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The Thermal Conductivity of Gases: Incorrect Results Due to Desorbed Air

W. Hemminger[1]

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Air desorbed from the measuring instrument can falsify the thermal conductivity of a gas measured by steady-state methods. For a guarded hot-plate apparatus the contamination effect was determined to depend on both the residence time in the system and the temperature. The investigation covered the gases H_2, He, Ne, CH_4, N_2, air, Ar, and Kr. For gases whose conductivity is better than that of air (H_2, He) the measured values are too small, and for gases of poorer conductivity they are too high. Corrections for the effect of impurity have been applied to the measurements presented. These impurity corrections are considerably larger than the precision of the measurements, but they are of the order of the estimated overall uncertainty of the measurements. The departures between the corrected thermal conductivities reported here and values taken from the correlations in the literature run up to 5% at the highest temperatures.

KEY WORDS: air; argon; helium; hot-plate apparatus; hydrogen; impurities; krypton; measurement error; methane; neon; nitrogen; steady-state method; thermal conductivity.

1. INTRODUCTION

When the thermal conductivity of pure gases of good conductivity (H_2, He) is measured by steady-state methods which require long measurement times, there is a risk of the gas being contaminated by air desorbed from the measuring instrument, and as a consequence, the thermal conductivity decreases. This risk has been pointed out by Guildner [1] for helium. For hydrogen a similar effect is to be expected. For gases whose thermal conductivity is lower than that of air (Ar, Kr), an increased thermal conductivity can be expected.

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[1] Physikalisch-Technische Bundesanstalt, Braunschweig, Federal Republic of Germany.

In order to determine quantitatively the influence of air as an impurity, the thermal conductivity of H_2, He, Ne, CH_4, N_2, air, Ar, and Kr was measured as a function of temperature between 30 and 190°C and as a function of residence time in the apparatus at temperatures of 160 and 190°C.

2. EXPERIMENTAL

The guarded hot-plate apparatus is described in the literature [2, 3]. The hot plate is 101 mm in diameter, and the guard ring 50 mm in width. Between the hot plate and the guard ring there is a gap 1 mm wide. The thickness of the gas layer between the hot and the cold plate is adjusted by three spacers made of glass. The spacers are 2.5 mm in diameter and 0.5, 1.0, and 2.0 mm high, with a thermal conductivity of $0.596\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$ at 20°C.

The temperature of the cold plate is adjusted with the aid of a liquid thermostat. The hot plate and the guard are heated electrically so that the desired temperature difference, ΔT, results in the gas layer and the temperature differences between the hot plate and the guard are generally less than 0.01 K.

It takes up to 5 h to reach the steady state and the originally pure gas becomes increasingly contaminated with air desorbed from the instrument. Prior to a measurement, the apparatus is therefore flushed with preheated pure gas and refilled. The resulting disturbance of the steady state disappears about 50 min after flushing and measurements which take about 20 min can than start. As a minimum, a residence time of the gas of 1 h prior to the first measurement must be accounted for.

The purity of the gases was 99.95 to 99.999% (cf. Table II). The air was obtained outside the laboratory building and dried prior to measurement. All measurements were performed at atmospheric pressure.

The measuring apparatus and the evaluation of the measured data are described in Refs. 3 and 4. The thermal conductivity λ is determined from the basic Fourier equation as follows:

$$\lambda = Pd/A\,\Delta T \qquad (1)$$

where P is the heat flux from the hot plate through the gas layer to the cold plate, A is the surface of the hot plate, d is the thickness of the gas layer, and ΔT is the temperature difference between the hot and the cold plate.

The following corrections were made:

- heat transfer between the hot plate and the guard heaters [4],
- heat transfer through the glass spacers,

- heat transfer by radiation between the hot and the cold plate [3],
- measurement errors due to the thermocouples [4],
- expansion of the hot plate and spacers with increasing temperatures,
- temperature difference between the site of temperature measurement in the hot or cold plate and the plate surfaces [4],
- temperature jump between the plate surfaces and the gas layer (cf. Section 2.1), and
- contribution of the contamination due to desorption of air from the instrument (cf. Section 2.2).

For layer thicknesses of 0.5 and 1 mm, the measured values were in good agreement. For a layer thickness of 2 mm, there were larger deviations between individual measurements, and at higher temperatures we suspect the onset of convection. Therefore, the mean value for layers 0.5 and 1 mm thick was taken as the measurement result. The scatter for repeated measurements for layers of equal thickness was 0.01% for the 0.5-mm layer and 0.05% for the 1-mm layer with air and 0.15% for the 0.5-mm layer with Kr. The precision is seen to be substantially smaller than the estimated overall uncertainty of the measured thermal conductivities (cf. Table I).

