U. S. Department of Commerce
Malcolm Baldrige
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National Bureau of Standards
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# National Bureau of Standards

# Certificate

## Standard Reference Material 720

## Synthetic Sapphire ($\alpha$-$Al_2O_3$)

This Standard Reference Material (SRM) is intended for use in calibrating or checking calorimeters used to measure either enthalpy or heat capacity within the range of 10 to 2250 K.

The material furnished is synthetic sapphire cylinders, cut from centerless-ground rods grown by the Vernieul process and obtained from the Union Carbide Corporation.

The enthalpy and heat-capacity data have been derived from high-temperature enthalpy and low-temperature heat-capacity measurements. These data are presented in both tabular and equation format.

The enthalpy values are accurate to ± 0.1 percent from 70 to 1173 K and the heat-capacity values have an accuracy ranging from ± 0.1 percent at 70 K to ± 0.3 percent at 1200 K. Below 70 K, the inaccuracy in heat-capacity and enthalpy values increase gradually to ± 10 percent at 10 K, because, with decreasing temperature, the heat capacity of sapphire diminishes at a much faster rate than does that of the sample container (mainly copper). The precision of the heat-capacity measurement between 100 and 380 K is estimated to be ± 0.02 percent. The precision of the enthalpy measurement from 273.15 to 1173 K is estimated to be 0.02 percent. For the temperature range 1173 to 2250 K, the precision of the enthalpy measurement is estimated to be ± 0.03 percent, and the accuracy of the measured enthalpy is estimated to be ± 0.2 percent, to a large extent reflecting the uncertainty in temperature measurements at these high temperatures. Above 1700 K, a detectable weight loss was observed in an open container due to evaporation of material.

Relative Enthalpy and Heat Capacity [b]

| Temp[a] | $H_T-H_{0\,K}$ | $C_p$ | Temp | $H_T-H_{0\,K}$ | $C_p$ |
|---|---|---|---|---|---|
| K | $J\cdot mol^{-1}$ | $J\cdot mol^{-1}\cdot K^{-1}$ | K | $J\cdot mol^{-1}$ | $J\cdot mol^{-1}\cdot K^{-1}$ |
| 10 | $0.02_3$ | $0.009_1$ | 70 | $74.6_8$ | $4.59_2$ |
| 15 | $0.11_5$ | $0.030_7$ | 80 | $131._7$ | $6.90_1$ |
| 20 | $0.36_4$ | $0.073_2$ | 90 | $214._2$ | $9.67_9$ |
| 25 | $0.89_8$ | $0.14_6$ | 100 | $326._6$ | $12.85_5$ |
| 30 | $1.90_5$ | $0.26_5$ | 110 | $472._4$ | $16.34_7$ |
| 35 | $3.64_6$ | $0.44_3$ | 120 | $654._3$ | $20.0_7$ |
| 40 | $6.46_0$ | $0.69_7$ | 130 | $874._3$ | $23.9_5$ |
| 45 | $10.7_7$ | $1.04_6$ | 140 | $1133._7$ | $27.9_3$ |
| 50 | $17.1_1$ | $1.50_7$ | 150 | $1433._1$ | $31.9_5$ |
| 60 | $38.1_8$ | $2.79_3$ | 160 | $1772._7$ | $35.9_5$ |

[a] Temperatures expressed on IPTS-68 scale

[b] Molecular Weight = 101.9613

(Table continued on page 2)

Heat-capacity measurements from 10 to 380 K were made by S.S. Chang in the Polymers Division of the Center for Materials Science. Enthalpy measurements from 273.15 to 1173.15 K were made by D.A. Ditmars and T.B. Douglas; those from 1173.15 K to 2250 K were made by S. Ishihara and E.D. West. The enthalpy measurements were made with facilities located in the Chemical Thermodynamics Division of the Center for Chemical Physics.

The technical and support aspects in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by J.L. Hague and R.K. Kirby.

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George A. Uriano, Chief
Office of Standard Reference Materials

(over)

