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Barbara Branstetter

October 1979

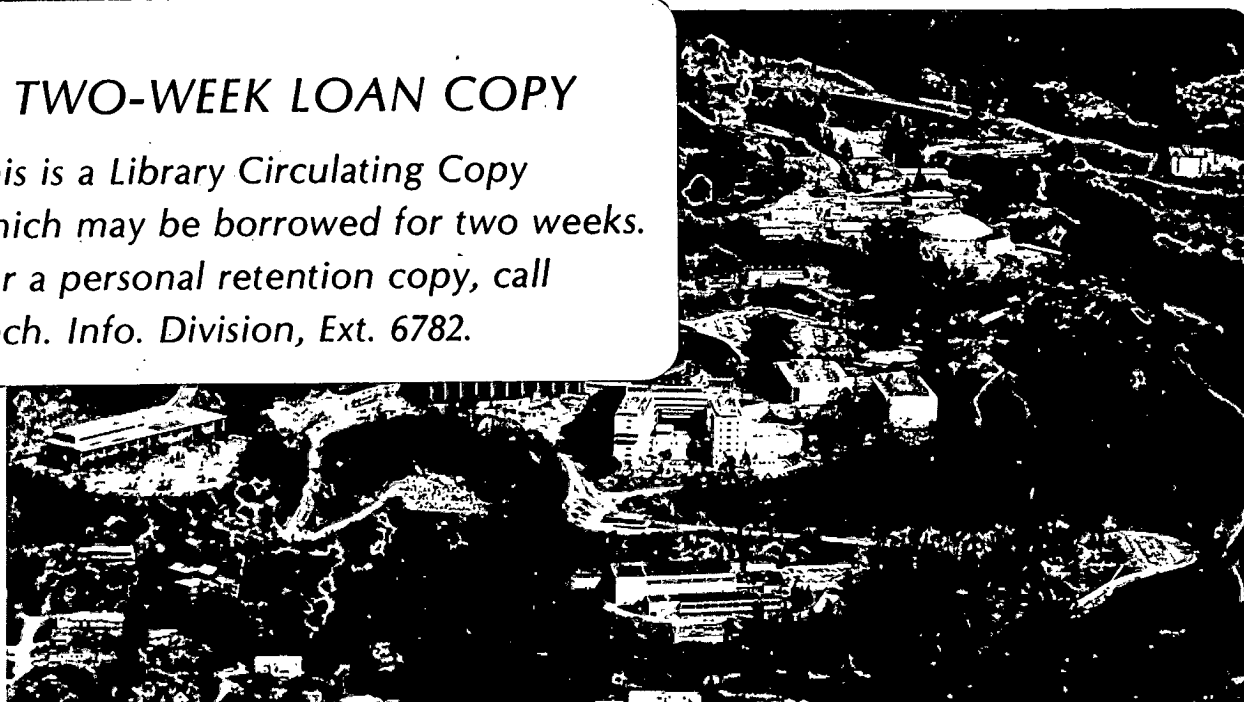
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Application of Zeeman Atomic Absorption Spectroscopy
For Measurement of Cadmium in Wheat

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Introduction

Since the development of the Zeeman technique,^(1,2,3,4,5) there have been accelerated efforts to utilize its unique capability of detecting atomic elements directly from a host of materials. Because these materials need no prior chemical treatment, the technique has caught the attention of environmental researchers who are increasingly called upon to monitor various toxic elements that might migrate into basic food staples. For instance, research on trace amounts of Cadmium in wheat, lettuce and potatoes is now being conducted at Battelle in Columbus, Ohio (M. McKown, personal communication 1979).

The purpose of this paper is two-fold: to explain the procedure used at LBL to calibrate the Zeeman Atomic Absorption system to measure Cd prior to its use at Battelle and to provide a data base independent of Battelle for future cross-comparison of results by both laboratories.

Principles of ZAA Spectroscopy

ZAA spectrophotometer utilizes the Zeeman effect to correct automatically for background interference. The source of light is a lamp emitting the characteristic emission of the Cd lines. The lamp is in a uniform magnetic field of about 10 KG. For Cd detection, the 228.8 nm line is used. The applied magnetic field splits the line according to

