

National Bureau of Standards

Certificate

Standard Reference Material 2213

2,2,4-Trimethylpentane

This Standard Reference Material (SRM) consists of 25 mL of commercially obtained 2,2,4-trimethylpentane and is intended for use as a liquid density standard, as a refractive index standard, and as a calorimetric heat of combustion standard.

DENSITY

The density of a sample of this material was measured in a U-shaped resonant comparator. The temperature of the bath was maintained constant to ± 0.015 °C. The uncertainties of the density values given below are ± 0.037 kg/m³.

Temperature	20 °C	25 °C	30 °C
Density, kg/m ³ (for air-saturated material)	691.929	687.680	683.432

The values of density are on the basis of weights in vacuum, with the sample at a pressure of 1 atmosphere and saturated with air.

The density measurements were made under the supervision of J.R. Whetstone in the Chemical Process Metrology Division, NBS Center for Chemical Engineering.

REFRACTIVE INDEX

The refractive index of four samples of this material was determined at seven wavelengths and at temperatures near 20, 25, and 30 °C. The measurements were made on a precision spectrometer by means of the minimum-deviation method. Emission lamps of Hg, He, H₂, and Na were used as wavelength sources. Each sample was contained in a prismatic cell with plane-parallel windows during the measurement process. Water from a temperature-controlled bath was circulated through the cell housing to maintain a constant temperature during the test. The temperature coefficient of refractive index was determined for each sample at each wavelength. An average temperature coefficient for the four samples at each wavelength was used to determine the refractive index value for the wavelength at 20, 25, and 30 °C.

Wavelength (nm)	Source	Refractive Index		
		20.0 °C	25.0 °C	30.0 °C
435.8	Mercury,g	1.4006	1.3979	1.3954
486.1	Hydrogen,F	1.3967	1.3940	1.3915
501.6	Helium	1.3957	1.3931	1.3906
546.1	Mercury,e	1.3934	1.3908	1.3883
589.3	Sodium,D _m	1.3917	1.3891	1.3866
656.3	Hydrogen,C	1.3897	1.3871	1.3847
667.8	Mercury	1.3894	1.3868	1.3844

The refractive index value given above for each wavelength at each temperature is an average of the values determined for the four samples at that wavelength and temperature. These average values are considered to be representative refractive index values accurate to within 2×10^{-4} for this lot of material.

The refractive index measurements were performed by M. Dodge of the Semiconductor Materials and Processes Division, NBS Center for Electronics and Electrical Engineering.

CALORIMETRIC HEAT OF COMBUSTION

The quantity of heat evolved by combustion of a sample of this lot of material in a closed bomb calorimeter has been found to be 47.712 absolute megajoules per kilogram mass (weight in vacuo) with an estimated uncertainty of about ± 0.01 percent, when the sample is used under the following conditions:

- The sample before combustion is all in the liquid phase and the combustion reaction is referred to 25 °C.¹
- The sample is burned in a bomb of constant volume in pure oxygen at an initial pressure of 30 atmospheres at 25 °C.
- The number of grams of sample burned is equal to three times the volume of the bomb in liters.
- The number of grams of water placed in the bomb before combustion is equal to three times the volume of the bomb in liters.

When the sample is burned under conditions differing from those given under A, B, C, and D above, the value given for the heat evolved should be multiplied by the following factor, if this factor is found to differ from unity to a significant extent:

$$1 + 10^{-6} [4(p - 30) + 45.5 \left(\frac{m_s}{v} - 3\right) + 21 \left(\frac{m_w}{v} - 3\right) - 81(t - 25)]$$

where p = initial absolute pressure of oxygen, in atmospheres, at the temperature t.

m_s = mass of sample burned in grams.

m_w = mass, in grams, of water placed in bomb before combustion.

v = volume of bomb in liters.

t = temperature to which the reaction is referred, in degrees.

In using the SRM to calibrate a bomb calorimeter for determining the heats of combustion of gasolines or other volatile liquids, the procedure followed should be the same as that followed in the measurement of heats of combustion, especially with respect to the kind of capsule used to enclose the liquids, the weighing of the samples, and the method of correcting for heat of combustion of the capsules, if these are combustible. If gelatin capsules are used to enclose the liquid, the method should take account of the effect of the variation of water content of the gelatin with varying relative humidity. For procedures involving sealed glass ampoules, reference is made to the following publications: J. Am. Chem. Soc. 37, 993 (1915); Precise Measurement of Heat of Combustion with a Bomb Calorimeter, NBS Monograph 7, 1960.²

The charge should be fired by passing electric current through a short length of iron or chromel wire (about No. 34 AWG) or a fine platinum wire. Correction should be made for the heat of combustion of the wire (7.5 kilojoules per gram of iron or 5.98 kilojoules per gram of chromel) and for the electric energy used in igniting the wire. The energy used to fire the charge (heat of combustion of iron or chromel wire plus electric energy) may be determined by blank calorimetric experiments on the fuse alone. A battery of 4 to 7 storage cells or 6 to 10 dry cells in series or a small transformer with a secondary voltage of about 10V can be used for ignition.

The charge should be burned in pure oxygen, or in commercially pure oxygen containing not more than 1 percent nitrogen and no combustible gases. The effect of combustible constituents (probably mostly hydrogen) in some samples of commercial electrolytic oxygen has been found to be of the order of 1 percent of the total heat produced by combustion in a bomb of about 0.37 liter capacity.

The amount of nitric acid formed in combustion may be determined by titration with a 0.1 molar solution of sodium hydroxide, using methyl orange as indicator. The correction for the formation of nitric acid is 58.7 kilojoules per mole, or 0.94 kilojoules per gram of HNO₃.

If the heat of combustion of the sample in calories per gram is desired, the following conversion factor may be used:

$$1 \text{ calorie} = 0.0041840 \text{ absolute kilojoules.}$$

The calorie thus defined is used in connection with practically all of the thermochemical work done in the United States. See Am. J. Phys. 12, 1 (1944).

The International Steam Tables calorie (I.T. cal.) and the corresponding Btu which are used in most modern tables of engineering data are defined by the relations

$$\begin{aligned} 1 \text{ I.T. cal} &= 0.0041868 \text{ absolute kilojoules} \\ 1 \text{ I.T. cal/gram} &= 1.8 \text{ Btu/lb.} \end{aligned}$$

In the ordinary use of the bomb calorimeter where an accuracy of not better than 0.1 percent is required, the factor given above for correcting for departures from the standard conditions of oxygen pressure, temperature, etc. may be omitted and the heat evolved expressed as 47.78₈ absolute kilojoules (kilowatt-seconds) per gram weight against brass weights in air (11,422 calories per gram weight in air, or 11,414 I.T. calories per gram weight in air, or 20.545 Btu per pound weight in air). In determining the heating values of fuels, it is desirable to follow a standardized procedure such as that specified by the American Society for Testing and Materials in their standard D-240 for liquid hydrocarbon fuels, or as given in NBS Monograph 7, Precise Measurement of Heat of Combustion with a Bomb Calorimeter.

The measurement of heat of combustion was made by D.R. Kirklin of the Chemical Thermodynamics Division, NBS Center for Chemical Physics.

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.

¹Methods of reducing bomb caloric data to a common temperature are described in NBS J. Research 10, 525 (1933) RP546. This paper also describes the calculation of heats of reaction at a constant pressure from the bomb calorimetric data.

²For sale by the National Technical Information Service, Springfield, VA 22161.