

**PHYSICAL PROPERTIES OF
HIGH TEMPERATURE MATERIALS**

**Part VI. Enthalpy and Heat Capacity of Magnesium Oxide,
Zirconium Oxide, and Zirconium Silicate from 0° to 900°C.**

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FOREWORD

This report was prepared by the National Bureau of Standards, under USAF Delivery Order No. AF 33(616) 56-21. The contract was initiated under Project No. 7360, "Materials Analysis and Evaluation Techniques", Task No. 73603, "Thermal Measurements". It was administered under the direction of the Materials Central, Directorate of Advanced Systems Technology, Wright Air Development Division, with Mr. Hyman Marcus acting as project engineer.

This report covers work conducted in the period March 31, 1957 to December 21, 1959.

The authors are indebted to the following members of the staff of the National Bureau of Standards: Martha M. Darr and H. A. Bright for the sample analyses, and F. P. Knudsen for preparing zirconium silicate specimens. The Norton Company of Niagara Falls, Ontario, Canada, kindly furnished the samples of magnesium oxide.

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The heat contents of three refractory substances, magnesium oxide, zirconium oxide, and zirconium silicate were measured relative to 0°C at 100-degree intervals up to 900°C, using a precise "drop" calorimetric method. The magnesium oxide sample was of high purity. The other samples, however, were of relatively low purity. No attempt was made to correct heat-content and heat-capacity values for impurities. The derived specific heats can be described by the following equations in calories per gram-deg C at t°C.

$$(170^{\circ}\text{-}900^{\circ}\text{C}) \text{ MgO: } C_p = 0.2889 + 2.94(10^{-5})t - 6.31(10^3)/(t+273.15)^2$$

$$(0^{\circ}\text{-}900^{\circ}\text{C}) \text{ ZrO}_2: C_p = 0.1524 + 2.98(10^{-5})t - 10.633/(t+273.15)$$

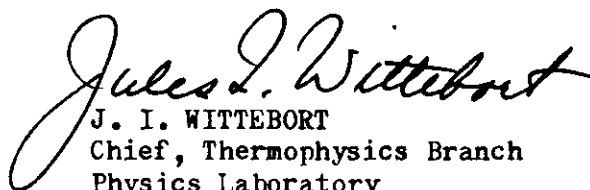
$$(0^{\circ}\text{-}900^{\circ}\text{C}) \text{ ZrSiO}_4: C_p = 0.17945 + 2.41(10^{-5})t - 4.598(10^3)/(t+273.15)^2$$

The specific heats from these equations are compared with values from the literature.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



J. I. WITTEBORT
Chief, Thermophysics Branch
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I. INTRODUCTION

During the past several years the National Bureau of Standards has contributed to the knowledge of the physical properties of metals, fused salts and salt mixtures, and ceramics of importance to the Wright Air Development Center by determining their specific heats and thermal conductivities at high temperatures. The current activity in this program has been the measurement of these properties of several ceramic materials. This report describes the determinations of the specific heats of magnesium oxide, magnesia-stabilized zirconium dioxide, and zirconium silicate.

The measured heat contents and derived specific heats have not been corrected for contained impurities. This will have little effect on the magnesium oxide results because of the high purity of the sample. For the other two samples, however, the impurity content was greater than one percent and hence subjects the final results to considerable uncertainty. Considerable difficulty was encountered in attempting to obtain quantitative analyses of the zirconium oxide and zirconium silicate samples. For this reason there was actually no basis for making reasonably accurate impurity corrections. The heat content measurements appear in general to be precise. The reproducibility of duplicate measurements is in general 0.1 percent or better.

II. SAMPLES AND THEIR COMPOSITIONS

The MgO sample was in the form of a single crystal supplied by the Norton Company, Niagara Falls, Ontario, Canada. Spectrochemical and spectrographic analyses showed that the sample had a high purity, with Mg comprising 99.93 percent of the included metallic elements. Heat content measurements were made on a specimen weighing 10.7578 grams.

