Chromium Analysis – Atomic Absorption Method: Chromic Oxide Determination in Feed and Feces

Materials:

5.5 cm aluminum pans
Drying oven, 100 °C
Muffle furnace, 470 °C
150 ml glass beakers
Hot plates
Watch glass
Volumetric flasks, 50 ml and 100 ml
Whatman 41 or 541 filter paper
25 ml screw cap tubes
Pipettes
Atomic Absorption Spectrophotometer
Desiccator

Reagents:

Standard Chromium Solution, (Perkin Elmer Cat # N9300112) 1,000 ug/ml

10% Manganese sulfate Solution: 11.19 g manganese sulfate monohydrate (Mallinckrodt Cat # 6192, FW 169.01, CAS # 10034-96-5) dissolved in 100 ml 18 MOhm water

Phosphoric acid-manganese sulfate solution: 30 ml 10% manganese sulfate in 1 liter 85% phosphoric acid (Fisher Cat # A-242, FW 98.00, CAS #7664-38-2)

Potassium bromate solution: 4.5% (w/v) potassium bromate (Mallinckrodt Cat # 0487, FW 167.00, CAS # 7758-01-2) in dd water (45 g $KBrO_3$ / liter 18 MOhm water)

Calcium chloride solution (4000 ppm): 14.68 g $CaCl_2$**.**$2H_2O$ (Sigma Cat # C-3881, FW 147.01, CAS # 10035-04-8 or Fisher Cat # S25221A, FW 147.02, CAS # 10035-04-8) dissolved in 1 liter 18 MOhm water

Procedure:

1. Dry duplicate samples (0.5000 g high pool samples, % recovery samples; and 1.0000 g for low pool samples, fecal, and duodenal samples) for 12-24 hours in a 100 °C drying oven. Cool in desiccator, weigh and record. Include in sample run 3 or each of the following: chromium blank, spiked control, high chromium control, and low chromium control.
2. Ash samples for 12-24 hours at 470 °C. Cool, weigh, and record weight.
3. Quantitatively transfer ash to 150 ml beaker.
4. Add 3 ml phosphoric acid solution and wet the ash.
5. Add 4 ml $KBrO_3$ solution; try to wash down the sides of the beaker.
6. Cover beaker with a watch glass and place on a preheated hot plate under a hood. Boil gently until frothiness subsides and no bubbling is evident. **DO NOT BOIL DRY.** Remove from hot plate and cool.

7. Wash residue from beaker into 100 ml volumetric flask with 18 MOhm water. Add 12.5 ml $CaCl_2$ solution. Bring to volume with 18 MOhm water. Mix well by inversion.
8. Filter through Whatman 41 filter paper into 25 ml screw cap tubes.
9. Determine concentration (or absorbance) on an atomic absorption spectrophotometer at a wavelength of 357.9.
10. Maximized AA Spec with the chromium standard of 2 ppm. This will typically give an absorbency reading of about 0.1 (about 20% absorption).

Note: Dilute samples that are out of range of the standard curve.

Standards:

1. Ash 4 samples (1.000 g) of chromium blank (fecal sample with no additional chromium).
2. Follow steps 3-7 of the above chromium procedure.
3. Filter the 4 volumetric flasks (containing Cr blank solution) into a 500 ml beaker.
4. Label six 50 ml volumetric flasks as follows: Blank, 1 ppm, 2 ppm, 3 ppm, 4 ppm, and 5 ppm.
5. To the volumetric labeled:
    a. 1 ppm: add 50 ul Cr stock standard solution (1,000 ug/ml)
    b. 2 ppm: add 100 ul Cr stock standard solution (1,000 ug/ml)
    c. 3 ppm: add 150 ul Cr stock standard solution (1,000 ug/ml)
    d. 4 ppm: add 200 ul Cr stock standard solution (1,000 ug/ml)
    e. 5 ppm: add 250 ul Cr stock standard solution (1,000 ug/ml)
6. Bring the six 50 ml volumetrics up to volume with the filtered Cr blank solution.
7. Make additional 2 ppm Cr standard for maximizing AA Spec. (200 ul Cr stock / 100 ml 18 MOhm water.)
8. Store standards in refrigerator.

Calculations:

$$Spiked\ sample = \frac{0.007\ g\ chromic\ oxide}{g\ fecal\ sample} = \frac{0.00479\ g\ chromium}{g\ fecal\ sample}$$

$$\%\ recovery = \frac{calculated\ \%\ recovery\ g/\ g\ x\ 100}{0.00479}$$

$$\frac{ug}{g} unknown = \frac{(abs\ in\ sample) \times (100) \times (dilution\ factor)}{Sample\ Dry\ Wt.}$$

Abs = use regression curve to calculate
100 ml = total sample volume
Dilution factor = 1 ml filtered sample diluted to a final volume of 10 ml gives a dilution factor of 10

Reference: Williams, C. H., David and O. Iismaa. The determination of chromic oxide in faeces samples by atomic absorption spectrophotometry. 1962. J. Agric. Sci. 59:381