

Microspectrophotometry

General Section

General Comments

- Why do we use MSP when there are other techniques available for colour comparison?
- Comparison Microscopy is important but is only a subjective technique when comparing one fibre with another. The lighting conditions on both microscopes must be identical and the operator must be capable of using the microscopes.

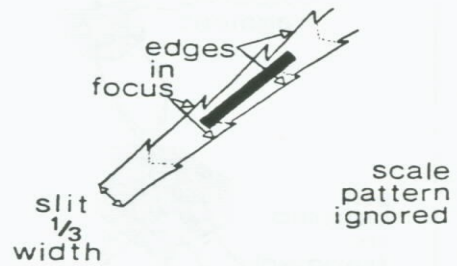
- HPLC, TLC, CE are techniques that can be used for colour comparison but some are problematical and all are destructive. The advantage over comparison microscopy is that they are objective techniques.

- MSP is also an objective technique and is non destructive.
- It is a technique that not only allows fibres to be classed as identical but also enables them to be differentiated.
- Metamerism is the condition where two colours appear similar under one set of lighting conditions but different under another.

- It can be difficult for the human eye to detect metameric pairs but instrumentation such as an MSP can do this.
- Natural fibres such as wool and cotton generally show a large amount of dye variation from one fibre to another. It can take time to find a fibre in a control item that matches a recovered fibre exactly.

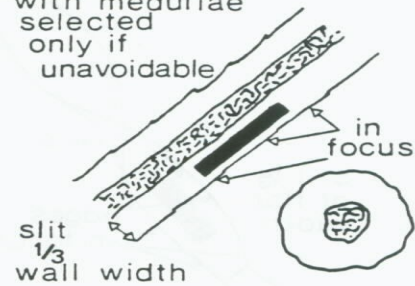
- When obtaining spectra from fibres it is important to take the measurement in a 'clean area' .
- Examples follow.

WOOL



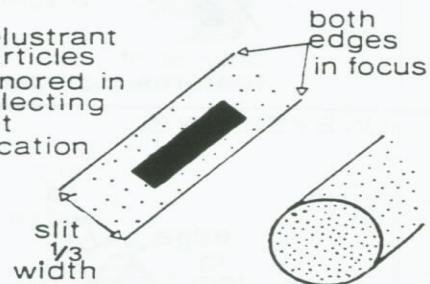
ANIMAL

fibres
with medullae
selected
only if
unavoidable

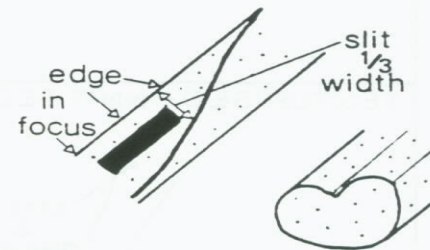


ROUND

delustrant
particles
ignored in
selecting
slit
location

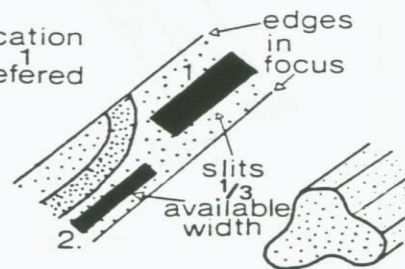


BEAN

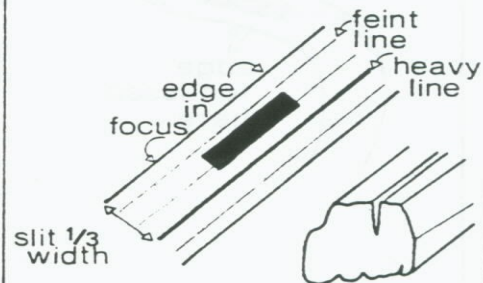


TRILOBAL

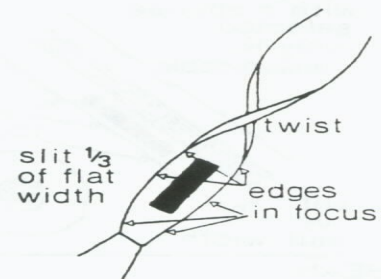
location
1
prefered



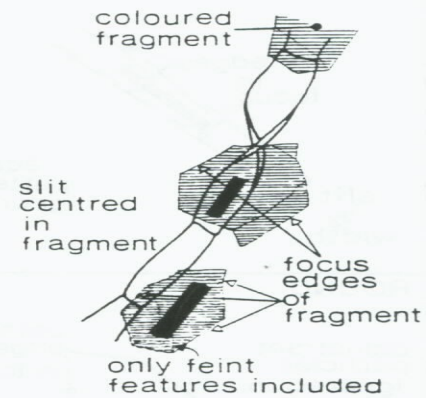
IRREGULAR



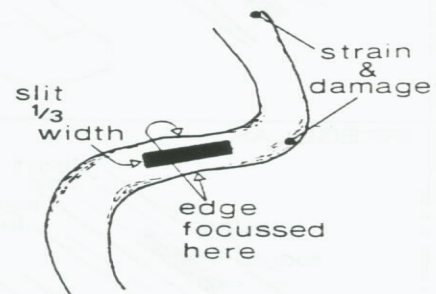
COTTON



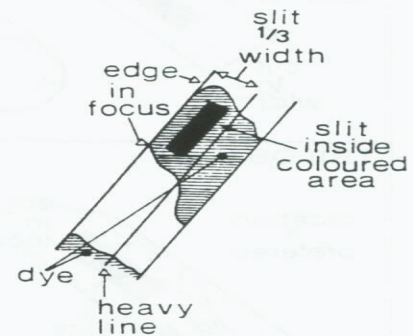
PRINT - COTTON



TEXTURISED - KNITTED



UNEVEN DYE



- The uptake of dye may also vary in man made fibres but does not occur as often as in natural fibres.
- However it is important to cover the complete range of dye variation when obtaining spectra from a control item.
- In a general situation it is advisable to measure 5 or 6 man made fibres and about 10 natural fibres.