The specimens of zirconium oxide were supplied by Corning Glass Works, Corning, N. Y., in the form of hollow cylinders about two inches O.D., one-half inch I.D., and six inches long. Specimens were obtained with as high a density as practical (5.4 g/cm³, which is about 96% of the theoretical), using magnesium oxide as the stabilizing material. This stabilizer was used because of an opportunity to compare heat-capacity results with those found earlier on a porous specimen of similar composition (4.1% 0.4% HfO₂, 0.4% SiO₂, plus smaller impurities) also furnished by Corning Glass Works. The manufacturer stated that (1) the dense specimen was "fired on a seven-day schedule to 1600°C, where it was held for twenty-four hours" and (2), X-ray analysis showed it to be "approximately 90% cubic and 10% monoclinic crystallization." A sample of the zirconium oxide other than that on which heat content measurements were made was heated to about 1100°C and subsequent X-ray analysis showed it to be about 60% cubic and 40% monoclinic. After thermal diffusivity measurements had been made on this same sample (up to about 1200°C) the composition was found to be about 33% cubic and 67% monoclinic. These results show that the specimens were not adequately stabilized.

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The zirconium oxide specimen used in heat content measurements was taken directly from the batch received from the Corning Glass Works without further heat treatment. This specimen was broken into small pieces and put into a container described in the next section. The total weight of the zirconium oxide specimen run was 11.2464 grams.

The zirconium silicate sample was obtained as a fine powder within the Bureau and was fired and sintered into the form of small cylinders (about 1" long and 3/8" diameter) by F. P. Knudsen of the Bureau's Engineering Ceramics Section.

The analyses of materials from the same batches as the samples on which heat measurements were made are given in table 1. It should be noted that only qualitative analyses were made for impurities in the zirconium oxide and zirconium silicate. The specimen of zirconium silicate on which heat-content measurements were made weighed 8.4811 grams. The analysis indicates an excess of Si over that for stoichiometric zirconium orthosilicate, and a deficiency of Zr. This would be the case if some of the sample were zirconium metasilicate. Because a gravimetric analysis was used to determine the amount of Zr + Hf, the higher atomic weight of the Hf would cause an even larger proportion of metasilicate than is indicated by the analysis in table 1.

III. SAMPLE CONTAINERS

During the heat measurements the samples were enclosed in sealed containers made of annealed silver of 999.5 fineness (cylinders 1.8 inches long and of 5/8 in. diameter). After introducing the sample and enough extra silver to adjust the total final mass of this metal to a standard value (exactly 12 g.), the protruding edges of the two end caps (each 0.02 in. thick) were flame-welded to the ends of the cylinder (wall thickness, 0.015 in.). Since the sample and container had an average temperature of approximately 700°C when the final sealing was accomplished, the amount of air sealed in was insufficient to distort the container by internal pressure at the highest temperatures subsequently used (900°C). A 0.025 in. Pt-10% Rh wire of known mass encircled a groove around the silver cylinder and served to suspend it in the furnace and calorimeter.

There are certain advantages in accurate work in using a container composed of silver instead of a hard base metal such as 80 Ni-20 Cr, which the authors have often used [8]. Unlike silver, the alloy mentioned undergoes a transition near 550°C, shows small but detectable changes in heat capacity attributed to minor annealing effects, and through traces of oxidizing gases accumulates surface coatings of oxides that must be measured by frequent reweighing and be corrected for. It appears that during the short time that a silver container is falling into the calorimeter the radiation at the highest temperatures, which must be constant to avoid error in the heat measurements, decreases slowly but

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Table 1. Analyses of Samples

Percent impurity in sample			
Element	MgO	"ZrO ₂ "	"ZrSiO ₄ "
Ag	< .001*	-	< 0.01*
Al	.004	0.1 - 1*	0.1 - 1*
Ca	.025	0.1 - 1*	< 0.01*
Cr	< .001*	-	-
Cu	< .001*	< .001*	< 0.01*
Fe	.020	0.01 - 0.1*	0.01 - 0.1*
Hf	-	1 - 10*	1 - 10*
Mg	>10 ^{*a}	1 - 10*	< 0.01*
Mn	.008	< .01*	< 0.0001*
Na	< .002	0.1 - 1*	-
Si	.009	0.1 - 1*	
SiO ₂			33.2 ^{ab}
Ti	-	0.1*	0.1 - 1*
Zr	-	>10 ^{*a}	
ZrO ₂ +HfO ₂			65.4 ^{ab}

^a Principle element.

^b Gravimetric chemical analysis was used here.

* Qualitative spectrochemical analysis.

- not detected.

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measurably from run to run. This effect presumably occurs through grain growth and consequent lowering of the emissivity of the silver surfaces, but the error appears to be negligible if the container is conditioned by being held for several hours in the furnace at 900°C before the heat measurements are begun. Two additional general disadvantages may be mentioned. The mechanically sealed base-metal containers frequently develop leaks. On the other hand, silver containers as described above would be inapplicable to samples which, unlike the refractory oxides, must be kept cold during the sealing to prevent volatilization or must be kept in an inert atmosphere to prevent reaction with air. Both disadvantages could obviously be avoided by substituting the superior sealing technique of induction welding.

