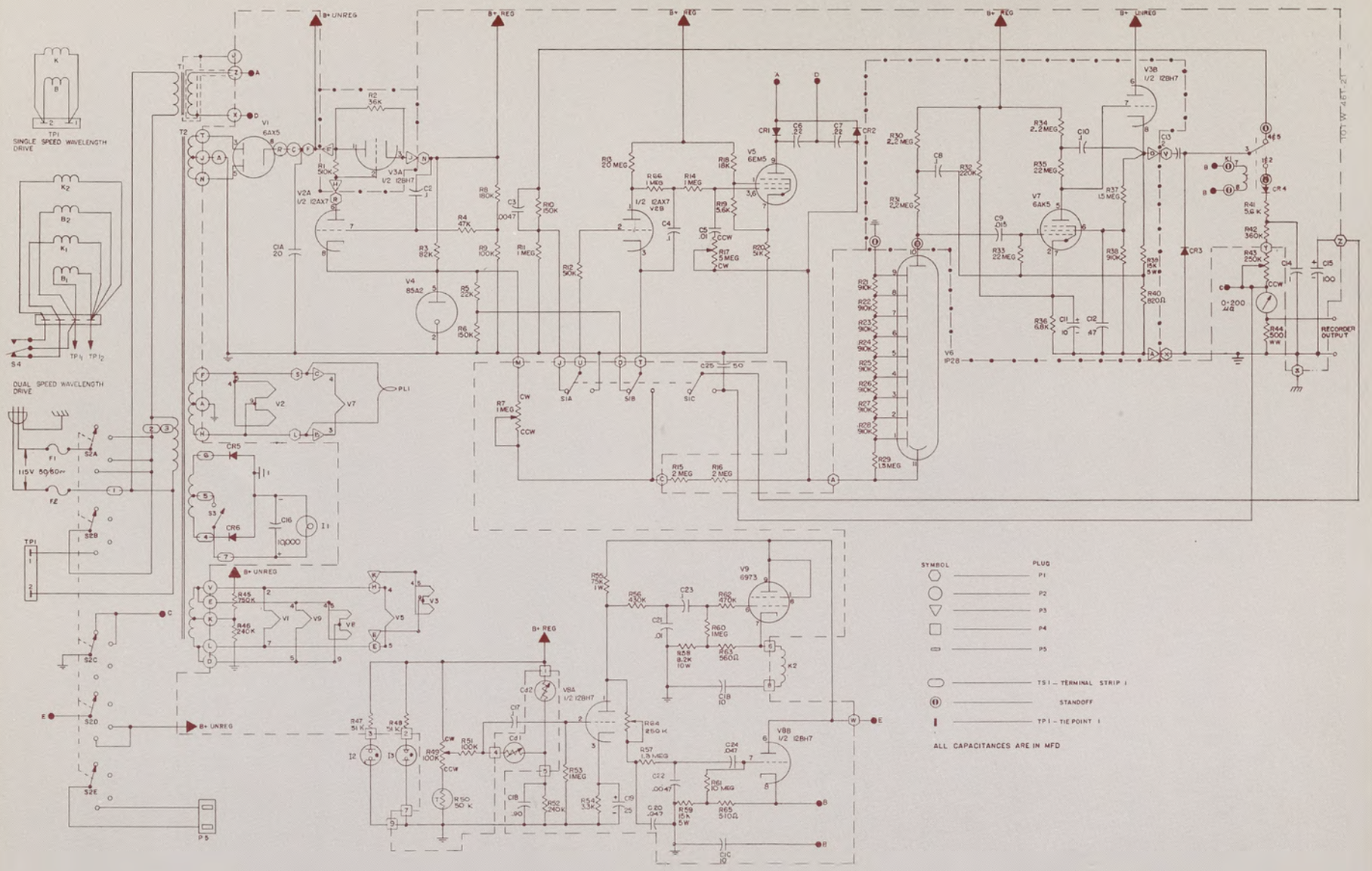


Figure 21. Model DB Circuit Diagram

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