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To: Members of the Lead
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We are attaching a reprint from the Journal of the American Ceramic Society entitled, "Dilatometric and X-Ray Data for Lead Compounds, II".

As you can see, this is the second in a series of papers on thermal-expansion properties of crystalline lead compounds. One of the authors of this paper is the LIA-Expanded Research Project fellow at The Pennsylvania State University, working under a ceramic research grant. The other author is his faculty supervisor and Chairman of the Dept. of Ceramic Technology.

Printing of this paper in the Ceramic Society Journal has, of course, given it wide distribution throughout the ceramic industry, and we have purchased only a limited number of reprints for distribution where we think it will be beneficial.

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Dilatometric and X-Ray Data for Lead Compounds, II

by J. F. ARGYLE and F. A. HUMMEL

College of Mineral Industries, The Pennsylvania State University, University Park, Pennsylvania

Linear thermal-expansion data are presented for $PbAl_2O_4$, Pb_2GeO_4 , Pb_2MoO_4 , $PbWO_4$, the perovskite-type structures Pb_2MgWO_4 , Pb_2FeTaO_4 , and Pb_2FeWO_4 , and the apatite-type structures $Pb_2(SiO_4)(VO)_2$, $Pb_2(GeO_4)(VO)_2$, and $Pb_2(GeO_4)(PO_4)_2$. X-ray powder data are given, and the behavior of the compounds is correlated with previous information from the literature.

I. Introduction

It was pointed out in Part I of this series¹ that more data on the thermal expansion behavior of crystalline lead compounds are necessary to predict better their behavior in complex ceramic systems composed of crystal mixtures or glass-crystal aggregates.

Data for ten compounds are presented in this paper, three of which have the perovskite type structure, three the apatite-type structure, and two the scheelite type structure.

II. Literature

(1) Perovskite-Type Compounds

Smolenskii, Agramovskaya, and Isupov² claimed that Pb_2MgWO_4 , $(2PbO \cdot MgO \cdot WO_3)$, Pb_2FeTaO_4 , $(1PbO \cdot Fe_2O_3 \cdot Ta_2O_5)$, and Pb_2FeWO_4 , $(3PbO \cdot Fe_2O_3 \cdot WO_3)$ all had a perovskite type structure.

(2) Apatite-Type Compounds

Merker and Wondratschek³ showed that $Pb_2(SiO_4)(VO)_2$, $(3PbO \cdot SiO_2 \cdot V_2O_5)$, $Pb_2(GeO_4)(VO)_2$, $(3PbO \cdot GeO_2 \cdot V_2O_5)$, and $Pb_2(GeO_4)(PO_4)_2$, $(3PbO \cdot GeO_2 \cdot P_2O_5)$ all had an apatite-like structure. They gave the a and c parameters, c/a ratios, and congruent melting points as follows:

Compound	a (Å)	c (Å)	c/a	Congruent melting point (°C)
$Pb_2SiO_4 \cdot V_2O_5$	9.199	7.35	0.799	882
$Pb_2GeO_4 \cdot P_2O_5$	9.388	7.32	0.741	1010
$Pb_2GeO_4 \cdot V_2O_5$	10.06	7.37	0.733	913

(3) Lead Molybdate and Lead Tungstate

Jager and Germs⁴ used heating curves and thermal analysis to determine melting points, inversions, and liquidus curves in the systems $PbO \cdot WO_3$ and $PbO \cdot MoO_3$. They claimed

congruent melting for $PbMoO_4$ at 1065°C and for $PbWO_4$ at 1121°C . $PbWO_4$ was reported to undergo a reversible polymorphic transition at 877°C .

(4) Lead Aluminate and Lead Germanate

Geller and Bunting⁵ worked in the system $PbO \cdot Al_2O_3$ and suggested the existence of a compound $PbO \cdot Al_2O_3$. This compound breaks down rapidly at 1000°C to form beta Al_2O_3 and liquid. These investigators pointed out that this breakdown may be true incongruent melting or it may be a decomposition accelerated at 950° to 1000°C by the volatilization of PbO .

Schutz⁶ claimed that $2PbO \cdot GeO_2$ melted in an Al_2O_3 crucible formed a homogeneous glass. Merker and Wondratschek³ showed that $2PbO \cdot GeO_2$ melted in a platinum crucible rapidly crystallizes, and suggested that the glass obtained by Schutz⁶ was probably stabilized by dissolved Al_2O_3 from the crucible.

