CHAPTER II

EXPERIMENTAL

1. INSTRUMENTS

Most of the measurements of both reflection and transmission were carried out by means of a Beckman DU quartz spectrophotometer\textsuperscript{210,211}. This well known instrument employs a prism monochromator and its light source, a tungsten filament lamp, works off a 6 V. battery supply which is maintained in constant charge by a battery power regulator\textsuperscript{212}. The charging current may be adjusted at will and generally the instrument is run on a steady battery voltage, the rate of charging being the same as that at which it is discharged. The optical system for transmission measurement is shown in Figure 1 (on page 49).

Two phototubes are provided to be used respectively up to and beyond 625 millimicrons, at which wavelength both have about the same sensitivity. The electronic circuitry involves electrometer tubes for amplification and a null method is employed to read off percentage transmission or optical density on two scales attached to the same variable resistor, which is calibrated in terms of both. The cells present an optical path length of 1 cm., and four of them could be placed in a sliding compartment; so that with one as a solvent standard of
Figure 1: OPTICAL SYSTEM OF BECKMAN MODEL DU SPECTROPHOTOMETER FOR MEASUREMENT OF TRANSMISSION.

Figure 2: OPTICAL SYSTEM OF BECKMAN MODEL DU SPECTROPHOTOMETER FOR MEASUREMENT OF DIFFUSE REFLECTION.
transmission, three could be used in a single set-up at any wavelength. Although the instrument monochromator is capable of providing all the wavelengths in the visible, in addition to extension into near ultraviolet or near infra red, those generally used in this work were 400 to 700 millimicrons, except in a few cases where the near infra red was also employed.

The optical system for "diffuse reflectance" in the Beckman instrument is shown in Figure 2 (on page 49). It may be noted that the system has normal incidence and 45° reflection in all azimuths focused on to the phototube. The transmission scale also works for reflection, and in both cases for values less than 0.1, the sensitivity could be increased ten times if desired.

For materials in the form of sheets, reflection measurements were relatively simple, and were carried out with the help of a suitable magnesium oxide (MgO) standard. In the initial part of the work a sub-standard vitrolite which had been calibrated in terms of a standard MgO surface at the National Bureau of Standards of U.S.A. (NBS) was used. This was later replaced by another sub-standard calibrated by means of a thick layer of freshly deposited MgO in the laboratory. This calibration sub-standard was tried several times with different layer thicknesses and the one with the most satisfactory surface was adopted for use. Reflection values of emulsions were taken
by placing plastic cuvettes, filled to the top, inside the sample chamber. All transmission values (for emulsions) were referred to water as the standard and all reflection values, to MgO.

Another instrument, a Reflection Meter, was also used which had been fabricated in this laboratory in 1953. Here too reflection from sheet materials could be directly measured, but for emulsions the instrument had to be placed front side down and set on a wooden mount made for this purpose. Plastic cuvettes were used in this case also. Light reflected at various angles was measured on a relative scale, the collimated incident beam falling at 45°. Its optical system is shown in Figure 3 (on page 52). This instrument was however used only at times, for a comparison of the reflection results against the corresponding Spectrophotometer values.

For the measurement of particle sizes of oil globules a Zeiss "Lancrometer" was used. It was calibrated with a stage micrometer and an occular scale, supplied with the instrument.

2. CALIBRATION OF THE BECKMAN SPECTROPHOTOMETER

The monochromator of the Beckman instrument was calibrated by means of the lines of known wavelengths in the arc spectrum of mercury, a mercury lamp with a special ultraviolet lamp power supply being used for the purpose. A comparison of standard wavelengths and scale readings is shown in Table I.
Figure 3: PLAN OF OPTICAL UNIT OF
ATIRA REFLECTION METER

A : Tungsten Lamp (25 W)
B : Lamp-housing
C : Circular slit
D : Collimating Lens
E : Tubular case
F : Light trap
G : Circular aperture
H : Housing
I : Phototube
J : Protractor
K : Cable connector
L : Hinged door
M : Shutter
TABLE I

