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Crystallographic Properties of Palladium

Assessment of properties from absolute zero to the melting point

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John W. Arblaster

Wombourne, West Midlands, UK

Email: jwarblaster@yahoo.co.uk

The crystallographic properties of palladium at temperatures from absolute zero to the freezing point are assessed following a review of the literature published between 1901 to date. However values above 1100 K are considered to be highly tentative since they are based on only one set of measurements. Selected values of the thermal expansion coefficient and measurements of length change due to thermal expansion have been used to calculate the variation with temperature of the lattice parameter, interatomic distance, atomic and molar volumes and density. The data is presented in the form of Equations and in Tables whilst a comparison between selected and experimental values is shown in the Figures.

This is the fifth in a series of papers in this Journal on the crystallographic properties of the platinum group metals (pgms), following two papers on platinum (1, 2) and one each on rhodium (3) and iridium (4). Like these metals palladium exists in a face-centred cubic (fcc) structure (Pearson symbol *cF4*) up to the melting point which is a secondary fixed point on ITS-90 at 1828.0 ± 0.1 K (5).

In the low-temperature region, high precision experimental thermal expansion values were available up to 283 K (6–9) and were correlated using a relationship between thermal expansion and specific heat as explained in the earlier review on platinum (1). The adoption of this procedure leads to a very close agreement with length change values obtained from both dilatometric (10) and lattice parameter measurements (11) and below 293 K the crystallographic properties of palladium can be considered as being known with a high degree of confidence.

In contrast, in the high-temperature region there have been a significant number of determinations of thermal expansion but few correlate with the low-temperature data and the selected thermal expansion curve must to a certain extent be considered as being tentative, especially above 1100 K where the values are essentially based on a single data point at the freezing point. Because of the quality difference between the high- and low-temperature data they are considered separately in this review, with the low-temperature data used to obtain the selected values at the pivotal temperature of 293.15 K.

Thermal Expansion

Low-Temperature Region

The thermodynamic thermal expansion coefficient, α, is based on a combination of the measurements of Waterhouse and Yates (6) and Bailey *et al.* (7) (from 30 K to 270 K) and White and Pawlowicz (8) (from 3 K to 85 K and at 283 K) except that the value at 283 K was amended by White (9) to $11.70 \pm 0.10 \times 10^{-6}$ K^{-1}. The correlated thermal expansion coefficients can be calculated from Equations (i) to (iii) where the specific heat (C_p) measurements for Equations (ii) and (iii) are those selected by the present author in an earlier review of the thermodynamic properties of palladium (12). Equation (i) is accurate to $\pm 1 \times 10^{-9}$ K^{-1}, Equation (ii) to $\pm 2 \times 10^{-8}$ K^{-1} and Equation (iii) to $\pm 4 \times 10^{-8}$ K^{-1} overall but with an accuracy of $\pm 1 \times 10^{-7}$ K^{-1} above 150 K. Because the use of Equations (ii) and (iii) requires a knowledge of the specific heat values then they can also be represented by a series of spline-fitted polynomials, Equations (iv) to (ix). The equations are given in the box below with derived values of low-temperature crystallographic properties given in **Table I**. The accuracies suggested for the low-temperature thermal expansion equations and for lattice parameter, density and atomic volume at 293.15 K can be considered as applying directly to all values in the low-temperature region due to the precision accuracy of the thermal expansion equations.

On the basis of the expression:

$$100 \times (\delta L/L_{293.15\ \mathrm{K\ (experimental)}} - \delta L/L_{293.15\ \mathrm{K\ (calculated)}})$$

