

SPECTROMETER / COLORIMETER - flame

Cat: CH3792-001 Flame emission and Atomic Absorption

DESCRIPTION: The IEC 'Flame & AA Spectrometer' is used for the teaching of analytical chemistry. It introduces instrumentation to the student which is similar in function to that used in the workplace. The 'IEC Spectrometer' can demonstrate the principles of both flame and atomic absorption spectrometry. It is used also as an accurate colorimeter.

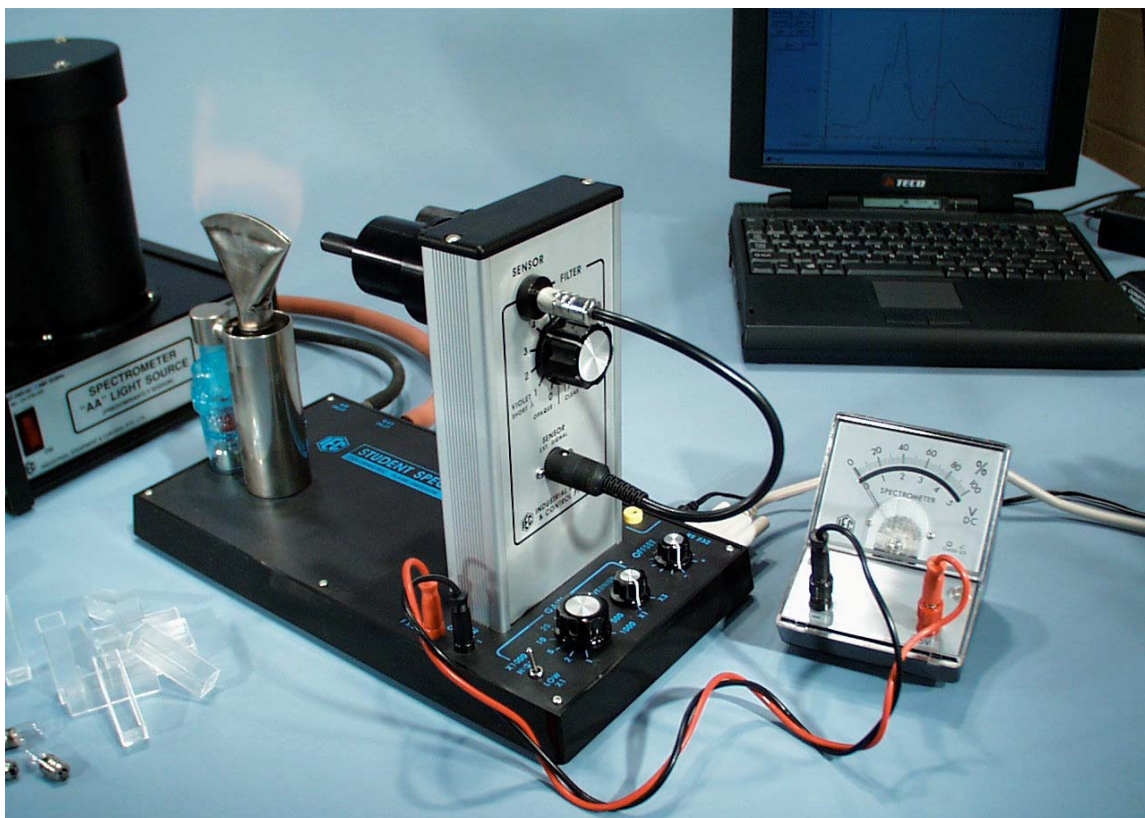
Results can be handled in two ways:

- **Manually:** by taking readings on the special meter provided in the kit and then calculating the results and plotting the various graphs manually or
- **Automatically:** by recording all data, readings and results on a computer disk and manipulating data graphically. The students' work can be saved for later reference.

This instrument is not limited to spectrometry. By removing the spectrometer's light sensor from the control panel and by inserting the external signal cable, the unit can be used as a convenient very high gain signal amplifier for other experiments. Signals can be applied from say a gas chromatograph and the results handled by either the meter or the software.

