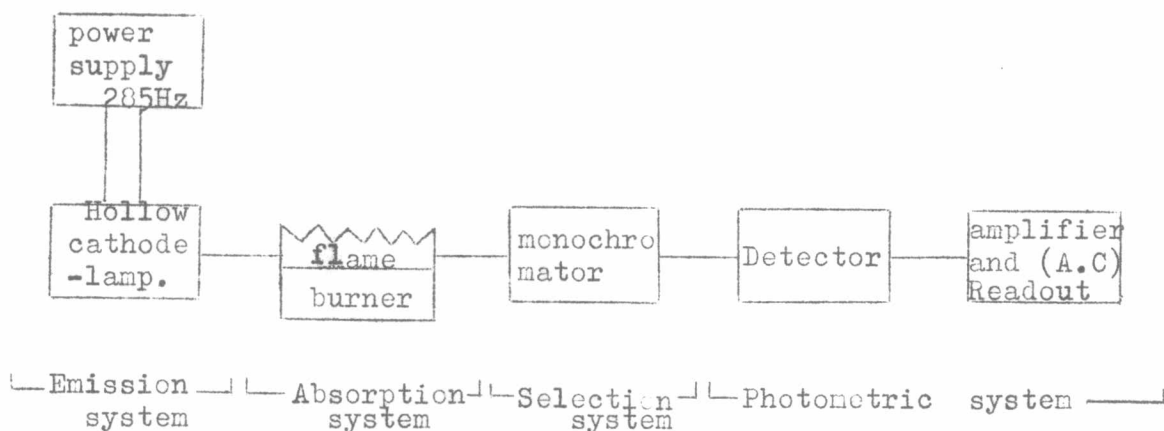


CHAPTER III

EXPERIMENTAL



Throughout the work a Varian Techtron A.A.5 atomic absorption spectrophotometer with a meter read out was used and its function can be simply represented by the following block diagram.



Emission system.

Varian Techtron hollow cathode lamps were used. These lamps are of standard design. For sodium and potassium a single lamp with multiple element cathode was used. A discussion for the theory of the hollow-cathode lamp design has been excellently discussed by Tolansky(5).

Such sources produce very narrow spectral emission lines, suitable for atomic absorption measurements.

Absorption system

The absorption system consists of three parts.

1. The Flame. The requirement is that it should give sufficient energy to promote efficient dissociation to provide atoms of the element of interest.

2. The Burner. It's main function is to introduce the sample solution into the flame. Further reading about the burner can be obtain from Christian and Feldman (16). In this work, a 4" x 0.020" slot burner designed for an air acetylene flame system and producing a flame of 10 cm. in length was used. An air acetylene flame was used. The air was supplied by a compressor while bottled acetylene purchased locally was used.

3. The Nebulizer. The most widely used nebulization process is pneumatic nebulization (16). Once the solution flows through the capillary into the nebulizer, the liquid droplets experience a sharp velocity change and the resulting effect is that some of them are broken into fine drops which are carried into the flame in the air stream. Larger droplets fall out and condensed from the flow system. A liquid trap remove excess solution

from the system. The Varian Techtron variable nebulizer used is of such a design. A constant setting for the nebulizer was maintained throughout.

Selection system

A monochromator is used to isolate the resonance line from the radiation from the hollow - cathode lamp. It should be of sufficient resolving power to isolate the resonance line from neighbouring non-absorbing spectral lines. In this work a grating monochromator of resolving power $3.3 \text{ \AA}^{\circ} / \text{m.m.}$ with dual variable central slit was used.

Photometric system

This is the part where the resonance radiation is detected and the corresponding impulses chopped at 285 Hz are amplified and measured. In atomic absorption photomultipliers have been used predominantly as detectors. The A.C. Signals from the detector are converted to a visual readout which appear as a deflection of a needle in a meter. The HTV (Hamamatsu TV Co. Ltd.) photomultiplier type R 213 was used. D.C. signals from the detector, arising from emission in the flame are filtered out by the amplifier.

