



Certificate of Analysis

Standard Reference Material 1079a 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 10.30

The standard deviation of this value is 0.02 percent; the 95 percent confidence limit ($t\sigma$) is 0.05 percent. Spectrochemical analysis indicates that interfering metals are not present in sufficient quantity to affect the accuracy of these determinations.

Iron was determined by wet-ashing a 0.5 g sample (dried for 1 hr at 110 °C) with nitric and sulfuric acids, precipitating with ammonium hydroxide, dissolving the precipitate in hydrochloric acid, reducing with stannous chloride and titrating with potassium dichromate. Determinations were also made by direct ignition of a 0.5 g sample, wrapped in filter paper and covered with oxalic acid, to Fe_2O_3 at 1050 °C.

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 impurities were each estimated to be less than 0.01 percent.

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.)

The tris(1-phenyl-1,3-butanediono)iron(III) was prepared by Distillation Products Industries of Rochester, N. Y. The material was sized and dried by Connie L. Stanley. Chemical analyses were conducted by B. B. Bendigo, and spectrographic analyses by Elizabeth K. Hubbard.

Washington, D. C. 20234
January 30, 1967

W. Wayne Meinke, Chief
Office of Standard Reference Materials

(Over)

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 smaller 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.