

U. S. Department of Commerce
Alexander B. Trowbridge,

Secretary
National Bureau of Standards
A. V. Astin, Director

Certificate of Analysis

Standard Reference Material 1077a Silver 2-Ethylhexanoate

(Standard for Determination of Silver in Petroleum Products)

This compound was prepared to ensure material that is essentially free from other metals and has suitable solubility, compatibility, and uniformity for use in the preparation of a standard of silver in lubricating oils.

CHEMICAL AND SPECTROGRAPHIC ANALYSES

Procedure and Results of Chemical Analysis

Silver, percent----- 42.60 \pm 0.05

The uncertainty shown represents the 95 percent confidence limit of the mean based on seventeen determinations and allows for the effects of possible sources of known error.

Silver was determined by ignition of 0.3 to 0.4 g samples of the silver 2-ethylhexanoate at 700 °C. The residues were weighed assuming complete combustion of the organic material and conversion to metallic silver. The residues were taken up in 2:1 nitric acid-water solution, diluted to 70 cm³ with water, and titrated with standard sodium chloride solution to a potentiometrically determined end point. Silver was also determined using a microgravimetric technique in which the silver was weighed as silver chloride after sealed tube (Carius) oxidation with nitric acid.

Procedure and Results of Spectrographic Analysis

The compound was examined spectrographically for metallic impurities. A 5 mg sample of the compound was excited in a direct-current arc and the photographed spectrum was examined for the characteristic lines of 51 elements. No significant impurities were found.

STABILITY.—Tests show that standard lubricating-oil solutions of this compound with concentrations of silver up to 500 ppm are stable for several weeks when prepared by 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 compounds in this series. Blends of several different compounds have been prepared by the procedures given in the certificates for the other compounds. (Tests have not been carried out to ensure compatibility with the various additives that may be in the oils to be analyzed.)

The silver 2-ethylhexanoate was prepared by Distillation Products Industries of Rochester, N.Y. Chemical analyses were conducted by R. A. Durst and R. A. Paulson, and spectrographic analyses by V. C. Stewart.

Washington, D.C. 20234
February 10, 1968

W. Wayne Meinke, Chief
Office of Standard Reference Materials

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DIRECTIONS FOR PREPARING LUBRICATING-OIL SOLUTIONS OF SILVER
2-ETHYLHEXANOATE

Transfer approximately 0.2 g of this compound from the bottle to a small beaker and dry over fresh phosphorus pentoxide in a desiccator for 24 hrs. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.117 g of this dried salt to a weighed 200 ml flask. (This weight of salt is equivalent to 50 mg of silver.) Add 2 ml of xylene and 4 ml of 2-ethylhexylamine and heat the flask on a hot plate, with swirling and without charring, until a clear solution forms. Add to the hot solution 2 ml of 2-ethylhexanoic acid and 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 ensure a homogeneous solution. The concentration of silver in this solution is 500 ppm.