where $\delta L/L_{293.15\ \mathrm{K\ (experimental)}}$ is the experimental length change relative to 293.15 K and $\delta L/L_{293.15\ \mathrm{K\ (calculated)}}$ is the relative length change calculated from the selected values for α, length change values calculated from the dilatometric measurements of Nix and MacNair (10) (from 86 K to 299 K) overall agree with the selected values given by Equations (i) to (ix) to within 0.002. A single measurement of Henning (13) at 82 K is 0.001 low and a value of Scheel (14) at 83 K is 0.002 low. Length change measurements calculated from the lattice parameter measurements of King and Manchester (11) (from 6 K to 293 K) overall agree to within 0.004 but other low-temperature lattice parameter measurements show poorer agreement. Values of Nagender-Naidu and Houska (15) at 80 K and 195 K are both 0.014 high, a measurement by Abrahams (16) at 4 K is 0.090 high whilst a single measurement by Balbaa *et al.* (17) at 77 K is 0.022 low. Of the lattice parameter measurements given over a range of temperatures, those of Smirnov and Timoshenko (18) (from 77 K to 300 K) were only shown graphically, with actual data points being given by Touloukian *et al.* (19) which

Table I

Low-Temperature Crystallographic Properties of Palladium

Temperature, K	Thermal expansion coefficient, α, 10^{-6} K^{-1}	Length change, $\delta a/a_{293.15\ \mathrm{K}} \times 100$, %	Lattice parameter, a, nm	Interatomic distance, d, nm	Atomic volume, 10^{-3} nm^3	Molar volume, 10^{-6} m^3 mol^{-1}	Density, kg m^{-3}
0	0	–0.2378	0.38810	0.27442	14.614	8.800	12093
10	0.085	–0.2378	0.38810	0.27442	14.614	8.800	12093
20	0.436	–0.2376	0.38810	0.27443	14.614	8.801	12092
30	1.23	–0.2368	0.38810	0.27443	14.614	8.801	12092
40	2.38	–0.2350	0.38811	0.27443	14.615	8.801	12092
50	3.63	–0.2320	0.38812	0.27444	14.616	8.802	12090
60	4.76	–0.2278	0.38813	0.27445	14.618	8.803	12089
70	5.76	–0.2225	0.38815	0.27447	14.620	8.804	12087
80	6.63	–0.2164	0.38818	0.27448	14.623	8.806	12085
90	7.36	–0.2094	0.38821	0.27450	14.626	8.808	12082
100	7.98	–0.2017	0.38824	0.27452	14.629	8.810	12079
110	8.47	–0.1935	0.38827	0.27455	14.633	8.812	12076

(Continued)

Table I (Continued)

Temperature, K	Thermal expansion coefficient, α, 10^{-6} K^{-1}	Length change, $\delta a/a_{293.15\ K}$ × 100, %	Lattice parameter, *a*, nm	Interatomic distance, *d*, nm	Atomic volume, 10^{-3} nm^3	Molar volume, 10^{-6} m^3 mol^{-1}	Density, kg m^{-3}
120	8.88	–0.1848	0.38830	0.27457	14.637	8.814	12073
130	9.23	–0.1758	0.38834	0.27460	14.641	8.817	12070
140	9.53	–0.1664	0.38837	0.27462	14.645	8.819	12067
150	9.80	–0.1567	0.38841	0.27465	14.649	8.822	12063
160	10.03	–0.1468	0.38845	0.27467	14.653	8.825	12060
170	10.24	–0.1367	0.38849	0.27470	14.658	8.827	12056
180	10.42	–0.1264	0.38853	0.27473	14.662	8.830	12052
190	10.59	–0.1159	0.38857	0.27476	14.667	8.833	12048
200	10.74	–0.1052	0.38861	0.27479	14.672	8.836	12044
210	10.89	–0.0944	0.38865	0.27482	14.677	8.838	12041
220	11.02	–0.0835	0.38870	0.27485	14.681	8.841	12037
230	11.14	–0.0724	0.38874	0.27488	14.686	8.844	12033
240	11.26	–0.0612	0.38878	0.27491	14.691	8.847	12029
250	11.37	–0.0499	0.38883	0.27494	14.696	8.850	12025
260	11.47	–0.0385	0.38887	0.27497	14.701	8.853	12020
270	11.56	–0.0270	0.38891	0.27500	14.706	8.856	12016
280	11.65	–0.0154	0.38896	0.27504	14.711	8.859	12012
290	11.74	–0.0037	0.38901	0.27507	14.717	8.863	12008
293.15	11.77	0	0.38902	0.27508	14.718	8.864	12007

