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# USE OF INCANDESCENT LAMPS TO MEASURE OPTICAL AND DETECTOR DRIFTS IN PHOTOELECTRIC SPECTROMETERS

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16. Abstract

An evaluation of incandescent lamps as photometric reference devices in the measurement of drifts in photoelectric spectrometers is reported. The selected lamps are stable to better than 0.4-percent relative standard deviation for at least 1000 on-off cycles, each of 30-sec duration. This is equivalent to a use time of about 1 year in the drift detection system. A procedure for routine monitoring of optical and detector drifts in spectrometers is suggested. The drift detection system provides an alternative way to correct for drifts affecting analytical calibrations without the need to analyze standard samples.

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nonreproducibility of results over extended time periods can be caused by instrument system drift. With multichannel photoelectric spectrometers, this problem is compounded by degradation of the individual photomultiplier tube sensitivities and the electronic drift of the entire digital readout system.

The internal standard technique is routinely used to correct for some types of fluctuations found in atomic emission spectroscopy. Compensation for variations in conditions from sample to sample is achieved by using this technique; however, the internal standard procedure offers no correction for the independent drifts of the separate photoelectric readout channels.

Another method of reducing the drift problem assumes that the entire spectroscopic analysis system will remain relatively stable for a short time period. If negligible drift occurs over a short time period, chemical analysis of unknown samples can be determined by comparison with standard sample data within that time. This technique requires preparing standard samples similar in composition and concentration to the unknown samples. The comparison standard is prepared separately but processed along with the unknown samples. This adds preparation time and effort to the analysis procedure.

Rather than using this technique, it is more expedient initially to prepare and record analytical calibration curves for each element over the concentration range of interest. Subsequent corrections are made for systematic drifts by rechecking a few calibration standards and shifting the analytical curves to compensate for drift. There are numerous variations of procedures for controlling analytical curve adjustments. A standardized procedure is found in reference 1.

Recent improvements in the direct-current arc sources (ref. 2) have resulted in more constant sampling, vaporization, and excitation conditions from sample to sample. Little is thus gained by using the internal standard technique mentioned earlier. When the improved source is used, widely varying sample compositions and concentrations produce useful data. The atomic emission line intensities of the unknown samples are compared with those line intensities of standard samples whose values are stored for long periods of time in the memory of a digital computer. For this system to operate without requiring recalibration for weeks, months, or years, it is mandatory to control or correct the instrumental drift. Two major types of drift necessitate correction: (1) drift which results from optical system transmission changes, optical misalinement, or changes in the analog to digital converter and affects all the readout channels in a proportional manner; and (2) drift which results primarily from individual photomultiplier sensitivity changes and individual channel electronic component changes and affects each of the channels differently.

A technique is described which allows for correction of time-dependent drifts in the optical system and the electronic system associated with a direct-reading spectrometer.

The primary photometric reference element is a tungsten-filament lamp located inside the spectrometer. The reference lamp illuminates all the photomultiplier detectors used for the readout of spectral line information. This constant irradiance source is used to calibrate each photometric readout channel and the electronic readout system prior to analyzing a group of samples. In addition, the constant intensity source is used to correct for drift in a specially positioned photomultiplier used to monitor changes in the optical system alone. This photomultiplier detector is illuminated by unused light from the emission source which is collected by the optical system but is reflected from the zero order of the diffraction grating.

The reference lamp has been shown experimentally to be stable to better than 0.4-percent relative standard deviation for a use equivalent to several months of operation. The reference lamp can itself be checked for drift by using a second identical lamp also located inside the spectrometer.. With this apparatus, corrections for changes in the optical system characteristics and electronics which are the same proportionally for all the detector channels can be made. The apparatus also provides a means of compensating for drifts which are different for each of the detector channels over long time periods without the need of running recalibration standards.

## APPARATUS AND PROCEDURE

### Experimental Arrangement of Monitor System

Figure 1 is a schematic diagram of the spectrometer showing the locations of the added components (heavy lines) in the spectrometer. The detector drift correction system uses tungsten-filament lamps as constant irradiance sources (General Electric lamp No. 1613). One reference lamp is used in routine checking of drifts, and the other is used sparingly to provide an occasional check on the stability of the first. This redundancy also provides insurance against loss of reference level in the event of catastrophic failure of one lamp. These lamps are mounted near each other inside the spectrometer. The light from the reference lamp passes through the exit slits and illuminates the photomultiplier tubes associated with 22 detection channels (only 2 channels are represented in fig. 1). Time integration of this light provides the necessary information for correction of individual channel drifts.