CALIBRATION OF WAVELENGTH SCALE IN THE BECKMAN DU SPECTROPHOTOMETER

<table>
<thead>
<tr>
<th>Wavelength of the mercury line (Millimicrons)</th>
<th>Observed scale reading (Millimicrons)</th>
<th>Specified tolerance</th>
<th>Actual deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>579.0</td>
<td>578.4</td>
<td>0.5</td>
<td>0.6</td>
</tr>
<tr>
<td>577.0</td>
<td>577.6</td>
<td>0.6</td>
<td>0.6</td>
</tr>
<tr>
<td>546.1</td>
<td>546.0</td>
<td>0.2</td>
<td>0.1</td>
</tr>
<tr>
<td>491.6</td>
<td>491.6</td>
<td>0.5</td>
<td>0.0</td>
</tr>
<tr>
<td>435.83</td>
<td>435.9</td>
<td>0.4</td>
<td>0.07</td>
</tr>
<tr>
<td>407.8</td>
<td>407.6</td>
<td>0.4</td>
<td>0.2</td>
</tr>
<tr>
<td>404.7</td>
<td>404.6</td>
<td>0.4</td>
<td>0.1</td>
</tr>
<tr>
<td>355.0</td>
<td>355.1</td>
<td>0.2</td>
<td>0.1</td>
</tr>
</tbody>
</table>

It would be seen that the actual deviations fall within the tolerance limit indicated by the manufacturers.

The photometric scale was calibrated by means of a standard cobalt glass the transmission values of which, at different wavelengths throughout the visible region, had been known from the NBS.* The observed and the known transmission values are shown in Figure 4 (on page 54).

3. MATERIALS

(a) Plain and Dyed Fabrics and Filter Papers

Plain woven cotton poplin was dyed with various direct colours like Duranol Yellow, Sky blue FF etc. For black dyeings

Figure 4: TRANSMISSION OF STANDARD COBALT GLASS IN THE VISIBLE REGION

WAVELENGTH IN MILLIMICRONS

TRANSMISSION

Continuous Line: NBS Results
Points: ATIRA Results
Cibanon black was used with various concentrations of the
dye to get a range of grey shades. Similar grey specimens
from Whatman No. 1 filter paper were also prepared. Besides
cotton, other fabrics, namely those made from silk, wool,
viscose rayon and nylon were also used.

(b) Grey and Coloured Cements

'Snowcem' powders supplied by the Associated Cement
Company, in all available nine colours, were used to make the
cement blocks. The colours were (a) White, (b) Pale Grey,
(c) Dark Grey, (d) Pale Blue, (e) Pink, (f) Apple Green,
(g) Pale Green, (h) Midcream, and (i) Yellow. Generally
35 gms. of powder were mixed with 14 ml. of distilled water;
but blocks were also made with a mixture of 30 gms. powder
and 20 ml. water.

(c) Wood, Asbestos, Hardboard etc.

Eleven different varieties of wood or hardboard and an
asbestos sheet were grouped together for a series of measurements.
These together with their respective relative density values
were: (i) Dealwood : 0.474, (ii) Sesame : 0.722, (iii) Plywood :
0.726, (iv) Brown Dealwood : 0.482, (v) Bulsar Teak : 0.649,
(vi) Jackwood : 0.731, (vii) Nashbar Teak : 0.523, (viii) Asbestos
Sheet : 1.659, (ix) Sitapur Plywood : 0.644, (x) Masonite : 1.058,
(xi) Hardboard: 0.962, and (xii) Bulsar (white) Teak: 0.553.

(d) Wetting Liquids

Water, Soap solution, Benzene, Paraffin, Aniline and Carbon bisulphide were used as wetting liquids in the case of most of the varieties of fabrics examined while for filter paper, most cellulose powder, cement blocks and wooden surfaces observations were carried out only with water as the wetting liquid.

(e) Colloid Dispersions

Oil emulsions with a range of concentrations were prepared by adding distilled water in requisite amounts to various commercial preparations containing mineral oils or some other organic liquids generally in presence of a suitable dispersing agent. They are widely used in textile industry as spinning aids. Some of these used in the present work were: (i) "Methyl Bornoxol Oil", (ii) "Fibricon 51", (iii) "Cirrasol SF 200", and (iv) "Strengthospin JR". In addition, emulsions of a disinfectant fluid "phenyl" and milk were also used. Besides these liquid/liquid systems, one solid/liquid system in the form of a commercial fifteen per cent silica suspension in water, "Syton W-20", also used as a spinning aid, was studied with all the others, for its light transmission and reflection characteristics at various concentrations and wavelengths. The particle size in all these cases was measured on the Zeiss Lennameter as mentioned before.