

PHOSPHORUS BEST PRACTICES GUIDELINES

AAFCO Laboratory Methods & Services Committee
Best Practices Working Group
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These guidelines are intended to assist laboratories in the selection of appropriate methods for use to determine phosphorus in various concentrations and in diverse feed matrices. This should be used together with the Phosphorus Methods Scope & Matrix Table.

Keep in mind the following when selecting the appropriate phosphorus method:

- Any method that is not being used within its scope should be validated for the extension or modification to the scope prior to the use of the method.
- Dry ash methods **should not** be used on mineral mix feeds.
- Dry ash methods should be used when organic matter is present in feeds.

The listed methods in this document are appropriate for specific matrix and phosphorus concentrations as used for the validation of the method. The scope of the method is determined by validation data from the original peer reviewed paper, single laboratory validation, collaborative study, or any technical communications. Modifications to these methods, such as detection via ICP-OES, may be used once it has successfully completed validation meeting specific criteria with regards to recovery, bias, reproducibility, and other statistical requirements as is necessary. Other methods besides those cited may be used once the method successfully meets verification requirements.

MAP, DAP, Super-&Triple-super Phosphate, Calcium meta-phosphate, Potassium dihydrogen phosphate, & Cottonseed

AOAC method 964.06 has demonstrated adequate recovery, reproducibility, and no bias for these matrices. It is an alkalimetric ammonium molybdophosphate method appropriate for all concentrations of phosphate.

AOAC method 962.02 is a detection method and refers to AOAC method 957.02 (B)(a, b, c, d, & e) and should be consulted to determine appropriate digestion is used prior to detection. Determination is by precipitation of the phosphate out of solution detected by weight.

AOAC method 968.08 (D)(b) is a wet digestion procedure intended for inorganic matrices. Detection is via atomic absorption. Atomic absorption is applicable to other minerals but not P. After this wet digestion, P would have to be determined by an appropriate spectrophotometric, gravimetric or titrimetric method.

DDGs, cottonseed, pet food, dry feed, laying rations, pig finishers, protein concentrates, ruminant feed

AOAC method 965.17 is appropriate for all animal feed and pet foods that have organic matrices. Samples are dry ashed, dilute acid mixture is added, transferred and further diluted. Detection of

phosphorus is by spectrophotometry. Samples containing monobasic calcium phosphate or with phosphorus concentrations at or greater than 16% **should not** be analyzed by this method.

AOAC method 968.08(D)(a) is appropriate for all animal feed and pet foods if the matrix is organic. Samples are dry ashed in a furnace followed by acid digestion and then analyzed via atomic absorption. Atomic absorption is applicable to other minerals but not P. After this ashing and digestion procedure, P would have to be determined by either an appropriate spectrophotometric, gravimetric or titrimetric method.

Pet food, corn leaves & stalk, peach leaves, and burmuda grass

AOAC method 985.01 is a dry ash method followed by a wet acid digestion on a hot plate. Detection is via ICP-OES.

Alfalfa, citrus, orchard, & tomato leaves, & pine needles

AOAC method 953.01 specifies the elements for analysis and instrumentation requirements. No specific digestion method is mentioned and none is referenced in the method. This defines standards needed for correct instrument results. It includes precision and accuracy requirements for unbiased, reliable, reproducible, accurate results.

Comparison of Wet Ash & Dry Ash Methods for Mineral-Mix Feeds

William Hoover of the Texas Agricultural Experiment Station wrote a technical communication addressing the low recoveries of phosphorus in mineral mix feeds (*JAOAC* **59**, 937(1976)). He digested 12 mineral feeds via dry ash and wet ashing methods. He found that low recoveries are due to some of the components of the mineral-mix feeds forming refractory compounds upon ashing that are not soluble in the dual acid digestion. These samples were not included in the original collaborative studies but several regulatory laboratories brought this low recovery to his attention. The results from his in-house study also demonstrated low recoveries for calcium, copper, iron, manganese, zinc. He stated only magnesium was not affected by the ashing temperature. He recommended that a statement be included for all AOAC dry ashing methods that states "Not applicable for mineral-mix feeds" for all minerals in feeds.

References:

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JAOAC **51**, 776(1968).
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