Operating conditions and instrument settings

The optimum conditions listed in Table IV were established with solutions of pure chloride salts of sodium, potassium and lithium.

Table IV

Optimum conditions for the study of lithium, sodium and potassium.

Element	Light source	Lamp current		lines slit width	Height
		mA	$^{\circ}$ A	mm.	mm.
Li	Li-lamp	5	6707	0.05	5
Na	Na-K lamp	5	5889	0.025	5
K	Na-K lamp	5	7665	0.05	5

The lamp current was set at the lowest possible value in order to conserve the life of the lamp. This also produces a slightly narrower emission line. Absorbance was measured at various slit widths and value chosen was that giving the highest absorbance as seen in Fig.2-4. The resonance lines of the alkali metals are

doublets of high intensity. A narrow slit width gave the best value of absorbance as seen in Fig.2. A single line, usually the more intense from each doublet was used with the exception of lithium where the doublet is not resolvable.

The other variables in the instrument which were investigated, were burner height and nebulizer setting. Absorbance was measured over a range of height above the burner surface and the height giving a maximum absorbance value was determined (see Fig.2-Fig.4). The nebulizer was set at the minimum value in order to supply the finest possible droplets to the flame. This setting was used throughout.

In this study two flames were used as shown in

Table V

Flame compositions

Fuel	Oxidant	Pressure of oxidant (psi)	Acetylene flow rate l/m	air flow rate l/m	
Flame I	C ₂ H ₂	air	15	1.5	11.5
Flame II	C ₂ H ₂	air	15	0.8	11.5

The stoichiometric ratio of C₂H₂: O₂ = 2 : 5

In both flames the flow rates of fuel and air were measured as using the rotameters provided with

the A.A.unit. The flow rates are converted to litres per minute by using standard flow meter calibration curve provided with the instrument. The fuel to oxygen ratios were computed (Appendix I).

The physical appearance of flame I is very luminous due to the presence of carbon particle in the flame, this characterizes a fuel rich flame. The fuel lean flame, flame II had a hazy blue flame, characteristic of a more oxygen rich mixture. However in both flames there is an unknown amount of entrained air coming in from the sides of the burner. This entrained air will result in an undetermined quantity of oxygen entering the flame which will cause a lowering of the calculated fuel oxidant ratio in both cases.

The instrument was set to read percent transmittance on the amplifier and readings were converted to absorbances value using conversion tables. For each solution an average of four readings was taken because of the small random variation in percent transmittance with time.

To quantify the reproducibility of the reading, for a series of eleven standards and a standard deviation of NaCl was calculated (Appendix II).



