

# 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 ( $\text{H}_2\text{SO}_4$ ), concentrated
  - a. Use straight from the bottle.
2. Potassium Sulfate ( $\text{K}_2\text{SO}_4$ )
  - a. Use straight from the bottle.
3. 30% Hydrogen Peroxide ( $\text{H}_2\text{O}_2$ ) solution
  - a. Use straight from the bottle.
4. Copper Sulfate ( $\text{CuSO}_4$ )

## 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  $\text{TiO}_2/\text{mL}$
2. 0.0200 mg  $\text{TiO}_2/\text{mL}$
3. 0.0400 mg  $\text{TiO}_2/\text{mL}$
4. 0.0600 mg  $\text{TiO}_2/\text{mL}$
5. 0.0800 mg  $\text{TiO}_2/\text{mL}$
6. 0.1000 mg  $\text{TiO}_2/\text{mL}$ 
  - Treat standards the same as unknown  $\text{TiO}_2$  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  $\text{TiO}_2$  for background correction.
2. Add a reaction catalyst containing 3.5g of  $\text{K}_2\text{SO}_4$  and 0.4g of  $\text{CuSO}_4$  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  $\text{H}_2\text{SO}_4$  to each tube and **swirl**. Digest samples at  $420^\circ\text{C}$  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%  $\text{H}_2\text{O}_2$  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.