Most laboratories have a sodium vapour light source however, if required, IEC can provide a suitable low-cost sodium vapour light source for atomic absorption work.

CH3792-001 flame spectrometer & colorimeter



Physical size: 320x167x220 LxWxH

Weight: 4.2 kg

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KIT CONTAINS:

- 1 pce spectrometer unit and light detector on cable.
- 1 pce flame spreader for top of burner
- 1 pce plug-pak power source, 12V.AC.
- 1 pce cable for connecting to the 9 pin serial port of a computer (9M/9F).
- 1 pce adaptor to convert the cable to suit a old type 25 pin serial port (9M/25F).
- 1 pce cable with DIN plug for external uV/mV signals.
- 1 pce bench meter (0-100% & 0-5V.DC. scales) for measurements.
- 1 pr connecting leads for meter, red / black.
- 1 pce atomiser (nebuliser) to feed a mist of sample fluid into the flame.
- 10 pcs cuvettes for colorimetric samples.
- 3 pce spare globes for colorimetric light source.
- 1 pce handbook of instructions and many experiments.
- 1 pce CD or 3-1/2" software floppy disc with instructions.

This manual is designed to provide basic explanations of the principles and should be read and fully understood before performing experiments using the main handbook.

A suitable light source for Spectrometry (not supplied with the Spectrometer):

CH3792-020 (sodium)

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THE IEC FLAME & AA SPECTROMETER / COLORIMETER

INTRODUCTORY AND EXPLANATORY NOTES:

HOW DOES IT WORK?

In the year 1900, Maxwell Planck put forward the theory that as electrons inside atoms shifted from one orbit to another due to the excitation of the atom, energy is either liberated or absorbed. Further, this liberation (emission) or absorption occurred not smoothly, but in small jumps.

In other words the electrons tended to remain in their original orbits until they suddenly jumped into a new orbit. These changes in energy always occurred in 'quantum' (whole number value) jumps.

The concept is similar to an article being able to alter price by only whole cents since the cent is the smallest denomination. A price cannot change by 1-1/2 or 255-1/4 cents, however, the difference between Planck's theory and a cent is that, in chemistry, the size of the quantum change in energy level is not always constant. It varies depending on the type of atom being excited because it depends on the strength of its orbital bond.

The usual method of exciting the atom is by heating the material containing the atoms. After excitation, the usual manner in which the energy change is detected is by the emission or absorption of light. The examination of the wavelength and strength of light either emitted or absorbed is called '**Spectrometry**'.

The term 'light' generally means the visible spectrum from violet through to deep red. In spectrometry work, this term can be extended outside the visible spectrum above violet up to ultraviolet and x-rays (short wave length and high frequency) and below red through infra-red down to microwaves (long wave length and low frequency). The IEC spectrometer deals only with the visible spectrum between violet and deep red. In fact, the instrument deliberately filters out ultra-violet and infra-red rays from the detection system.

EMISSION: We said before that when atoms are excited, energy is emitted and that this emission is in the form of light. The exact level of emission determines the exact wavelength or frequency of light that is emitted when the excitation takes place. When molecules of most materials are excited there are multiple changes in energy that produce several wavelengths of light at different strengths, thus it may be required to observe a set of different wavelengths and strengths to obtain a result.

ABSORPTION: Not only do changes in energy of excited atoms emit certain exact wavelengths of light, but also the same action absorbs the exact same wavelength of light should that wavelength be trying to pass through the energy emitting area (usually a flame).

Thus, if white light (the whole visible spectrum) is passed through the energy producing area, some wavelengths would be absorbed on the way through and an examination of the spectrum after the white light had passed through the area would reveal gaps in the spectrum which would be the exact wavelengths of light absorbed in the energy producing area (usually the flame).



WHY IS THE SPECTROMETER USEFUL ?

