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ATOMIC ABSORPTION SPECTROMETRY PRINCIPLES AND METHOD

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HCMC, JULY 2012

I. PRINCIPLES AND INSTRUMENT

1.1 PRINCIPLES

- Theory of Bunsen and Kirchoff (1859): The structure of spectrum of coloured flames:
- Atoms of certain gases can: emit a certain spectrum + absorb light having the same wavelength as the wavelength of the emitted ones.
- Alan Walsh (Australia, 1955) developed atomic absorption spectrometry (AAS) method used to quantitatively determine most metals in the periodic table.

Table 1.1 Periodic table

- Qualitative and quantitative determination of up to 70 elements (pink squares)
- Sensitivity: $10^{-3} - 10^{-6}$ mg/L
- Moderate instrument cost

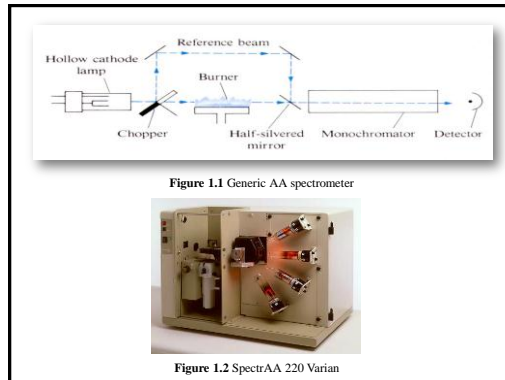
CONTENT

- 1) Principles and Instrument
- 2) Sample Preparation
- 3) Analytical program

- In term of AAS's principles: gaseous atoms of each metal (in atomic cloud) get excited at a specific resonant wavelength which is different from other metals.
- Characteristic wavelength: $\Delta E = E_1 - E_0 = hc/\lambda$
- E_1 – excited state
- E_0 – ground state
- h – Planck's constant
- c – velocity of light
- λ – wavelength
- The exchange of light intensity is referred to atomic absorption spectrum of the metal in question.

1.2 INSTRUMENT

- To process the method, several conditions are required:
- The gaseous metal atoms (atomic cloud)
- Specific resonant wavelength
- Photospectrometry system called light resolving unit (monochromator)
- Consequently, the AAS instrument must adequately correspond to the requirements above: light source, sampler, monochromator, detector (see Figure 1.1)



Beer's law

$$A = \log(1/T) = \log(P_0/P) = e \cdot b \cdot c$$

A : absorbance

T = P/P_0 : transmittance

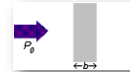
P₀ : initial radiant power of a beam

P : radiant power of the beam after being absorbed

e : the molar absorptivity (L/mol.cm)

b : the path length of the sample (path length of the cuvette in which the sample is contained)

c : the concentration of the compound in solution (mol/L)



2.2 SUMMARY OF METHOD

- 2.2.1 A representative 0.3 g (wet weight) sample is heated with repeated additions of nitric acid (HNO₃) and hydrogen peroxide (H₂O₂).
- 2.2.2 Hydrochloric acid (HCl) is added to the digestate. This digestate will be filtered. Then the filter paper and residues are rinsed with reagent water. After all, the digestate is diluted to a final volume of 100 mL.

Brief: key considerations in quantitative determination of heavy metal by AAS instrument:

- Flame temperature
- Beam height
- Aspiration rate and aerosol quality
- Interferences
- Correlation between concentration and absorbance relied on Beer's Law

II. SAMPLE PREPARATION: ACID DIGESTION OF VEGETABLE SAMPLES

2.1 SCOPE AND APPLICATION

- 2.1 This method is the digestion procedure of vegetable samples used to analyze heavy metal by flame atomic absorption spectrometry (FLAA).
- 2.2 This method is not an ideal digestion technique for most samples. Because, there is strong acid mixture that dissolves almost all elements being available in the environment.

