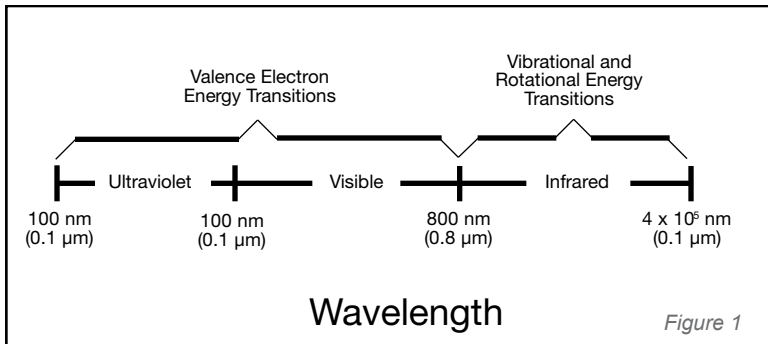


# How AMETEK's Western Research® Model 4000 Photometric Analyzer Works

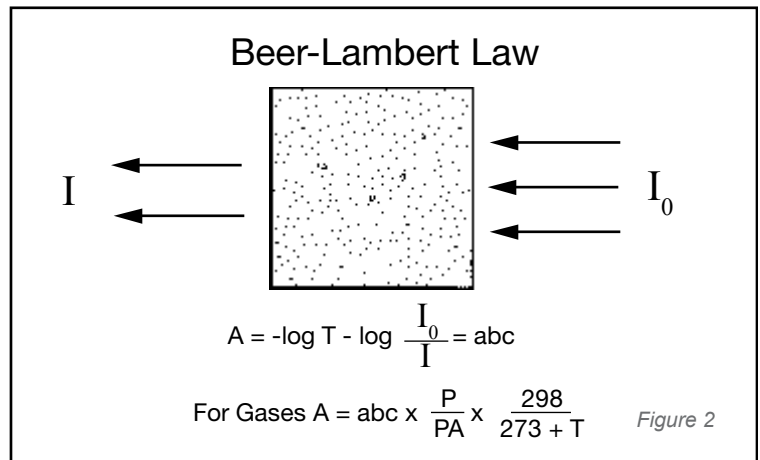
In photometric analyses, the interaction of electro-magnetic radiation energy with the component to be measured results in a loss of radiation (light) intensity. The AMETEK 4000 analyzer operates primarily in the near ultraviolet and visible region of the electromagnetic spectrum (200 to 700 nm) and extends into the near infrared to about 1,100 nm. Based on Planck's Law ( $E=h\nu$ ), the lower the wavelength of radiation (the higher the frequency), the higher the energy of the photon.

As shown on figure 1, photoelectron energies in the UV and visible regions are at levels corresponding to energy transitions of valence electrons. In general, these transitions



are broader and not as common as the rotational and vibrational patterns in the infrared region. With the relatively high photoelectron energy associated with the UV/visible region, metallic discharge lamps with intense line emissions and long life can be used as light sources. Photodiodes, which are sensitive, highly stable, and exhibit very low noise, temperature coefficients, and fatigue characteristic, are used as detectors.

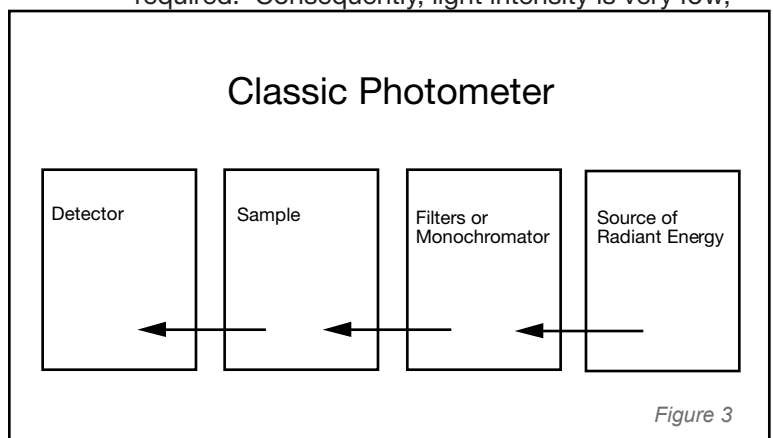
Beer's Law shows the quantitative way the loss of light intensity varies with concentration and pathlength (figure 2). Basically, light intensity transmitted through a windowed cell is an



exponential function of pathlength and concentration. To provide a function linear with pathlength and concentration, the nondimensional absorbance

(A) is defined on the figure; "a" is the molar extinction coefficient and is constant for a given compound at a given wavelength and within a given matrix (liquid, solid, or gas), "b" is the pathlength in centimeters, and "c" is the concentration in moles per liter.