Chemicals and their preparation

Chemicals

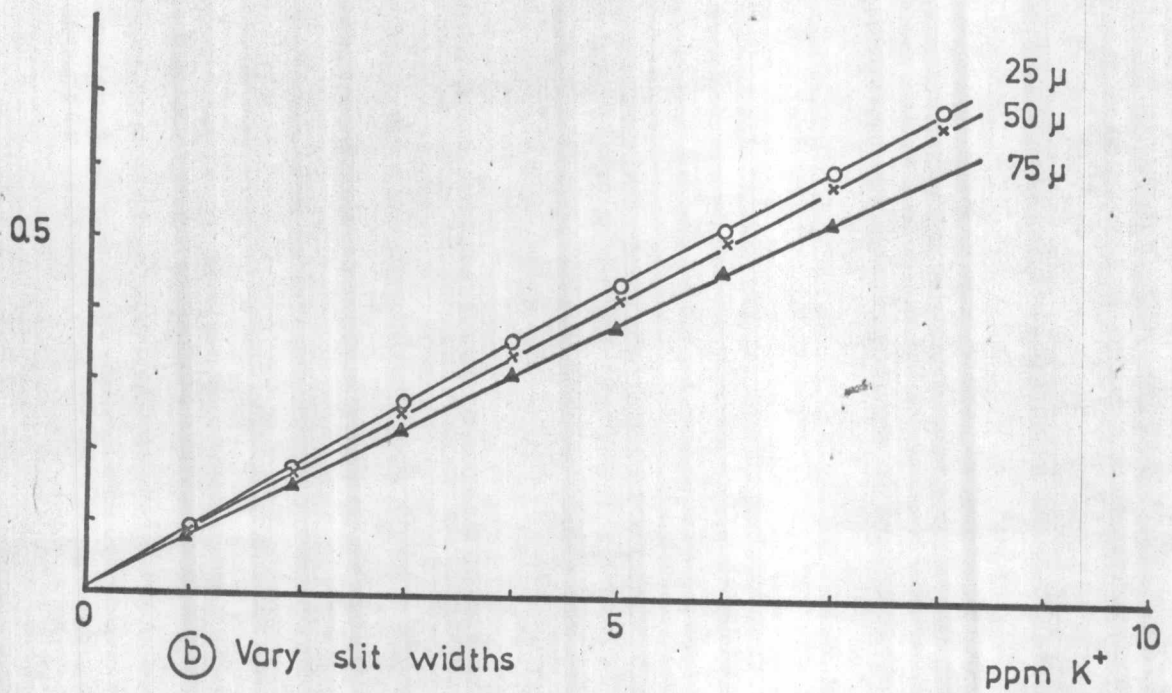
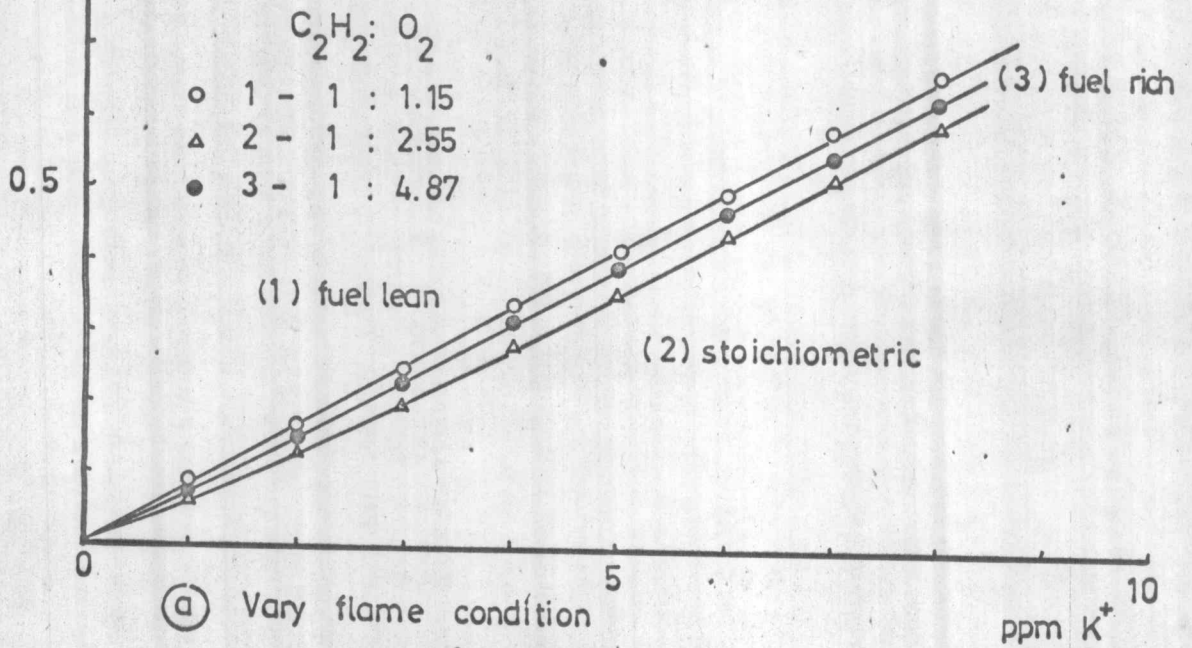
1. The analytical reagent grade $\text{Li}_2\text{SO}_4 \cdot \text{H}_2\text{O}$, NaCl , KCl , CsCl (B.D.H) and $\text{LiCl} \cdot \text{H}_2\text{O}$ (atomic absorption spectroscopic grade).
2. Reagent grade RbCl (B.D.H) and $\text{LaCl}_3 \cdot 6\text{H}_2\text{O}$ (Fisher Scientific Co.)
3. The atomic absorption spectroscopic grade NaCl in solution of 10^3 ppm (B.D.H).
4. The microanalytical reagent grade KNO_3 (B.D.H)

