

Certificate

Standard Reference Material 1651

for

Heat-Source Powder Calorimetry

ZIRCONIUM-BARIUM CHROMATE FORMULATION

(Nominal Heating Value – 350 cal g⁻¹)

This material was evaluated at the National Bureau of Standards for use as a Standard Reference Material for calorimetry of gasless heat-source (thermite-type) materials.

The heating value of this Standard Reference Material was found to be 1460.0 Jg⁻¹ (weight in air) (348.9 cal g⁻¹). An uncertainty of ± 4.8 Jg⁻¹ (1.1 cal g⁻¹) is to be associated with this heating value. The calorie used in this work is defined as 4.184 joules.

The above heating value is obtained when no air, oxygen, or nitrogen is present in contact with the sample. To obtain reliable calorimetric measurements with the sample, air should be excluded or purged from the reaction vessel by an inert gas.

This heating value was determined calorimetrically by comparison with electrical energy measured in terms of the National Standards of resistance, voltage, and time. The calorimetric measurements were made using a specially designed electrical heater and a small calorimeter submerged in an isothermal calorimeter jacket that has been described by Prosen, Johnson, and Pergiel, [J. Research NBS 62, 43-47 (1959)].

The barium chromate-zirconium mixture was prepared by a commercial supplier from barium chromate meeting the specifications given by Comyn, Couch, and McIntyre (DOFL Report No. TR-635, 17 September 1958), and zirconium meeting the specifications given by McIntyre and Couch (DOFL Report No. TR-821, 9 March 1960). The mixed powders were agglomerated, to prevent resegregation by material or by size, following a procedure described by Comyn, Marcus, and McIntyre [Ind. Eng. Chem. 52, 995-998 (1960)]. The agglomerate was dried, screened, and separated into 2.4 kg segments which were separated into smaller parts for safety in handling.

The calorimetric measurements on this material were carried out in the NBS Institute for Materials Research by J. I. Minor, Jr. of the Thermochemistry Section, G. T. Armstrong, Chief.

Washington, D. C. 20234
November 12, 1968

W. Wayne Meinke, Chief
Office of Standard Reference Materials

OBSERVE SAFETY PRECAUTIONS ON REVERSE PAGE

CAUTION

This material requires special handling for safety, and the following instructions should be observed carefully:

1. Zirconium–barium chromate mixtures can be ignited readily by flame, spark, friction, and the discharge of static electricity. They burn rapidly generating intense heat and solid ash. Both the heat from the reaction and the hot slag which may be thrown from the reaction zone can cause severe burns. However, if proper precautions are taken, this heat powder is no more dangerous than solvents such as acetone and ether.

2. No flame, smoking, or matches should be permitted in the heat powder area.

3. In order to prevent accumulation of static charges, working surfaces should be conductive and grounded; personnel should wear grounded wrist straps and avoid wearing silk or synthetic fabrics that can generate static electricity. Cotton clothing is preferred.

4. The area should be kept clean, and the amount of heat powder stored in a container or exposed at one time should be limited to 50 grams. It is advisable to break 50 gram samples down to smaller quantities for laboratory use.

5. The heat powder should be kept in covered conductive rubber containers or small metal containers with rubber stoppers. Friction lids must not be used. Desiccators should be made of metal, should contain only solid desiccants, and should be placed on a grounded surface before cover is removed to insert or remove a sample.

6. Heat powder may generate static when poured. It should be transferred with a grounded metal scoop.

7. Personnel should wear kid gloves and either safety glasses or a face shield while handling heat powder to prevent injury if the powder ignites accidentally.

8. Before use, heat powder should be dried for two hours at 71 °C and 10 mm pressure in a flat metal container in a vacuum oven then cooled in a desiccator. It should never be dried in an oven with open heating coils. The standard samples should be stored in a desiccator over any adequate, dry desiccant such as indicating Drierite, etc.