indicated a bias of 0.011 low below 220 K. Values of Lawson *et al.* (20) (from 15 K to 300 K) are also shown only graphically but estimates suggest deviations of 0.012 to 0.019 high, whilst the measurements of Schröder *et al.* (21) (from 91 K to 1809 K) are initially 0.044 low before converging to selected values in the room temperature region. Similarly the dilatometric measurements of Masumoto and Sawaya (22) (from 125 K to 1184 K) show the same behaviour being initially 0.084 low. Deviations of the last two sets of measurements are shown in **Figure 1**.

High-Temperature Region

There have been a significant number of determinations of thermal expansion in the high-temperature region but of these only length change measurements derived from the lattice parameter measurements of Owen and Jones (23) (from 283 K to 559 K), Dutta and Dayal (24) (from 298 K to 1151 K) and Rao and Rao (25) (from 296 K to 1063 K) show any agreement with the low-temperature data. It was found that continuity with the low-temperature selected values could best be achieved if Equation (iii) was extrapolated to 500 K and length change values obtained by integrating this equation were combined with the measurements of Rao and Rao for the range 873 K to 1063 K. In order to extend beyond this temperature limit it was noted that length change values derived from the lattice

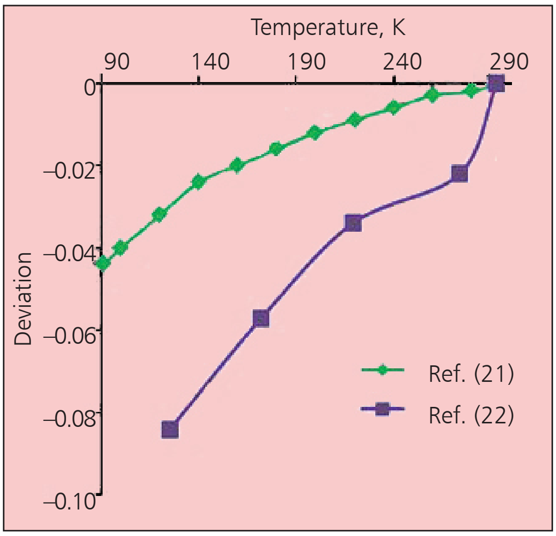

Fig. 1. The difference between length change values calculated from Equations (i) to (ix) and experimental length change values of Schröder et al. (21) and Masumoto and Sawaya (22) for the low-temperature region

parameter measurements of Filipponi *et al.* (26) (from 1249 K to 1769 K), direct density measurements of Stankus and Tyagel'skii (27) (from 293 K to 1827 K) and volume ratio measurements of Hüpf and Pottlacher (28) (from 1300 K to 1828 K), although disagreeing with the selected values at 1100 K, converged when approaching the freezing point which on the basis $100\delta L/L_{293.15\text{ K}}$ leads to values of 2.41, 2.35 and 2.46 respectively. Since the last two values straddle the lattice parameter value then the latter is selected. However, possibly because of the large interval from 1100 K to the freezing point, it was found that standard polynomial fits showed abnormal behaviour with a sudden very sharp increase in the thermal expansion coefficient above about 1300 K, which is considered to be unnatural behaviour. Therefore, in this special case, a non-standard polynomial, Equation (x), was selected in which the thermal expansion coefficient increases in a more natural manner.

On the same basis as used to represent deviations of the low-temperature data, i.e. lattice parameter measurements not used in the fit, those of Owen and Jones (23) on average agree with the selected values to within 0.002 whilst the measurements of Dutta and Dayal (24) trend to a maximum of 0.031 high. The measurements of Schröder *et al.* (21) initially agree closely to 500 K and then deviate sharply to 0.39 low at 1809 K. A single measurement of Bidwell and Speiser (29) at 1173 K biases 0.029 low.

