CHAPTER V.

EXPERIMENTAL II- INTENSITY MEASUREMENTS AND RESULTS.

1

Introductory

The principal methods generally used for intensity measurements are (i) the Photographic method and (ii) the Photoelectric method. For a full treatment of these and other methods of comparing intensities reference may be made to Harrison, Lord and Loofbourow (1955). Both the methods have their merits and limitations. Photographic method is definitely superior because of the advantage of recording the spectra and maintaining it permanently. With the spectra recorded photographically, it is very convenient to measure wave-lengths of lines and bands. But the response of a photographic plate to light intensities being not linear, the plate requires calibration and this makes the method a laborious one. It has the added inconvenience of having to adhere strictly to uniform developing and fixing. Especially in the case of spectra to be recorded from flame radiations, another source of error is the background intensity of the continuum, ever present, because for some exposures the background might have intensity below the threshold resulting in its non-recording and consequential nonaccounting. Further a selected plate
may not be useful to compare intensities of bands in far off different wavelength regions, as no single plate with uniform intensity response over all the wavelengths is generally available.

All these factors will increase the labour and possibly the error, so that it becomes difficult to achieve accuracy much greater than 10 per cent with all the necessary precautions.

On the other hand photoelectric method though poor in recording the spectra and preserving it, it has a unique advantage over photographic method as far as intensity measurements are concerned. It responds linearly to intensity at a single wavelength. Besides this the photoelectric arrangement can be made highly sensitive, limited only by the noise level of the electronic equipment involved, to pick up even the weakest continuum. So no large time spans need to be spent to obtain a record of intensity. Furthermore the photoelectric cell may be used over all wavelength regions except the infrared, only by changing the sensitivity of the whole set-up to compensate for the changes in sensitivity of the photoelectric tube. However, a great disadvantage of the photoelectric method is that the source to be recorded (flame) must remain uniform and steady over the entire period of scanning,
otherwise spurious results would be obtained. In this score the photographic method is preferable to photoelectric one because it averages over time the variations in intensity and will avoid erratic results.

Lewis R.K. (1937) discusses the difficulties of using phototube for the measurement of intensity. Some of the common prerequisites are that the sensitivity of the tube and the D.C. amplification should remain constant and the leakage current should be negligible. The modern high vacuum phototube with drying tower on top of the glass enclosure will assure constant sensitivity and negligible leakage current. The D.C. amplification generally has the tendency of drifting the null point and this could be rectified by introducing in the recording unit a standardising device which sets the null point.

Even then errors might still arise on account of

(i) the width of the exit slit which sets a fixed width for intensity measurement and in a way limits the full utilisation of the resolving power of the spectrograph,
(ii) the finite scanning speed in relation to circuit constants and (iii) the circuit noise (Schubert 1958). The errors introduced on account of these factors are, however, expected, quite justifiably, to be relatively much lower in comparison to those due to uncertain factors in the method of photographic photometry.
In the method of photoelectric recording, to devise an arrangement for scanning the spectrum when the focal plane of the spectrograph is curved is not generally easy. It is a technical handicap but efforts are being made to overcome it for the method to be adopted for its outweighing merits.

As a result, the photoelectric method has been greatly used in recent years in particular by Mcnolls and his group (1956) for the intensity determinations of many band systems. Phillips (1954, 1957) has used the photoelectric method for his intensity profiles and it appears that the method has been established as of better accuracy in such cases where it could be applied.

In the case of present experiments the flame source was possible to be maintained in a steady state for hours together and hence it was thought worthwhile to employ both the methods to obtain intensities so that the results could be verified between themselves and used as checks upon each other. The methods are described and discussed in the following sections one by one.

2

Photographic photometry.

In this method quantitative measure of light intensities is dependent upon the relations which the blacken-
ing density or optical density bears with intensities of light. These relations are briefly reviewed here.

(i) **Optical density and intensity**: Optical density is a function of intensity of light $I$, causing the blackening, the time of exposure, $t$, the wavelength of light falling on the plate, $\lambda$, kind and age of the plate, temperature and development. The last three factors can be kept constant in any experiment with great care. Thus the optical density $S$ is expressed as $S=f(I, t, \lambda)$.