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The writers are, respectively, Lead Expanded Research Program fellow and professor, Department of Ceramic Technology, College of Mineral Industries, The Pennsylvania State University.

¹ J. F. Argyle and F. A. Hummel, "Dilatometric and X-Ray Data for Lead Compounds, I," *J. Am. Ceram. Soc.*, **43** [9] 452-57 (1960).

² G. A. Smolenskii, A. I. Agramovskaya, and V. A. Isupov, "New Ferroelectrics of Complex Composition: III. Pb_2MgWO_4 , Pb_2FeWO_4 , and Pb_2FeTaO_4 ," *Fiz. Tverd. Tela*, **1**, 90-92 (1959).

³ Ludwig Merker and Hans Wondratschek, "Compounds of Apatite Type Structure," *Fortschr. Mineral.*, **35**, 15-46 (1957).

⁴ P. M. Jager and H. C. Germs, "The Sulphate, Chromate, Molybdate and Tungstate Binary Systems of Lead," *Z. anorg. u. allgem. Chem.*, **119**, 145-73 (1921).

⁵ R. F. Geller and E. N. Bunting, "Report on the Systems Lead Oxide-Alumina and Lead Oxide-Alumina-Silica," *J. Research Natl. Bur. Standards*, **51** [5] 255-70 (1943); RP 1594; *Ceram. Abstr.*, **23** [3] 50 (1944).

⁶ Wolfgang Schutz, "Die Kristallchemische Verwandtschaft zwischen Germanium und Silicium," (Crystal Chemical Relation Between Germanium and Silicon), *Z. physik. Chem.*, **B31**, 292-308 (1930); *Ceram. Abstr.*, **18** [1] 15 (1939).

⁷ Ludwig Merker and Hans Wondratschek, "Einige Bemerkungen zum Bleisulfidat und germanat und deren Glasigkeit" (Lead Orthosulfate and Germanate, and Their Glassy Character), *Glastech. Ber.*, **30** [11] 671-73 (1957).

Table I. Caking and Firing Treatments for Lead Compounds

Compound	Calcination			Firing of bars (Temp., °C)
	First (Temp., °C)	Second (Temp., °C)	Time (hr)	
2PbO·MgO·WO ₃	800	900	24	900
4PbO·Fe ₂ O ₃ ·Ta ₂ O ₅	800	900	24	900
3PbO·Fe ₂ O ₃ ·WO ₃	600	700	24	700
5PbO·SnO ₂ ·V ₂ O ₅	400	700	24	470
5PbO·GeO ₂ ·V ₂ O ₅	400	700	24	400
3PbO·GeO ₂ ·P ₂ O ₅	450	700	24	510
PbO·WO ₃	850	1000	24	1000
PbO·MoO ₃	850	1050	24	1050
PbO·Al ₂ O ₃	750	900	12	900
2PbO·GeO ₂	400	400	12	400

III. Experimental Procedures

All compositions were made from cp chemicals. After weighing, the batches were hand mixed in a glass mortar with acetone for 15 to 20 minutes, air dried, and then heated slowly in a Gaylor furnace to 300°C and held for 4 hours to expel volatiles. The batches were remixed, given a "first calcination" for 24 hours, cooled, remixed, and given a "second calcination," as shown in Table I.

After the second calcination, the batches were ground, mixed with a Carbowax solution, modulated through a 20-mesh sieve, and pressed into 10- by 1- by 1-cm bars at 1000 psi. The specimens were fired slowly to the temperatures indicated in Table I and held for 6 hours until the bar was sintered to a relatively dense condition.

Note that several volatile oxides (PbO, WO₃, V₂O₅, P₂O₅, MoO₃, and GeO₂) were involved in the preparation of these compositions. By the use of repeated low-temperature calcining, repeated grinding and mixing, and carefully chosen final sintering temperatures for each composition, it is highly probable that losses due to volatilization were negligible.

Thermal expansion runs were made in a vertical fused silica dilatometer, using a heating rate of 120°C per hour. The expansions were measured by dual gages which were accurate to 1/10,000 in. At least two runs were made on each specimen. Any flattening of the expansion curve near its high-temperature extremity is probably due to deformation of the specimen under the weight of the fused silica transmission tube (about 55 g).