Preparation of solutions

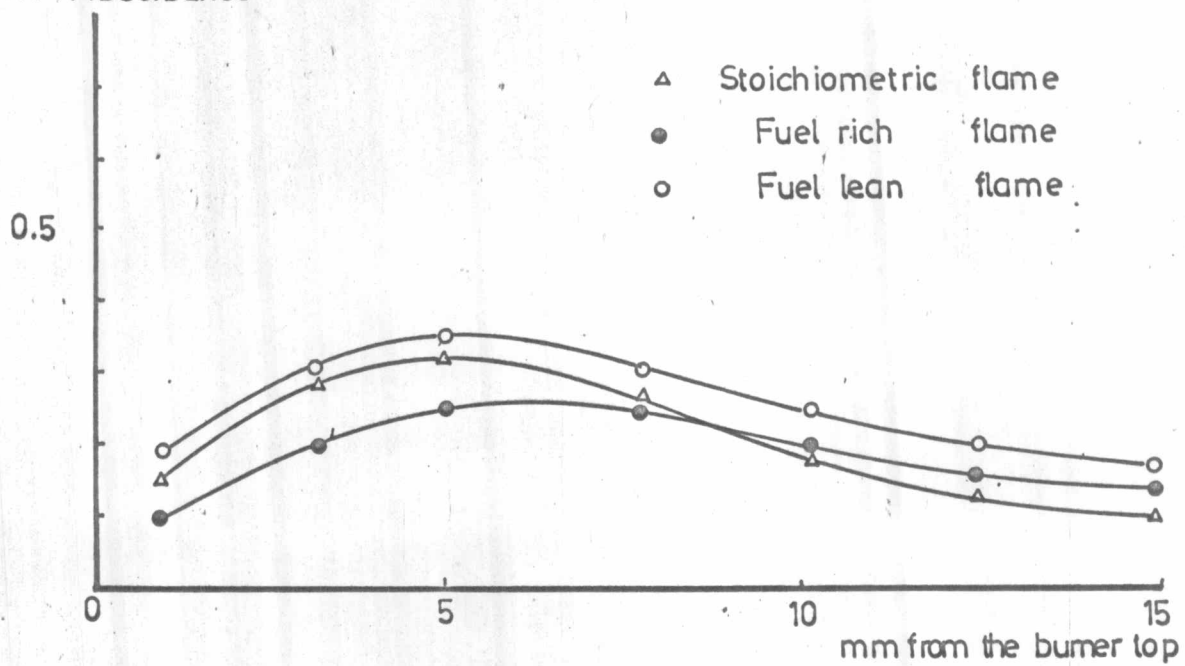
Standard stock solutions of 10^4 ppm were prepared with conductivity water and were kept in the polyethylene bottles. In these experiments, the chloride salts were predominantly used except for some studies done on anionic interference. The KCl and NaCl salts contained 0.05 % Na and 0.01 % K respectively, the M.A.R. grade KNO_3 and atomic absorption spectroscopic grade NaCl were used instead. The effect of anion on absorbance were also determined between the nitrate and chloride salts of potassium as shown in Fig.8 Since the effect is extremely small, the nitrate was used without correction to the result.