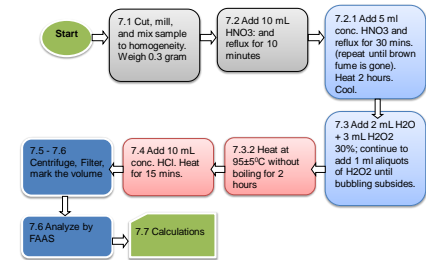


Figure 2.1 Acid digestion of vegetables

Calculation

$$\text{Conc. Mg in sample} = \frac{C_m \times V}{m} \quad (\text{mg/Kg})$$

- C_m : Mg conc. according to standard curve.
- V : the final volume of extract (ml).
- m : weight of sample (g).

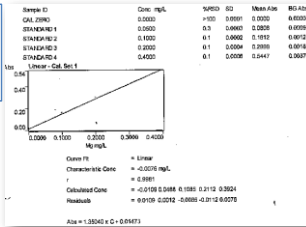


Figure 2.2 Typically analytical report

2.3 QUALITY CONTROL AND QUALITY ASSURANCE PROCEDURES FOR TRACE METALS IN FRUITS

- 2.3.1 For each batch of samples processed, a method blank should be carried throughout the entire sample preparation and analytical process. These blanks will be useful in determining if samples are contaminated.
- 2.3.2 Spiked duplicate samples should be processed on a routine basis. Spiked duplicate samples will be used to determine precision and bias.

- Soaking them in acid bath overnight
- Rinsing with DI water and drying in oven.
- **Note:** Wearing gloves, laboratory coat and eye-protect glasses.



Figure 2.3 Clean up

BLANK	-0.0107	94.4	0.0000	0.0000	-0.0001
TA	-0.0050	3.3	0.0004	0.0107	-0.0004
TA-0	0.0407	0.7	0.0008	0.0887	0.0001
C-1	0.0802	0.0	0.0000	0.1097	0.0004
C-2	0.0700	0.3	0.0004	0.1100	0.0004
C-3	0.1104	0.2	0.0004	0.1029	0.0009
SR-1	0.0884	0.2	0.0000	0.1041	0.0004
SR-2	0.0902	0.8	0.0011	0.1400	0.0000
SR-3	0.0858	0.3	0.0000	0.1306	0.0011
V-1	0.0970	0.3	0.0004	0.1487	0.0000
V-2	0.0924	0.8	0.0008	0.1409	0.0001
V-3	0.0948	0.4	0.0006	0.1400	0.0000
O-1	0.3050	0.1	0.0000	0.8117	0.0001
O-2	0.0336	0.4	0.0000	0.0801	0.0001
O-3	0.2414	0.7	0.0000	0.4758	0.0000
ST1	0.0499	0.9	0.0004	0.0801	0.0000

Figure 2.2 (cont.) Typically analytical report

2.3.3 Restricting contamination

- 2.3.3.1 Controlling water quality
- Using distilled water and Deionised water
- Checking filter every month
- Replace a new one when contamination is determined

2.3.3.2 Cleaning up

- Removing residues
- Washing these tubes by distilled water

III. ANALYTICAL PROGRAM

3.1 SAMPLE PREPARATION

3.1.1 SAMPLE REPLICATION

- There are 3 distinct species of fruit. Each fruit is sampled 3 times (3 replications)
- Prepare 2 blanks controlling this batch of digestion.



Figure 3.1 Chéri, guava, and litchi

3.1.2 RECOVERY

- To test the recovery of the procedure, replicate a certain fruit 3 times. Each digestion tube containing recovery sample is spiked 1 mg/L magnesium.
- **NOTE:** The replications and recoveries of this batch would follow the same procedure, at the same time.

IV. REVIEWING NOTES

- Strictly comply the method manuals
- Implement blanks, replications, and recoveries
- Avoid cross contamination
- In case OVER signal is presented, dilute this sample
- Be careful in diluting concentrated samples



Figure 3.4 Analytical equipments

3.2 Standard Preparation

From an aqueous stock standard of Mg 1000 mg/L, prepare 04 concentrations suggested by SpectrAA software.

3.3 Controlling analytical instrument

- Standard curve is linear
- RSD < 10%

*** Note:**

- Reslope the curve every 50 samples
- Renew standard solutions every day

ACTUAL PROCEDURE



Figure 3.2 Step 7.1



Figure 3.3 Step 7.2 to 7.6

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