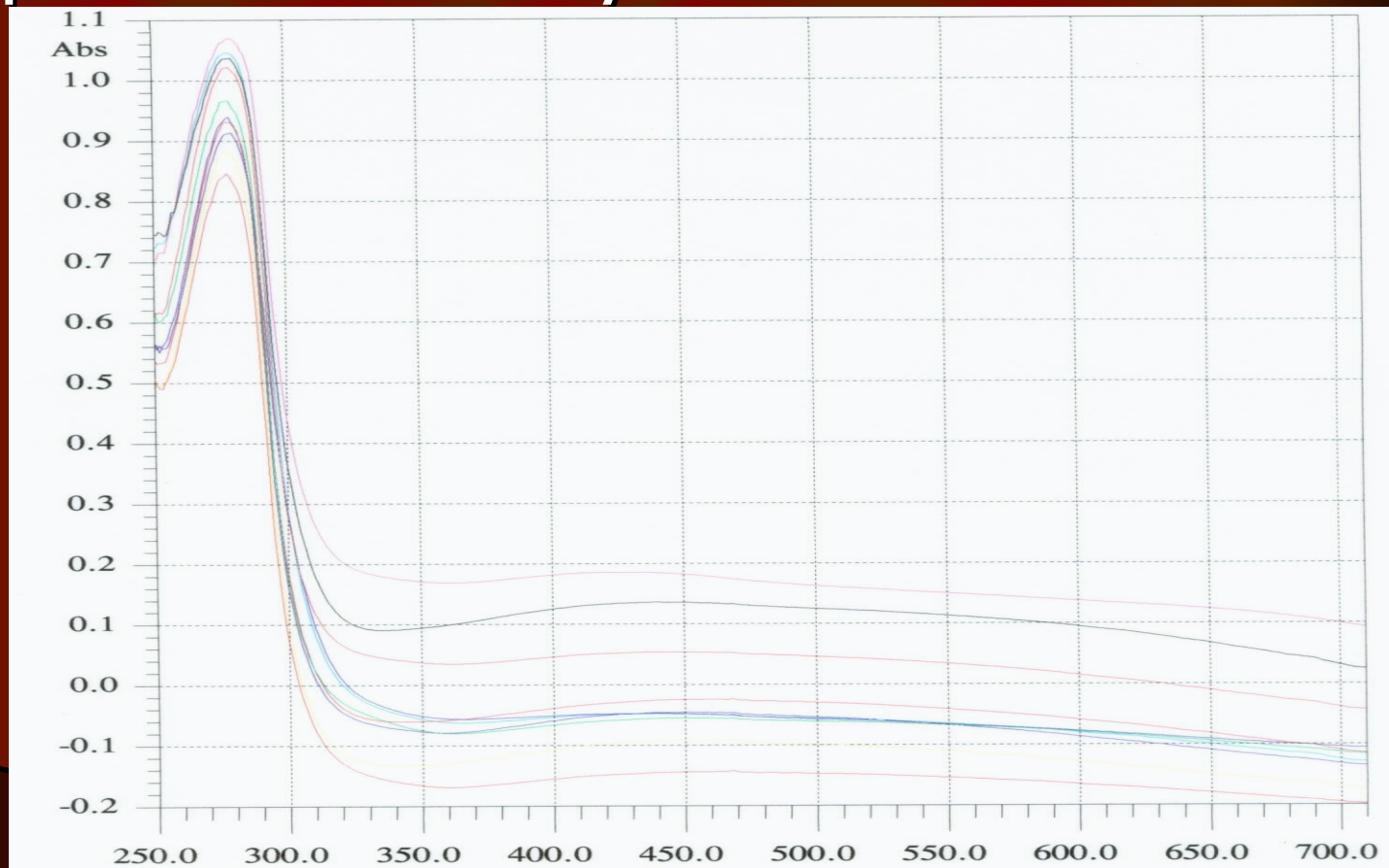
- The number of recovered fibres measured will vary from one laboratory to another but most will test at least 33% up to a maximum of 10 recovered from one item.
- The normal way of comparing spectra is either on screen or by producing hard copies and overlaying them.

- The comparison involves peak positions, peak shapes, absorbance values and peak ratios.
- In my opinion there are 4 end results:
 - +ve (positive)
 - -ve (negative)
 - +WR (positive within the range of the control spectra)
 - Inc (inconclusive)

- A positive result is where there is an exact spectral match.
- A negative result is where the spectra are totally different or the recovered fibre falls outside the spectral range of the control. If the latter happens it may be advisable to run additional control fibres.

- A fibre which is said to be positive within the range of the control fibres means there is no exact match with one spectrum but all the parts of the recovered fibre spectrum matches parts of the control spectra. The fibre therefore falls within range of the control.

- Inconclusive is only applied if the spectrum has very little detail.



Instrumentation Problems

- In order to obtain good spectra it is important to be aware of instrument problems that may occur.
- The instrument should be set up for Kohler illumination before use and calibration standards should be used.
- Calibration will be covered in more detail later.

- If strange spectra are obtained after the correct set up and calibration procedures have been used then operators need to know what the problem is.
- Some examples will follow.