Fig 2 Variation of absorbance of K solution with :

K-Absorbance



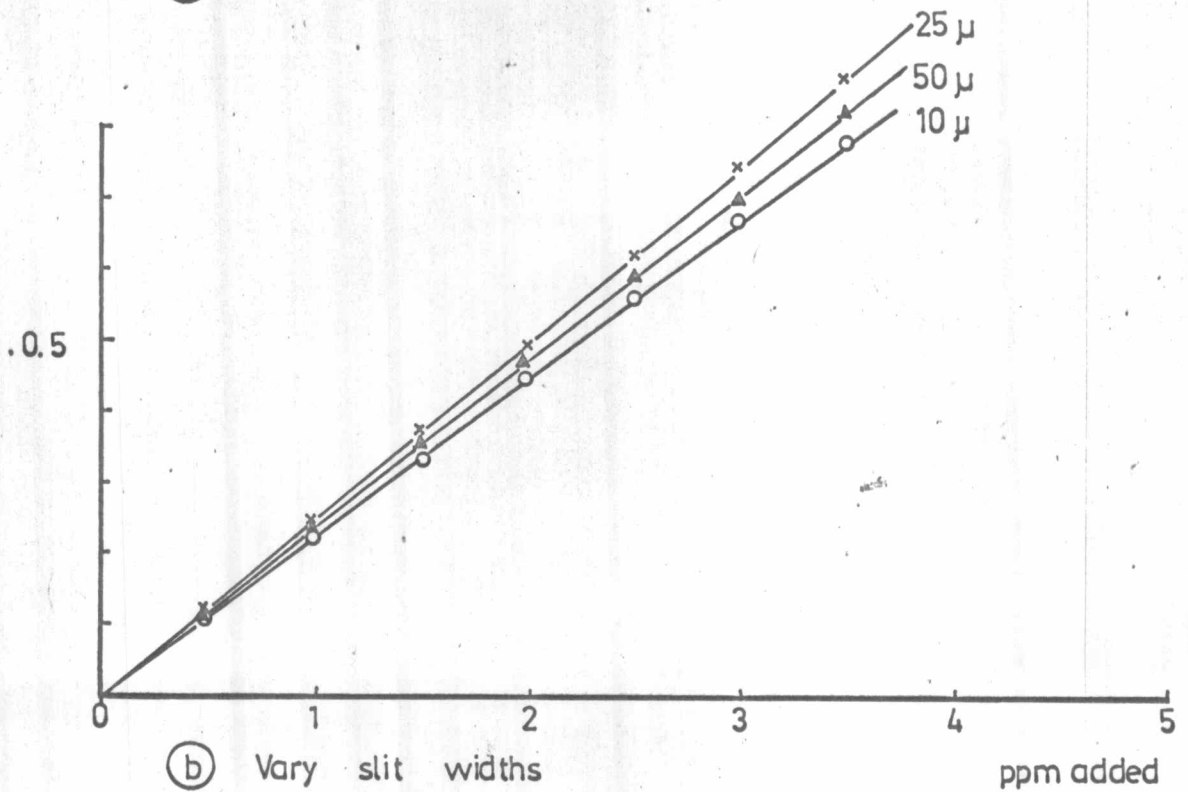
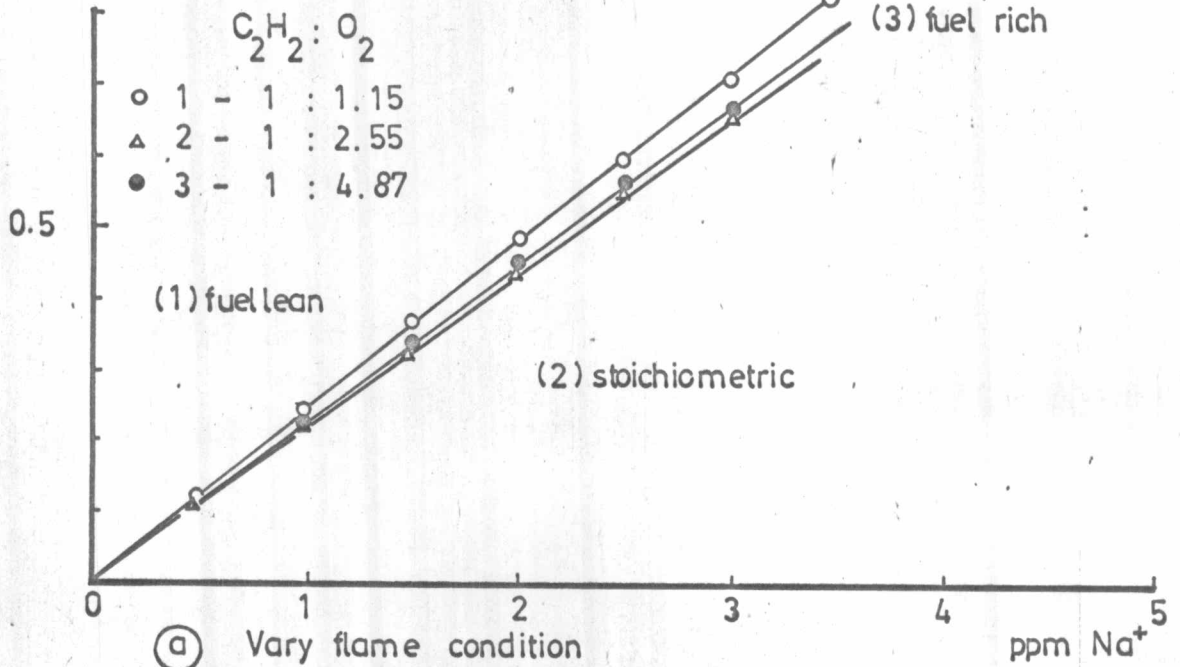
K- Absorbance