The relation between $S$, $I$, and $t$ is extremely complicated and is ultimately connected with photochemical processes and cannot be adequately fitted in the exact mathematical expression.

(ii) **Intensity and Spectral Sensitivity**: The photographic emulsion has widely varying spectral sensitivity and has to be taken into account while measuring densities at different wavelengths. Various devices are used to produce calibrated blackening on a photographic plate by varying the light intensity in known amounts.

Some of the common arrangements are, the use of a step-slit, a step filter or a long-step-sector in front of the slit of the spectrograph. In this investigation long-step-sector has been used as it is convenient and
accurate also. It has been the subject of much controversy because the average intensity of light falling on the plate is varied, rather than the actual intensity. The investigations of Twyman (1931-32), of O'Brien (1929 and 1931) and especially of Webb (1933) have justified its use under controlled circumstances. Webb's results indicate that an intermittent exposure is equivalent photographically to a continuous exposure when the rate of flash is so great that each grain of the emulsion receives on the average not more than one quantum of light per flash. So there exists a critical frequency for a given type of plate. For ordinary conditions the critical frequency is of the order of 10 flashes per second but it is preferable that the rotating sector should be run at a speed greater than 1200 R.P.M.

Errors to be guarded against are slow rotation of the disc and bad collimation both of which can be conveniently avoided. In the present investigation they are avoided by rotating the disc at a speed of 1400 R.P.M., where the light is chopped twice per rotation and also by proper collimation. Step-sector has 5 steps with widths in multiple of two. Intensities corresponding to these steps will be taken proportional to the width of the steps.
The optical densities are calculated from the microphotometer trace obtained by running across any selected wavelength, using the formula,

\[ S = \log \frac{I_0}{I} \]

where, \( I_0 \) is the distance from the absolute darkness limit to the mean background limit and \( I \) is the distance between absolute darkness and peak of the trace.

The densities thus obtained, in the case of a log-step-sector, correspond to intensities which are proportional to the widths of the steps of the sector i.e., 1, 2, 4, 8 and 16. From these a graph of optical densities as a function of logarithm of intensities is prepared. (Fig. 6).

(iii) Comparison with a standard lamp: All the intensities measured as indicated above are not in terms of a particular standard. They merely express results which have relative values in terms of blackening densities in the calibration marks. In order to fix a standard unit for intensities for different wavelengths, source of continuum radiation in the wavelength region of interest is needed. The calibrated blackening produced on a plate by this source is dependent upon the sensitivity of the plate, dispersion of the spectrograph and the energy distribution
of the lamp. Plate sensitivity will be eliminated indirectly if calibration curve is prepared for each different wavelength. The energy distribution of the lamp and the dispersion of the spectrograph should, however, be known beforehand.

In the present work a substandard lamp whose spectral energy distribution (\(E_\lambda - \lambda\) curve) is known, is used.

Hilger small quartz spectrograph whose dispersion was determined earlier has been used in this investigation. How to express intensities in terms of given standard, the intensity scales of the characteristic curves will be multiplied by (i) the energy density \(E_\lambda\) and (ii) dispersion at the corresponding wavelengths. The first factor is obvious. The second multiplying factor becomes necessary because the energy densities are on the basis of uniform dispersion.

The energy distribution curve and the calibration curve are presented graphically in Figs. 5 and 6, respectively.

(iv) **Method as applicable to present problems:** The photometric method outlined above, when applied to band, presents certain special difficulties. Peak intensities are
not exact indices of the true relative intensities of the vibrational bands in the electronic transition. Differences exist in the quantum number of rotational line at which the peak is occurring and also in the concentration of rotational lines at the peak. Johnson and Tawde (1932) and many earlier workers have taken the peak value to be the real band intensity on the assumption that moment of inertia of a molecule varies but little with vibration quantum number. Recently King (1948) has taken peak values for C₂(Swan) system on the ground that the structure is similar in all the bands. Further in a later publication Floyd and King (1955) have corrected the peak values for change in quantum number of rotational line forming the head and the change in the density of lines at the same point. The corrected values are not much different from the uncorrected ones if values of intensities are to be taken on a relative scale.

