

DEPARTMENT OF COMMERCE

**Bureau of Standards****Certificate**

FOR

STANDARD SAMPLE No. 39b

**BENZOIC ACID**

(Calorimetric and Acidimetric use)

**DIRECTIONS FOR USE AS A CALORIMETRIC STANDARD**

The total heat of combustion at constant volume, per gram weight in air against brass weights, of standard sample No. 39b of Benzoic Acid, has been found to be 6329 calories<sup>20</sup>.

The definition of "total heat of combustion" may be found in Bureau of Standards Circular No. 11, footnote 1, and also in B. S. Scientific Paper No. 230, Combustion Calorimetry and the Heats of Combustion of Cane Sugar, Benzoic Acid, and Naphthalene. Circular No. 11 contains detailed directions relating to the use of standard heat samples, as well as general information on bomb calorimeters.

In using the standard sample, it is desirable to observe the following procedure:

1. The material should be weighed in the form of a briquet not too large for complete combustion in the bomb in which it is to be burned, usually from 1.0 to 1.5 grams. The briquet should be placed in the bomb immediately after weighing.

2. The briquet should be fired by a short length of iron wire of about No. 34 B. & S. gage (about 0.15 mm diameter) and a correction (1600 calories per gram) should be applied for the heat of combustion of the wire. A battery of 3 to 5 storage cells or 6 to 10 dry cells in series should be used for ignition.

3. The briquet should be burned in pure oxygen or in commercially pure oxygen, containing preferably not over 5% of nitrogen, and no combustible gases. To secure complete combustion the total quantity of oxygen should be not less than three times that which will combine with the combustible charge. This usually requires a pressure of from 20 to 40 atmospheres in the bomb.

4. A correction of 230 calories per gram should be applied for the heat of formation of the nitric acid formed as the result of combustion.

When burning most fuels it is not necessary to briquet the material, and platinum wire may be used for ignition in place of iron wire; otherwise the conditions specified above, as well as the details of observing and of computing results, should be as nearly as possible identical in fuel combustions and in calibration observations.

**DIRECTIONS FOR USE AS AN ACIDIMETRIC STANDARD**

In using this sample of benzoic acid as an acidimetric standard the following precautions should be carefully observed:

1. The acid should be carefully fused before use in a covered glass or platinum vessel placed in an air-bath. The temperature during fusion must not rise above 140° C; it is best to keep it below 130° C. and to cease heating as soon as fusion is complete. (Fusion is unnecessary unless a high degree of accuracy is desired. Titrations carefully made check to 1-2 parts in 10 000 and consistent differences as great as 6-7 parts in 10 000 have been observed between fused and unfused materials which have stood in the laboratory for 1-2 years.)

2. The acid should be dissolved in alcohol (about 20 cc for a 1-gram sample) and a blank test made upon the same volume of alcohol so that the necessary correction may be applied in the subsequent titration.

3. The titration must be made in the cold with carbonate-free alkali and the necessary precautions taken to prevent the access of carbon dioxide from the air during titration. Phenolphthalein is the most satisfactory indicator, and 75 cc is a convenient volume in which to make the titration.

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Director.

Washington, D. C., January 7, 1922.