The optical monitor detector in figure 1 is installed to intercept the zero-order (nondispersed) light reflected from the grating during excitation of samples in the arc chamber. The detector provides a measurement of this light which indicates the changes in optical transmission between the arc source and itself. Since the optical monitor detector is a photomultiplier tube, it is also subject to long-term drift. This source of

drift is kept under control by illuminating the optical monitor detector and calibrating it with the reference lamp at the same time the element detector channels are illuminated. A light baffle is mounted between the reference lamps and the optical drift monitor tube to reduce the light intensity on the optical monitor detector to a more suitable level. The same baffle allows the reduction of reference light intensity on the most sensitive photomultiplier readout channels so that the output signals stay within the dynamic readout range.

## Routine Calibration Procedure

In operation, one reference lamp is used to calibrate the detector-readout system immediately before a series of samples is analyzed. Figure 2 is a flow chart summarizing the data treatment for the drift correction procedure. The reference lamp is turned on for 15 seconds and allowed to warm up. Then a 15.00-second integration of the light is made by all the detection channels and the optical monitor detector, as indicated in figure 2 by calibration step 1. The data from all detectors are subsequently recorded in digital form. These data are then compared with initial reference data taken at an arbitrary point in time. A drift correction factor is then calculated and applied to the data for each separate readout channel and for the optical monitor channel as indicated by the correction blocks A and B, respectively. This drift correction procedure can be used to correct for any type of change in the detector-readout system.

The calibration of the external optics, including changes in reflectivity of the grating, is done in a second calibration step of figure 2 in which a blank sample is excited in the arc source. The signal from the optical monitor detector previously calibrated is compared with initial arc source reference data taken at an arbitrary point and tested for significance using standard statistical procedures not discussed herein. If the indicated drift is significant at an arbitrary level, a proportionate correction is made to all detection channels in subsequent analytical calculations. This step is diagrammed in block C.

The excitation of a blank sample in the arc also serves to establish and to control baseline responses of the total system in the absence of analytical signals. The light emitted by the arc in this step, in addition to being sensed by the optical monitor detector, is simultaneously sensed by all detector channels. The recorded signals from these channels can be used to correct for drifts in the baseline response. This step is indicated in block D of figure 2.

## Stability Tests on Incandescent Lamps

The long-term stability of the photometric monitor system was estimated by determining drift of the lamps in cyclic operation over a period of about 25 hours. During this time the lamps were in actual operation for a period of about 9 hours. Assuming four drift checks per 8-hour working day, this is equivalent to operation of the monitor system for about 1 year.

The warmup time of the reference lamp was determined to be about 15 seconds from the data in figure 3. These data showed that repeated 15.00-second integrations of light intensities are within 1 percent of the mean values irrespective of the warmup time. A warmup time of approximately 15 seconds was used in the long-term test because after this time the integrated intensities were relatively independent of warmup time.

The accelerated long-term test of the drift correction system was conducted by turning the lamps on for a 15-second warmup time and a subsequent 15.00-second integration time. The recording of the integrated intensities required about 1 minute during which time the lamp was off. This cycle was repeated over 1000 times.

The lamp intensities were read out by integration of the photomultiplier tube currents stored on capacitors shown in figure 1. The charge integrated on the capacitor was digitally recorded on punched paper tape.

The analog drifts associated with this system are primarily in the power supplies for the incandescent lamp and the photomultiplier detectors and in the analog-digital converter. During the time of the long-term tests, the lamp power supply provided 1.1235±0.0003 ampere, and the photomultiplier supply was stable to 1020±0.1 volt. In addition, the gain of the analog-digital converter was checked for drift on every cycle by application of a constant test voltage, and the relative readout error from this source remained less than 0.1 percent.

