Instrumental Changes for Increasing the Precision of the Beckman Spectrophotometer, Model DU

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An account is given of instrumental changes made in the Beckman Spectrophotometer, model DU, for increasing the precision of the instrument and for facilitating its use and maintenance. Among the changes may be mentioned the use of a mains operated power supply, an external housing for the C-batteries, a constant voltage transformer stabilizing the filament voltage of the hydrogen lamp, the use of more stable tungsten lamps, insertion of high quality switches and desiccant boxes with glass windows.

When judging the reliability of an instrument, its accuracy and precision are to be considered. The closer the reading of the instrument is to the correct value, the better is the accuracy and the smaller is the systematic error. If, however, several measurements of the same quantity are performed, the individual readings differ in magnitude. The smaller these deviations are, the better is the precision and the smaller the random error.

In spectrophotometry there are several ways of increasing the accuracy and precision by arranging the experimental factors so as to give the most favourable condition for measurement (choosing correct wavelength, suitable absorbance of reference and sample solutions, cleaning of cuvettes, purifying of solvents, etc.). The aim of this article is to show that some instrumental changes of the Beckman spectrophotometer itself may increase the precision of the instrument.

Measurements in the Beckman spectrophotometer are performed by adjusting the dark current potentiometer, the slit or the sensitivity control and the density scale potentiometer, interrupted by the turning of the selector and of the shutter switches. From the foregoing it is evident that during these operations all factors influencing previously made adjustments should remain unaltered. Let us call this the instrumental condition for precision.

The operation of an instrument always means a certain strain on the operator resulting in operator’s fatigue. This in turn results in decreased accuracy and precision of the measurement. The more the performance of the measurement
is facilitated, the less the operator's fatigue will be and the smaller the errors. Let us call this the psychological condition for precision.

The instrumental and psychological factors are often rather closely connected to each other. The separation of these factors made below should thus not be taken too literally.

INSTRUMENTAL CHANGES

The amplifier in the Beckman spectrophotometer, model DU, is a dc amplifier. A drawback of these amplifiers is their drift in amplification. Part of this drift can be eliminated by supplying the amplifier with constant voltages. An accumulator is used for the filament and bias voltages and built in dry cells for the phototube and anode voltages. In the visible range the tungsten lamp is also supplied from the accumulator, and as the drain of the lamp is considerable (5 A), the bias and filament voltages will gradually decrease thus requiring readjustment of the dark current and sensitivity controls. This can be avoided by providing separate accumulators for the instrument and the lamp.

Recently a voltage regulator, STAVOLT-A, has been developed specially for the Beckman DU*. It consists of three separate voltage regulators, one giving 2 volts dc for the filaments, one giving 6 volts dc for the bias and one giving 6 volts ac for the tungsten lamp. 115 volts unregulated ac is also supplied for the hydrogen lamp power supply. The use of separate circuits for the 2 and 6 volts dc supplies makes this regulator superior to previous commercial regulators for this purpose as the STAVOLT-A makes the settings of the sensitivity and dark current controls independent of each other, i.e. allows a "fine adjustment" of the slit width with the sensitivity control.

The 6 volts ac circuit of the STAVOLT-A may not be left unloaded when the regulator is in operation. To avoid burning the tungsten lamp when the hydrogen lamp is in use, a two-pole-two-position switch has been inserted in the STAVOLT-A. By means of this switch a resistance (three 5 ohms, 12 watt w.w. resistors in parallel) replaces the tungsten lamp when the hydrogen lamp is used. A 50 ohms resistor and a 0.05 μF capacitor in series have been connected in parallel with each pair of switch contacts to avoid spark generation resulting in oxidation.

The General Electric automobile lamps originally supplied for the Beckman instrument are not suited for ac. However, the Philips automobile lamp 6905-C works quite satisfactorily on ac and as it has the same socket as the General Electric bulb, they are directly interchangeable. The Philips lamp is not etched and may hence be used for the fluorescence adapter as well. As the Philips lamp gives a more intense light than the General Electric bulb, it may be used further down in UV, provided the visible stray light be filtered off.

The C-batteries of the Beckman instrument are located underneath the monochromator housing. In spite of the small drain, the batteries gradually become exhausted and the connections have to be altered. As this means turning the whole apparatus upside-down to and fro between each alteration

* Made by H. Struers Chemiske Laboratorium, Skindergade 38, Copenhagen, Denmark.

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and checking, it is a cumbersome operation. Cecchini and Eicher recently described the construction of an external C-battery supply, where the author has made some alterations. Two such external C-battery supplies with Pertrix 1.5 E6 batteries have now been in daily use for 22 months at our institute without changes of batteries.

Finally the hydrogen lamp regulated power supply has been provided with a 2.8 volts, 10 amps, constant voltage transformer controlling the filament current of the hydrogen lamp.