## Relative Enthalpy and Heat Capacity

| Temp | $H_T$-$H_0$ K | $C_p$ | Temp | $H_T$-$H_0$ K | $C_p$ |
|---|---|---|---|---|---|
| K | J·mol$^{-1}$ | J·mol$^{-1}$·K$^{-1}$ | K | J·mol$^{-1}$ | J·mol$^{-1}$·K$^{-1}$ |
| 170 | 2152.$_0$ | 39.9$_0$ | 600 | 4012$_1$ | 112.5$_5$ |
| 180 | 2570.$_3$ | 43.7$_5$ | 610 | 4124$_9$ | 113.0$_6$ |
| 190 | 3026.$_7$ | 47.5$_0$ | 620 | 4238$_2$ | 113.5$_5$ |
| 200 | 3519.$_9$ | 51.1$_2$ | 630 | 4352$_0$ | 114.0$_2$ |
| 210 | 4048.$_7$ | 54.6$_1$ | 640 | 4466$_3$ | 114.4$_8$ |
| 220 | 4611.$_6$ | 57.9$_5$ | 650 | 4581$_0$ | 114.9$_2$ |
| 230 | 5207.$_1$ | 61.1$_4$ | 660 | 4696$_1$ | 115.3$_5$ |
| 240 | 5833.$_9$ | 64.1$_8$ | 670 | 4811$_7$ | 115.7$_6$ |
| 250 | 6490.$_3$ | 67.0$_8$ | 680 | 4927$_6$ | 116.1$_6$ |
| 260 | 7175.$_0$ | 69.8$_2$ | 690 | 5044$_0$ | 116.5$_5$ |
| 270 | 7886.$_3$ | 72.1$_2$ | 700 | 5160$_7$ | 116.9$_2$ |
| 273.15 | 8115.$_6$ | 73.2$_1$ | 720 | 5395$_3$ | 117.6$_4$ |
| 280 | 8622.$_8$ | 74.8$_7$ | 740 | 5631$_3$ | 118.3$_2$ |
| 290 | 9383.$_2$ | 77.2$_0$ | 760 | 5868$_5$ | 118.9$_6$ |
| 298.15 | 1002$_0$ | 79.0$_1$ | 780 | 6107$_1$ | 119.5$_6$ |
| 300 | 1016$_6$ | 79.4$_1$ | 800 | 6346$_8$ | 120.1$_4$ |
| 310 | 1097$_1$ | 81.5$_1$ | 820 | 6587$_6$ | 120.6$_9$ |
| 320 | 1179$_6$ | 83.4$_9$ | 840 | 6829$_5$ | 121.2$_1$ |
| 330 | 1264$_1$ | 85.3$_7$ | 860 | 7072$_4$ | 121.7$_1$ |
| 340 | 1350$_3$ | 87.1$_6$ | 880 | 7316$_3$ | 122.2$_0$ |
| 350 | 1438$_3$ | 88.8$_4$ | 900 | 7561$_2$ | 122.6$_6$ |
| 360 | 1528$_0$ | 90.4$_5$ | 920 | 7807$_0$ | 123.1$_1$ |
| 370 | 1619$_2$ | 91.9$_7$ | 940 | 8053$_6$ | 123.5$_5$ |
| 380 | 1711$_9$ | 93.4$_1$ | 960 | 8301$_1$ | 123.9$_7$ |
| 390 | 1806$_0$ | 94.7$_8$ | 980 | 8549$_5$ | 124.3$_7$ |
| 400 | 1901$_4$ | 96.0$_8$ | 1000 | 8798$_6$ | 124.7$_7$ |
| 410 | 1998$_2$ | 97.3$_2$ | 1020 | 9048$_6$ | 125.1$_6$ |
| 420 | 2096$_1$ | 98.5$_0$ | 1040 | 9299$_2$ | 125.5$_3$ |
| 430 | 2195$_1$ | 99.6$_2$ | 1060 | 9550$_7$ | 125.9$_0$ |
| 440 | 2295$_3$ | 100.6$_9$ | 1080 | 9802$_8$ | 126.2$_6$ |
| 450 | 2396$_5$ | 101.7$_1$ | 1100 | 1005$_{60}$ | 126.6$_1$ |
| 460 | 2498$_7$ | 102.6$_8$ | 1120 | 1030$_{90}$ | 126.9$_5$ |
| 470 | 2601$_8$ | 103.6$_0$ | 1140 | 1056$_{40}$ | 127.2$_9$ |
| 480 | 2705$_9$ | 104.4$_8$ | 1160 | 1081$_{80}$ | 127.6$_1$ |
| 490 | 2810$_8$ | 105.3$_3$ | 1180 | 1107$_{40}$ | 127.9$_4$ |
| 500 | 2916$_5$ | 106.1$_3$ | 1200 | 1133$_{00}$ | 128.$_{25}$ |
| 510 | 3023$_0$ | 106.9$_0$ | 1250 | 1197$_{30}$ | 129.$_{01}$ |
| 520 | 3130$_3$ | 107.6$_4$ | 1300 | 1262$_{00}$ | 129.$_{74}$ |
| 530 | 3238$_3$ | 108.3$_5$ | 1350 | 1327$_{10}$ | 130.$_{43}$ |
| 540 | 3347$_0$ | 109.0$_2$ | 1400 | 1392$_{40}$ | 131.$_{08}$ |
| 550 | 3456$_3$ | 109.6$_7$ | 1450 | 1458$_{10}$ | 131.$_{70}$ |
| 560 | 3566$_3$ | 110.2$_9$ | 1500 | 1524$_{10}$ | 132.$_{29}$ |
| 570 | 3676$_9$ | 110.8$_9$ | 1550 | 1590$_{40}$ | 132.$_{84}$ |
| 580 | 3788$_1$ | 111.4$_6$ | 1600 | 1657$_{00}$ | 133.$_{36}$ |
| 590 | 3899$_8$ | 112.0$_2$ | 1650 | 1723$_{80}$ | 133.$_{85}$ |