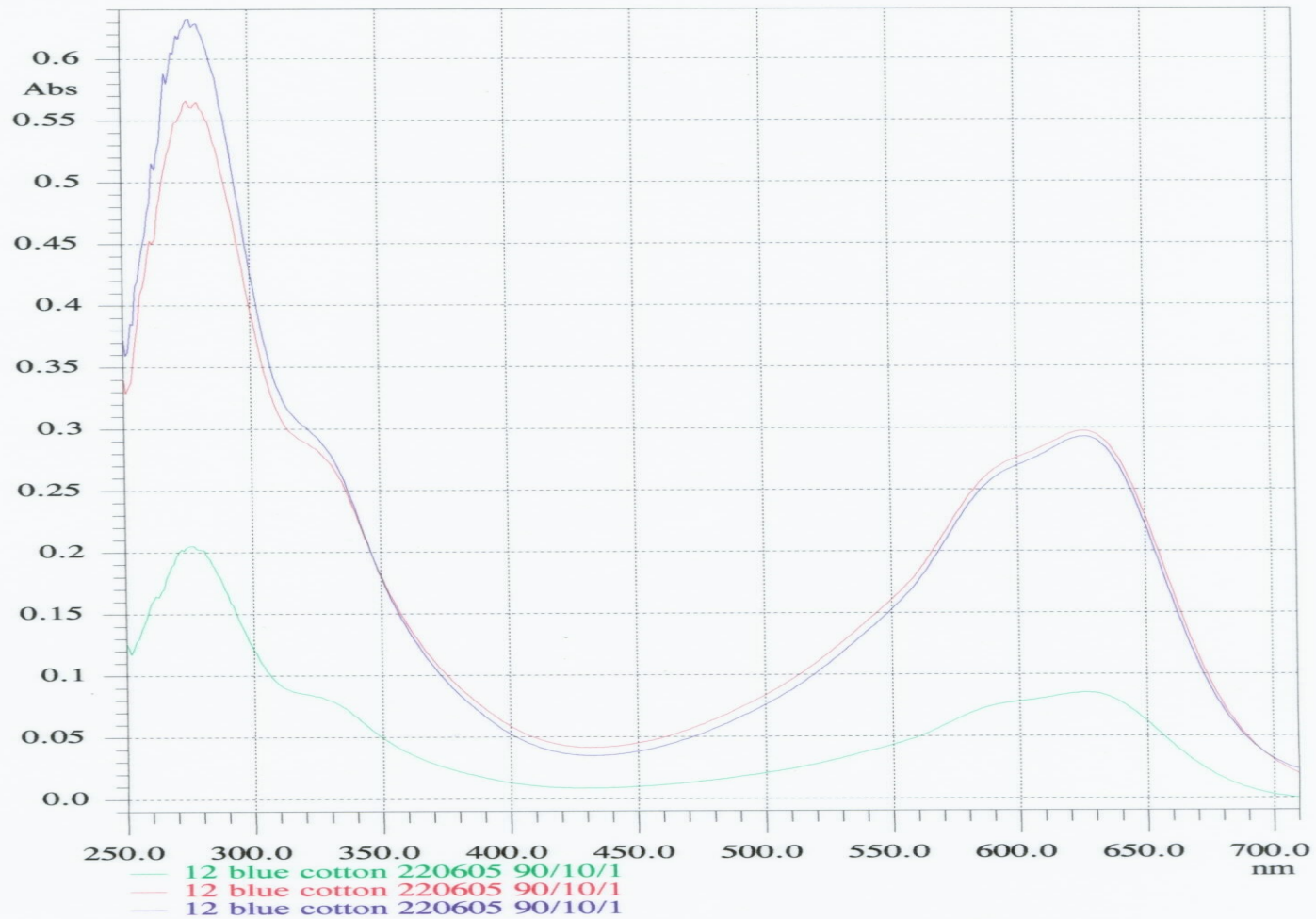
Degrading UV objective



EXAMPLE (1)
SET A (1)

Degrading
Objective

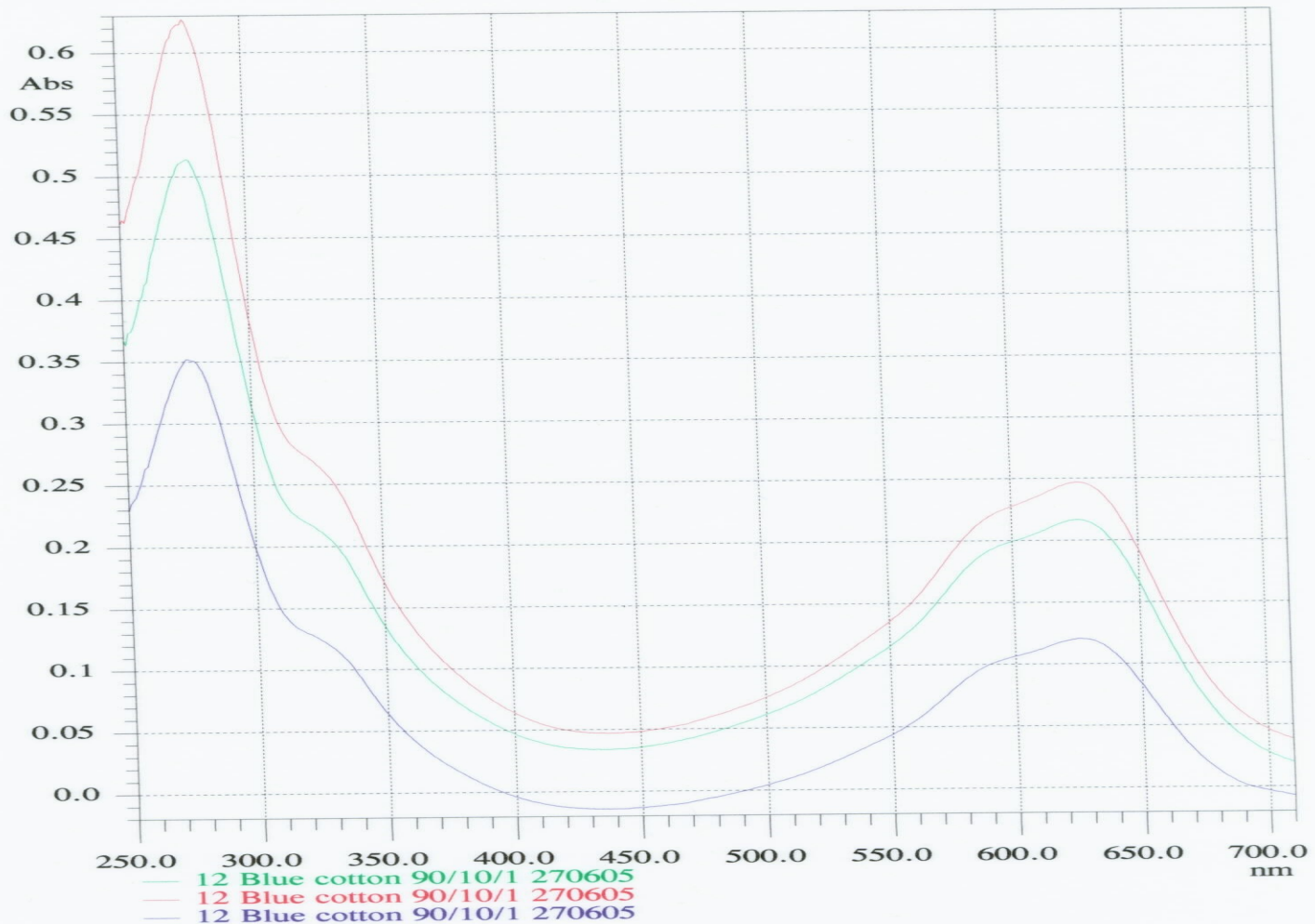
New objective but lamp not aligned



EXAMPLE (1)
Set A (2)