However, if the measured intensities are to be used for determining transition probabilities, it is imperative to use the integrated values of the band intensity. But in the present experiment the aim being not to calculate transition probabilities, but to see how the intensity of different radicals observed in the flame change their intensity with air/fuel ratio, it is fairly satis-
factory to use peak intensity for representing the relative intensities of the bands. From the experimental point also, this has to be preferred in respect of the methyl alcohol flames. This is sufficiently amplified in the following paras.

$C_2$ (Swan) is obtained in these flames in traces. Being very weak, it required very long exposures to be recorded, in a qualitatively measurable condition. So it was considered not quite feasible to obtain accurately, integrated intensities of $C_2$ on account of the errors inherent in very long exposure. In addition such long exposures bar the possibility of precise correlation with the other band systems. So only peaks were measured in the case of $C_2$ and were taken to represent intensity of $C_2$.

The CH $\left(4315 \text{ Å} \right)$, is heavily masked by the CO-continuum and the diffuse cool flame bands. In addition CH has got open rotational structure even on small dispersion instruments. Both the factors affect adversely any effort to obtain reliable integrated intensity values for CH also. So again the CH was measured only at its peak after properly accounting for the background. The band CH $\left(3900 \right)$ was too weak to yield any useful measurements.

The OH $\left(2\Sigma^+ - 2\Pi \right)$ being a band of open rotational
structure, is possible to be subjected to process of integration by measuring the intensity of each line and then adding. But this could not be done in the present case as the radiations from this flame are too weak, to be studied with very high dispersion spectrograph. Under the circumstances in order to make a systematic and thorough study of the problem in hand it was found adequate to take peak intensities to represent the intensities of the bands.

\( C_2 \text{(Swan)} \) and \( \text{GH-} (4315-4318) \) are very weak in comparison to OH. The former two being covered by the diffuse bands, it was not possible to satisfactorily correlate them with OH in intensities. Hence their intensities stand independently. But this does not reduce the utility of the data because the variation of intensity of a radical with air/fuel ratio is more significant than its comparison with other radicals. It may also be mentioned that this is of the nature of a handicap in photographic photometry because for correlation we need a band or a line whose intensity is intermediate to the two intensities to be correlated. The spectrum does not contain such a band or a line. So if photographic photometry is to be used then one has to introduce some material, which can emit lines or bands of required intensity, in to the flame. This is troublesome and also changes the condition
in the flame. However, the intensities will be correlated by the photoelectric method to be described in the next section.

(v) Cool-flame bands and CO-flame bands: The bands were roughly located at 3700 Å, 3847 Å, 3952 Å, 4044 Å, 4121 Å, 4230 Å, 4337 Å, 4434 Å and 4566 Å and these were identified as belonging to cool-flame spectrum by taking the photograph on a contrast plate and processing it with contrast developer. Owing to diffuse characters of these bands it was difficult to make precise wavelength measurements. However, from the rough measurements and their comparison with Brand's (1951 and 1956) values of prominent heads and with the available photographs of cool-flame spectrum the presence of the bands in this flame was confirmed.

A direct comparison with the inner-cone spectrum of ethyl alcohol-air flame revealed the absence of HCO bands. Flames with all air/fuel ratios were tried. In none of them the HCO could be traced with any certainty.

CO-flame spectrum is observed as a continuum extending from below 3300 Å to beyond 5000 Å with a large number of narrow headless bands superposed on it. Here also it was not possible to make precise measurements of wavelength for identification, but direct comparison
with available spectrum pictures leaves no doubt about the emitter.

The region 3300 Å to 5000 Å is overcrowded by the presence of CO-flame bands, cool-flame bands and CH (4315 Å and 3900 Å). Hence it was extremely difficult to make any quantitative investigation of the variation of cool-flame spectrum or CO-flame spectrum independent of each other. So a general qualitative description of the variation of intensities of these systems is attempted.

At low air/fuel ratios cool-flame bands predominate over the CO-flame bands. The continuum is very weak. When the content of air is increased, the strength of both goes on increasing; the continuum gradually gaining in intensity. Roughly at air/fuel ratio 5.5, cool-flame bands appear to be at their maximum and the band system is very well defined. This is better revealed in the photometric record of this region under high amplification.