X-ray diffraction patterns were obtained on a Norelco diffractometer using Cu K α radiation. The specimens were ground to pass a 200-mesh sieve and packed into sample holders in such a way as to minimize the possibility of any preferred orientation of the crystal aggregate. The samples were run between 10° and 80° 2 θ at 1° per minute, this rate being adequate to give patterns suitable for qualitative identification purposes. The strongest peak of each pattern was fully accommodated on the recording chart by placing thin nickel foils in front of the counter tube when peak intensities required it. High-temperature X-ray patterns were obtained with a platinum wound furnace mounted on a Norelco diffractometer. A finely ground specimen was packed into a Pt depression slide and sintered in place for several hours at a temperature about 50°C higher than the temperature subsequently used to obtain the diffraction pattern. This procedure was used to prevent any effect of sintering on the positions or intensities of the X-ray reflections at elevated temperatures.

IV. Experimental Results and Discussion

(1) Perovskite-Type Structures

The linear thermal expansion curves of 2PbO·MgO·WO₃, 4PbO·Fe₂O₃·WO₃, and 4PbO·Fe₂O₃·Ta₂O₅ are shown in Fig.

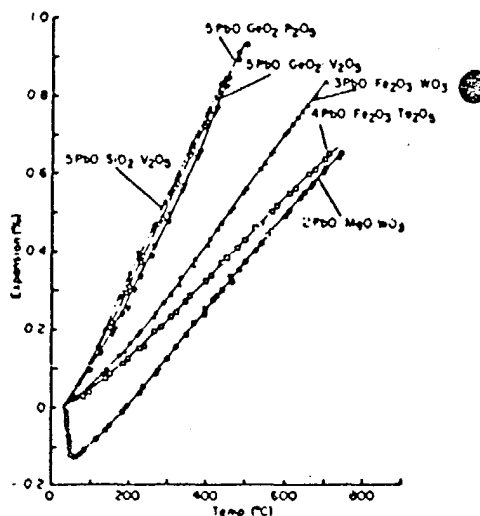


Fig. 1. Linear thermal expansion of perovskites and apatites.

1. 3PbO·Fe₂O₃·WO₃ and 4PbO·Fe₂O₃·Ta₂O₅ have continuous expansion curves, but 2PbO·MgO·WO₃ shows a discontinuity, with a marked negative expansion beginning at about 40°C. Smolenskii, Agranovskaya, and Isupov¹ claimed that 2PbO·MgO·WO₃ undergoes an antiferroelectric transition at 39°C, which corresponds to the temperature below which this compound shows a marked negative expansion.

The X-ray diffraction data for these three compounds are shown in Table II.

(2) Apatite-Type Structures

The linear thermal expansion characteristics of 5PbO·GeO₂·P₂O₅, 5PbO·SnO₂·V₂O₅, and 5PbO·GeO₂·V₂O₅ are

Table II. X-Ray Diffraction Data for Compounds with Perovskite-Type Structure

3PbO·Fe ₂ O ₃ ·WO ₃		4PbO·Fe ₂ O ₃ ·Ta ₂ O ₅		2PbO·MgO·WO ₃	
d(A)	I, %	d(A)	I, %	d(A)	I, %
3.99	15	4.02	9	4.82	26
				4.10	6
				3.26	2
3.01	5	3.05	9		
2.81	100	2.84	100	2.83	100
2.61	3	2.64	4	2.55	3
				2.42	7
2.30	13	2.32	9	2.32	18
				2.15	4
				2.01	15
1.890	31	2.00	27	1.989	14
1.841	2	1.863	3	1.834	4
1.774	3			1.781	2
				1.716	2
1.626	35	1.637	31	1.657	21
				1.631	18
1.570	1	1.580	3		
		1.526	1		
1.408	15	1.417	11	1.410	10
				1.356	1
				1.274	5
1.250	10	1.268	9	1.250	4
1.150	3	1.158	2	1.155	6

