4.2.08 - Animal Feed / Protein

AOAC Official Method 990.03
Protein (Crude) in Animal Feed

Combustion Method
First Action 1990
Final Action

(Applicable to solid feeds containing 0.2-20% N.)

Results of Interlaboratory Study:
$s_r = 0.28$; $s_R = 0.52$; $RSD_r = 0.59\%$; $RSD_R = 1.10\%$

A. Principle

Nitrogen freed by combustion at high temperature in pure oxygen is measured by thermal conductivity detection and converted to equivalent protein by appropriate numerical factor.

B. Apparatus

Any instrument or device designed to measure nitrogen by combustion may be used, which is equipped to provide following conditions. (*Caution:* Follow manufacturer's recommendation for safe operation of instrument.)

(**a**) *Furnace*.—To maintain minimum operating temperature of 950°C for pyrolysis of sample in pure (99.9%) oxygen. Some systems may require higher temperature.

(**b**) *Isolation system*.—To isolate liberated nitrogen gas from other combustion products for subsequent measurement by thermal conductivity detector. Device for converting NO_x products to N_2 or measuring N as NO_2 may be required and included in the design.

(**c**) *Detection system*.—To interpret detector response as percent nitrogen, w/w. May include features such as calibration on standard material, blank determination, and barometric pressure compensation. Any required calibration must be based on theoretical percent nitrogen in pure primary standard organic material such as EDTA.

C. Performance Requirements

System equipped as in **B** must meet or exceed following minimum performance specifications.

(*1*) System must be capable of measuring nitrogen in feed materials containing 0.2-20% nitrogen.

(*2*) Accuracy of system is demonstrated by making 10 successive determinations of nitrogen in nicotinic acid and 10 successive determinations in lysine·HCl. Means of determinations must be within ±0.15 of the respective theoretical values, with standard deviations 0.15. Standard tryptophan may be substituted for lysine·HCl.

(*3*) Suitable fineness of grind is that which gives relative standard deviation (RSD) 2.0% for 10 successive determinations of nitrogen in mixture of corn grain and soybeans (2 + 1) that has been ground for analysis. RSD, % = (SD/mean %N) 100.

Fineness (ca 0.5 mm) required to achieve this precision must be used for all mixed feeds and other hetergeneous materials.

(*4*) Properly blanked system is demonstrated by analyzing suitable N Blank material, such as powdered cellulose, and obtaining zero reading. Atmospheric blank corrections with software (constants) introduce error and are not necessary when the system is properly configured (purged) and sample is properly introduced to minimize trapped atmospheric nitrogen.

D. Calculation

$$\text{Crude protein, \% (w/w)} = \%\ \text{N} \times 6.25$$

or

$$\%\text{N} \times 5.70 \text{ in case of wheat grains}$$

Reference:
JAOAC **72**, 770(1989).

Revised: March 1998