IV. MEASUREMENTS OF HEAT CONTENT

The calorimetric apparatus and method used to measure the enthalpy have been described in detail in several reports and papers from this Laboratory during the past thirteen years [1]*.

A drawing of the apparatus is shown in Figure 1. Briefly, the experimental method was as follows. The weighed specimen, in a thin-wall silver container, was suspended in the furnace for a sufficient length of time to ensure its reaching the known constant temperature of the furnace. Sample plus container were then dropped (with almost free fall) into the Bunsen ice calorimeter, which, by absorbing mercury equivalent in volume to the shrinkage of the ice melted, measured the heat evolved by the sample and container in cooling to 0°C. A similar measurement on an empty container at the same furnace temperature provided correction for the contribution for the heat the container itself delivered and for the small amount of heat lost during the fall, thus enabling a simple calculation of the net heat-content change of the specimen alone. Repetition at a sufficient number of furnace temperatures establishes the heat content as a function of temperature, and differentiation of this function with respect to temperature gives the true heat capacity at any temperature in the range of the measurements.

* Numbers in brackets refer to literature references at the end of this report.

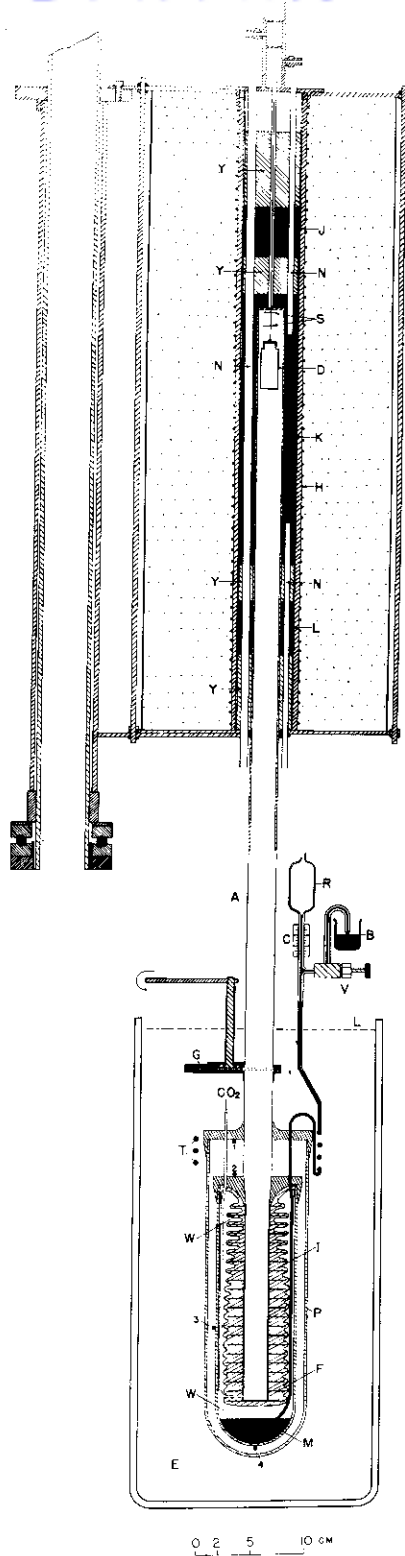


Figure 1. Calorimetric Apparatus Used

A, Calorimeter well; B, beaker of mercury; C, glass capillary; D, sample container; E, ice bath; F, copper vanes; G, gate; I, ice mantel; JH, KH, LH, furnace heater leads; J, K, L, silver cylinders; M, mercury; N, Inconel tubes; P, Pyrex vessels; R, mercury reservoir; S, platinum shields; T, mercury "tempering" coil; V, needle valve; W, water; Y, porcelain spacers.

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The temperature of the furnace was measured by an accurately calibrated strain-free platinum resistance thermometer up to 600°C and by a calibrated platinum--platinum-10% rhodium thermocouple above 600°C. During a run the furnace temperature was held constant usually to within ± 0.01 deg C. The 10-inch-long silver pipe of 1/2-inch wall thickness surrounding the specimen, together with end silver pieces, reduced temperature gradients to negligible amounts and ensured that the specimen assumed the same temperature as that measured. That the time of the specimen in the furnace was adequate was established by determining the relaxation time in a special run deliberately made too short to give thermal equilibrium, and also by varying the time in most sets of duplicate measurements at the same furnace temperature in order to detect any systematic trend with time. The furnace core and calorimeter were filled with pure helium gas, which flowed through during the measurements to minimize not only the times required for reaching thermal equilibrium but also the oxidation of the specimen container. Corrections (all relatively very small) were applied for such non-standard conditions as deviation of the furnace temperatures from the nominal values and the heat leak of the calorimeter, the latter averaging several tenths of a calorie per hour. The calibration factor of the ice calorimeter had been determined electrically by several hundred experiments in this laboratory several years ago to be 270.48 absolute joules per gram of mercury absorbed, and is believed to be accurate to ± 0.01 percent.