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changes in the quantum number M . The transition with $\Delta M = 0$ has the same frequency as the transition in zero-magnetic field (i.e., $\nu = \nu_0$, same as conventional AA). This light is linearly polarized with its electric vector parallel to the magnetic field. The transitions with $\Delta M = \pm 1$ emit light linearly polarized perpendicular to the magnetic field and are shifted to $\nu_0 \pm \Delta\nu$, where

$$\Delta\nu = (eh/2 \mu c)/2\pi \text{ sec}^{-1}.$$

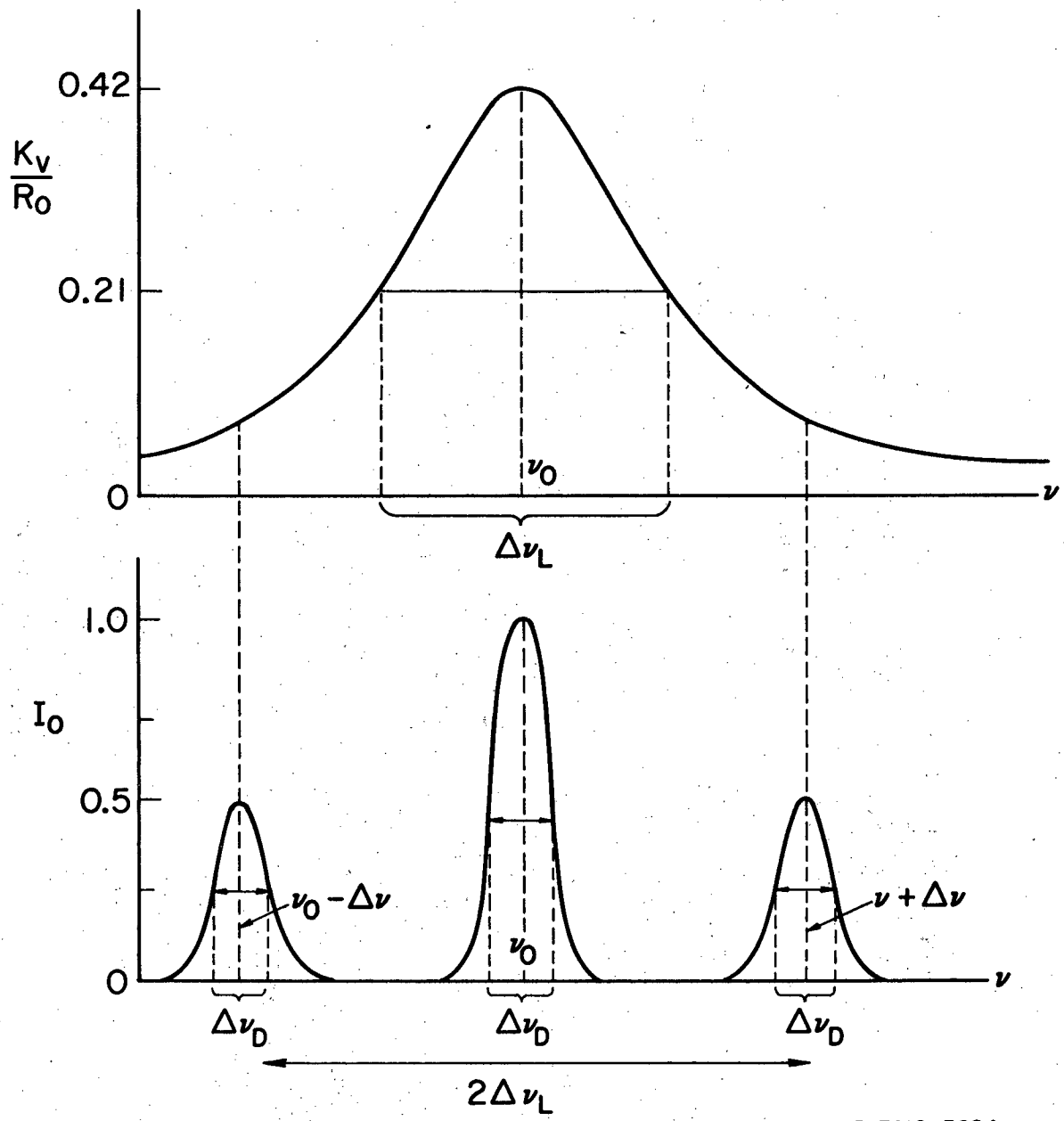
Thus, by means of a linear polarizer, either the unshifted Zeeman component (π component) polarized parallel to magnetic field or the shifted components polarized perpendicular to the magnetic field (σ components) can be selected. Under such conditions, the difference in absorption between π and σ components is proportional to the concentration of atomic vapor with the complete background rejection because the presence of background molecules and particles in the atomic vapor essentially affect both π and σ components equally.

Figure 1 shows the graphic representation of 228.8 nm Cd emitted in presence of magnetic field with a typical absorption profile. Figure 2 shows the block diagram of the instrument.