Knowing the exact wavelengths of light emitted or absorbed and knowing the intensity or strength of each particular wavelength provides us with a method of:

- Identifying which elements are present in a material and
- Knowing the concentrations of those elements present.

Both spectral emission and spectral absorption techniques are very commonly used methods of chemical analysis and their study is an integral part of the understanding of chemistry.

HOW IS IT DONE IN PRACTICE ?

SPECTRAL EMISSION TECHNIQUE: Requirements are as follows:

We need a sample of the material to be analysed:

BY EMISSION means we are measuring the light being emitted by the flame into the viewing tube. The sample is usually a solution containing the material and created by dissolving or reacting the material in some pure chemical (or distilled water) so that it is not contaminated with other materials and can be handled in liquid form.

Often, a material can be burned directly in the flame and, if the energy level changes are such that certain emissions take place, there is an obvious change in the colour of the flame. For example, calcium gives a brick red flame, potassium gives a soft lilac flame, strontium gives a crimson flame and copper is green etc..

Many elements and compounds do not alter the flame colour to such a degree that the human eye can detect the differences, therefore more sensitive methods of colour discrimination are necessary.

We need a method of exciting the atoms of the material to be analysed:

The normal method is by burning the solution containing the atoms in a non-luminous gas flame.

We need a method of passing the solution containing the material to be analysed into the flame for burning:

This is done by placing the solution into an **Atomiser** or **Nebuliser** which is fed constantly with clean, non-contaminated air. The resulting continuous atomised mist or cloud is fed into the air stream of the burning device and so is burned simultaneously with the gas.

We need a method of viewing the light emitted by the flame:

A **Collimator** or viewing tube points directly at the flame and through this tube, the flame is viewed either by eye or with a sensitive electric detector.

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We need a method of observing only various sections of the visible spectrum so it can be discovered exactly which wavelengths are affected by the burning of the sample:

This is done by placing accurate colour filters in the path of the viewing tube. The filters begin at violet (shortest wavelength) and, over ten steps, change progressively to deep red. The user rotates a knob to select the filter required. **Remember, a filter absorbs all wavelengths (colours) except for the band of colour which is permitted to pass through.**

A narrow band filter permits the passage of a very small part of the spectrum and excludes all else. A broad band filter, permits a wider area of the spectrum and excludes all else. For accurate analysis using spectroscopy, the narrower the filter band the better.

We need a method of measuring the presence and strength (intensity) of light that has passed through the filter:

The IEC Spectrometer uses an electronic sensor that provides an electric signal which varies depending on the strength of the light falling upon it. Its sensitivity is almost constant across the whole visible spectrum from violet through to deep red.

This signal is amplified, measured and plotted on a sheet of paper, or on a computer screen thus creating a graph relating wavelength to intensity. Thus one can see which wavelengths and the intensity of each wavelength that is emitted by the flame whilst it is exciting atoms in the material under study.

Therefore, if we have prior knowledge of which materials produce which emission spectra, we can identify the elements in the sample being burned.

If we have prepared a set of sample solutions of known concentrations, we can prepare a graph covering say three or four different concentrations and, by comparing the results of the unknown sample against the graph, the concentration of the sample can be found.

We need a method of measurement and documentation:

The IEC Spectrometer has inbuilt amplification and various controls to set the level of data to be measured. Measurements can be made in the following ways:

- Visually: by observing the flame either directly or through the viewing tube.
- Electrically: by using a 0-5V.DC. voltmeter in the sockets provided. This method is perfectly acceptable and is as valid as the computer method. The meter provided in the IEC Spectrometer kit is calibrated from 0-100% and 0-5 V.DC. The % scale is used for **comparison** in percentage of light intensity readings.
- By computer: the spectroscope is provided complete with a computer interface and program which, when running on any IBM compatible computer, permits measurements to appear on the screen. They can be 'frozen' on the screen, stored, printed and recalled for future use.

