U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

WASHINGTON, D.C. 20234

National Bureau of Standards Certificate

Standard Sample 1079

Tris(1-phenyl-1,3-butanediono)iron(III)

(Standard for Determination of Iron 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 iron in lubricating oils. The compound is certified to one part per hundred of iron, and 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

Iron, percent	10.3

Iron was determined by the direct ignition of a 0.5-g sample (dried for 1 hr at 110 °C), wrapped in filter paper and covered with oxalic acid, to Fe_2O_3 at 1050 °C. 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 51 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 aluminum, 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 iron 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 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(1-PHENYL-1,3-BUTANEDIONO)IRON(III)

Transfer approximately 0.5 g of this compound from the bottle to a small beaker and dry in an oven at 110 °C for 1 hr. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.485 g of this dried salt to a weighed 200-ml flask. (This weight of compound is equivalent to 50 mg of iron.) 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 solution forms. Add 3 ml of 6-methyl-2,4-heptanedione, and continue heating and swirling the solution for 3 min. 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 iron in this solution is 500 ppm.

A. V. ASTIN, *Director.*

WASHINGTON, D.C. 20234
April 24, 1964.