It may be of interest to cite here a few tests of the accuracy of the present apparatus. The heat capacity of standard-sample aluminum oxide was measured in both this apparatus and also a precise low-temperature adiabatic calorimeter at the Bureau over the range 0° to 100°C, the maximum difference in heat capacity from the two apparatuses being 0.25 percent [1]. A similar comparison with a new precise high-temperature adiabatic calorimeter at the Bureau showed discrepancies in heat capacity of approximately 0.1 percent at about 200° and 400°C. The mean heat capacity of water was compared [1] with the results from still other adiabatic calorimeters at the Bureau, the discrepancy amounting to 0.05 ± 0.14 percent over the interval 0° to 25°C, and 0.02 ± 0.02 percent over the interval 0° to 250°C. These other calorimetric apparatus are all of the highest precision and are believed to be a little more accurate than the present "drop" method apparatus except possibly at temperatures as high as 400°C. A general discussion of the errors of the "drop" method by one of the present authors has been published [2].

V. SPECIFIC HEATS OF MAGNESIUM OXIDE, ZIRCONIUM OXIDE

AND ZIRCONIUM SILICATE

The mean observed heat-content values for magnesium oxide given in table 2, column 4, were smoothed by using them to derive (by the method of least squares) the coefficients of an empirical equation to represent the heat content as a function of the temperature. The form of equation was chosen to blend most smoothly with existing low temperature specific heat data, although the low temperature values were given no weight in deriving the coefficients. Actual blending of low- and high-temperature specific heats will be discussed in the next section. The least-squares equation representing the heat content of the sample in excess of the heat content at 0°C, in calories* per gram at t°C, is

$$H_t - H_0 = 0.33985t - 5.573 (10^{-7})t^2 - 81.0286 \log_{10} [(t+273.15)/273.15] \quad (1)$$

Differentiation of equation (1) with respect to temperature gives the heat capacity in calories per gram-degree C or specific heat (which is the same as in BTU per lb.-deg. F),

$$C_p = 0.33985 - 1.1146 (10^{-6})t - 35.190/(t+273.15) \quad (2)$$

The mean observed heat-content values for zirconium oxide given in table 2, column 6, were used to derive coefficients of equation (3) in calories per gram.

$$H_t - H_0 = 0.15238t + 14.90 (10^{-6})t^2 - 24.484 \log_{10} [(t+273.15)/273.15] \quad (3)$$

Differentiation of equation (3) with respect to temperature gives equation (4) for specific heat in calories per gram-degree C.

$$C_p = 0.1524 + 2.98 (10^{-5})t - 10.633/(t+273.15) \quad (4)$$

* One calorie = 4.1840 absolute joules.

Table 2. Individual Measurements of Heat Content

Furnace Temperature t (°C)	Mean Heat content of empty container (calories)	MgO		ZrO ₂		ZrSiO ₄	
		Observed heat content cal/g	Mean heat content cal/g	Observed heat content cal/g	Mean heat content cal/g	Observed heat content cal/g	Mean heat content cal/g
100	73.62	22.91 22.89	22.90	11.92 11.93	11.92	13.54 13.54	13.54
200	149.57	48.62 48.64	48.63	25.27 25.20	25.24	29.26 29.27	29.27
300	227.98	75.90 75.84	75.87	39.38 39.20 39.28	39.28	46.01 46.02	46.02
400	306.94	104.04 104.26 104.02	104.11	53.72 53.77	53.74	63.66 63.61	63.64
500	387.64	133.32 133.21 133.35	133.29	68.87 68.93	68.90	82.01 81.90	81.96
600	469.70	162.71 162.65	162.68	84.42 84.37	84.39	100.45 100.44	100.45
700	554.40	192.90 192.79	192.85	100.45 ^a 100.48 100.42	100.45	119.48 119.57 119.45	119.50
800	642.08	223.40 223.44	223.42	116.82 116.75	116.78	138.57 138.67	138.62
900	731.02	254.11 254.14	254.12	134.10 133.87 133.90 133.70 133.58	133.96 133.80 ^b 133.64	158.34 158.38	158.36

^a Two other values 100.81 and 100.65 were discarded because of inferior precision.