## RESULTS AND DISCUSSION

The relative standard deviations for the measured light from the two lamps tested were 0.26 and 0.31 percent for 1000 cycles with an on time of 30 seconds per cycle. These results indicate that the lamps are satisfactory as a photometric reference for monitoring the long-term drift in a photoelectric spectrometer. In extrapolating the short-term data of the lamps to estimate long-term stability, it is assumed that changes in intensity occur only when power is applied to the filament. Therefore, the repeatability obtained when operating the lamps for 1000 cycles over a 24-hour period is indicative of the stability that can be expected for 1000 cycles of operation over a longer period of time, that is, 1 year in the intended application.

The primary cause of intensity degradation from incandescent lamps is the evaporation of tungsten and its deposit on the lamp glass. This process can be dramatically retarded by operation of the lamp at less than rated voltage. Figure 4 is a nomogram showing the relations between the fraction of rated voltage applied to the lamp and the life of the lamp. Reducing the operating voltage to one-half the rated voltage extends the lamp life by about 4000 times compared with operation at rated voltage. The nomogram also shows that at the reduced voltage the decrease in light detected by the photomultipliers is a factor of about 50. This decrease in intensity is not limiting in this application. The dependency of current factor on applied lamp voltage is also indicated on the nomogram for comparison purposes.

The experimental error in making the 1000-cycle tests included the drifts in the photometric readout system, as well as changes in lamp intensity. The important causes of measurement error were the power supplies for the lamp and for the photomultiplier detectors. A 0.1-percent change in current applied to the lamp filament caused an intensity change of about 2 percent. This relatively large change was caused by the shift of spectral distribution of the lamp intensity, in addition to the change in total spectral irradiance with changes of lamp current. In the cyclic tests, the lamp current was kept within about ±0.03 percent of the operating current. This magnitude of current change would cause maximum relative errors in the readout of 0.6 percent. Changes of 0.1 percent in photomultiplier supply voltage produced changes in readout of about 1 percent. Since this supply voltage was monitored and kept within 0.01 percent of the applied voltage, the maximum error expected from drifts in the photomultiplier supply was about 0.1 percent.

These error estimates indicate the importance of maintaining close control of the current through the reference lamp. If the lamp current is properly controlled, drifts in the photomultiplier supply voltage can be tolerated during the actual experimental operation because they will be compensated in the routine use of the photometric monitoring system. In the spectrometric system at the Lewis Research Center, the lamp current is automatically recorded for every lamp cycle as a check on lamp stability.

In addition to long-term stability, several other requirements need to be met for an irradiance standard for photometric calibration of spectrometers. The light intensity must provide sufficient intensity in the blue region of the spectrum so that blue-sensitive photomultipliers give sufficient response at the same gain as is used for chemical analysis. This constraint is important because it is not desirable to change the voltages applied to the photomultipliers for the purpose of adjusting their gains when making photometric calibration checks. Figure 5 shows plots of the approximate spectral distributions of the lamps at color temperatures of about $1700^{\circ}$ and $2500^{\circ}$ C, corresponding to filament currents of 1.163 and 1.730 amperes, respectively. Also shown is the spectral response characteristic of the photomultiplier type used in spectrochemical analysis.

Because most of the lamp light is in the red region of the spectrum and the detector sensitivity is in the blue, only the relatively small fraction of total light, shown by the blackened area in figure 4, is detected by the photomultipliers. With the high gains available with photomultiplier tubes, the light available is more than sufficient. Comparison of the overlapping areas between the two color temperature curves with the photomultiplier spectral response curve indicates the disproportionately large changes in the amount of light measured by the photometric monitoring system for relatively small changes in lamp current.

The location of the lamps inside the spectrometer must be such that each of the photomultipliers receives enough incident light so that the integrated tube currents are in the range normally measured in analysis. In the spectrometer at the Lewis Research Center, the integrated output currents of the 22 detection channels differed by a factor of about 200, from the least sensitive to the most sensitive, when illuminated for 15.00 seconds by the reference lamp. (This variation in channel sensitivity included the attenuating effect of special spectral cutoff filters in front of some of the photomultipliers as well as varying detection sensitivities and gains of the tubes.) This range of sensitivities is about the maximum to fall within the dynamic range of the readout system, defined by a precision of 1 percent or better. In addition to proper positioning of the lamps, the absolute light level can be made to fall within the readout detection range by small adjustments in lamp current, positioning of baffles, and proper selection of integration time. For convenience in automating the photometric calibrations along with analysis of samples, the lamp integration time was made the same as the analytical integration time.

