SPECTREX

REPRINT FROM:
JOURNAL OF CHEMICAL EDUCATION

Qualitative Emission Spectroscopy in Freshman Chemistry

Leonard F. Druding
Rutgers University
Newark, New Jersey

Atomic spectra represent a key portion of the experimental evidence behind Bohr's original atomic theory, and at least a brief discussion of the origin of such spectra is included in most introductory chemistry texts. Aside from a few text illustrations or lecture slides, the students are given little further exposure to spectroscopy. For qualitative and quantitative elemental identification, emission spectroscopy has long been a very important technique, yet it is ignored in all but a few advanced analytical courses.

The principal reason for the exclusion of atomic emission spectroscopy has been the lack of suitable instruments. For the introductory courses, there have been such visual spectroscopes as those of the Bunsen-Kirchoff types or the Fisher Duo-Spectranal. These have such low energy excitations that for practical purposes they can only be used for the alkali and alkaline earth elements. The instruments more normally used for analytical work are complex instruments which use various kinds of electric arc sources and have photographic or photocell recording of the spectra. These instruments are frequently too expensive or unnecessarily complex for use in the undergraduate laboratories. The development of photographic plates or positioning of the photocells is too time-consuming for more than a few students.

Recently, there has been available a relatively low cost, easy-to-operate instrument that is suitable for student use in the introductory courses, the Vreeland Direct Reading Spectroscope, Model 6A, manufactured by the Spectrex Company, Redwood City. Cal. This instrument has a grating optical system with an AC carbon arc excitation of solid or liquid samples. This source allows for the observation of the spectra of most of the metals and many of the nonmetals. Visual observation of the spectra in the region 390—700 nm is the usual mode, but accessories for camera attachments, video spectral scanning, as well as for semi-quantitative analytical spectroscopy are available.

The key feature of this instrument which commends it to student use is a set of standard spectrograms on special transparent mylar films built into the instrument. These are illuminated by the continuous spectrum of the carbon arc and their lines appear in true colors. Two such films are mounted on either side of the observed spectrum, each containing 40 spectra. They cover 57 elements, and include Angstrom scales, key line scales, and band scales for certain elements. Each standard spectrum includes the D lines of sodium at 5889.95 and 5895.9 A which are aligned with those observed as a consequence of sodium incorporated in the carbon electrodes.

There will then be a direct correlation between the observed lines and those on the standard spectrograms in the visible region. The key lines scales contain the strongest or most persistent lines for each of the elements and are used for a quick qualitative scanning to identify which elements might be present.

This instrument has been used in the general chemistry laboratory in the second semester as an optional part of qualitative analysis. The unknown samples usually used are from a collection of metal alloys from a long-defunct qualitative unknown supplier. Smith and Underwood Laboratories [Troy, Mich.] have both quantitative metal alloys and qualitative unknown solutions which could be used. The instrument is operated in a fume hood because some of the elements generate toxic vapors and small amounts of cyanogens are generated by the electrodes.

In student operation, the unknown sample is placed in the appropriate sample holder, the arc is ignited and sample moved into the arc. The student observes the resulting spectrum and positions one of the Key Lines standard spectrograms so that the sodium D lines on the film coincide with those observed in the spectrum. He then looks for correlations between the strong lines of the various elements on the Key Lines spectrogram and his experimental spectrum. With the two sets of Mylar film spectra on either side of the observed spectrum, a Key Lines spectrum can be left in place on one side while the other film is turned to the spectrum of the suspected element. If there is correct registry between the lines on the spectrogram and the unknown spectrum, that element is identified. The student then turns to the film spectrograms of other suspected elements until the unknown has been identified.

About 20% of the students in the course elected to do this spectroscopic experiment, and almost all observed a spectrum at least once during the course. Most of those students completing the experiment correctly identified all of the elements in their alloy sample. A number of students were motivated to bring in their own samples of metals or minerals for analysis.

The use of this spectroscope has enabled this department to not only demonstrate atomic spectra to the students but also to show some very practical uses of this method of analysis. The time, convenience, and ability to analyze about twice as many elements as the traditional qualitative analysis scheme, using far smaller samples also demonstrates to the students why the equal scheme is no longer commercially used for elemental identification.