^b The value 133.80 is chosen as the average of two sets.

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Equation (5), representing the heat content of zirconium silicate in calories per gram, was derived in the same manner as equations (1) and (3).

$$H_t - H_0 = 0.17945t + 12.05 (10^{-6})t^2 - 16.833 (t/(t+273.15)) \quad (5)$$

Differentiation of equation (5) with respect to temperature gives equation (6), which represents the specific heat of zirconium silicate in calories per gram-deg C.

$$C_p = 0.17945 + 2.41 (10^{-5})t - 4.598 (10^3)/(t+273.15)^2 \quad (6)$$

In table 3 the heat-content and specific-heat values obtained from equations (1) through (6) are tabulated every 50°C. One requirement which is put on heat-content equations is that the differences between observed and calculated values of heat content should be comparable in magnitude to the differences between duplicate observed results at the same temperature. Comparison of tables 2 and 3 shows that this requirement is generally satisfied by the equations used in this report.

VI. COMPARISON OF SPECIFIC HEAT VALUES

WITH THOSE OF OTHER WORKERS

The solid lines in figures 2 and 3 represent the specific heats as obtained from equations (4) and (6) respectively. The open circles represent the "mean specific heats" of the present investigation. These are obtained from the differences between adjacent mean heat contents in table 2 with small corrections for curvature.

The "dashed" lines in figures 2 and 3 represent the heat capacity values of Coughlin and King [3] for zirconium oxide and zirconium silicate respectively. Coughlin and King's data on zirconium oxide have been adjusted by the additive contribution of 4.1 percent magnesium oxide to correspond to the "stabilized" composition used in the present study. The "dot-dash" line in figure 2 represents the specific heat of a zirconium oxide sample similar in composition to, but of lower density than, the one described in this report. The specific heat of this low density specimen was measured at the National Bureau of Standards several years ago.

The specific heats of zirconium oxide and zirconium silicate from the present study have not been compared with low-temperature data. It was felt that the impurity of the specimens would negate the value of such a comparison.

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Table 3. Heat Content and Specific Heat of Magnesium Oxide, Zirconium Oxide, and Zirconium Silicate from 0° to 900°C.

Temperature °C	Heat Content			Specific Heat		
	MgO	ZrO ₂	ZrSiO ₄	MgO	ZrO ₂	ZrSiO ₄
	calories/gram			calories/gram-deg C		
0	0.00	0.00	0.00	.2110	.1134	.1178
50	11.08	5.87	6.40	.2309	.1210	.1366
100	23.00	12.07	13.55	.2454	.1269	.1488
150	35.56	18.54	21.22	.2565	.1317	.1574
200	48.61	25.23	29.26	.2652	.1359	.1637
250	62.06	32.12	37.57	.2723	.1395	.1687
300	75.82	39.17	46.11	.2781	.1428	.1727
350	89.86	46.39	54.83	.2830	.1458	.1760
400	104.11	53.74	63.70	.2871	.1485	.1789
450	118.56	61.24	72.72	.2907	.1511	.1815
500	133.17	68.85	81.85	.2938	.1535	.1838
550	147.93	76.59	91.09	.2965	.1558	.1859
600	162.82	84.44	100.44	.2989	.1581	.1879
650	177.81	92.39	109.88	.3010	.1602	.1897
700	192.91	100.46	119.41	.3029	.1623	.1915
750	208.10	108.62	129.03	.3046	.1643	.1931
800	223.37	116.89	138.72	.3061	.1663	.1947
850	238.72	125.25	148.50	.3076	.1682	.1963
900	254.13	133.72	158.35	.3088	.1701	.1978