Before Xenon
Lamp re-
Alignment

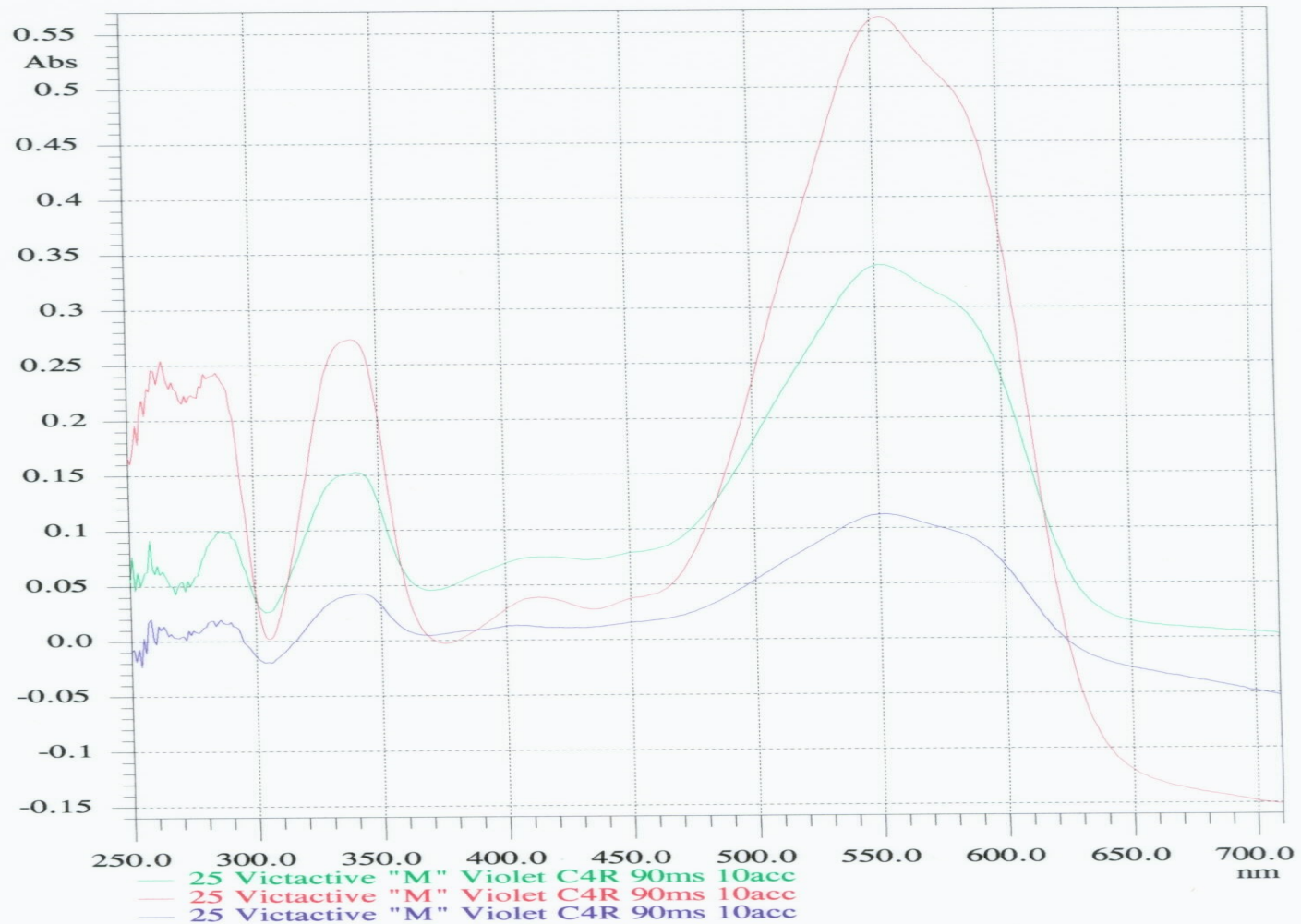
New objective and lamp properly aligned



EXAMPLE (1)
SET A (3)

Optimum Results

Degrading UV objective

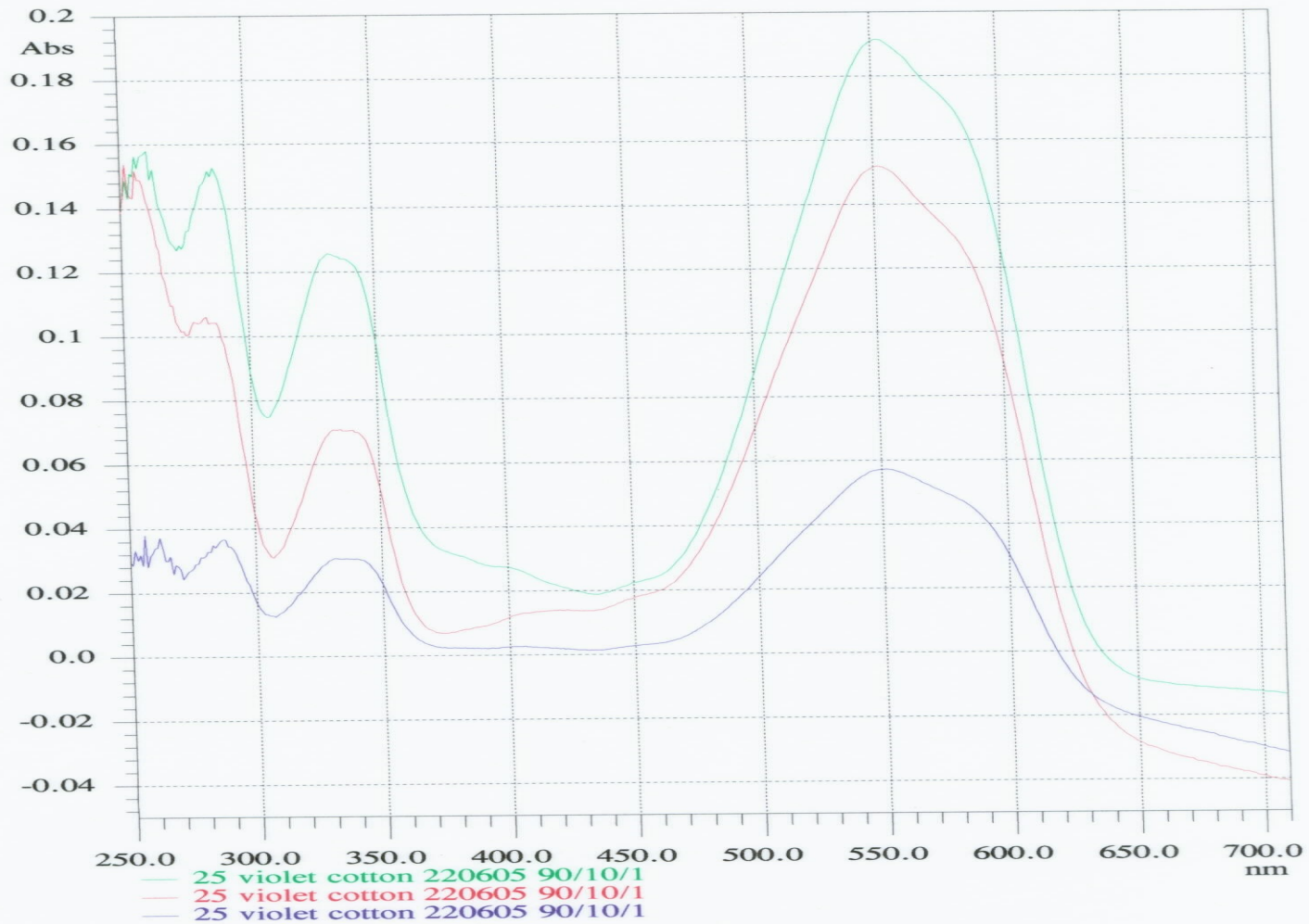


EXAMPLE (2)

Set A (1)