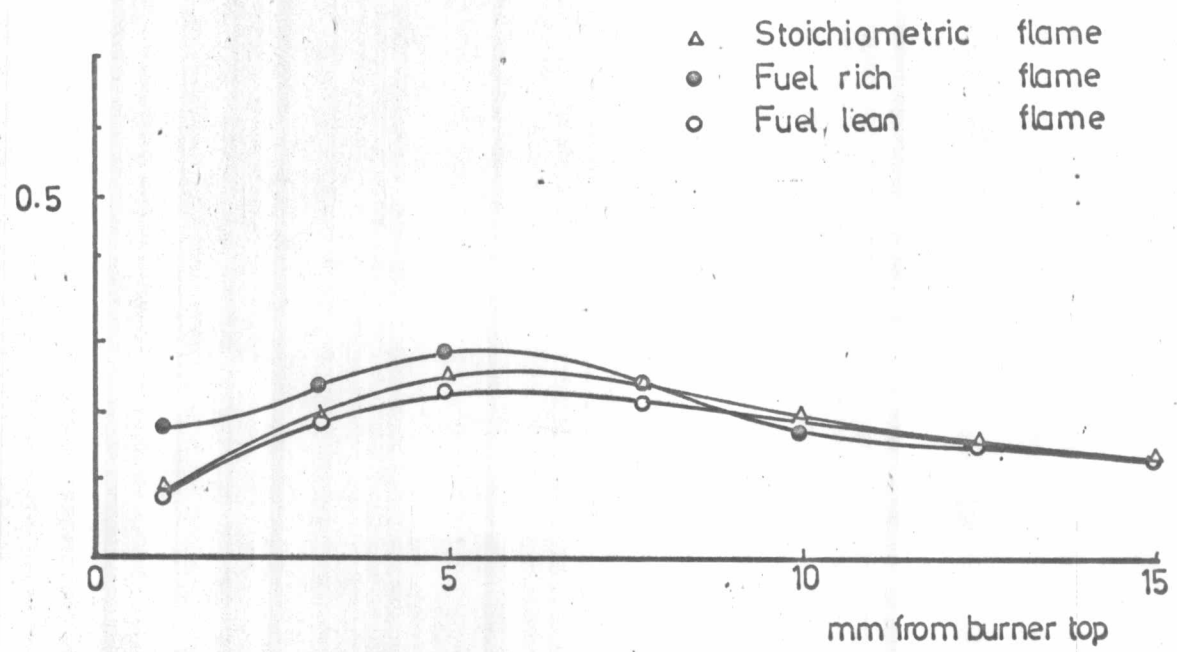
(c) Vary height above burner top traversed by light beam