Calibration of ZAA System

The Cd system was found to be stable and reproducible at the instrument setting in Table 1 below. These adjustments were determined by prior experimentation with standard solutions of Cd of 0.1 ppm, 0.5 ppm and 0.025 ppm⁽⁶⁾. The well-analyzed NBS Standard Reference Material (SRM) 1571, "Orchard Leaves" was chosen because of its obvious agricultural and organic similarities to wheat. A locally distributed

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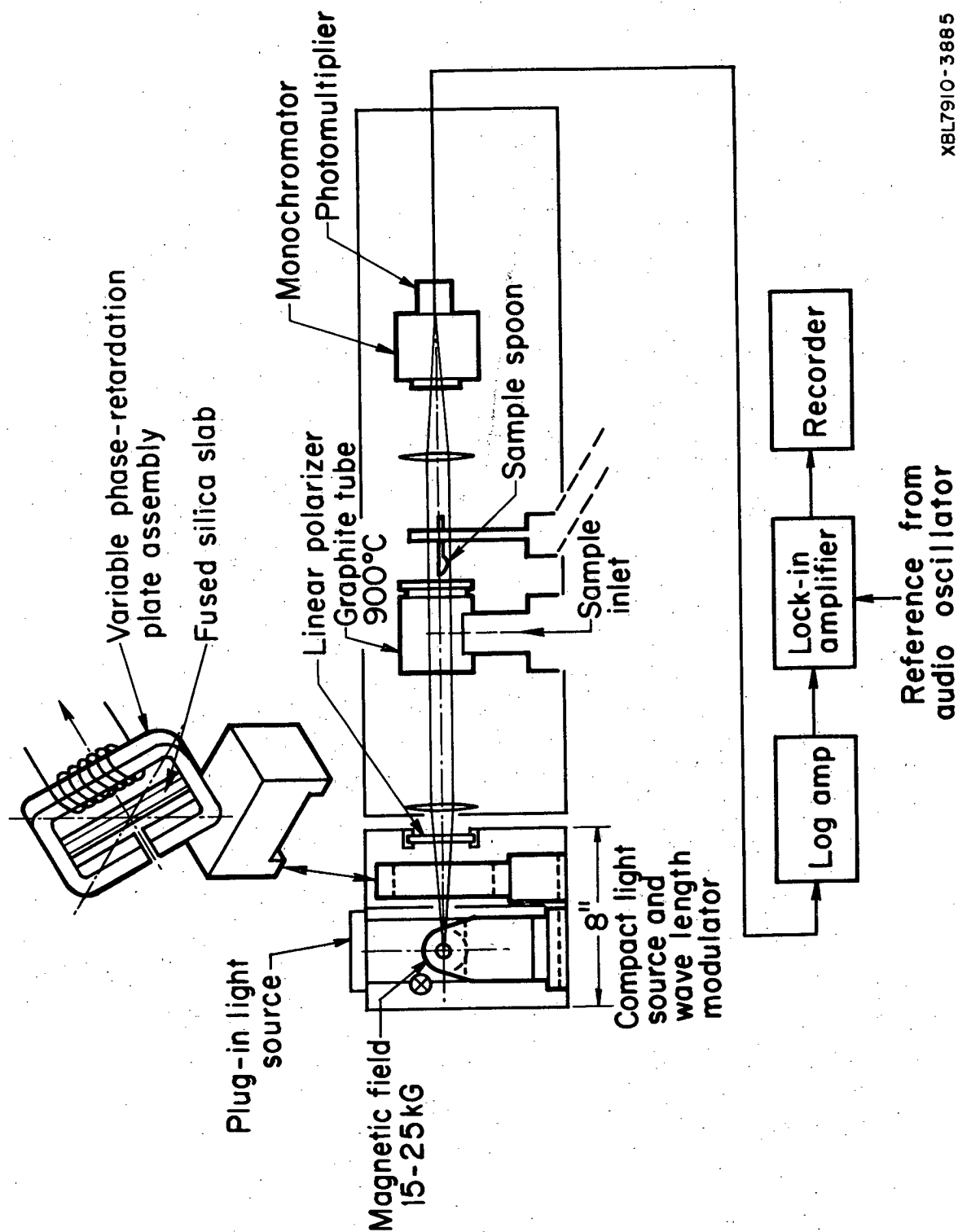


XBL7910-3884

Figure 1

Figure 2

Block Diagram of ZAA Instrument



XBL7910-3885

wheat sample was randomly collected to be tested for its behavior during various stages of the analysis. The weight of the sample needed to produce a Cd peak was determined and effects due to the direct insertion of wheat into the furnace without additional sample preparation and of possible interference due to smoke resulting from the charring stage were checked.