The computer method is particularly useful because the previously created calibration curves from the various sets of sample solutions, can be recalled to the screen at a later time or printed for student use as required.

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**ATOMIC ABSORPTION SPECTROMETRY (AAS):**

In many ways this is similar to EMISSION Spectrometry. All the above mentioned points are valid and the method is almost identical.

The differences are as follows:

Instead of measuring and identifying the colours being emitted by the flame, a bright light source of a known exact wavelength (eg: sodium light source) is beamed THROUGH the flame and the setting of the filters selects the sodium band of yellow. Electrical settings are adjusted to the correct levels (the experiment manual supplied with the unit explains everything). The controls are set so that the meter or the computer shows zero with no light (viewing tube covered), then the controls are set so the light source shows a reading of say 80 on the meter (the point chosen is not important) and this point is indicating a reading of the light source without the flame.

When a sample containing sodium is burned in the flame, the sodium part of the spectrum partly disappears from view because of absorption of the bright sodium light into the flame. The reading on the meter or the computer falls suddenly. This demonstration of AAS is very dramatic and of course other monochromatic light sources can be used for other elements.

A white light source with say a yellow filter in front (so that it looks, to the eye, exactly like the colour of a sodium lamp) can be used as an important demonstration. The student will soon discover that there is no reduction in signal level when the sodium sample is burned. This effect clearly demonstrates the necessity of the light source being **EXACTLY** the same wavelength as the wavelength emitted/absorbed by the sample being burned. If sodium is the element to be detected, only a sodium lamp can be used as the light source.

Experiments may be performed with a potassium light source or other light sources and compounds, but sodium is usually the most convenient.

IMPORTANT NOTE: The concentration of the sodium solution being burned should be very low so that the flame very slightly changes colour to the eye. If the flame burns too brightly at the colour of sodium, the bright colour **emitted** from the flame will be seen by the sensor and this will reduce the measured effect of the sodium light source being absorbed in the flame. Details of the best concentrations will be found in the manual.

**COLORIMETRY: used as method of measuring concentration of a solution:**

Another important part of chemistry study is that of Colorimetry. The sodium light source, the flame and the atomiser are not used and the white electric light source to be used is supplied as part of the instrument. The viewing tube is removed from its socket and the white light source is fitted into the viewing cavity in its place. The instrument has a pair of sockets to provide the light source with a regulated voltage so that the light intensity and therefore its reasonably white colour is perfectly stable.

Between the light source and the selectable filters there is a receptacle to accept a small rectangular vial (cuvette) that contains the solution under study. The colours being transmitted through the sample (the ones not absorbed by the sample) are identified and their wavelengths and strengths are plotted in the same way as previously described.

Beer-Lambert's Law states that: "The extent of light absorption in a substance for a given wavelength depends on the thickness of the absorbing layer and the concentration of the substance in the layer".

Since the cuvette is 10mm internal dimension, the 'thickness' of the absorbing layer is 10mm. Since the wavelengths being transmitted are not the wavelengths being absorbed and since we wish to measure the absorption, the filters used in the path of the light from the source to the light sensor must be the **complement** of the transmitted colours (as seen by eye). **The filter that provides the maximum absorption (lowest light output) is chosen.**

As in flame spectrometry, the measurement of quantitative values of transmission or absorption requires calibrating pre-defined sample solutions of known concentrations. The 100% transmission (or zero absorption) setting is adjusted by using distilled water in the cuvette.

The calibration graph is a line joining the points measured from several known concentration samples. When this 'best fit' line is drawn, using the several pre-defined samples, the unknown concentration sample can be measured from the graph.

If a computer is used, when the graph is saved, it is remembered in the computer's program and the calibration curve for that material need not be repeated except as another exercise for students.

To convert 'transmission' readings into 'absorption' readings refer to the manual supplied with the instrument.