Table III. X-Ray Diffraction Data for Compounds with Apatite-Type Structure

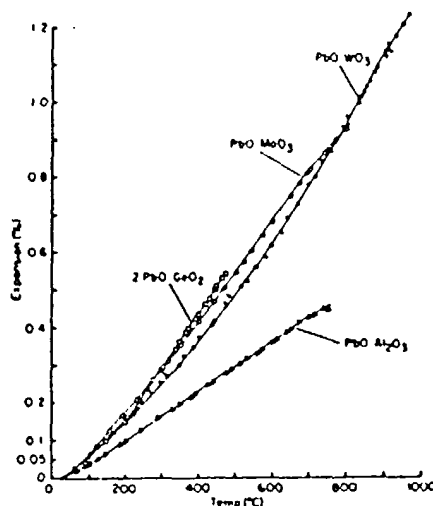
SPMO GLEN PAW		SPMO GLEN PAW		SPMO GLEN PAW	
d(A)	I/I ₀	d(A)	I/I ₀	d(A)	I/I ₀
8.51	5	8.67	4		
		6.37	8	5.34	5
4.90	3	4.46	3		
4.25	20	4.33	32	4.31	34
4.08	20	4.12	30	4.19	5
3.66	8	3.98	15	4.11	35
3.36	20	3.39	30	3.96	6
3.22	34	3.29	42	3.47	3
		3.19	18	3.38	31
		3.13	27	3.27	43
2.92	100	2.99	100	3.00	11
		2.97	99	3.04	5
2.84	47	2.90	56	2.98	92
2.77	4	2.85	67	2.96	55
		2.76	3	2.92	3
2.25	4	2.39	4	2.37	3
2.19	9	2.24	11	2.26	5
2.14	5	2.21	9	2.23	9
		2.18	8	2.16	8
				2.11	5
2.04	20	2.08	20	2.06	21
1.940	13	2.02	13	2.01	15
		2.01	6	1.985	6
1.901	7	1.960	20	1.901	22
1.915	20	1.929	22	1.914	22
		1.903	13		
1.892	14	1.877	27	1.884	18
1.846	10			1.866	25
1.845	20	1.845	18	1.838	8
1.831	10	1.749	11		
1.681	4	1.662	6		
1.663	4	1.659	5		
		1.679	6	1.634	8
1.613	5			1.623	9
1.605	5	1.608	9	1.603	9
1.593	5	1.593	9	1.580	2
		1.578	6		
		1.598	4	1.568	5
1.549	5	1.554	16	1.551	12
1.542	10	1.545	9	1.544	9
1.530	9	1.530	13	1.519	12

shown in Fig. 1. All the curves are continuous and the three compounds have almost identical linear thermal expansions. During the final firing of these bar specimens it was noted that the edges became slightly rounded above about 500°C; the firing therefore was halted at this temperature since good sintering had already produced a suitable expansion specimen. Under the optical microscope only the apatite phase was observed in these specimens, making it highly likely that the rounding process was due to loss of oxide components from the specimen surface. Differential thermal analysis runs were made on small portions of the bar specimens, giving melting temperatures of 875°C (5140: $\text{SrO} \cdot \text{V}_2\text{O}_5$), 995°C (5140: $\text{GeO}_2 \cdot \text{P}_2\text{O}_5$), and 905°C (5140: $\text{GeO}_2 \cdot \text{V}_2\text{O}_5$); each temperature was 7° to 8° below those reported by Merker and Wondratschek.³

The X-ray diffraction data for these compounds are shown in Table III.

(3) Lead Tungstate and Lead Molybdate

The linear thermal expansion curves of PbWO_4 and PbMoO_4 are shown in Fig. 2. Jaeger and Gerns⁴ claimed that PbWO_4 has a reversible inversion at 877°C, but the present expansion curve does not show an inflection at this point and a DTA run showed no heat effect.

Fig. 2. Linear thermal expansion of scheelite-type compounds, $\text{PbO} \cdot \text{Al}_2\text{O}_3$ and $2\text{PbO} \cdot \text{GeO}_2$.

X-ray diffraction data were then obtained between room temperature and 925°C as shown in Table IV. It is interesting to note that both PbWO_4 and PbMoO_4 , after relatively rapid cooling to room temperature (or near room temperature) do not give patterns which agree with the original room temperature patterns (which are in essential agreement with ASTM data⁵ for the two compounds). If, however, the specimen is left overnight, very close agreement with the original pattern is obtained. These data indicate a probable rearrangement of the heavy metal atoms in these compounds at high temperatures and a very pronounced dependence on time in returning to the room temperature configuration.⁶ The line splitting observed in PbMoO_4 at high temperatures may indicate a transition from the tetragonal scheelite structure to one of lower symmetry.

