

POTASSIUM SALTS OF PHOSPHORUS ACID
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Analytical Method Validation in Freshwater

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DATA EVALUATION REPORT

MRID# & TITLE OF STUDY: MRID 43905815, Potassium Salts of Phosphorus Acid: Analytical Method Validation in Freshwater.

DB BARCODE: D226397
REG./FILE#: 069579-R

CASE: 046750
CHEMICAL/BIOL#: Potassium salts of phosphorus acid

COMPANY/SPONSOR: U.I.M. Agrochemicals, Pty. Ltd., Brisbane Markets, Queensland 4106, Australia.

TEST MATERIAL: Potassium salts of phosphorus acid (Foli-r-Fos 400)

REVIEW CONCLUSION: The analytical method presented for determination of phosphorus acid in freshwater is suitable over the concentration range tested, 15.05 to 62.29 mg/L (detection limit 4.26 mg/L), and appears suitable for concentrations tested in acute toxicity tests with wildlife. This concentration range corresponds to 50.7 to 209.7 mg/L of Foli-r-Fos 400 expressed as whole material.

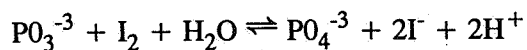
RECOMMENDATIONS: None

ADEQUACY OF STUDY: Supplementary; this study was not intended to fulfill a guideline requirement but was intended to validate the analytical method for determination of phosphorus acid in freshwater samples at concentrations utilized in toxicity tests.

MATERIALS & METHODS: The study conformed to Method LTM-017 of U.I.M. Consolidated Limited (not provided); the study was modified by Toxikon Environmental Sciences with the objective of optimizing sample quantitation and using standard laboratory materials (details not provided). Signed and dated GLP, Quality Assurance, and Data Confidentiality statements were provided. The test substance was characterized by U.I.M. Agrochemicals in Australia and was not performed under U.S. Environmental Protection Agency GLP. The test material, Foli-r-Fos 400, was identified in the following manner: CSI No. 64-F5B, Batch No. 2244-3, 41.0% w/v, 29.7% w/v as phosphorus acid, pH 5.7.

A reference stock solution of 4255 mg active ingredient (ai) was prepared by diluting 0.4272 g of phosphorus acid (99.6%, Aldrich Chemical Co.) to volume in a 100 mL flask with pure water; reagent solution confirmation samples at a lower concentration (42.55 mg/L) were prepared in triplicate. Test solutions were prepared by initially diluting Foli-r-Fos 400 (29.7% purity) to 10381 mg ai/L (primary test solution) and diluting the primary test solution with water to 1038 mg ai/L (secondary test solution). Method validation samples, also referred to as spike samples, were prepared from the secondary test substance solution by diluting with laboratory water resulting in concentrations of 15.05 and 62.29 mg ai/L. These were prepared in triplicate. Matrix blanks of unspiked water were included to determine the limit of detection and a method blank was included to determine background.

Quantitation of potassium salts of phosphorus acid was performed by titrimetric analysis. The determination of the phosphorus acid in freshwater utilized the chemical reaction below:



A known amount of excess iodine was mixed with the test solution. The excess iodine was then back-titrated with sodium thiosulfate using starch as the indicator. The difference between the total iodine and excess iodine was used to calculate the amount of PO_3^{-3} :

$$\text{mg/L H}_3\text{PO}_3 = \frac{[(\text{mL iodine}) (\text{N iodine}) - (\text{mL Na}_2\text{S}_2\text{O}_3) (\text{N Na}_2\text{S}_2\text{O}_3)] \text{EW} \times 10^6}{\text{sample weight (mg)}}$$

where N = normality of the iodine and thiosulfate solutions and EW = the equivalent weight of phosphorus acid (41.0 mg/meq). The method blank was calculated first and subtracted from all samples. Active ingredient concentrations were converted to whole material (wm) concentrations by dividing by the active ingredient percent of Foli-r-Fos 400 (29.7% w/v).

REPORTED RESULTS: The method blank concentration was equivalent to 8.07 mg/L phosphorus acid. This was subtracted before calculation of the following recoveries. The percent recovery in the reagent solution was $84 \pm 1\%$ (range 84-85%). Recoveries for the low and high spike samples were $102 \pm 4\%$ (range 98-105%) and $95 \pm 1\%$ (range 94-95%), respectively (total mean, $98 \pm 4\%$). The limit of detection was calculated as three times the concentration of the highest matrix blank, 1.42 mg ai/L (4.78 mg wm/L), resulting in 4.26 mg ai/L (14.3 mg wm/L).

DISCUSSION: The range and detection limit for potassium salts of phosphorus acid appear to be appropriate for the concentration ranges being tested in wildlife toxicity tests as evidenced by the no-effect concentration of ~100 mg wm/L of Foli-r-Fos 400 in toxicity tests with the invertebrate *Daphnia magna* (MRID 43905816) and the rainbow trout *Oncorhynchus mykiss* (MRID 43905814). The toxicity tests for daphnids and fish were conducted with solutions based on wm/L concentrations. The testing solution of ~100 mg wm/L is within the range of the analytical detection, 50.7 to 209.7 mg wm/L.

Although the background as measured by the method blank was fairly high in relation to the lowest concentration tested, the matrix blanks were reasonably low in relation to the concentrations in the dosing solutions. The recovery for the reagent grade phosphorus acid was only 84% which appears low. However, recoveries of spiked solutions averaged 98% and variations among triplicate samples were low as was the range among all spiked samples, 94-105%.

It should be noted that sodium sulfate is usually designated $\text{Na}_2\text{S}_2\text{O}_3$.