Low thermal output is another important characteristic required with the lamps located inside the spectrometer. An increase in ambient temperature inside the spectrometer can change the optical alinement and thus affect the accuracy of analytical determinations. The system described herein uses 3 watts of power for 30 seconds for each calibration cycle. This small quantity of input heat did not raise the interior temperature of the spectrometer $\pm 0.1^{\circ}$ C during the 1000-cycle tests as determined by experimental monitoring.

During the course of study of the problem, several photometric reference devices were considered and evaluated. A summary of these results is presented in table I.

## CONCLUSIONS

An incandescent tungsten-filament lamp, operated at about one-half the rated voltage and at two-thirds the rated current, can serve as a precise irradiance source for monitoring electronic drift of a photoelectric spectrometer. The lamps are stable to within

0.4-percent relative standard deviation for a period of at least 8 hours. This time period is equivalent to a use time of approximately 1 year of cyclic operation in a photometric monitor system. The error reported also includes the 24-hour drift of the photometric system used in recording the data.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, January 28, 1971,
129-03.

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TABLE I. - SUMMARY OF EVALUATIONS OF VARIOUS PHOTOMETRIC REFERENCE DEVICES

| Photometric reference device | Description | Remarks on evaluation |
|---|---|---|
| Photodiode detector | Photodiode, 1 $cm^2$ active area (United Detector Technology) | Good stability if temperature compensated. Dark current doubles for $10^{\circ}$ C temperature change. Not sufficiently sensitive to accurately measure zero-order light through 25-$\mu$m entrance slit of spectrometer. |
| Radioactive beta ray light source | 400 $\mu$Ci $Kr^{85}$, 10 ft-L, green phosphor, No. 5000 (American Atomics Corp.) | Decay rate very constant but not theoretical; use of empirical decay rate necessary. Insufficient light intensity in blue. Temperature coefficient of 0.2 percent/$^{\circ}$C. |
| Halogen-filled tungsten-filament lamp | 12 W, 2 A at 6 V, No. 1973, quartz envelope, bromine filled (G. E. Co.) | Excessive heat output at full power; halogen cycle inoperative at less than 85 percent of full rated power when envelope temperature drops below about $350^{\circ}$ C. |
| Tungsten-strip lamp | Pyrometer lamp, 120 W, 20 A at 6 V, glass envelope (G. E. Co.) | Excessive heat output for location inside spectrometer; external location limited by critical alinement of emitting surface with small-aperture optical instrument. |
| Tungsten-wire-filament lamp | 9 W, 1.5 A at 6 V, No. 1613, glass envelope (G. E. Co.) | Good long-term stability when operated at reduced voltage and current. Adequate intensity in blue. Requires close control of current through lamp. |
| Tungsten-wire-filament lamp | Miniature optical pyrometer lamp, 0.15 W, 1.5 V at 0.1 A max, glass optical windows (Micro Optical Pyrometer Co.) | Insufficient light output for calibration of photomultipliers in application described in this report. |

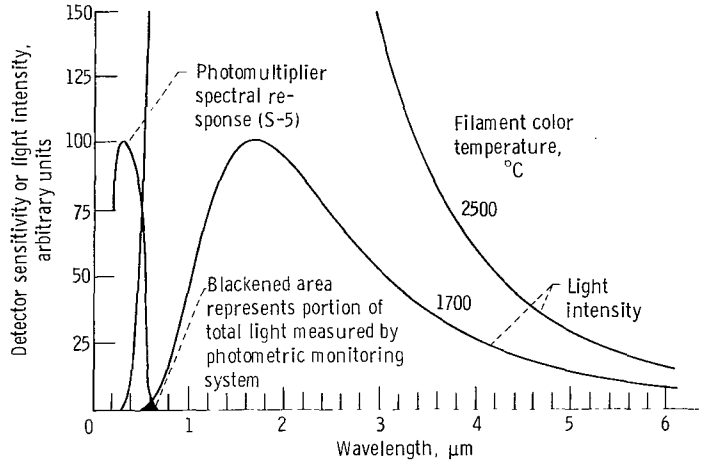


Figure 5. - Light intensity at color temperatures of 1700° and 2500° C for tungsten filament voltages of 2.75 and 6.00, respectively, and photomultiplier spectral response for S-5 photocathode.



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