Fig. 3 Variation of absorbance of Na solution with :

Na - Absorbance



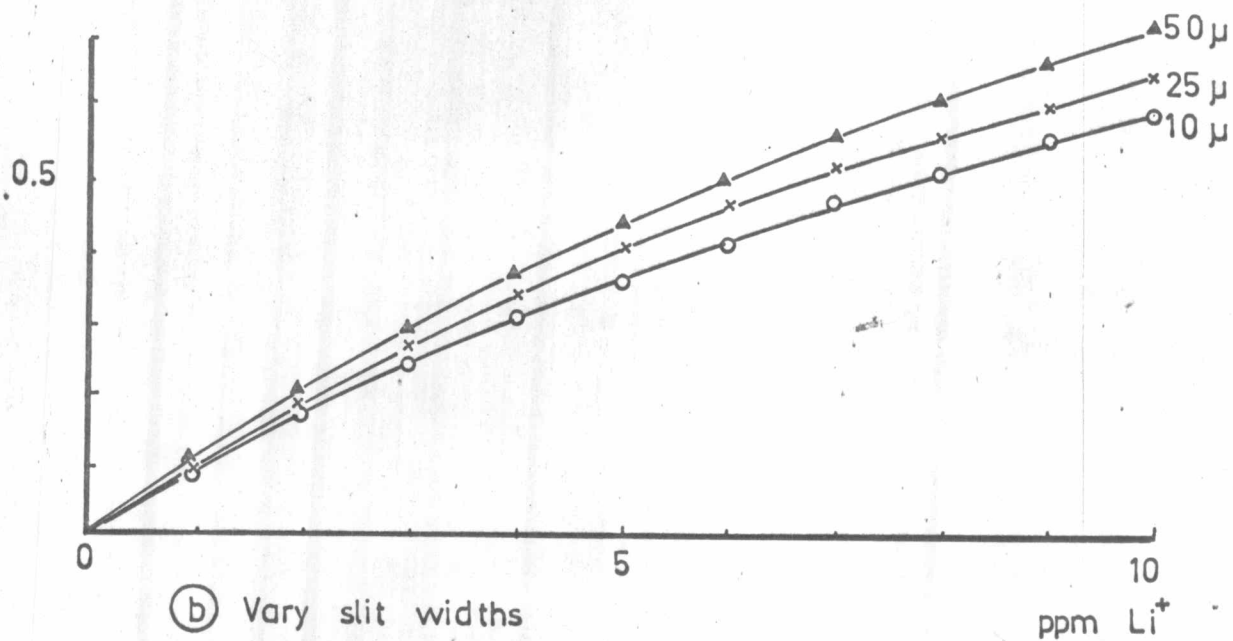
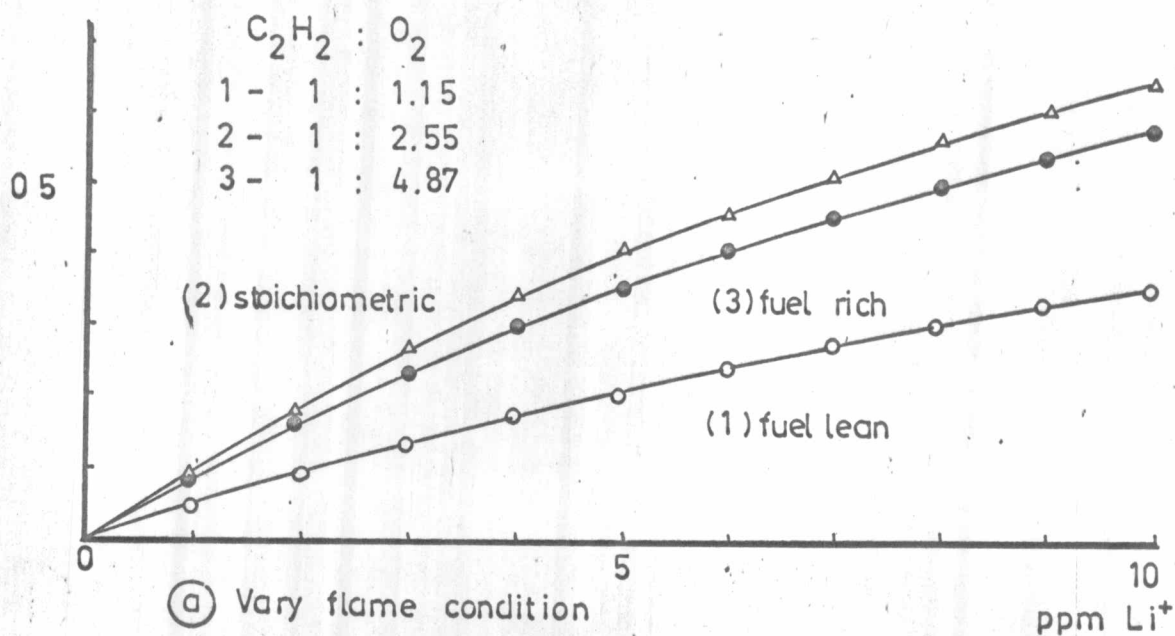
Na - Absorbance