Dilatometric measurements in the high-temperature region generally show satisfactory agreement with each other and form a consistent set of data. They are nevertheless incompatible with the low-temperature values and lead to deviations from Equation (x) as shown in **Figures 2** and **3**, except for the measurements of Scheel (14) (from 83 K to 373 K) which agree with the selected values. The measurements of Holborn and Day (30) (from 273 K to 1273 K) and Henning (13) (from 289 K to 1273 K in the high-temperature region) both shown an identical trend to 0.067 low whilst the measurements of Holzmann (31) (from 293 K to 1257 K) trend to an average of 0.091 low above 1064 K and those of Krikorian (32) (from 298 K to 1273 K) trend to 0.063 low. The measurements of Vest (33) (from 293 K to 1068 K) trend to 0.085 low whilst smooth values derived from the measurements of Masumoto *et al.* (34) (from 296 K to 862 K) trend to 0.084 low. The density measurements of Stankus and Tyagel'skii (27) (from 293 K to 1827 K) converted to length change values show a maximum deviation of 0.099 low at 1200 K but are still 0.061 low at the freezing point. The exceptions to these trends are the measurements of Masumoto and Sawaya (22) (from 318 K to 1184 K in the high-temperature region) which deviate to a maximum of 0.057 high at 877 K whilst the volume ratios of Hüpf and Pottlacher (28) (from 1300 K to 1828 K) trend from initially 0.25 high to 0.050 high. The measurements of Masumoto and Sawaya (22) and Masumoto *et al.* (34) were only shown graphically with actual data points being given by Touloukian *et al.* (19). **Figure 2** shows high-temperature values that differ from Equation (x) by less than 0.12, whilst **Figure 3** includes those values that differ by more than 0.12.

In their review of the thermal expansion of palladium, Touloukian *et al.* (19) appeared to have favoured the same set of lattice parameter measurements as used in the present review but although acknowledging in a table that the thermal expansion at 293 K was $11.8 \times 10^{-6}\ \text{K}^{-1}$, in agreement with the present selection, their actual equations representing length change values led to a value of $12.1 \times 10^{-6}\ \text{K}^{-1}$ at 293 K which appears to have no basis. However in spite of these different approaches the agreement between the presently selected values and those of Touloukian *et al.*

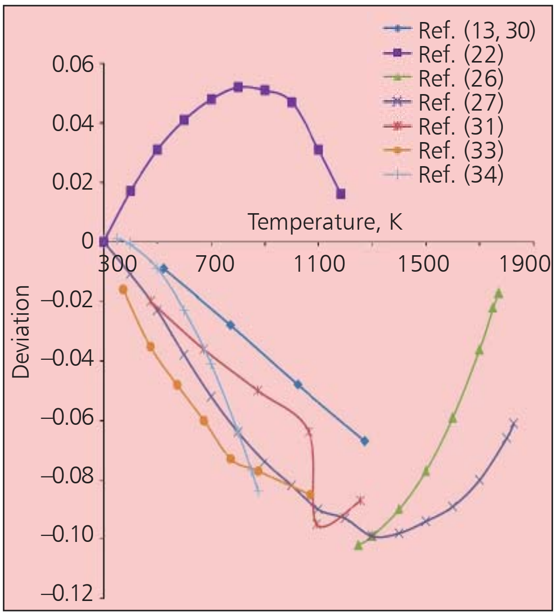

Fig. 2. The difference between length change values calculated from Equations (x) and experimental length change values of Henning (13), Masumoto and Sawaya (22), Filipponi et al. (26), Stankus and Tyagel'skii (27), Holborn and Day (30), Holzmann (31), Vest (33) and Masumoto et al. (34) for the high-temperature region