A laboratory photometer or spectrophotometer usually has a broadly emitting source such as a hydrogen discharge or tungsten lamp (figure 3). The wavelength usually is isolated by a prism, grating, or interference filters. For good monochromaticity, narrow bandpasses are required. Consequently, light intensity is very low,



and usually highly sensitive detectors such as photomultipliers must be used. Photomultipliers have high temperature coefficients and fatigue, and require well stabilized high voltage power supplies. Light from the monochromator passes through a window cell (a cuvette) before striking the detector.

### Design Criteria For Photometric Analyzers

Early in the development of process stream analyzers, it was realized that one could not take a laboratory instrument and put it in the field. You had a laboratory instrument sitting in a hostile environment for which it was not designed. Over the years, a design criteria philosophy was developed. Among these are:

- || Ruggedness
- || Serviceability
- || Accuracy, Reproducibility, Stability
- || Linear Readout
- || Fast Response
- || Ease of Calibration
- || Simplified Sampling Requirements
- || The instrument must be rugged; that is, very well built. It is installed in a very hostile environment, often with high vibration, and is not treated with tender loving care one so often finds in the laboratory. Associated with ruggedness is the need to meet the electrical classification of the area in which it is installed.
- || The instrument must be readily maintained. It is designed so that parts are easily accessible and is not too complicated. The technician taking care of it then will have a very positive feeling toward it, and the unit will be maintained.
- || Stability and reproducibility of a process stream analyzer often must well exceed that of a laboratory instrument. Since analyzers often are discharged to the atmosphere, accuracy often is limited in the gas phase measurement because of variations in absolute pressure. Unless an absolute pressure control is used, these variations can amount to  $\pm 2$  percent, often adequate for most monitoring applications. Reproducibility must be high, particularly for good process control when the analyzer is often integrated in a control loop. Stability in a plant installation, where temperatures can vary greatly and voltage fluctuations and vibrations can occur, must be such that the instrument provides reliable readings day in and day out.

Our experience has shown that most operators do not like to use calibration curves, particularly if highly nonlinear. They like to have a direct readout, and will use the instrument more readily if the output is linear with concentration of the component measured.

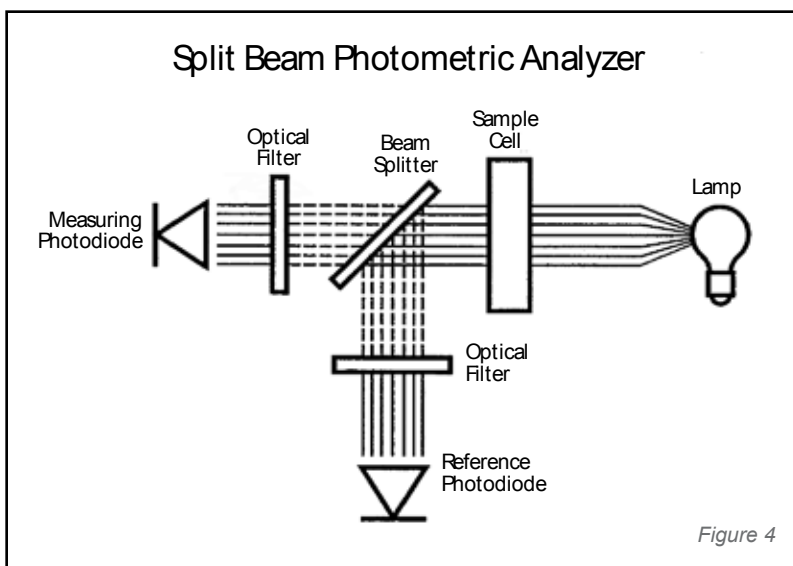
- || Fast response is needed particularly for good process control. Analyzer response usually is adequate; however, sample lag, the time it takes the sample being extracted from the process to reach the analyzer, most often limits response time. A fast "slip" stream with a high sample flow bypassing the sample cell often is used to increase speed of response. In monitoring and/or controlling film thickness, a millisecond response often is required because of the high speed at which the film is being produced.

From experience we have found that operators retain a great deal more confidence in an instrument which can easily be checked for response and calibration with a minimum of effort.

- || Last, but not least, is a simple sample system. Those who have been in the field for a long time have found that sampling problems account for some 80 to 90 percent of the problems in a process stream analyzer installation. A design minimizing the need for extensive sample conditioning to the analyzer minimizes down time and gives higher reliability.