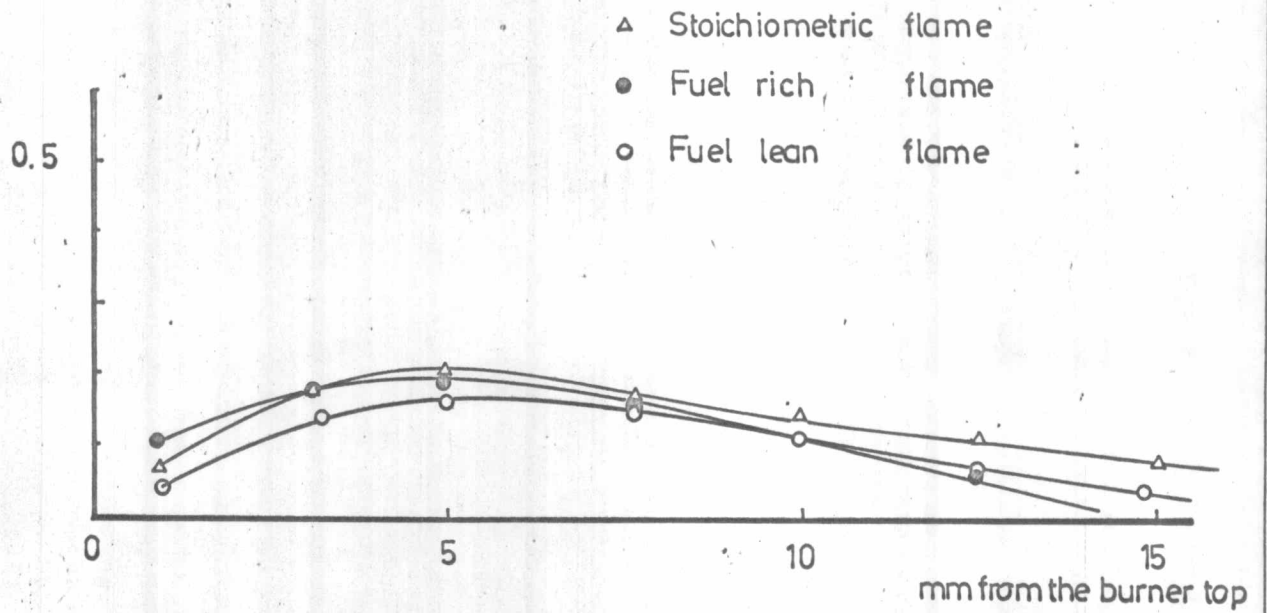
(C) Vary height above burner top traversed by light beam

Fig 4 Variation of absorbance of Li solution with :

Li - Absorbance



Li - Absorbance



© Vary height above burner top traversed by light beam