As air is increased further the cool-flame bands decrease in intensity, whereas the continuum continues to gain in intensity and at air/fuel ratio 6.4 nearly all the radiation in this region is CO-flame bands with very weak CH (4315 Å) and almost no trace of cool-flame bands.
At this stage the CO-flame bands, especially the narrow headless bands, are very well developed and it seems that the strength of CO-flame bands has increased and cool-flame bands even if they are present are not easily detectable. It is also noticed that at this stage the strength of continuum has slightly decreased.

(vi) Results. All the results on intensity measurements of various bands are collected in table I and represented graphically in Fig. 7. Photograph of the spectrum at various air/fuel ratios is attached on the next page (Fig. 6a).

Table I.

*Intensities of bands

<table>
<thead>
<tr>
<th>Air fuel</th>
<th>C2</th>
<th>OH</th>
<th>OH</th>
<th>OH</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.40</td>
<td>1.8</td>
<td>2.8</td>
<td>5.2</td>
<td>1.2</td>
</tr>
<tr>
<td>4.80</td>
<td>2.0</td>
<td>3.3</td>
<td>8.1</td>
<td>2.4</td>
</tr>
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<td>5.20</td>
<td>2.0</td>
<td>3.7</td>
<td>11.0</td>
<td>3.2</td>
</tr>
<tr>
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<td>1.7</td>
<td>4.0</td>
<td>13.6</td>
<td>4.3</td>
</tr>
<tr>
<td>5.87</td>
<td>0.9</td>
<td>4.4</td>
<td>16.3</td>
<td>5.0</td>
</tr>
<tr>
<td>6.17</td>
<td>-</td>
<td>4.1</td>
<td>17.5</td>
<td>5.3</td>
</tr>
<tr>
<td>6.48</td>
<td>-</td>
<td>3.6</td>
<td>16.4</td>
<td>5.2</td>
</tr>
<tr>
<td>6.76</td>
<td>-</td>
<td>2.9*</td>
<td>14.9*</td>
<td>4.6*</td>
</tr>
</tbody>
</table>

* Extrapolated values.

** The intensities are not on the same scale.
3

**Photoelectric Method.**

(i) **Equipment**: Hiller (B.49&) Medium Quartz Spectrograph was used for the study of various band systems emitted by the flames. A quartz spherical lens (F 958) was used to focus the flame on to the slit of the spectrograph. For inner cones a slightly magnified image was taken with the centre of the image on the centre of the slit. For the study of outer cones the portion of the flame about 1.5 cms. from the grid was chosen.

For intensity measurements, Hilger (F 730) scanning unit was used with (R.C.A.) 1 P 28 photomultiplier tube in conjunction with Hilger F.A.7 power unit and a Brown Recorder. Scanning unit contains the travelling photomultiplier and slit assembly with appropriate motor drive and manual controls for setting the position of slit and operates in conjunction with Hilger F.A.7 power supply and Recorder. In principle the spectrum is scanned by a slit moving exactly in the focal plane of the spectrograph and the light passing through the slit is reflected from a front piece mirror on to the cathode of the photomultiplier. The signal currents from the Photomultiplier responding to the incident radiation are fed to the d.c. amplifier which in turn deflects the pen of the Recorder.
The slit and the photomultiplier assembly are traversed at a pre-scaled constant speed by a synchronous motor-drive and the recorder chart is also rotated by a synchronous motor actuated by the same main supply. The recorded trace is, therefore, virtually a record of the signal response of the photomultiplier corresponding to the position of the scanning slit over the spectrum.

In order to take account of the variation with wavelength of the angle of the light emerging from the spectrograph, the whole multiplier slit assembly is rotated slightly (about a total of 6°) during traverse by means of a cam mechanism. Rotation takes place about the slit axis in the focal plane so that the correct optical conditions are maintained. This helps further to minimise the selective effect (Lewis, R.K., 1937).

The F.A. 7 power unit supplies the necessary voltages both for photomultiplier and recorder besides amplifying the signal from the photomultiplier and feeding it to the recorder. The recorder used is Honeywell Brown type 153 electronic Recorder. It incorporates a continuous balance system in potentiometer circuit. The whole system works automatically once the proper conditions are set to obtain the desired results.
The whole unit, comprising of spectrograph, scanning unit, power unit and recorder, though meant for absorption work can be used with equal facility for emission measurements.