Depending on the thermal conductivity of the gas, values of ΔT were adjusted between 5 and 20 K. The dependence of the measured values on ΔT was checked with air at 110°C for $\Delta T = 5$, 10, and 20 K and with He at 100°C for $\Delta T = 2.5$, 5, and 10 K, the layer thickness being 1 mm. A depen-

Table I. Maximum Correction P_{corr}/P for the Heat-Flux Density P, Maximum Temperature-Jump Correction $(2g/d)$, and Maximum Correction $\Delta T_{corr}/\Delta T$ for the Temperature Difference Between the Hot and the Cold Plate ΔT[a]

	P_{corr}/P	$2g/d$	$\Delta T_{corr}/\Delta T$	$\Delta\lambda/\lambda$
H_2	1.3	0.7	0.9	0.8
He	1.3	0.9	0.9	0.8
Ne	3.7	0.3	0.5	0.8
CH_4	4.7	0.1	0.3	0.8
N_2	6.4	0.1	0.4	1.0
Air	6.5	0.1	0.3	1.0
Ar	9.3	0.1	0.3	1.2
Kr	17.2	0.1	0.1	2.1

[a] All corrections as percentages. $\Delta\lambda/\lambda$ is the estimated overall uncertainty of thermal conductivity as a percentage.

dence upon ΔT was not found, and we therefore conclude that convection was not present. In these tests the individual values scattered by 0.12% for air and by 0.03% for He around the respective mean thermal conductivity value.

2.1. Temperature-Jump Correction

The temperature-jump distance g was calculated as indicated, for example, in Ref. 1 or 5. The mean free path of the gas atoms (molecules) as a function of temperature was determined according to Ref. 7. The accommodation coefficients were taken from Ref. 6; for air and methane the value for nitrogen (0.8) was used. For the surfaces of the cold and the hot plate (both nickel-plated), equal accommodation coefficients were assumed so that the correction is $2g/d$ ($d =$ the thickness of the gas layer). The uncertainty of the temperature-jump correction is estimated to be 30%.

The maximum temperature-jump corrections for the 0.5-mm layer at 190°C are 0.9% for helium, 0.7% for hydrogen, and 0.3% for neon. For the other gases the correction is smaller than 0.1% and is disregarded.

2.2. Impurity Corrections

Air was left for 4 days in the measuring apparatus and heated several times to 200°C during this period. Air did not show any change in its thermal conductivity. Helium was kept in the system for 4 days at 190°C during the day but cooled to ambient temperature during the night. During the 4 days of observation, the thermal conductivity of helium decreased linearly as a function of the residence time of the gas in the apparatus. When helium was treated in the same way at 110°C, the thermal conductivity also decreased linearly but the absolute decrease was smaller than at 190°C. In order to determine quantitatively the influence exerted by time and temperature, the thermal conductivity of helium was measured at 190°C for residence times of 1 and 6 h. At layer thicknesses of 0.5, 1, and 2 mm, a systematic influence of the layer thickness was not discovered, and the mean value served as the measurement value. At 160°C, the thermal conductivities of helium were also determined for residence times of 1 and 6 h with a 0.5-mm layer. For a difference in the residence time of 5 h, the thermal conductivity of helium decreased by 1.06% at 190°C and by 0.63% at 160°C. At temperatures less than 160°C, the contamination effect was obscured by the scatter of the individual measurements.

For the lower temperatures, the impurity effect is calculated on the assumption that it decreases exponentially with the temperature for the same residence time. Such a dependence is valid for the desorption from surfaces [8]. Furthermore, it is assumed that the contamination of each gas is subject to the same temperature function. This is why for all gases except air and nitrogen, only the effect of contamination for a time difference of 5 h was measured at 190°C. The assumptions appear to be justified, as the desorption of air from parts of the apparatus will be seen to be the single source of contamination.

For equal desorption times t, the desorption can be described as follows [8]:

$$I(T) = K\exp(-E/RT) \tag{2}$$

where I is the impurity correction, K is the constant, E is the activation energy, and R is the gas constant.

The constants K and E/R of the exponential function are determined on the basis of measurements with helium at 190 and 160°C:

$$I(T, \Delta t) = I_0(\Delta t)\exp[-8.275(T_0 - T)/T] \tag{3}$$

where $I_0(\Delta t) = I(T_0, \Delta t)$, the (maximum) impurity correction measured at $T_0 = 463$ K (190°C) for the respective gas.