Degrading
Objective

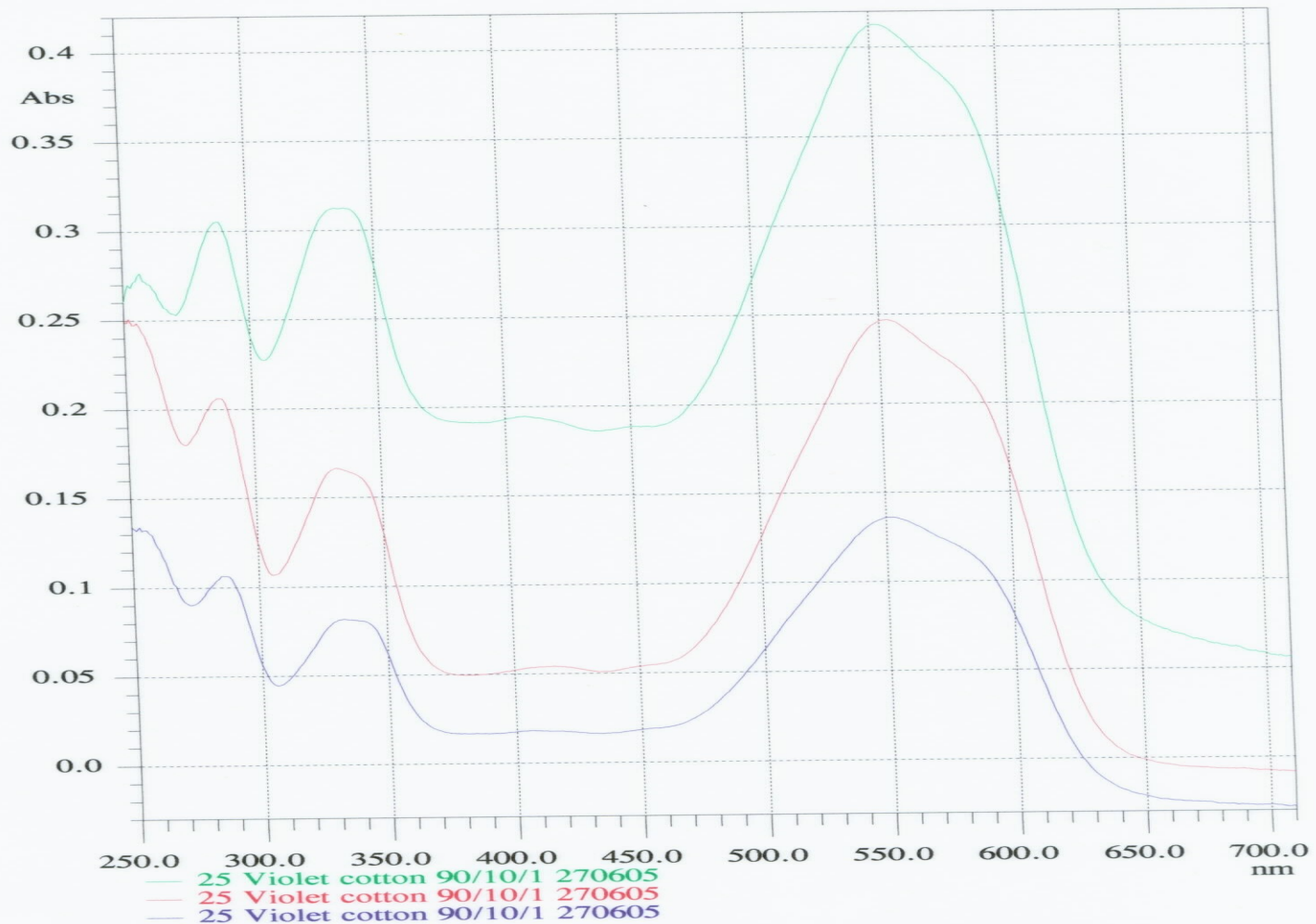
New objective but lamp not aligned



EXAMPLE (2)
SET A (2)

Before Xenon
Lamp re-
Alignment

New objective and lamp properly aligned

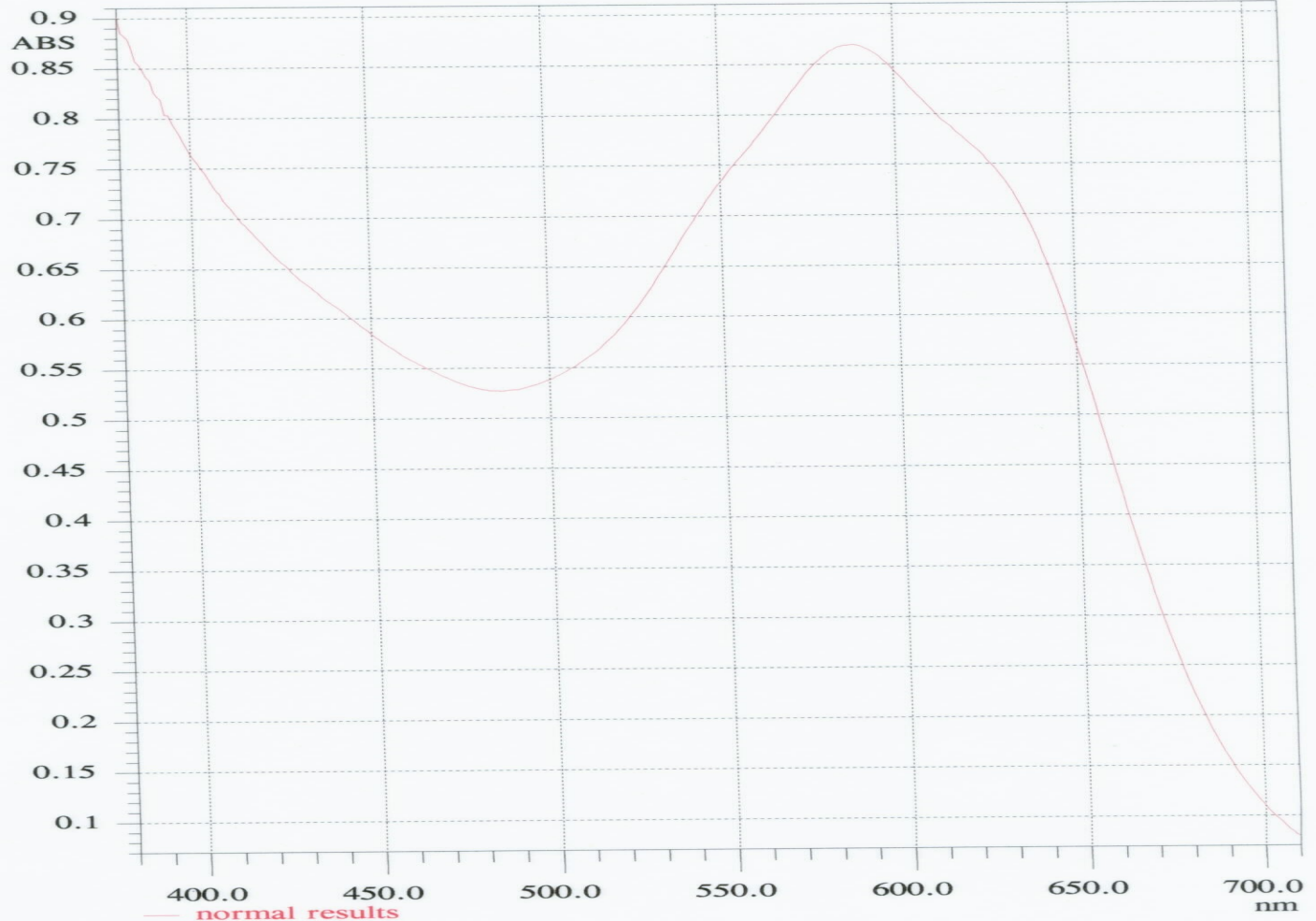


EXAMPLE (2)