The two compounds have the same space group as CaWO_4 ($C_4h/a - 14/a$) and similar lattice parameters as follows:

	a	c	Reference
CaWO_4	5.242	11.372	(8)
PbMoO_4	5.435	12.11	(8)
	5.41	12.08	(9)
PbWO_4	5.4010	12.046	(8)
	5.44	12.01	(9)

Araki¹⁰ has shown that the Pb ions are asymmetrically surrounded by oxygens in PbMoO_4 , similar to other lead com-

⁵ ASTM Data Files, American Society for Testing and Materials, Philadelphia, Pa.

⁶ L. G. Silfen and Anna Lisa Nylander, "Oxygen Positions in Tungstates and Molybdates with Scheelite Structure," *Arkiv. Kemi, Mineral. Geol.*, 17A (4) 1-27 (1943).

¹⁰ Takaharu Araki, "Crystal Structure of Wulfenite, PbMoO_4 ," *Mem. Coll. Sci. Univ. Kyoto, Ser. B*, 24, 155-69 (1957) (in English).

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Table IV. X-Ray Diffraction Data for PbWO_4 and PbMoO_4

PbWO_4											
Room temp		170°C (heating)		170°C		300°C (cooling)		325°C (cooling)		Room temp (after cooling)	
d (Å)	$1/d$	d (Å)	$1/d$	d (Å)	$1/d$	d (Å)	$1/d$	d (Å)	$1/d$	d (Å)	$1/d$
3.24	100	3.28	100	3.29	100	3.28	100	3.28	100	3.24	100
3.01	29	3.08	54	3.09	73	3.08	10	3.08	10	3.01	29
2.72	28	2.75	100	2.75	90	2.74	100	2.74	100	2.72	67
2.39	2	2.40	7	2.41	6	2.41	7	2.41	4	2.39	6
2.37	1										
2.08	1										
2.02	33	2.04	51	2.05	48	2.04	93	2.04	32	2.02	11
1.929	15	1.941	47							1.929	2
		1.911	47	1.917	25	1.921	14	1.907	4		
1.781	34									1.781	44
1.658	22	1.672	37	1.675	35	1.678	10	1.672	18	1.658	74
1.623	15	1.639	26	1.647	40	1.645	18	1.642	27	1.623	13
				1.547	19						
1.503	3									1.503	31
1.365	4	1.375	12			1.377	4			1.365	4
				1.347	19	1.342	4	1.344	4		
1.318	9	1.328	28	1.331	29	1.328	4	1.328	75	1.318	31
1.298	10									1.298	6
1.259	5	1.269	54							1.259	4
1.247	3	1.255	19	1.256	8	1.256	4			1.247	11
1.221	4									1.221	40
		1.207	21								
1.187	5			1.180	8	1.183	6			1.187	13
						1.175	12	1.172	15		
PbMoO_4											
Room temp		170°C (heating)		300°C		300°C (cooling)		350°C for 2 hr (cooling)		300°C for 2 hr (cooling)	
d (Å)	$1/d$	d (Å)	$1/d$	d (Å)	$1/d$	d (Å)	$1/d$	d (Å)	$1/d$	d (Å)	$1/d$
4.93	15	4.98	15	5.01	8	5.01	3	5.01	13	4.98	20
3.23	100	3.27	100	3.31	50	3.29	100	3.28	80	3.24	100
				3.29	54						
3.02	20	3.07	20	3.12	100	3.11	23	3.08	26	3.03	42
						3.10	64				
2.71	19	2.74	19	2.75	33	2.75	27	2.74	18	2.71	21
2.37	7	2.40	7	2.41	9	2.40	11	2.40	12	2.38	28
2.21	4	2.24	4	2.28	5			2.20	8	2.21	6
										2.11	3
2.08	3	2.10	3	2.11	12	2.11	34	2.11	41	2.09	46
								2.10	37	2.08	44
2.02	24	2.04	24	2.06	68	2.05	63	2.05	90	2.02	38
1.907	8	1.917	8	1.941	21	1.941	16	1.945	100	1.922	28
								1.937	92		
1.784	13	1.814	13	1.828	26	1.821	26	1.821	18	1.787	51
				1.811	9	1.811	9				
1.653	17	1.662	17	1.672	11	1.672	9	1.670	20	1.653	50
1.621	11	1.637	11	1.645	12	1.645	7	1.642	7	1.623	5
1.512	4	1.540	4	1.558	17					1.514	3
				1.375	9						
PbMoO_4 for 1 hr											
d	$1/d$										
3.29	100										
3.13	33										
3.10	33										

pounds, such as the oxide, for example. Introduction of lead into the structure not only expands the lattice (especially in the c direction) but also brings about a change in the local environment of the lead ion and in the symmetry of the structure at high temperature relative to room temperature.