Before proceeding to the measurement of intensities proper, it was necessary to calibrate (i) the position of the phototube for wavelengths and (ii) phototube for intensity and wavelength response. The methods of calibrations are described below:

(ii) Calibration for wavelengths: The position of the photomultiplier can be read by the numbers on the drum. These numbers were to be correlated with wavelength so that the peaks due to the respective lines or bands could be identified. For this purpose a record of mercury-cadmium arc lamp was taken. The peaks were then marked with corresponding numbers on the drum and compared with the spectrum photographed and identified earlier. The number of lines therein being few and far between, an auxiliary spectra of copper arc was scanned and compared in a similar way with the Hg-Cd spectrum. The identifications were made taking mercury-cadmium lines as guides and two graphs prepared as follows:

(a) Wavelength is plotted against corresponding num-
ber on the drum vide Fig.9. This represents calibration curve in terms of scanning motor meter.

(b) In the record some suitable point was selected as reference point and graph of wavelength against distance of the peak from the reference point was plotted. This represents calibration curve in terms of wavelengths and distance from the point for the selected scanning and chart speeds. This graph is shown in Fig.8.

The second graph is used to construct the dispersion curve for the instruments i.e. \( \frac{dl}{d\lambda} \propto \lambda \). This is useful in correcting for dispersion while measuring the response of photo tube.

(iii) Calibration for intensity: Photomultiplier tube was positioned at the required wavelength and the spectrograph was collimated to receive radiation from a standard lamp whose spectral energy distribution is known (Fig.5). A rotating sector was interposed between the slit and source. The sector with a certain width was rotated with 1400 revolutions/minute. Since the sector was open at diametrically opposite ends the light was chopped at the rate of 2800/minute. With this speed the photomultiplier gave a signal as if with a fixed intensity. So for the purpose of intensity calibration the different widths of
the rotating sector are taken to represent relative intensities of light passing through them and falling on the photomultiplier tube, as the light intensity can be taken to be proportional to the widths. The output of the tube recorded with different sector widths were plotted against the corresponding intensities. The resulting graph was found to be a straight line.

A straight line graph is a great advantage because intensities of both weak and strong lines can be measured with the same accuracy. These curves have been used to obtain the intensity of OH, CH and O2 relative to each other as well as with respect to the air/fuel ratios.

(iv) Wavelength response of the tube and correlation of calibration curves at different $\lambda$: To determine the wavelength response of the tube the entire spectral region of the lamp radiation was scanned. Then the record was compared with energy distribution curve for the lamp at regular wavelength intervals. Then at each such wavelength, deflection ($D$), for 10 units of intensity was determined and a graph of $D$ against corresponding $\lambda$ was plotted. The dispersion correction was then applied using dispersion curve of Fig. 10.

The final graph so obtained is given in Fig. 12.
It represents the response for light intensity of a fixed band-width of the spectrum entering the photomultiplier slit. From this curve two intensities at different wavelengths could be correlated by the rule of 3. This procedure introduces a slight error as the calibration curve does not pass exactly through origin and hence the deflection in this region are not precisely in direct proportionality. To avoid this a straight forward procedure was adopted. The intensity calibration curves at the wavelengths of C₂, CH and OH are obtained in terms of the intensity of the standard lamp at the corresponding wavelengths. Then the intensities measured with the help of such curves stand on the same scale (Fig.11).

(v) Derivation of intensities: The entire spectrum of the flame (inner cones) at one ratio was scanned repeatedly to see whether the record was consistent and the flame reliably steady. The records obtained agreed within 1 per cent indicating a high degree of stability for the flame and reproducibility of results. The next problem was to correlate the intensities of C₂, CH and OH. With slit widths adjusted for large deflection to get C₂ or CH, the recorded OH was very small and a small error in its measurement would result in high percentage error. So an
alternative procedure was used. The slit width of the photomultiplier was kept adequate enough to get the CH bands as high peaks. This slit width was adjusted to another suitable value at C₂ and CH, all the while taking care to avoid back-lash error of the screw head of the photomultiplier slit assembly. Necessary corrections were applied for differing widths of the slits. This way the intensities of CH, C₂, CH and of the background (at 4110 A°) were measured at all the air/fuel ratios. The results of intensity measurements are entered in Table II and are presented graphically in Fig.13.