### Functional Diagram of Split Beam Photometric Analyzer

The AMETEK 4000 photometric analyzer (figure 4) was designed after an evolution of a large number of photometric analyzers extending from about 1942



- the design still is evolving. The split beam configuration is shown here, and, as seen later, meets many of the criteria listed earlier. Light from the source, usually a metallic lamp discharge with discrete lines of emission, passes through the windowed sample cell, usually with quartz windows to provide good ultraviolet light transmission. Light transmitted through a cell is split into two channels by a beam splitter, a quartz plate coated with Inconel. The transmitted portion of light passes through a filter bundle which isolates the one wavelength where the component to be measured absorbs strongly, so that light of this wavelength is the only light striking the photodiode. The photodiode produces a current precisely proportional to the intensity of light striking it. Photodiode current is then fed to a logarithmic amplifier, converting the current to a voltage varying as the logarithm of the current and, in turn, as the intensity of light at the measuring wavelength. This logarithmic conversion linearizes the Beer's Law characteristic described previously. The reference channel is functionally identical to the measuring channel in that it has a similar filter bundle, photodiode, and amplifiers. However, this filter bundle isolates a different wavelength, one where the material measured does not absorb or absorbs very weakly compared to that of the measuring wavelength. The amplifier outputs are subtracted from one another, providing the logarithm of light intensities at the two wavelengths. This differential signal is fed to the controller where the instrument is electronically zeroed and spanned. The controller output is then signal fed to the recorder and/or process controller. The advantage of the split beam configuration is derived from looking at two different wavelengths simultaneously. The instrument inherently has the means of compensating for particles or bubbles passing through the cell, or light fluctuations which affect the intensity of light at both wavelengths equally and tend to be canceled out.

Figure 5 shows some of the optical components used in this system. On the left is a 2-cm thick quartz bottle with chlorine used to block out wavelengths where chlorine absorbs (300 to 375 nm). Clockwise from the chlorine bottle there is a quartz bottle with a

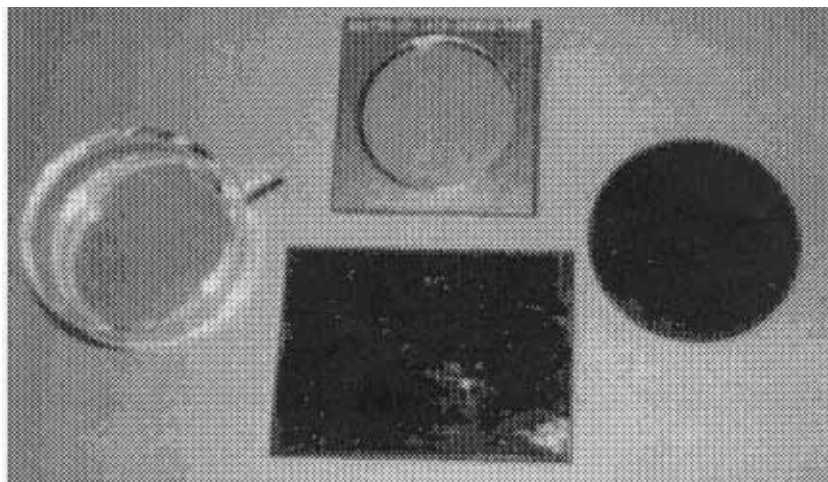


Figure 5

nickelous sulfate solution, a glass filter, and nickelous sulfate crystal sealed between two quartz discs, again all used for blocking wavelengths. The next element is a beam splitter, and the last is an interference filter, which has become very common. Interference filters have blocking built in; but for ultimate isolation of a specific wavelength, external blockers are often added to improve linearity over wide absorbance ranges. Incidentally, thin film coating technology has been greatly extended in the past 30 years, and interference filters with excellent band pass and blocking characteristics now can be purchased fairly reasonably.

Figure 6 shows some of the light sources used. From left to right; an ST49 UV lamp is the most commonly used. It is a medium pressure mercury discharge lamp used to produce a large variety of