FORMULAE: relating Transmission and Absorption:

$$\text{Absorption} = \log(1/T) = \log_{10}(100 / \%T) = 2 - \log_{10}\%T \quad (\text{unit for Absorption is 'A'})$$

$$\text{Absorption} = (2 - \log \text{ of } \% \text{transmission}) \text{ or } A = 2 - \log \%T \quad (\log \text{ to base } 10)$$

If transmission is 0%, Absorption is infinity

If transmission is 1%, Absorption is 2A

If transmission is 10%, Absorption is 1A

If transmission is 100%, Absorption is zero

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**WAVELENGTHS AND FILTERS:**

The filters in the IEC Spectrometer are as follows:

(XXXnm) = peak transmission wavelength in nanometers ($m \times 10^{-9}$).

The table below indicates the peak of the wavelength transmitted and the last column indicates the frequency of each wavelength in Hz.

FILTER	POSITION on dial	WAVELENGTH in nanometres metres x 10^{-9}	FREQUENCY in Hertz x 10^{14}
Opaque	0	Zero transmission	
Deep Violet	1	435nm	6.89×10^{14} Hz
	2	445nm	6.74×10^{14} Hz
	3	465nm	6.45×10^{14} Hz
	4	488nm	6.15×10^{14} Hz
	5	519nm	5.78×10^{14} Hz
	6	552nm	5.43×10^{14} Hz
	7	573nm	5.23×10^{14} Hz
	8	590nm	5.08×10^{14} Hz
	9	675nm	4.44×10^{14} Hz
Deep Red	10	700nm	4.28×10^{14} Hz
Transparent	11	All wavelengths	

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CONSTRUCTION OF THE IEC SPECTROMETER:

The vertical pillar at one end of the instrument carries on its inner face either the colorimetry cuvette and electric light source (for colorimetry) or the viewing tube (for flame emission or Atomic Absorption). The light sensor and colour filter selection knob is mounted on the outer face. The light sensor may be removed from its receptacle at any time for viewing either the flame or the cuvette directly by eye.

The base of the unit has two inlet tubes. One for compressed air for the atomiser together with an air flow regulator screw and the other for LP or Natural gas for the burner. The burner's chimney may be unscrewed for removal and it will be seen at the lower end of the chimney that a spring clip is provided to convert the system from LP gas (bottle with regulator) to natural gas (regulator not required). The unit is supplied suitable for natural gas with the spring clip completely covering the two large air holes at the lower end of the burner chimney. For use on LP gas be sure that the clip is slid along the tube so that all air holes are unobstructed.

- The base provides a switch for the incandescent lamp used for colorimetry and a simple parking facility is provided for either the incandescent lamp for colorimetry or the viewing tube when not in use.
- 4mm Sockets for connection to the 0-100% / 0-5V.DC. voltmeter for measurements when a computer is not used. The 0-5V output can be used on a normal logger.
- 4mm sockets for 12V.AC. power in, say from any 12V transformer or power supply in the lab.
- Special socket for 240/12V.AC. PlugPak supplied with the unit.
- 'D9' type socket for data communication cable to the computer.

Other simple controls include **on/off** switch and **sensitivity** and **offset** (for setting null reading on the meter or computer). These are used for setting the amplification of the signal and the thresholds of measurements and calibration. A separate cable is provided for connecting an external signal source to the unit when data capture and analysis is required for other types of experiments not involving Spectrometry.

USEFUL HINTS:

- Since the instrument is very sensitive to the presence of elements in solutions, care must be taken in **cleanliness** of all equipment used.
- Always rinse the atomiser with distilled water thoroughly before use and between changes of samples. **Always wash and dry after use.**
- Never put samples left over from experiments back into storage bottles - they may be contaminated.
- Use only **distilled water (not tap water)** for cleaning atomiser and for dilutions.
- The contaminants in tap water is one of the student experiments. Always use 'AR' grade chemicals for the creation of reference sample solutions.
- **It is recommended to initially use the Voltmeter for measurements so that students are familiar with the data and with plotting the information. It is important in early study that the computer does not draw the students' attention from the experiment itself.**

Designed and manufactured in Australia

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