SET A (3)

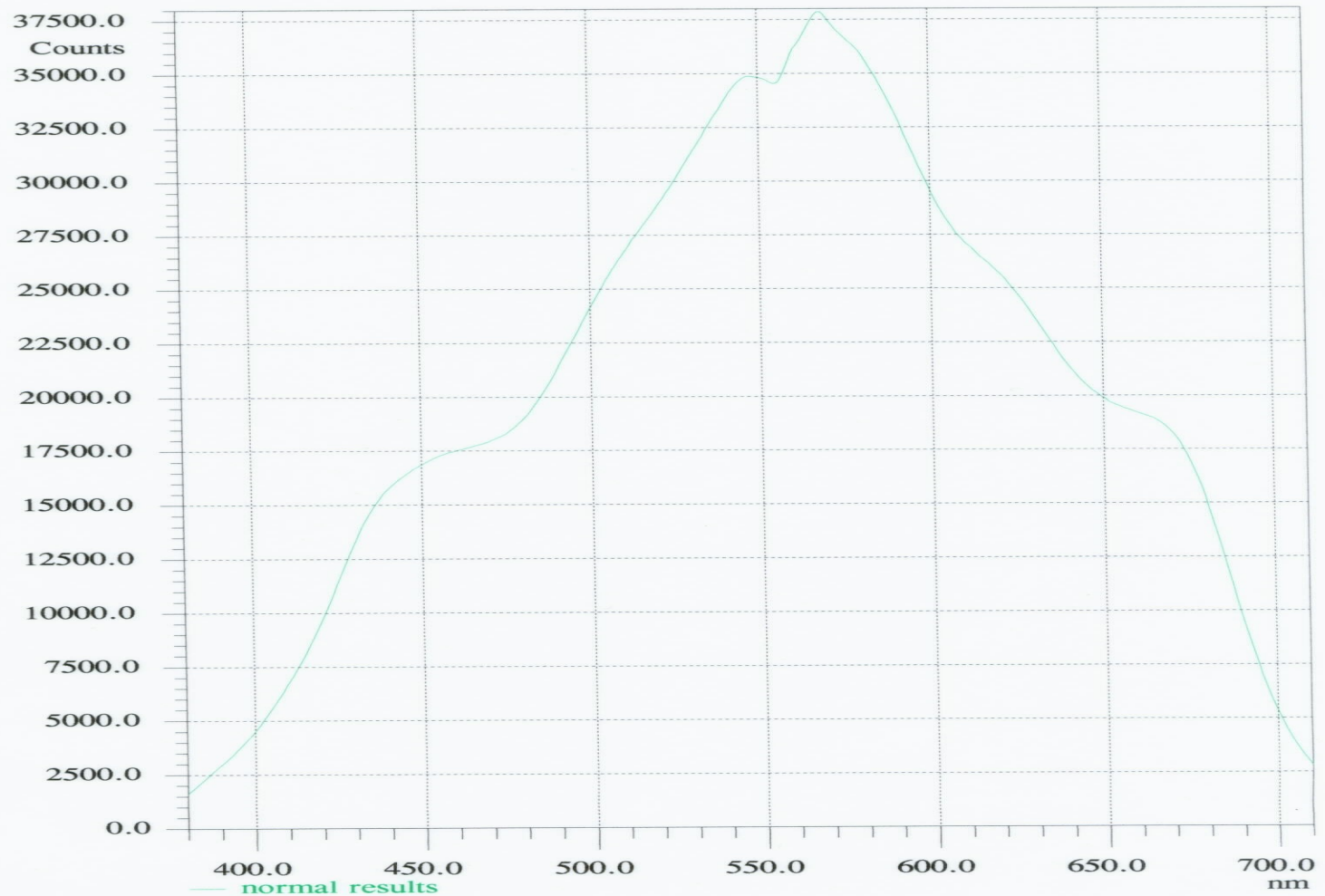
Optimum Results

Normal absorbance spectrum of a fibre

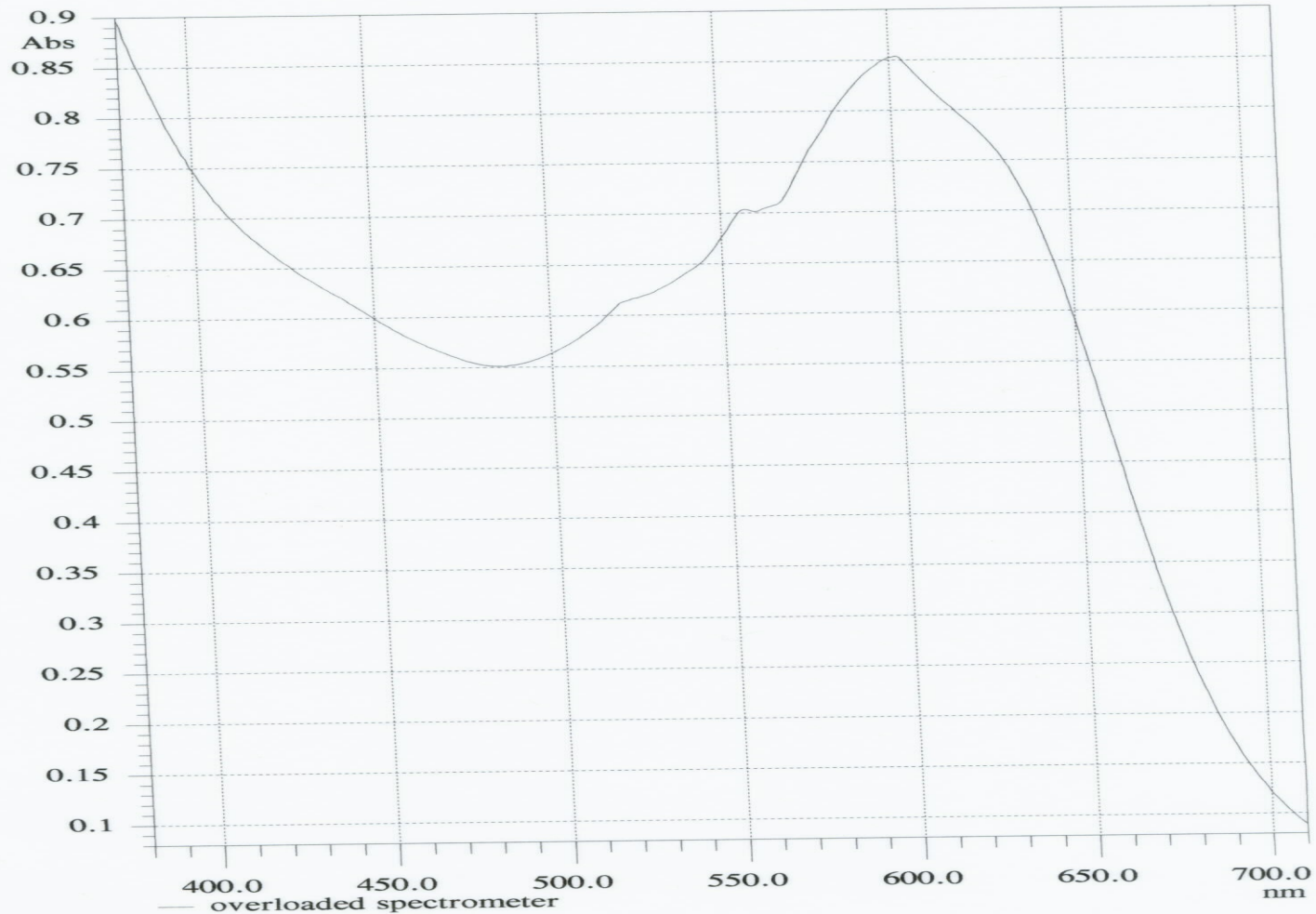


Set B(1)