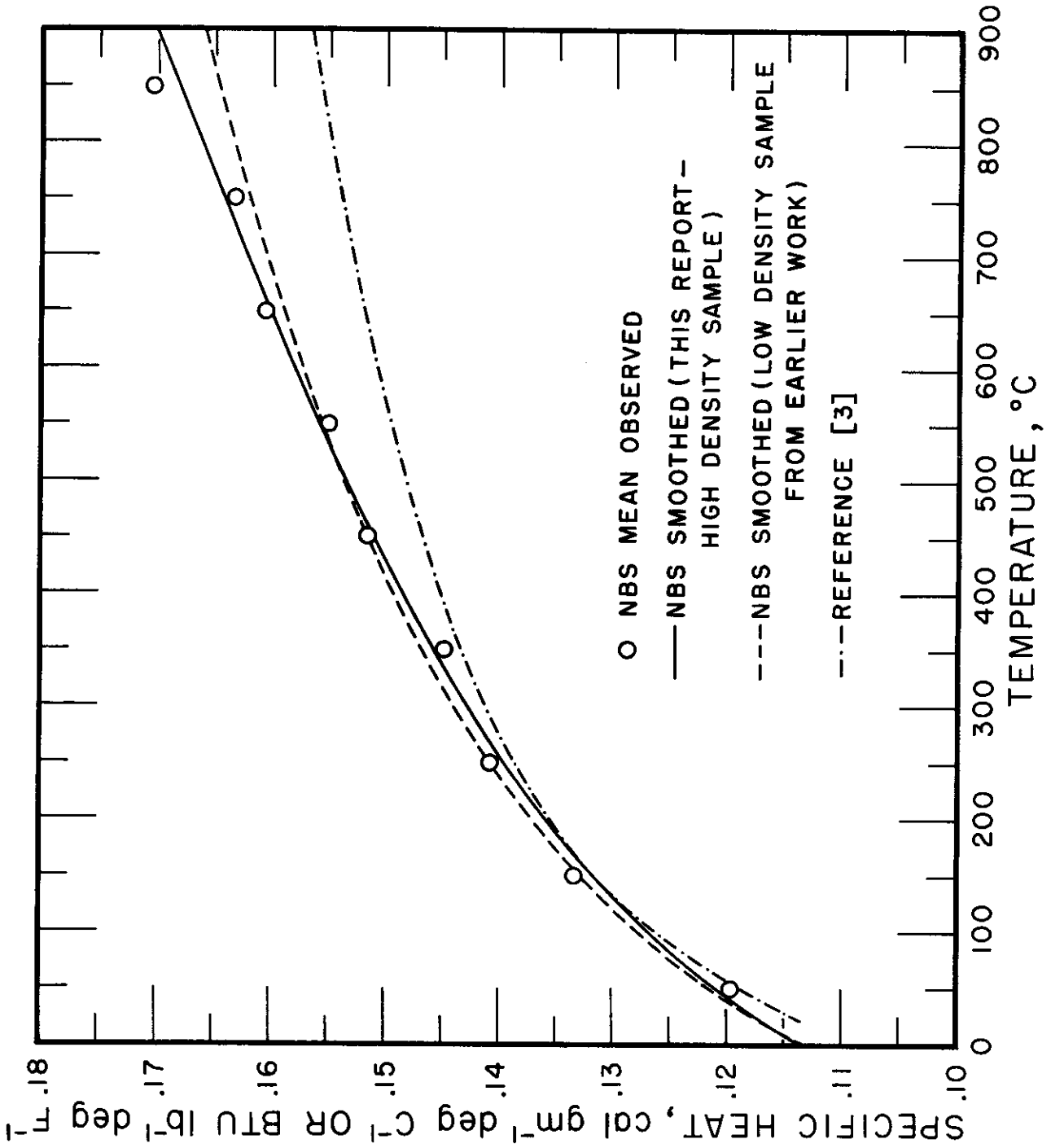


Figure 4. Specific Heat of Zirconium Oxide

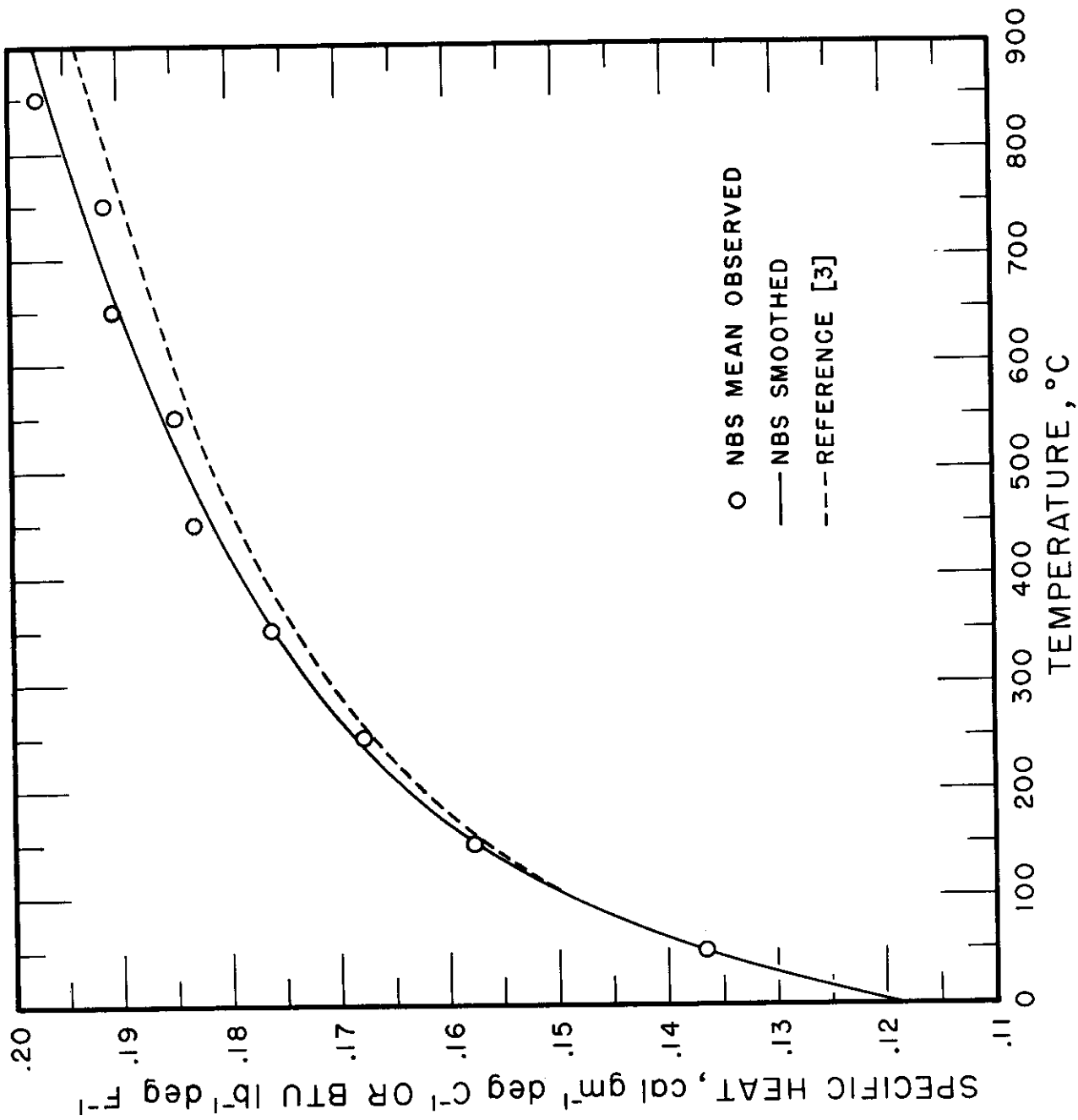


Figure 3. Specific Heat of Zirconium Silicate

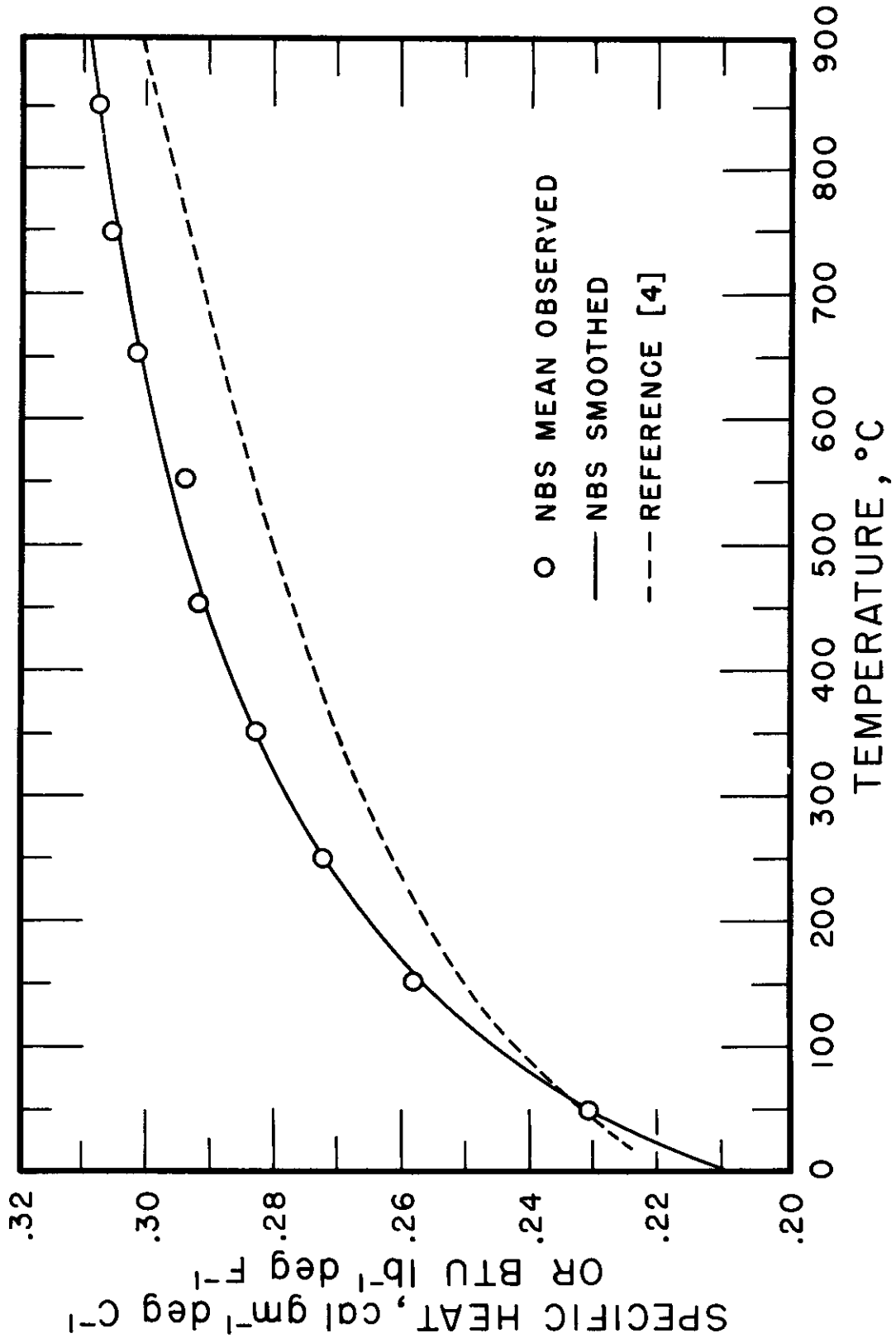


Figure 4. Specific Heat of Magnesium Oxide

Table 4. Molar Heat Capacity of Magnesium Oxide
from 273.15° to 450°K.

T °K	Cp cal/mole °K	T °K	Cp cal/mole °K	T °K	Cp cal/mole °K
273.15	8.409	310	9.107	390	10.058
275	8.450	320	9.261	400	10.148
280	8.556	330	9.401	425	10.355
285	8.658	340	9.530	450	10.541 ^a
290	8.756	350	9.649		
295	8.850	360	9.760		
298.15	8.906	370	9.865		
300	8.939	380	9.964		

^a

Above 450°K use equation (2) with the following modifications: Multiply eq (2) by 40.32 (mol. wt. of MgO₂); note that T°K = 273.15 + t°C.

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The specific heat of MgO obtained in the present work is compared with the values chosen by