

Titanium Dioxide Analysis-Wet Ash

September, 2013

References:

Myers, W. D., Ludden P.A., Nayigihugu, V., and Hess, B. W. 2004. Technical Note: A procedure for the preparation and quantitative analysis of samples for titanium dioxide. J. Anim. Sci., 82:179-183.

Personal Protective Equipment:

1. Lab coat
2. Safety glasses/goggles
3. Designated acid specific gloves
4. Perform in designated titanium wet ash fume hood

Reagents:

1. Sulfuric Acid (H_2SO_4), concentrated
 - a. Use straight from the bottle.
2. Potassium Sulfate (K_2SO_4)
 - a. Use straight from the bottle.
3. 30% Hydrogen Peroxide (H_2O_2) solution
 - a. Use straight from the bottle.
4. Copper Sulfate (CuSO_4)

Standard Curve Solutions:

NOTE: Color is stable so it can be made in advance and reused for multiple analyses, but protect from evaporation. Store in sealed container.

1. 0 mg TiO_2/mL
2. 0.0200 mg TiO_2/mL
3. 0.0400 mg TiO_2/mL
4. 0.0600 mg TiO_2/mL
5. 0.0800 mg TiO_2/mL
6. 0.1000 mg TiO_2/mL
 - Treat standards the same as unknown TiO_2 samples in procedure below.

Procedure:

Required: Make sure to wear appropriate attire to conduct this procedure: lab coat, gloves, safety goggles and designated acid specific gloves.

1. Weigh duplicate 0.5g samples into 250 mL macro Kjeldahl digestion tubes. Include a baseline sample of feces (or duodenal, ileal digesta or forage) devoid of TiO_2 for background correction.
2. Add a reaction catalyst containing 3.5g of K_2SO_4 and 0.4g of CuSO_4 to each tube.
3. **Make sure to perform the following steps in the appropriate fume hood wearing appropriate gloves and eye protective equipment.** Add 13mL of concentrated H_2SO_4 to each tube and **swirl**. Digest samples at 420°C for 2 h; make sure to **swirl** at the 1 h mark.
4. Be sure to wear thermal gloves over nitrile gloves, remove tubes from heat and allow cooling for a minimum of 30 min.
5. Add 10mL of 30% H_2O_2 to each tube, **swirl**, and allow cooling for 30 min.
6. Bring the total liquid weight up to 100g using distilled water and **swirl**.
7. Filter liquid through Whatman No. 541 filter paper to remove any precipitate.
8. Measure absorbance at 410 nm. Calibrate spectrophotometer with working standards listed above. Use the 0 mg standard to zero the instrument.