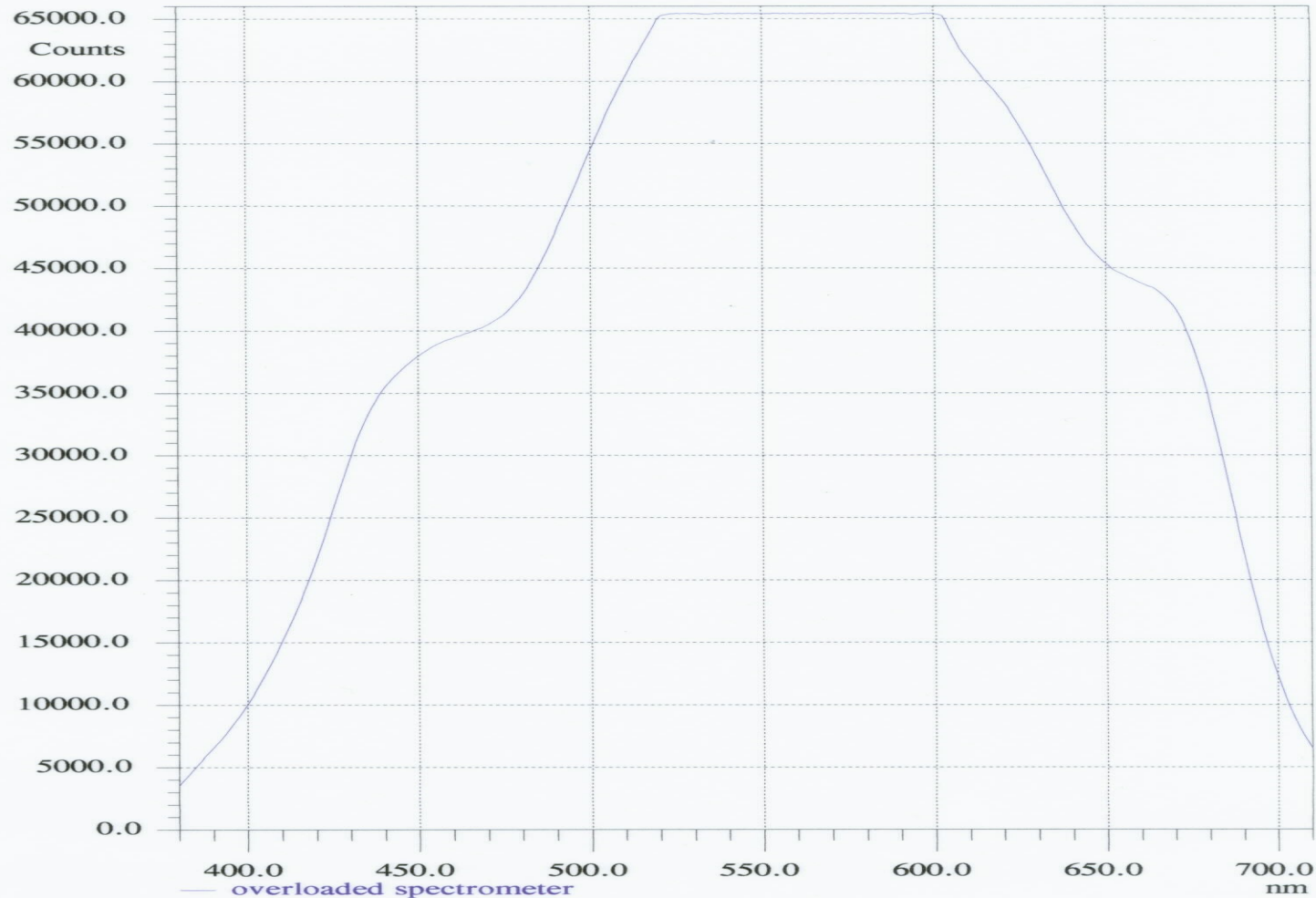
Normal absorbance spectrum of the lamp



Overloaded absorbance spectrum of a fibre



Overloaded absorbance spectrum of the lamp



Normal fibre spectra

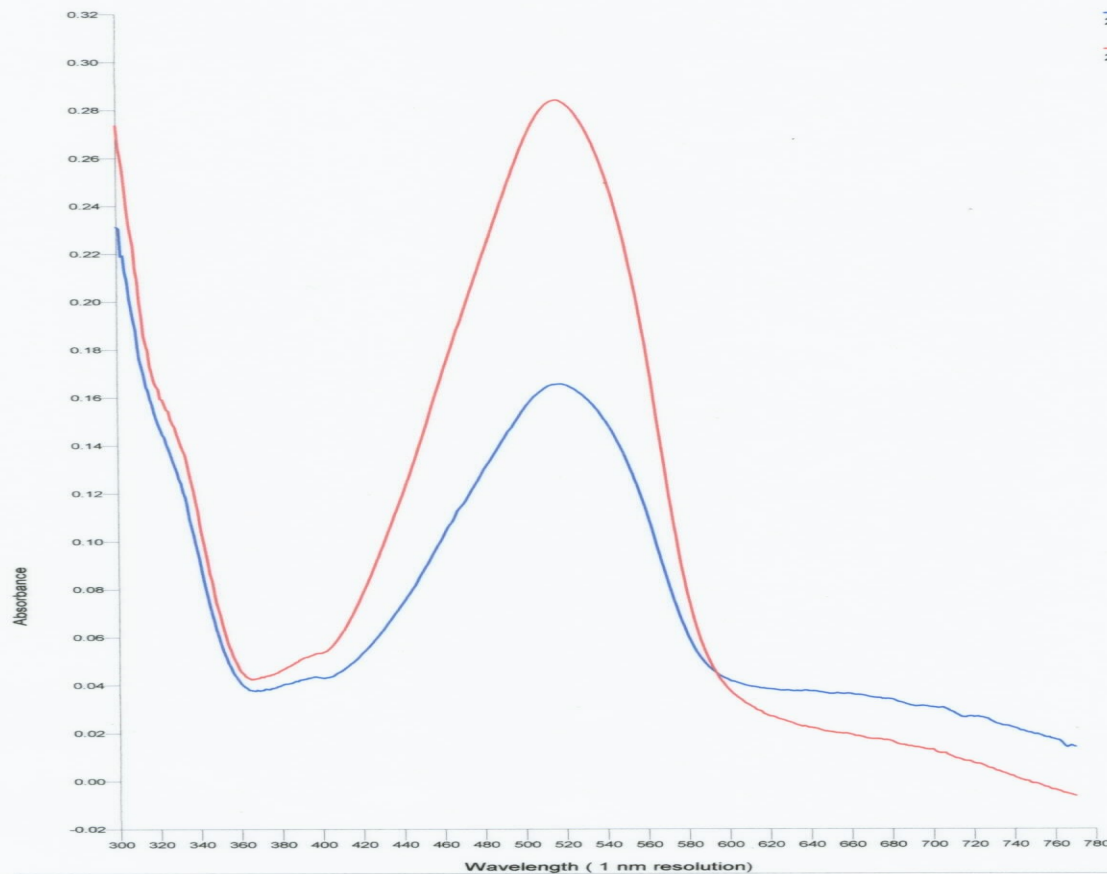
Fibre Microspectrophotometry - Control Sample Spectra