The electrical power sources thus being altered for the better still does not mean that the spectrophotometer will work satisfactorily. During measurements and between the different adjustments of the controls the selector and the shutter switches are switched to and fro. The zero position of the microamperemeter is often altered during these manipulations due to changed resistances between the switch contacts. An expedient is of course to substitute the switches by new ones.

Examination of the selector switch shows that it is a sixpole, four-place switch, whereas according to the circuit diagram a four-pole, four-place switch would suffice. The explanation is that four of the sections are paralleled, two by two, and these sections are only used as an on-off switch of the instrument. When turning the switch further from "Check" to "1" and "0.1" during measurements, the two paralleled pairs simply traverse short circuited contacts. Provided the contact resistances in these positions be changed during measurements, the instrument is supplied with altering voltages, i.e. different zero positions of the micro-amperemeter occur during switching.

The functions of switching the instrument on or off, and switching it between "Check", "1" and "0.1" were therefore separated. This was made by the inclusion of a standard two-pole, two-position on-off switch of good quality. It was mounted on the end of the monochromator housing just below the dark current control and the selector switch.

The original selector switch is of the Yaxley type. It has been substituted by an ELGE brush type instrument switch **. The switch used was a commercial one, a two-pole, seven-place, make-before-break switch.

A stop had to be soldered on the switch in order to change it to a three-place switch. All interconnections were then made on the switch and short connections soldered to the contacts by which the switch was to be connected to the instrument. After this the switch was washed first in carbon tetrachloride and then in grain alcohol. Thus all soldering paste, rosin and contaminations from handling the switch during soldering were removed from the contacts. Two contact strips, a four-pole for the on-off switch and a six-pole for the selector switch were mounted on the end of the monochromator housing below the on-off switch and the connections from the instrument and the switches were brought to these strips. In this way any contamination of the switch contacts with rosin during connecting it to the instrument could be avoided.

The new selector switch has a smaller angle between the steps than the old one. The old scale is thus not quite correct, but as the positions are brought

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* Raytheon, Model W 5855, made by Raytheon Manufacturing Co., Chicago, Ill., U.S.A.
** Made by ELGE, Vienna, Austria. A Panton Winkler Switch, Type A8/4P/3Rh/1B + K21 made by Panton Co., Ltd., Northampton, England may be even better.
down from four to three, this has not caused any trouble. On the other hand, by turning the switch one position too far to the left one cannot switch off the instrument by mistake, a blunder that previously led to a loss of at least 5 to 10 minutes in the measuring procedure.

A similar two-pole six-place ELGE brush switch with both sections paralleled has been inserted as a filament regulator switch in the hydrogen lamp power supply in order to obtain a more reliable operation. The resistance wire connected to the original switch was disconnected and transferred to the new one.

In spite of a thorough search the author has had no success in finding an instrument switch of the brush type small enough to use as a substitution for the original Yaxley type shutter switch. Discarded Beckman shutter switches were as a consequence rebuilt to brush type switches. The lever by which an arm of this switch moves the shutter to and fro is made of brass in its original design. As this lever was soon worn out, it was substituted by a replica in steel thus transferring the wear to the arm of the shutter switch, a detail that has to be exchanged at regular intervals.

**SOME MINOR CHANGES FACILITATING THE OPERATOR'S WORK**

The micro-amperemeter used as a zero instrument on the Beckman DU Spectrophotometer has a fine graduation. The reading is facilitated if a magnifying lens is placed over the scale. Another lens is tilted against it to concentrate the light from the window on the scale. This arrangement increases precision rather than accuracy. The parallax error of the system ought to be negligible.

As the first stage valve of the amplifier works as an electrometer valve it is important that no leakage currents occur in the amplifier. This is accomplished by keeping the air in the phototube housing dry with silica gel as drying agent in an desiccant box. Another desiccant box is placed in the monochromator housing. The condition of the desiccant may only be seen by opening the phototube or the monochromator housings. Desiccant boxes with glass windows for inspection of the condition of the silica gel have therefore been installed *. These boxes are suitably fastened to the housings from the outside for facilitating the change of silica gel.

For the insertion of these boxes, holes, 31—35 mm in diameter, have to be made in the housings, preferably with a radio chassis punch. Before the punching of the holes, the phototubes and amplifier valves have to be removed from the phototube housing, and the monochromator housing has to be removed as described in the instruction manual by changing the Helipot potentiometers. After punching all filings have to be carefully removed from the housings.

The disadvantage that this desiccant box has a smaller capacity than the original one in the monochromator housing is well counterbalanced by its advantages.

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This work was undertaken during a comparative study on diffuse reflectance using different optical geometries where a high precision of the values was needed.

REFERENCES


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