

National Bureau of Standards Certificate Standard Reference Material 1062b Manganous Cyclohexanebutyrate

(Standard for Determination of Manganese in Petroleum Products)

This material was prepared to insure material that is essentially free from other metals and has suitable uniformity, solubility, and compatibility for use in the preparation of a standard for manganese in lubricating oils. Every effort should be made to maintain a uniform procedure by following the directions in this certificate.

Chemical and Spectrographic Analyses

Results and brief description of Chemical Analyses

Manganese, percent 13.2 ± 0.2

The uncertainty expressed above is twice the standard deviation of the mean based on 12 determinations with allowances for known sources of possible error.

After drying the samples over phosphorus pentoxide for 72 hours*, manganese was determined by the two following methods:

A. Neutron Activation Analysis

Samples were analyzed by neutron activation techniques using pure manganese metal (99.99% purity) as the reference standard and SRM 1062a as a control standard. Both instrumental and destructive techniques were used for these analyses.

B. Potentiometric Titration

Samples were wet-ashed using a mixture of nitric and sulfuric acids. The manganese was oxidized using ammonium persulfate in the presence of a silver nitrate catalyst and then determined by potentiometric titration with sodium arsenite. High purity electrolytic manganese was used as a primary standard.

C. Spectrographic Analysis

This material was examined spectrographically for metallic impurities. A 5-mg sample of the compound was excited in a high voltage alternating-current arc and the photographed spectrum was examined for the characteristic lines of 35 elements.

Several impurities were found, but none is considered to be present in sufficient concentration to interfere with the intended use. The following values (in percent) are not certified but are given for information only: Na (0.3), Ca (0.02), Si (0.02), Mg (0.005), Fe (0.005), Cu (0.002), Ag (0.001). All others are below limits of detection.

The manganous cyclohexanebutyrate was prepared by the Eastman Kodak Company of Rochester, N.Y. Activation analyses were performed by T. E. Gills, chemical analysis by S. A. Wicks, and spectrographic analysis by J. A. Norris, all of the NBS Analytical Chemistry Division.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of T. E. Gills.

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 R. W. Seward.

*A weight loss of 0.1% was observed after drying.

Stability - Tests show that standard lubricating-oil solutions of this compound with concentrations of manganese 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 MANGANOUS CYCLOHEXANEBUTYRATE

Transfer approximately 0.5 g of this compound from the bottle to a small beaker and dry over fresh phosphorus pentoxide in a desiccator for 72 hours. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.379 g of this dried salt to a weighed 200-ml flask. (This weight of salt is equivalent to 50 mg of manganese.) Add 3 ml of xylene and 5 ml of 2-ethylhexanoic acid and heat flask on a hot plate; swirl without charring until clear solution forms. Add to hot solution 80 to 90 ml of lubricating oil and gently shake flask to mix contents. Allow flask to cool to room temperature, then add enough lubricating oil to bring the total weight of the contents of the flask to 100 ± 0.5 g. Stopper flask and shake gently to insure a homogeneous solution. The concentration of manganese in this solution is 500 ppm.