SERVICE ORDER NUMBER: UV PROBLEMS

EXHIBIT NUMBER: Red Wool

EXHIBIT DESCRIPTION: 90ms 10acc

SAMPLE DESCRIPTION: Measured from bulk sample



OPERATOR:

DATE:

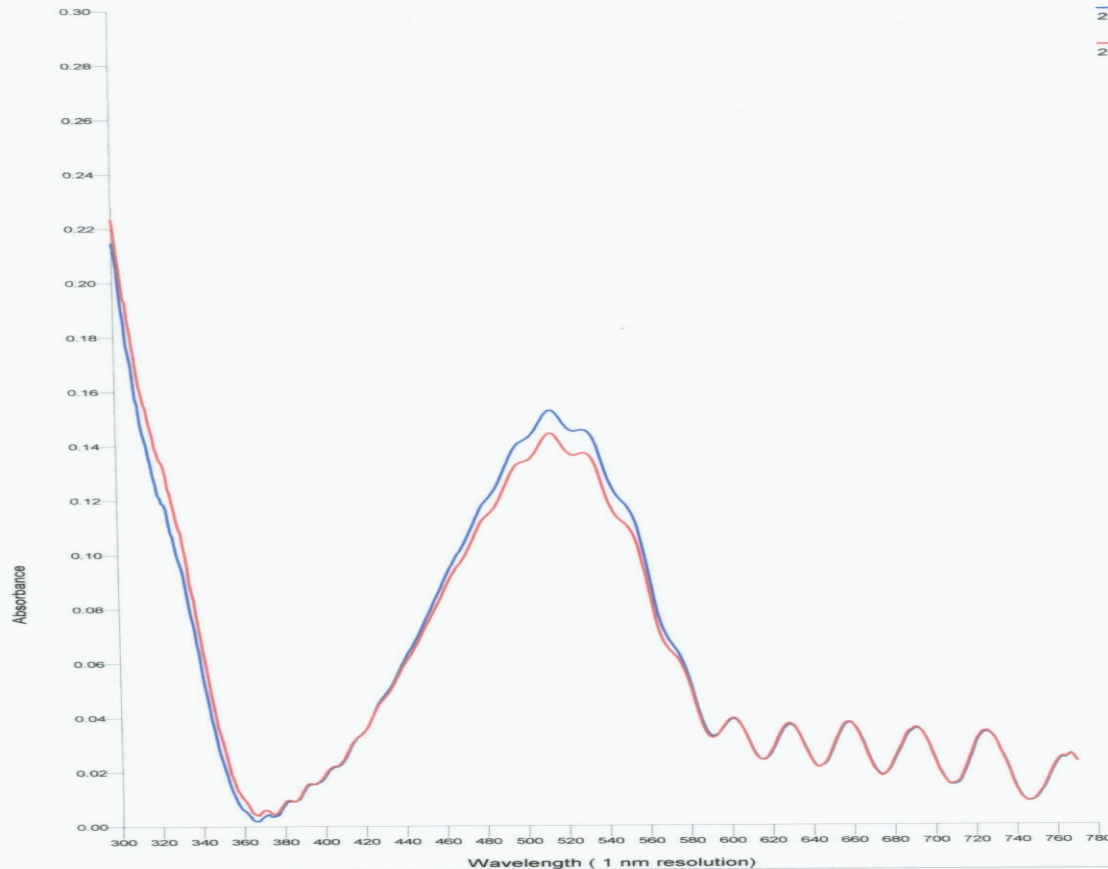
CHECKED BY:

DATE:

Spectral waves caused by light source being in contact with the slide

SERVICE ORDER NUMBER: UV PROBLEMS
EXHIBIT DESCRIPTION: 90ms 10acc
SAMPLE DESCRIPTION: Measured from bulk sample

EXHIBIT NUMBER: Red Wool



OPERATOR:

DATE:

CHECKED BY:

DATE:

Value of UV Spectra

- UV MSP will not provide additional information for all fibres but will in many cases.
- In 1994 Grieve mentioned that grey and black cotton fibres often gave additional information in the UV region.
- He also said that turquoise green cotton fibres can be difficult to discriminate in the visible region of the spectrum.

- He compared 27 fibres of this colour and type – 351 comparisons.
- When comparing them over the range 390-760nm 10 pairs had indistinguishable spectra. However when compared over the range 240-760nm the number of matching pairs was reduced to 6.

- In 2005 myself and two colleagues did a complete validation and evaluation of the J&M MSP800 instrument but this was only published as an internal document.
- Part of the project was to examine both coloured and undyed fibres from a range of fibre types.
- As wool and polyester absorb all light below the 300nm region approximately the only region in which additional information is available is between 300-380nm.

- Acrylic, cotton, nylon, polypropylene and viscose fibres were examined and showed that more than 70% of the samples within each fibre type offered information in the UV region. The average figure for all the coloured fibre types above was 87%.
- The wool samples gave 21% additional information and the polyesters 29%.

- An additional part of the project was to examine samples from manufacturers that started as undyed fibre through to the finished product.
- The fibre types investigated were viscose, polyester and wool.
- It was only after dyeing that any of the samples gave information in the UV region.

Additional Information about the UV region

- In my experience information above 250nm is worth collecting.
- Fibres exposed to UV light for any period of time begin to bleach.
- The bleaching of the fibre can cause the absorbance spectrum to change with respect to the absorbance levels and peak ratios and as a result the overall shape.

- Changing the lighting conditions i.e. turning the lights on and off in the laboratory can make very minor changes to the UV region of the spectrum.
- Therefore when comparing fibres in casework the lights should always be on or off.