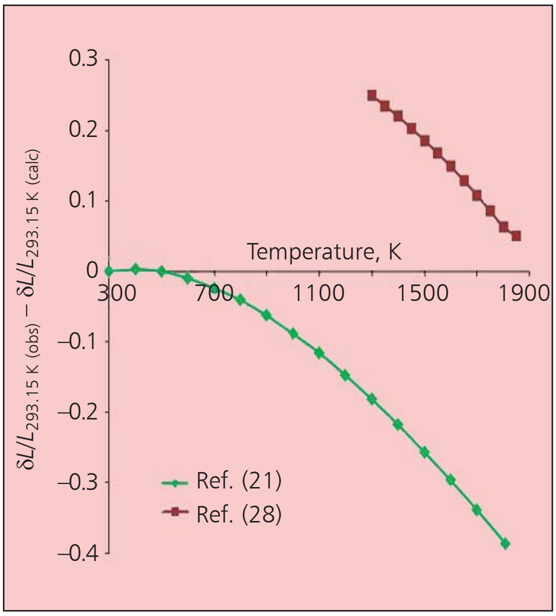

Fig. 3. The difference between length change values calculated from Equations (x) and experimental length change values of Schröder et al. (21) and Hüpf and Pottlacher (28) for the high-temperature region (expanded scale)

which extend to 1200 K show a reasonable agreement with a maximum deviation of their values of only 0.012 low at 1000 K.

The Lattice Parameter at 293.15 K

The values of the lattice parameter, a, given in **Table III** represent a combination of those values selected by Donohue (35) and more recent measurements. Values originally given in kX units were converted to nanometres using the 2010 International Council for Science: Committee on Data for Science and Technology (CODATA) Fundamental Constants (36) conversion factor for $CuK\alpha_1$, which is 0.100207697 ± 0.000000028 whilst values given in ångströms (Å) were converted using the default ratio 0.100207697/1.00202 where the latter value represents the old conversion factor from kX units to Å. Lattice parameter values were corrected to 293.15 K using the values of the thermal expansion coefficient selected in the present review. Density values given in **Tables I** and **II** were calculated using the currently accepted atomic weight of 106.42 ± 0.01 (37) and an Avogadro constant (N_A) of (6.02214129 ± 0.00000027) × 10^{23} mol^{-1} (36). From the lattice parameter value at 293.15 K selected in **Table III** as 0.38902 ± 0.00006 nm, the derived selected density is 12007 ± 6 kg m^{-3} and the molar volume is (8.8635 ± 0.0041) × 10^{-6} m^3 mol^{-1}. In **Tables I** and **II** the interatomic distance, d, is equal to $a/\sqrt{2}$ and the atomic volume to $a^3/4$. The molar volume is calculated as $N_A\, a^3/4$ which is equivalent to atomic weight divided by density.

Table II

High-Temperature Crystallographic Properties of Palladium

Temperature, K	Thermal expansion coefficient, α, 10^{-6} K^{-1}	Length change, $\delta a/a_{293.15\ K}$ × 100, %	Lattice parameter, a, nm	Interatomic distance, d, nm	Atomic volume, 10^{-3} nm^3	Molar volume, 10^{-6} m^3 mol^{-1}	Density, kg m^{-3}
293.15	11.77	0	0.38902	0.27508	14.718	8.864	12007
300	11.83	0.0081	0.38905	0.27510	14.722	8.866	12004
400	12.63	0.131	0.38953	0.27544	14.776	8.898	11960
500	13.24	0.260	0.39003	0.27579	14.834	8.933	11913
600	13.76	0.396	0.39056	0.27617	14.894	8.969	11865
700	14.24	0.537	0.39111	0.27655	14.956	9.007	11815
800	14.68	0.682	0.39167	0.27695	15.021	9.046	11764
900	15.10	0.832	0.39226	0.27737	15.089	9.087	11712
1000	15.50	0.987	0.39286	0.27779	15.158	9.128	11658
1100	15.88	1.145	0.39347	0.27823	15.230	9.172	11603
1200[a]	*16.24*	*1.308*	*0.39411*	*0.27868*	*15.303*	*9.216*	*11548*
1300	*16.58*	*1.474*	*0.39475*	*0.27913*	*15.379*	*9.261*	*11491*
1400	*16.91*	*1.644*	*0.39542*	*0.27960*	*15.456*	*9.308*	*11433*