## Relative Enthalpy and Heat Capacity

| Temp | $H_T-H_{0\,K}$ | $C_p$ | Temp | $H_T-H_{0\,K}$ | $C_p$ |
|---|---|---|---|---|---|
| K | $J \cdot mol^{-1}$ | $J \cdot mol^{-1} \cdot K^{-1}$ | K | $J \cdot mol^{-1}$ | $J \cdot mol^{-1} \cdot K^{-1}$ |
| 1700 | $1790_{80}$ | $134._{31}$ | 2000 | $2197_{20}$ | $136._{50}$ |
| 1750 | $1858_{10}$ | $134._{73}$ | 2050 | $2265_{50}$ | $136._{80}$ |
| 1800 | $1925_{50}$ | $135._{13}$ | 2100 | $2334_{00}$ | $137._{10}$ |
| 1850 | $1993_{20}$ | $135._{50}$ | 2150 | $2402_{60}$ | $137._{41}$ |
| 1900 | $2061_{00}$ | $135._{85}$ | 2200 | $2471_{40}$ | $137._{73}$ |
| 1950 | $2129_{00}$ | $136._{18}$ | 2250 | $2540_{30}$ | $138._{06}$ |

Below 273.15 K, the heat-capacity values were calculated from a spline function fitted to the heat-capacity data over three temperature intervals and employing polynomials, Pn (n = 1,2,3) of the form,

$$Pn = \sum_{i=0}^{6} \frac{A_i}{i!} (T - T_0)^i$$

$45.0 \text{ K} > T \geq 8.61 \text{ K}$:

$C_p = \exp(P1)\ J \cdot mol^{-1} \cdot K^{-1}$

$T_0 = 8.61$ K　$A_3 = +0.450764E-02$
$A_0 = -0.5147E+01$　$A_4 = -0.51464E-03$
$A_1 = +0.34127E+00$　$A_5 = +0.397864E-04$
$A_2 = -0.333446E-01$　$A_6 = -0.152136E-05$

$125.0 \text{ K} > T \geq 45.0 \text{ K}$:

$C_p = P2\ J \cdot mol^{-1} \cdot K^{-1}$

$T_0 = 40.0$ K　$A_3 = +0.95173E-04$
$A_0 = +0.6966E+00$　$A_4 = -0.35910E-05$
$A_1 = +0.59387E-01$　$A_5 = -0.6498E-07$
$A_2 = +0.40357E-02$　$A_6 = +0.4089E-08$

$273.15 \text{ K} > T \geq 125.0 \text{ K}$:

$C_p = P3\ J \cdot mol^{-1} \cdot K^{-1}$

$T_0 = 125.0$ K　$A_3 = -0.83967E-04$
$A_0 = +0.21993E+02$　$A_4 = +0.19133E-05$
$A_1 = +0.38853E+00$　$A_5 = -0.31778E-07$
$A_2 = +0.13955E-02$　$A_6 = +0.29562E-09$

Above 273.15 K, enthalpy and heat-capacity values were derived from the following equation

$$H_T - H_{273.15} = AT^{-2} + BT^{-1} + C\ln T + K + DT + ET^2 + FT^3 + GT^4 + HT^5\ J \cdot mol^{-1}$$

$A = +6.6253E+07$　$E = -8.57516E-02$
$B = -4.54238E+06$　$F = +4.299063E-05$
$C = -5.475599E+04$　$G = -1.15192E-08$
$K = +2.5819702E+05$　$H = +1.26351E-12$
$D = +2.574076E+02$

Low-temperature measurements from 10 to 380 K were made with a vacuum adiabatic calorimeter [1,2] operated automatically under the control of a minicomputer. Enthalpy measurements at eighteen temperatures from 273 to 1173 K were made by the drop method using a Bunsen ice calorimeter [3,4]. From 1173 to 2250 K, enthalpy measurements were made with an adiabatic receiving calorimeter [5,6]. All temperatures are expressed on the IPTS-68 scale. In the correction of specimen mass data for atmospheric buoyancy, a density of 3.97 $g \cdot cm^{-3}$ for $\alpha$-$Al_2O_3$ was assumed. The functions presented were fitted by the method of least squares to these data. The tabulated values were calculated using these functions.

An occasional particle may contain an end smear due to the method employed in cutting the material. These smears do not contribute significantly to the enthalpy values given in this certificate. However, it is recommended that the material be heated to 1000 °C in air prior to heat-capacity measurements below 350 K. Microprobe analyses indicate small quantities of chloride, titanium, calcium, silicon, iron, copper, and zinc on the surfaces. Spectrographic examination indicates the purity of the bulk material to be at least 99.95 + percent, with the major impurities being magnesium, calcium, chromium, iron, silicon, and titanium. Examination by atomic absorption spectrometry for magnesium, indicated as the major impurity by the above tests, shows that the surface contamination by this element amounts to 1 ppm, or less, and that the bulk material contains 10 ppm, or less, of magnesium. In addition, the material absorbs a small amount (30 ppm or less, presumably moisture) of weight on the ground surfaces on exposure to room air, and may require heating in an inert atmosphere, if this amount of moisture is of concern. Combustion analysis in oxygen indicates the material contains on the order of 10 ppm or less of carbonaceous material calculated as carbon.