



National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1514

Thermal Analysis Purity Set

This Standard Reference Material (SRM) is a set of four vials containing "pure" phenacetin and phenacetin doped with nominal 0.7, 2, and 5 mole percent of p-aminobenzoic acid. SRM 1514 is intended for use in evaluating methods of determining purity by differential scanning calorimetry. The actual amounts of p-aminobenzoic acid present were determined by liquid chromatography and confirmed by fluorescence spectrometry. The certified values for p-aminobenzoic acid in mole percent are given below.

<u>Nominal Level</u> p-ABA, mole %	<u>Certified Level</u> p-ABA, mole %
0.0	0.00
0.7	0.69 ± .07
2.0	1.91 ± .12
5.0	5.02 ± .46

The listed uncertainties are \pm two standard deviations of the certified values. These uncertainties include estimates of variability between the methods as well as heterogeneities observed. A minimum 3 mg size sample for each measurement is recommended for the certified values to be valid.

The measurements were performed in the NBS Organic Analytical Research Division by D.K. Hancock, J.M. Brown-Thomas and L. Watts.

The technical direction of the measurements was provided by W.E. May and D.J. Reeder, Organic Analytical Research Division.

Statistical analysis of the data was performed by R.C. Paule, NBS National Measurement Laboratory.

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 L.J. Kieffer.

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Gaithersburg, MD 20899

Stanley D. Rasberry, Chief
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The materials for SRM 1514 were prepared by Hoffman-LaRoche and donated to the National Bureau of Standards. ASTM Committee E-37 on thermal measurements requested that NBS certify these materials.

Information regarding the purity of the undoped phenacetin was obtained from ASTM Committee 37.02.01, as well as results of differential thermal analysis of this material. Any impurity in the form of a solid solution cannot be observed by the differential thermal analysis. The undoped phenacetin appeared to be 99.9 mole percent pure by thermal analysis. However, liquid chromatography by both NBS and ASTM showed the undoped phenacetin impurities to be between 0.3 and 0.6 percent. Thus the thermal analysis results indicate that these impurities, which are not p-ABA, are predominantly in solid solution form. The NBS homogeneity studies using liquid chromatography also suggest that these impurities are evenly dispersed throughout the sample. These impurities should not greatly interfere with the intended use of this SRM. The non-solid solution impurities, over and above the p-ABA, appear to be about 0.1 mole percent.

The following purity values are provided for information only; they are *not certified*. By combining the p-ABA and the non-solid solution impurity results, the phenacetin "purity levels" for differential thermal analysis are estimated to be:

<u>Nominal Level</u> <u>p-ABA, mole %</u>	<u>Purity, mole %</u>
0.0	99.9 ± 0.2
0.7	99.2 ± 0.2
2.0	98.0 ± 0.2
5.0	94.9 ± 0.5