U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS
WASHINGTON 25, D. C.

PROVISIONAL CERTIFICATE
STANDARD SAMPLE 1068
SILVER CYCLOHEXANEBUTYRATE

(Standard for Determination of Silver in Petroleum Products)

This compound was prepared to insure 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. The compound is being certified provisionally to one part per hundred of silver, pending further studies of analytical procedures, of loss of weight on drying, and of changes in weight when exposed to air. Every effort should be made to maintain a uniform procedure, by following the directions in this certificate.

CHEMICAL AND SPECTROGRAPHIC ANALYSES

Procedure and Results of Chemical Analysis

Silver, percent - - - - - 38.7

Silver was determined by direct ignition of a 1-g sample (dried for two hours over phosphorus pentoxide), wrapped in filter paper and covered with oxalic acid. The oxide was ignited under hydrogen, and weighed as the metal. Determinations were also made by dissolving the ignited oxide in nitric acid, precipitating with hydrochloric acid, and weighing the silver chloride. Analysts, L. A. Machlan and B. B. Bendigo.

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 50 elements. Several impurities were found, but none is considered to be present in sufficient concentration to interfere with the intended use. The principal impurity was silicon, estimated to be less than 0.01 percent. Analyst, Elizabeth K. Hubbard.

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 below.

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 provisional certificates for the other compounds. (Tests have not been carried out to insure compatibility with the various additives that may be in the oils to be analyzed.)

DIRECTIONS FOR PREPARING LUBRICATING-OIL SOLUTIONS OF SILVER CYCLOHEXANEBUTYRATE

Transfer approximately 0.2 g of this compound from the bottle to a small beaker and dry over phosphorus pentoxide in a desiccator for two hours. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.129 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 $\pm$ 0.5 g. Stopper the flask and shake gently to insure a homogeneous solution. The concentration of silver in this solution is 500 ppm.

Merrill B. Wallenstein

MERRILL B. WALLENSTEIN, Chief
Division of Chemistry II

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