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

PROVISIONAL CERTIFICATE
STANDARD SAMPLE 1050
ALUMINUM CYCLOHEXANEBUTYRATE

(Standard for Determination of Aluminum 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 aluminum in lubricating oils. The compound is being certified provisionally to one part per hundred of aluminum, 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

Aluminum, percent - - - - - - - 6.9

Aluminum was determined by wet-ashing a 1-g sample (dried for 2 hours over phosphorus pentoxide) with sulfuric and nitric acids, precipitating with ammonium hydroxide, and igniting the filtered precipitate to Al_2O_3 at 1100°C. Determinations were also made by direct ignition of a dried 1-g sample, wrapped in filter paper and covered with oxalic acid, to Al_2O_3 at 1100°C. Analysts, B. B. Bendigo and L. A. Machlan.

Procedure and Results of Spectrographic Analysis

This compound was examined spectrographically for impurities present in sufficient concentrations to interfere with the intended use. The principal impurities are iron and silicon, but each is 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 aluminum 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 metallo-organic standards in this series. Blends of several different compounds have been prepared by use of 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 ALUMINUM CYCLOHEXANEBUTYRATE

Transfer approximately 1.0 g of this compound from the bottle to a small beaker and dry over fresh phosphorus pentoxide in a desiccator for two hours. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.725g of this dried salt to a weighed 200-ml flask. (This weight of salt is equivalent to 50 mg of aluminum.) Add 3 ml of xylene and 4 ml of 2-ethylhexanoic acid and heat the flask on a hot plate, with swirling and without charring, until a clear gel forms. Add 3 ml of 6-methyl-2,4-heptanedione and continue heating and swirling until the gel disappears and 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 $\pm$ 0.5 g. Stopper the flask and shake gently to insure a homogeneous solution. The concentration of aluminum in this solution is 500 ppm.

Merrill B. Wallenstein

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