(Continued)

Table II (Continued)

Temperature, K	Thermal expansion coefficient, α, 10^{-6} K^{-1}	Length change, $\delta a/a_{293.15\,K} \times 100$, %	Lattice parameter, *a*, nm	Interatomic distance, *d*, nm	Atomic volume, 10^{-3} nm^3	Molar volume, 10^{-6} m^3 mol^{-1}	Density, kg m^{-3}
1500	*17.22*	*1.818*	*0.39609*	*0.28008*	*15.536*	*9.356*	*11375*
1600	*17.52*	*1.995*	*0.39678*	*0.28057*	*15.617*	*9.405*	*11316*
1700	*17.80*	*2.175*	*0.39748*	*0.28106*	*15.700*	*9.455*	*11256*
1800	*18.06*	*2.358*	*0.39819*	*0.28157*	*15.784*	*9.506*	*11196*
1828.0	*18.13*	*2.410*	*0.39840*	*0.28171*	*15.808*	*9.520*	*11179*

[a] *The values above 1100 K are considered to be tentative and are given in italics*

Low-Temperature Thermal Expansion Equations for Palladium

0–28 K: $$\alpha = 4.15367 \times 10^{-9}\,T + 4.27891 \times 10^{-11}\,T^3 + 7.05954 \times 10^{-15}\,T^5 - 9.39675 \times 10^{-18}\,T^7 \quad \text{(i)}$$

28–96 K: $$\alpha = C_p\,(4.30784 \times 10^{-7} + 1.79706 \times 10^{-10}\,T + 1.63606 \times 10^{-7} / T) \quad \text{(ii)}$$

96–283 K: $$\alpha = C_p\,(3.93325 \times 10^{-7} + 1.69782 \times 10^{-10}\,T + 3.85873 \times 10^{-6} / T) \quad \text{(iii)}$$

Low-Temperature Thermal Expansion Equations (Spline-Fitted Equations above 28 K)

28–42 K: $$\alpha = 1.62297 \times 10^{-5} - 1.74247 \times 10^{-6}\,T + 6.98420 \times 10^{-8}\,T^2 - 1.16942 \times 10^{-9}\,T^3 + 7.40073 \times 10^{-12}\,T^4 \quad \text{(iv)}$$

42–67 K: $$\alpha = 8.38484 \times 10^{-7} - 1.64721 \times 10^{-7}\,T + 8.74617 \times 10^{-9}\,T^2 - 1.11101 \times 10^{-10}\,T^3 + 4.87713 \times 10^{-13}\,T^4 \quad \text{(v)}$$

67–96 K: $$\alpha = -5.11937 \times 10^{-7} + 2.36230 \times 10^{-7}\,T - 1.43355 \times 10^{-9}\,T^2 + 4.31403 \times 10^{-12}\,T^3 - 4.71931 \times 10^{-15}\,T^4 \quad \text{(vi)}$$

96–150 K: $$\alpha = -2.14978 \times 10^{-6} + 1.47086 \times 10^{-7}\,T - 3.46315 \times 10^{-10}\,T^2 - 1.96244 \times 10^{-12}\,T^3 + 8.49737 \times 10^{-15}\,T^4 \quad \text{(vii)}$$

150–270 K: $$\alpha = -1.39162 \times 10^{-6} + 1.59438 \times 10^{-7}\,T - 8.50840 \times 10^{-10}\,T^2 + 2.25095 \times 10^{-12}\,T^3 - 2.32845 \times 10^{-15}\,T^4 \quad \text{(viii)}$$

270–293.15 K: $$\alpha = 5.04531 \times 10^{-6} + 5.65325 \times 10^{-8}\,T - 2.05622 \times 10^{-10}\,T^2 + 3.97366 \times 10^{-13}\,T^3 - 2.96804 \times 10^{-16}\,T^4 \quad \text{(ix)}$$