Table 1: ZAA Instrument Setting

| <u>Function</u> | <u>Setting</u> |
|----------------------------|--|
| Cd Lamp Setting. | Adjust the Vapor pressure of Cd by temperature control so that 2288 Å line is as intense as possible with minimum self-reversal. |
| Heating Cycle A. | Char time: 25 sec |
| | Char temp: 600°C |
| B. | Atomization Time: 25 sec |
| | Atomization Temp: 1500°C |
| Carrier Gas Flowrate (Ar) | 0.3 l/m |
| Wavelength | 228.8 nm |
| Strip Chart Recorder Speed | 2.5 cm/m |

The instrument settings for the heating cycle in Table 1 proved to be compatible with the Cd solutions, SRM 1571, and the wheat. The linearity of the system was determined by measuring 5 µl samples of 0.1 ppm, 0.05 ppm and 0.025 ppm of Cd standard solutions dispensed with an eppendorf pipette. Trial runs of both SRM 1571 and wheat, (dispensed into the furnace by means of a stainless steel sample spoon) were required to find a compatible heating cycle for combustion of both samples and to determine the weight range under which predictable and reproducible results could be obtained. In every experiment a second set of Cd standard solutions was run to continuously monitor the system for

possible drift of the light source, and problems of smoke interference or contaminations.

Sample Preparation

Solid samples require weighing prior to their being analyzed.

Previous experiments with orchard leaves proved successful if the ranges of the sample weight was between 1.0 mg and 2.0 mg (Table 2).

Table 2: Cd Measurements of SRM (1571) by ZAA

| <u>Sample No.</u> | <u>Sample Weight mg</u> | <u>ppm</u> | <u>σ</u> | <u>NBS</u> |
|-------------------|-------------------------|------------|----------------------------|---------------------|
| 1 | 1.6 | 0.11 | | |
| 2 | 1.3 | 0.12 | 0.12 ± 0.02 ppm | |
| 3 | 1.3 | 0.12 | | 0.11 ± 0.02 ppm |

Several runs of whole wheat berries proved unsatisfactory; therefore, the decision was made to pulverize the wheat in a porcelain mortar and pestle. In this manner the transfer of the sample and sample weight were more easily controlled. The smoke interference was minimized in the weight range between 4.0 mg and 5.0 mg (Table 3). It should be noted that insertion of the samples by means of the stainless steel sample spoon results in momentarily blocking off the light beam. Therefore, the light beam should be allowed to correct itself to "zero" before the charring phase is initiated.

Table 3: Cd Measurements of SRM (1571) and wheat by ZAA

| <u>Sample No.</u> | <u>Sample Weight mg</u> | <u>ppm</u> | <u>σ</u> | <u>NBS</u> |
|-------------------|-------------------------|------------|----------------------------|---------------------|
| RM 1571 1 | 1.7 | 0.17 | | |
| " 2 | 1.5 | 0.12 | 0.14 ± 0.02 ppm | 0.11 ± 0.02 ppm |
| " 3 | 1.6 | 0.14 | | |
| Wheat 4 | 4.4 | 0.08 | | |
| " 5 | 4.5 | 0.07 | 0.07 ± 0.01 ppm | |

Results

Table 2 illustrates the results of measurements of NBS Standard Reference Material 1571, "Orchard Leaves" comparing the ZAA and NBS results. A second run illustrated in Table 3 shows the results of both SRM 1571 and pulverized wheat. In a third experiment the husk or hull of the pulverized wheat was analyzed separately from the pulp or interior. The values resulting from both measurements indicate that Cd concentrations may selectively migrate to the hard husk as opposed to the wheat pulp (Table 4).

These results are preliminary and further testing should be conducted on several varieties of wheat to determine whether the same is true of all wheat varieties. The implications of this finding could be far reaching with respect to wheat bran and comestibles utilizing wheat bran.

Table 4: Cd Measurements of Wheat Husks vs. Pulp

| <u>Sample No.</u> | <u>Sample Weight mg</u> | <u>ppm</u> | <u>σ</u> | <u>NBS</u> |
|-------------------|-------------------------|------------|----------------------------|---------------------------|
| 1 | 5.0 | 0.13 | | |
| Husk 2 | 5.5 | — | $0.12 \pm 0.01\text{ppm}$ | $0.11 \pm 0.02\text{ppm}$ |
| 3 | 5.3 | 0.12 | | |
| 4 | 4.3 | 0.03 | | |
| Pulp 5 | 4.2 | 0.03 | $0.03 \pm 0.00\text{ppm}$ | $0.11 \pm 0.02\text{ppm}$ |
| 6 | 4.2 | 0.03 | | |

Acknowledgements

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