Maurice H. Stans
Secretary
National Bureau of Standards
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Certificate of Analysis

Standard Reference Material 1079b
Tris(1-phenyl-1,3-butanediono)iron(III)

(Standard for Determination of Iron in Petroleum Products)

This compound was prepared to ensure 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 or iron, and every effort should be made to establish a uniform procedure by following the directions in this certificate.

CHEMICAL AND SPECTROGRAPHIC ANALYSES

Procedure and Results of Chemical Analysis

Iron, percent..... 10.45 ± 0.04

The uncertainty shown represents the 95 percent confidence limit of the mean based on 16 determinations made by two methods and on allowances for the effects of known sources of possible errors.

Iron was determined on samples of tris(1-phenyl-1,3-butanediono)iron(III) (dried for 1 hour at 110 °C) by two methods:

a) A 0.5 g sample was wet-ashed with nitric and sulfuric acids and precipitated with ammonium hydroxide. The precipitate was dissolved in hydrochloric acid, the iron reduced with stannous chloride, and then titrated with potassium dichromate solution.

b) Samples were nondestructively analyzed using the 14 MeV neutron activation technique. The radioactivity induced in these samples and in a standard of known iron content was compared. The determination made use of the 1.81 MeV photopeak of ^{56}Mn (2.576 h half life) produced by the ^{56}Fe(n,p)^{56}Mn nuclear reaction.

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.

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 ensure compatibility with the various additives that may be in the oils to be analyzed.)

Washington, D. C. 20234
February 26, 1969

W. Wayne Meinke, Chief
Office of Standard Reference Materials

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The tris(1-phenyl-1,3-butanediono)iron(III) was prepared by Distillation Products Industries, Rochester, New York. Chemical analyses were conducted by B. B. Bendigo, activation analysis by S. S. Nargolwalla, J. Suddueth and G. W. Smith, and spectrochemical analyses by V. C. Stewart.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of P. D. LaFleur.

The technical and support aspects involved in the preparation, certification and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

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 hour. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.478 g of this dried salt to a weighed 200-cm^3 flask. (This weight of compound is equivalent to 50 mg of iron.) Add 3 cm^3 of xylene and 4 cm^3 of 2-ethylhexanoic acid and heat the flask on a hot plate, with swirling and without charring, until a clear solution forms. Add 3 cm^3 of 6-methyl-2,4-heptanedione, and continue heating and swirling the solution for 3 minutes. Add to the hot solution 80 to 90 cm^3 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 ensure a homogeneous solution. The concentration of iron in this solution is 500 ppm.