The high temperature diffraction patterns of PbWO_4 and PbMoO_4 were obtained by running at 1° per minute, and at some temperatures the specimens may not have been fully equilibrated to the extent needed for good thermal expansion data. The high temperature X-ray data therefore were not used for the calculation of α and ϵ expansion coefficients or for comparison with the linear dilatometric data.

(4) Lead Germanate and Lead Aluminate

The linear thermal expansion characteristics of $2\text{PbO} \cdot \text{GeO}_2$ and $\text{PbO} \cdot \text{Al}_2\text{O}_3$ are shown in Fig. 2.

The X-ray diffraction data for $2\text{PbO} \cdot \text{GeO}_2$ and $\text{PbO} \cdot \text{Al}_2\text{O}_3$ are shown in Table V. The data for $\text{PbO} \cdot \text{Al}_2\text{O}_3$ are in good agreement with the previous work of Celler and Huntington.⁵

V. Summary and Conclusions

(1) Aggregate thermal expansion data on $2\text{PbO} \cdot \text{WO}_3$, $3\text{PbO} \cdot \text{Fe}_2\text{O}_3 \cdot \text{WO}_3$, $4\text{PbO} \cdot \text{Fe}_2\text{O}_3 \cdot \text{Ta}_2\text{O}_5$, $5\text{PbO} \cdot \text{SnO}_2 \cdot \text{V}_2\text{O}_5$, $5\text{PbO} \cdot \text{GeO}_2 \cdot \text{V}_2\text{O}_5$, $5\text{PbO} \cdot \text{GeO}_2 \cdot \text{P}_2\text{O}_5$, $\text{PbO} \cdot \text{WO}_3$, $\text{PbO} \cdot \text{MoO}_3$,

Table V. X-Ray Diffraction Data for $2\text{PbO} \cdot \text{GeO}_2$ and $\text{PbO} \cdot \text{Al}_2\text{O}_3$

d (Å)	h	k	l
8.83	7	1.961	7
5.44	4	1.937	8
4.22	3	1.929	14
4.85	2	1.898	21
4.09	1	1.877	27
4.12	26	1.844	25
3.77	3	1.794	11
3.64	22	1.774	7
3.53	12	1.719	4
3.34	49	1.695	7
3.29	26	1.670	8
3.14	47	1.656	10
3.11	72	1.616	12
3.02	78	1.608	12
2.94	75	1.595	11
2.91	109	1.570	22
2.82	41	1.551	4
2.73	32	1.535	3
2.62	27	1.521	16
2.61	41	1.512	8
2.54	4	1.498	8
2.42	7	1.433	3
2.34	7	1.413	3
2.21	4	1.389	3
2.19	4	1.379	3
2.16	4	1.363	3
2.12	4	1.348	7
2.07	34	1.315	8

(continued next column)

Table V (concluded)

d (Å)	h	k	l
2.01	14	1.307	6
1.949	10	1.275	7
4.02	24	1.026	5
4.45	63	1.015	4
4.23	16	1.005	9
4.06	28	1.001	2
3.12	48	1.519	9
2.07	100	1.533	5
2.63	29	1.517	5
2.54	13	1.480	6
2.51	13	1.461	8
2.39	4	1.441	1
2.31	5	1.425	1
2.23	24	1.412	1
2.18	11	1.397	3
2.15	5	1.344	9
2.12	8	1.329	2
2.03	6	1.308	5
1.980	22	1.302	2
1.922	12	1.299	2
1.910	14	1.264	3
1.787	5	1.249	1
1.701	11	1.244	4
1.675	4	1.230	4
1.664	3	1.212	5
1.650	9	1.192	3

$2\text{PbO} \cdot \text{GeO}_2$ and $\text{PbO} \cdot \text{Al}_2\text{O}_3$ show that the expansions range from about 0.3 to 1.0% in the interval from room temperature to 500°C.

(2) X-ray diffraction patterns have been provided which can be used to identify the foregoing compounds, and some observations on the high temperature structural changes in

$2\text{PbO} \cdot \text{GeO}_2$ and $\text{PbO} \cdot \text{Al}_2\text{O}_3$ have been made which emphasize the need for detailed X-ray studies of these compounds.

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