

Bureau of Standards

Certificate

FOR
STANDARD SAMPLE No. 39c

BENZOIC ACID

(Calorimetric or Acidimetric Use)

TREATMENT OF MATERIAL

The sample as issued by the bureau may be considered free from moisture. After long standing, particularly if the container has been opened, there is a possibility that the material may contain some moisture. If there is any doubt as to the integrity of the sample, fusion may be resorted to. The acid should be fused 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. (melting point about 122° C.) and to cease heating as soon as fusion is complete. In one case, material which had stood in the laboratory for about two years showed differences before and after fusion of 6 to 7 parts in 10,000 when tested for acid equivalent.

CALORIMETRIC STANDARD

The total heat of combustion at constant volume, of standard sample No. 39c of Benzoic Acid, per gram weight in air against brass weights, has been found to be

6,329 calories_{20°} = 26.47 international kilowatt-seconds.

A definition of "total heat of combustion" may be found in Bureau of Standards Circular No. 11, also in B. S. Scientific Paper No. 230, "Combustion Calorimetry and the Heats of Combustion of Cane Sugar, Benzoic Acid, and Naphthalene." The calorie is the 20° gram calorie. The use of the international kilowatt-second as a heat unit offers some advantages. It is about 5 per cent smaller than the British thermal unit. 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. If the material has not been fused it should be made into a briquet and weighed in this form. The charge should not be too large for complete combustion in the bomb in which it is to be burned, usually from 1.0 to 1.5 grams. The charge should be placed in the bomb immediately after weighing.

2. The charge 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 (1,600 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. A toy transformer with secondary voltage of about 10 is more convenient, if alternating current is available.

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

4. The formation of nitric acid as a result of combustion yields 230 calories per gram of acid formed and a correction for the heat so produced should be applied.

Most fuels can be burned without briqueting, 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.

ACIDIMETRIC STANDARD

The following directions for the standardization of a 0.1 *N* solution of sodium hydroxide indicate the precautions that must be observed in the standardization of alkaline solutions by the use of benzoic acid.

1. The sodium hydroxide solution must be free from carbonate.

2. A 1 gram sample of the acid should be weighed, placed in a 300 cc flask which has been swept free from carbon dioxide, and treated with 20 cc of alcohol. The flask should then be stoppered and allowed to stand until the sample has dissolved. Three drops of a 1 per cent solution of phenolphthalein should then be added and the solution titrated as a current of air free from carbon dioxide is bubbled through the solution.

3. The effect of the alcohol on the end point should be determined by a blank experiment with the same amount of alcohol, water and indicator and the result subtracted.

For full directions regarding the use of benzoic acid in acidimetry see articles by George W. Morey, *Jour. Am. Chem. Soc.*, **34**, 1027 (1912) and E. R. Weaver, *Ibid.*, **35**, 1309 (1913).

GEORGE K. BURGESS,
Director.