

Agri 201 – Lab Techniques

ATOMIC ABSORPTION- CALCIUM

PURPOSE: to gain experience using the atomic absorption spectrophotometer and to learn how to extract elements using dry ashing.

Materials:

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| (1) 1000 ppm Ca standard | 2% Lanthanum solution |
| (5) 50 ml vol. flasks | (3) Whatman #1 filter paper, 11 cm |
| (3) 100 ml vol flasks | |
| (1) 1 <u>N</u> nitric acid | (22) Scintillation vials |
| (1) GBC Atomic Absorption Spectrophotometer | (3) Ashed alfalfa in crucible (1 g ashed for 2 hours @550 C)- |
| (1) electric pipettor | (1) Funnel rack |
| (10) 10 ml pipet tips | (3) Funnels |
| (1) Unknown Ca solution | (4) 100 ml plastic cups w/ lids |

Procedure:

- I. Make 50 ml of 0, 2, 10, 20, and 40 ppm Ca standards from 1000 ppm Ca standard.
This will be made up in DI water and 5 ml of 1 N nitric acid.
- II. Extract Ca from dry ashed alfalfa.
 - A. Add 10 ml of 1 N HNO₃ to crucible with ashed alfalfa.
 - B. Mix with a stir rod.
 - C. Filter into a 100 ml volumetric flask.
 - D. Wash funnel into volumetric flask with DI water until almost full
 - E. Add DI water to line.
 - F. Pour contents into a labeled 100 ml plastic cup
- III. Determine absorbance of samples and standards. (without La)
 - A. Pipette 5 ml of standard, alfalfa or unknown into a Scintillation vial.(9 tubes)
 - B. Add 5 ml of DI water.
 - C. Turn on AA, light flame and adjust wavelength. (Follow directions on instrument)
 - D. Zero AA using DI water.
 - E. Record three readings for each solution. The integration time should be at least 1 second.
 - F. Graph results and determine Ca concentration in unknown and alfalfa.
- IV. Determine absorbance of samples and standards. (with 1% La)
 - A. Pipette 5 ml of standards, alfalfa or unknown into a Scintillation vial.(9 tubes)
 - B. Add 5 ml of 2% La to each tube.
 - C. Turn on AA, light flame and adjust wavelength. (Follow directions on instrument)
 - D. Zero AA using 1% La 0 ppm Ca standard.
 - E. Record three readings for each solution.
 - F. Graph results and determine Ca concentration in unknown and alfalfa.

Questions:

1. What is the resolution of the instrument in Absorbance units, and ppm Ca.
2. What is the resolution of the procedure in % Ca in alfalfa.
3. What is the detection limit of the AA in ppm Ca?
4. Did using the La effect your results? How?
5. Discuss the precision of your measurements?

6. What is the working range of the AA for Calcium. (Assume that the highest usable absorbance reading is .400 Abs.)
7. What is the working range of the procedure?

Note: Dilute samples that have Abs readings higher than your highest standard.