K. K. Kelley [4] in figure 4. The solid line represents the specific heat of equation (2) modified by Table 4 below 177°C (details later in this section). The authors believe that the values given in column 5 of table 3 are accurate to ± 0.5 percent at and above 200°C and hence a great improvement over the earlier values (shown in figure 2), from which they differ by as much as 4 percent.

The low temperature specific heat of MgO has been measured by Parks and Kelley [5] (94° to 290°K), Giaugue and Archibald [6] (20° to 301°K) and Barron, Berg, and Morrison [7] (10° to 270°K). Giaugue and Archibald made their measurements on finely divided powder. The specific heat of the powder is about one percent higher at 25°C than that of the macro crystal. The present work was done with macro-crystalline MgO and for this reason should be compared with low temperature work on the same form [5,7].

The data of Barron, Berg, and Morrison join fairly smoothly with the extrapolated MgO results of this report although equation (2) yields a specific heat 1% higher at 0°C than the low temperature work. Slight adjustment of the high temperature data below 180°C to the low temperature values [7] must be made to give a smooth, continuous heat capacity curve from -273°C to 900°C when coupled. The results of such adjustment, which was done by G. Furukawa, M. Reilly and J. H. Piccirelli of the Bureau staff, are given in table 4. For some purposes the data are likely to be more useful in calories per mole-deg. K at temperature T°K; for this reason they have been tabulated in these units in table 4. In these units the heat-capacity equation in calories per mole-deg. K becomes (for use above 450°K)

$$C_p = 13.715 - 4.494 (10^{-5}) T - 1.4189 (10^3)/T. \quad (7)$$

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