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

PROVISIONAL CERTIFICATE
STANDARD SAMPLE 1072
TRIS(2'-HYDROXYACETOPHENONO)CHROMIUM(III)

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

Chromium, percent - - - - 10.6

Chromium was determined by wet-ashing an 0.5-g sample (dried for two hours over phosphorus pentoxide) with sulfuric and nitric acids, oxidizing the chromium with ammonium persulfate, and titrating the chromium with ferrous ammonium sulfate solution. Analyst, 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 impurities were: barium, estimated to be less than 0.1 percent, and aluminum, silicon, and strontium, each 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 chromium 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

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 TRIS(2'-HYDROXYACETOPHENONO)CHROMIUM(III)

Transfer approximately 0.5 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 0.472 g of this compound to a weighed, 200-ml flask. (This weight of compound is equivalent to 50 mg of chromium.) Add 3 ml of 2-ethylhexanoic acid and 3 ml of 6-methyl-2,4-heptanedione, and heat the flask on a hot plate, with swirling and without charring, until the solid dissolves. 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 chromium in this solution is 500 ppm.

Merrill B. Wallenstein

MERRILL B. WALLENSTEIN, Chief
Division of Chemistry II

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