



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1071b

Triphenyl Phosphate

(Standard for Determination of Phosphorus in Petroleum Products)

This Standard Reference Material (SRM) is primarily intended for use in preparing standard oil solutions containing phosphorus. SRM 1071b is essentially free from other metals and has suitable solubility, compatibility, and uniformity for use with most lubricating oils or petroleum products. The SRM consists of 5 grams of triphenyl phosphate that is certified to one part per hundred of phosphorus. The certified phosphorus content is given below.

Phosphorus-----9.48 ± 0.08 weight percent

The uncertainty shown is expressed as two times the standard deviation for a single measurement based on 12 determinations.

Phosphorus was determined by the two following independent methods on samples that had been dried overnight over phosphorus pentoxide (a weight loss of less than 0.1 % was observed on drying.):

(a) Gravimetric Procedure, Quinolinium Phosphomolybdate

Dried samples weighing 1 gram were dissolved in 40 mL of 3:1 nitric-perchloric acid. The phosphorus was precipitated with quinoline in the presence of sodium molybdate. This precipitate was dried to a constant weight and weighed as $(C_9H_7N)_3 \cdot H_3PO_4 \cdot 12MoO_3$.

(b) Differential Spectrophotometric Procedure

Aliquots of the dissolved samples from procedure A were combined with a mixed reagent (ammonium molybdate and ammonium metavanadate) and the phosphorus was determined by differential spectrophotometry using the mixed complexation reaction between phosphate molybdate and vanadate. The phosphorus standards used as reference solutions were prepared from dihydrogen phosphate (SRM 186-1-c).

Stability: Tests show that standard lubricating-oil solutions of this compound with concentrations of phosphorus up to 500 ppm are stable for several weeks when prepared according to the directions given on the reverse side of this certificate.

Compatibility: Lubricating-oil solutions of this compound have been found to be compatible with lubricating-oil solutions of the other metallo-organic SRMs in this series. However, tests have not been carried out to insure compatibility with the various additives that are commonly used in many petroleum products.

This Certificate of Analysis has undergone editorial revision to reflect program and organizational changes at NIST and at the Department of Commerce. No attempt was made to reevaluate the certificate value or any technical data presented in this certificate.

Gaithersburg, MD 20899
September 1, 1991
(Revision of Certificate
dated 2-26-76)

William P. Reed, Chief
Standard Reference Materials Program

(over)

The triphenyl phosphate was prepared by the Eastman Kodak Company of Rochester, NY. Chemical analyses were performed by B.I. Diamondstone and spectrographic analysis by J.A. Norris.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of I.L. Barnes.

The technical and support aspects involved in the original preparation, certification, and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by R.W. Seward. Revision of the Certificate was coordinated through the Standard Reference Materials Program by T.E. Gills.

DIRECTIONS FOR PREPARING LUBRICATING-OIL SOLUTIONS OF TRIPHENYL PHOSPHATE

Transfer approximately 0.6 g of this compound from the bottle to a small beaker and dry over fresh phosphorus pentoxide in a desiccator for 2 hr. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.527 g of this dried compound to a weighed 200 mL flask. (This weight of compound is equivalent to 50 mg of phosphorus.) Add 5 mL of xylene and heat the flask on a hot plate, swirling without charring, until a clear solution forms. Add to the hot solution 80 to 90 mL of lubricating oil and gently shake the flask to mix the contents. Allow the flask to cool to room temperature and add enough lubricating oil to bring the total weight of the contents of the flask to 100 ± 0.5 g. Stopper the flask and shake gently to insure a homogeneous solution. The concentration of phosphorus in this solution is $500 \mu\text{g/g}$ (ppm).