Table II.

Intensities of bands.
Photo-electric method.

<table>
<thead>
<tr>
<th>Air/fuel</th>
<th>G₂</th>
<th>CH</th>
<th>CH</th>
<th>*B.G.</th>
<th>OH (Outer cone)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.40</td>
<td>3.6</td>
<td>3.8</td>
<td>13.4</td>
<td>5.2</td>
<td>4.4</td>
</tr>
<tr>
<td>4.80</td>
<td>4.0</td>
<td>7.4</td>
<td>20.6</td>
<td>9.5</td>
<td>4.8</td>
</tr>
<tr>
<td>5.20</td>
<td>2.3</td>
<td>7.2</td>
<td>28.0</td>
<td>13.3</td>
<td>5.9</td>
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<td>0.6</td>
<td>6.2</td>
<td>33.2</td>
<td>16.0</td>
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</tr>
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<td>-</td>
<td>5.5</td>
<td>39.8</td>
<td>17.8</td>
<td>10.2</td>
</tr>
<tr>
<td>6.17</td>
<td>-</td>
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<td>44.6</td>
<td>17.4</td>
<td>11.8</td>
</tr>
<tr>
<td>6.48</td>
<td>-</td>
<td>4.0</td>
<td>36.4</td>
<td>15.0</td>
<td>10.6</td>
</tr>
</tbody>
</table>

* background
The OH was partly resolved showing fine structure of the bands. This would be rather inconvenient for the measurement of intensity. So a faster scanning speed which will smooth over all the resolved structures was used. With the selected speed only two peaks, one corresponding to $R_2$ and the other to $R_1$, were obtained. The $R_1$ peak was taken to represent the intensity of OH. However, considering the statement of Gaydon (1957), that the peak intensity might be in serious error if the rotational temperatures of the bands are different, it was thought worthwhile to measure also integrated intensity of the band.

The area under the entire OH band profile on the record is taken to represent the integrated intensity. By smooth extrapolation of the band profiles, overlapping due to other bands has been eliminated. However, a small error may persist on account of some of the lines of $(1,1)$ band getting mixed with the $(0,0)$ band. But these are few and also weak and hence their contribution is almost negligible. Thus it is hoped that the area under the curve, with the overlapping eliminated, will represent reasonably, the integrated intensity of OH. These values are expressed in arbitrary units and entered in table III. The peak values are also entered side by side. Their variation with air/fuel ratios is shown graphically in Fig.13. It
may be noted that the nature of variation of integrated intensity of OH remains the same as that for peak values. To consider this point more carefully, the ratios of peak intensity to integrated intensity is calculated in each case and entered in Table III (represented as a function of air/fuel ratios in Fig.17).

**Table III.**

**Intensities of OH—(Peak and Integrated).**

<table>
<thead>
<tr>
<th>Air/fuel</th>
<th>Peak ( R_1 )</th>
<th>Area under the profile ( A )</th>
<th>( \frac{R_1}{A} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.40</td>
<td>13.4</td>
<td>9.0</td>
<td>1.49</td>
</tr>
<tr>
<td>4.80</td>
<td>20.6</td>
<td>16.8</td>
<td>1.23</td>
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<td>6.48</td>
<td>36.4</td>
<td>25.0</td>
<td>1.46</td>
</tr>
</tbody>
</table>

* The values are in arbitrary units.

A similar measurement of intensities for OH could also have been undertaken, but for the fact that the area
under the curve (for CH) obtained may not be quite representative of integrated intensity for CH. The reasons are: (i) CH has more open rotational structure than that of OH and (ii) CH is very heavily masked by diffuse bands and continuum, whose overall intensity distribution changes with air/fuel ratio. The two reasons make it impossible to obtain purely the contribution of rotational lines that constitute the band, a good number of them being always in the continuum. If the contribution from the background strengthens then the area under the curve will not be true representative of real integrated intensity.

(vi) Results: The results of intensity measurements by photoelectric method are collected in tables II and III against corresponding air/fuel ratios. Graphical representation of the same will be found in Figs. 13 and 17.
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