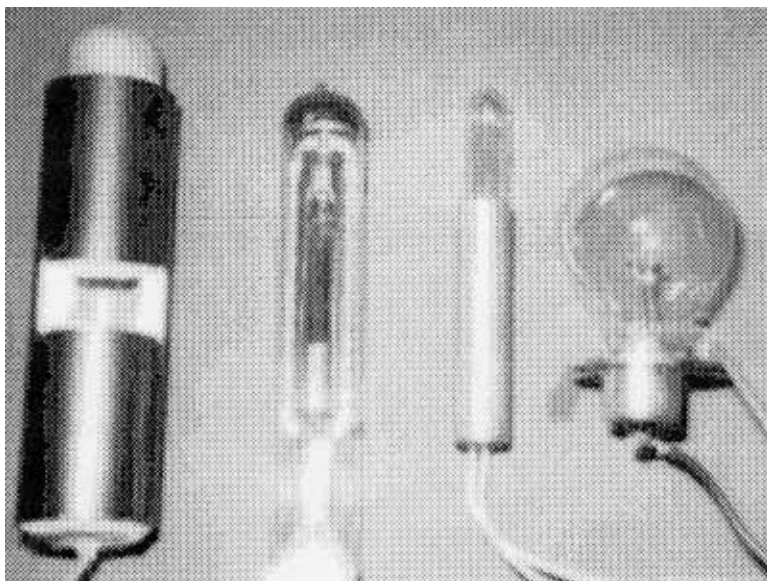
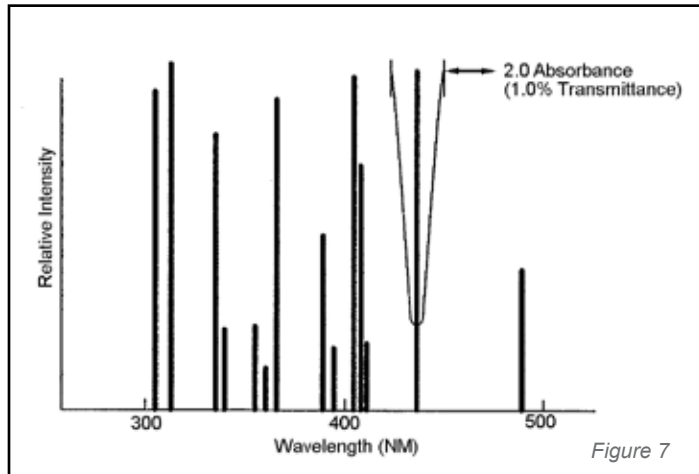


Figure 6

# How AMETEK's Western Research® Model 4000 Photometric Analyzer Works

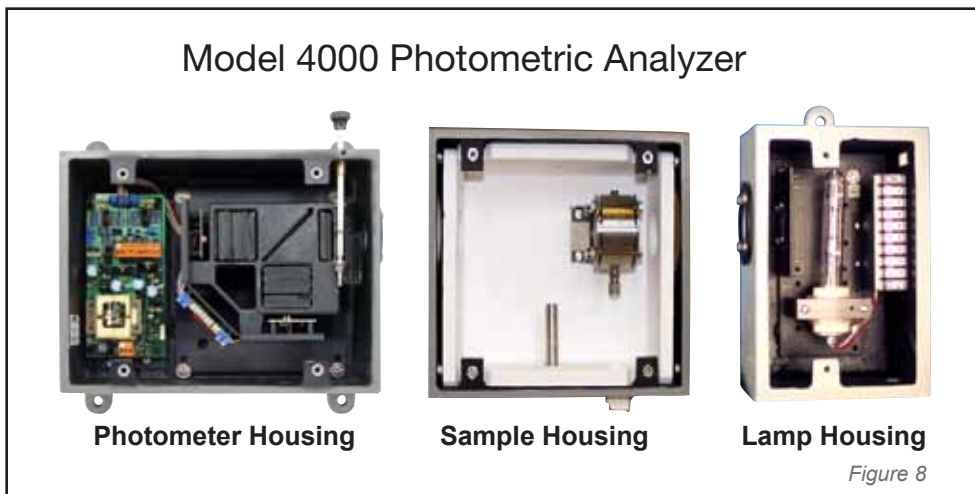
lines for monitoring in the UV region. The next lamp is made by UV Products and provides the 228-nm cadmium discharge line in several applications. The third is a low pressure mercury discharge lamp, a Penray lamp used primarily for the mercury resonance line at 253.7 nm. The last is a rugged tungsten lamp used to provide the light for color and near infrared measurements.

Figure 7 shows how a line emitted by a metallic discharge lamp can be isolated readily to provide highly monochromatic light without a monochromator. The advantage of using metallic discharge lamps is that the discharge lines are often several orders of magnitude in intensity above background light, and very narrow. We cannot even talk in terms of half-bandwidth. Discharge lines have one-tenth bandwidths less than 0.1 angstrom. Furthermore, wavelengths are fixed and unchanging. Based on energy levels available for a valence electron to move, which are fixed regardless of temperature, etc, the light wavelength emitted results from the fixed difference in valence electron energy levels.



To summarize split beam design advantages :

- || There is internal compensation for particulate matter and bubbles in the sample stream.
- || There is internal compensation for dirty windows.
- || There is internal compensation for variations of light intensity.
- || No moving parts increases reliability.
- || Modular design (figure 8) is highly flexible and allows for analyzing high temperature and high pressure samples without potential damage to electronics or optics.



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