High-Temperature Thermal Expansion Equations for Palladium (293.15 K to 1828.0 K)

$$\delta a/a_{293.15\,\mathrm{K}} = -3.67831 \times 10^{-3} + 1.10122 \times 10^{-5}\,T + 2.69121 \times 10^{-9}\,T^2 - 2.25680 \times 10^{-13}\,T^3 + 6.58134 \times 10^{-2} / T \quad \text{(x)}$$

$$\alpha^* = 1.10122 \times 10^{-5} + 5.38242 \times 10^{-9}\,T - 6.77040 \times 10^{-13}\,T^2 - 6.58134 \times 10^{-2} / T^2 \quad \text{(xi)}$$

α^* is the thermal expansion coefficient relative to 293.15 K. All thermal expansion coefficients given in the tables are in units of K^{-1}

Table III
Lattice Parameter Values at 293.15 K

Authors (Year)	Reference	Original temperature, K,	Original units	Lattice parameter, *a*, corrected to 293.15 K, nm	Notes
Stenzel and Weerts (1931)	(38)	293	kX	0.38890	
Owen and Yates (1933)	(39, 40)	291	kX	0.38906	
Owen and Jones (1937)	(23)	293	kX	0.38906	
Swanson and Tatge (1953)	(41)	298	Å	0.38898	
Coles (1956)	(42)	295	kX	0.38909	
Rayne (1960)	(43)	300	kX	0.38895	
Dutta and Dayal (1963)	(24)	298	Å	0.38899	
Anderson (1964)	(44)	293	kX	0.38896	
Goetz and Brophy (1964)	(45)	rt	Å	0.38891	
Rao and Rao (1964)	(25)	296, 298	Å	0.38906	
Bidwell and Speiser (1964)	(29)	298	Å	0.38900	
Maeland and Flanagan (1964)	(46)	293	Å	0.38898	
Catterall and Barker (1964)	(47)	295	kX	0.38910	
Rao (1965)	(48)	rt[a]	Å	0.38908	
Humble (1968)	(49)	rt	Å	0.38905	
Siller *et al.* (1969)	(50)	298	Å	0.38900	
Nagender-Naida and Houska (1971)	(15)	298	Å	0.38900	
Schröder *et al.* (1972)	(21)	296	Å	0.38903	
King and Manchester (1978)	(11)	292.7	Å	0.38876	(a)
Balbaa *et al.* (1987)	(17)	296	Å	0.38917	(a)
Stankus and Tyagel'skii (1992)	(27)	293	–	0.38905	(b)
Filipponi *et al.* (2000)	(26)	293	Å	0.38909	

[a] *rt = room temperature*

Notes to Table III

Lattice parameter value selected for the present paper = 0.38902 ± 0.00006

(a) Not included in the average

(b) Calculated from direct density measurement 12004 kg m^{-3}

Summary

Whilst thermal expansion data for palladium below 293.15 K is of high quality, above this temperature there is a marked lack of agreement between lattice parameter and dilatometric determinations, with the latter showing a particular incompatibility with the low-temperature data. Whilst selected high-temperature values up to about 1100 K can be said to be reasonably determined, above this temperature the selected values are highly tentative and essentially based on the selection of a single value at the freezing point. This may be of concern since it is noted that the thermal expansion derivative $d\alpha/dT$ is decreasing at high-temperatures whereas this value would be expected to be at least constant or even increasing. However rather than suspect that this selected value is too low it could be considered that the number of acceptable data points are far too few and that further high quality measurements are required in the high-temperature region to replace the current speculation.

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The Author

John W. Arblaster is interested in the history of science and the evaluation of the thermodynamic and crystallographic properties of the elements. Now retired, he previously worked as a metallurgical chemist in a number of commercial laboratories and was involved in the analysis of a wide range of ferrous and non-ferrous alloys.