Figure 1 shows the impurity corrections for a residence time of 1 h at 190°C as a function of the thermal conductivity of the gases. The thermal conductivity of H_2, He, Ne, and CH_4 decreases on account of the contamination (positive correction), whereas the thermal conductivity of Ar and Kr increases. A correction of zero results within the uncertainty of the measurements for air and nitrogen. From Fig. 1 and from the result

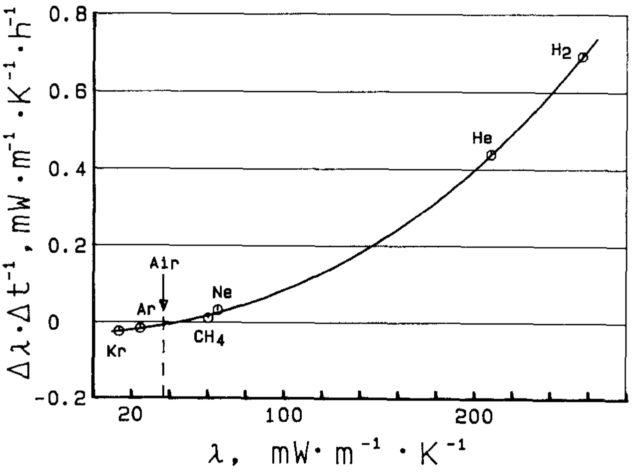

Fig. 1. Impurity correction $\Delta\lambda\Delta t^{-1}$ for a residence time of the gas in the apparatus of 1 h at 190°C as a function of the respective thermal conductivity.

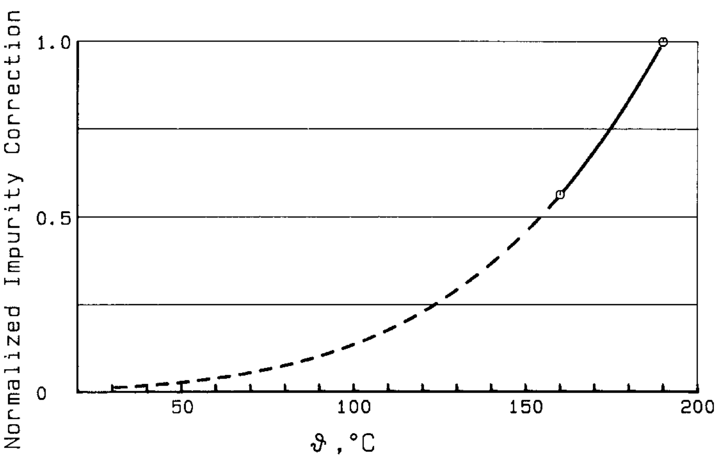

Fig. 2. Contamination effect for a residence time of the gas in the apparatus of 1 h as a function of temperature, normalized to 1 and related to the respective thermal conductivity as a percentage. Unit values per hour for the various gases are as follows: H_2, 0.27; He, 0.21; Ne, 0.05; CH_4, 0.02; Ar, −0.06; and Kr, −0.17.

obtained in measurements with air lasting several days, it is concluded that the desorption of air from parts of the hot-plate apparatus is the source of the contamination. Further, the contamination gives rise to a time- and temperature-dependent change in the measured thermal conductivity. The amounts of the impurity correction are, of course, apparatus specific.

Figure 2 shows the temperature dependence of the impurity correction normalized to 1, with the relative corrections for the various gases for a residence time of 1 h given in the legend.

RESULTS

We first consider the experimental corrections.

For each gas, Table I states the maximum corrections as percentages:

(a) the sum of all maximum individual corrections for the heat-flux density, P_{corr}/P, comprises all heat-transfer losses and allows for the thermal expansion of the hot and cold plate and spacers;

(b) the maximum temperature-jump correction, $2g/d$; and

(c) the sum of the maximum individual corrections for the temperature difference between the hot and the cold plate, $\Delta T_{corr/\Delta T}$.

The impurity corrections are dealt with in Section 2.2; they are not contained in Table I.

Each of the individual corrections is subject to an uncertainty. These uncertainties were determined by estimate. The estimated uncertainties of the correction and the uncertainties of measurement were added and grouped as follows.

(a) The overall uncertainty of P is expressed as the sum of the uncertainties of the individual corrections of the various heat-transfer losses and the uncertainty of measurement of the electric heating power. The overall uncertainty for P is 0.4 to 2.0%, depending on the gas concerned.

(b) The overall uncertainty of d is expressed as the sum of the uncertainty of the temperature-jump correction and the uncertainty of measurement for the height of the glass spacers. The overall uncertainty for d is 0.3 to 0.6%, depending on the gas concerned.

(c) The overall uncertainty of ΔT is expressed by the sum of the uncertainties of the individual corrections and the general uncertainty of temperature measurement using thermocouples. The overall uncertainty for ΔT is 0.1 to 0.4%, depending on the gas concerned.

(d) The overall uncertainty of A is expressed by the uncertainty of measurement for the determination of the hot-plate surface, 0.1%.

From the uncertainties of the indivicual quantities the maximum uncertainty was calculated according to the law of error propagation. To this uncertainty the uncertainty of the impurity correction was added, roughly 0.03% for all gases. The sum is the estimated overall uncertainty $\Delta\lambda/\lambda$ given in Table I.

The thermal conductivities are listed in Table II. They have been corrected with regard to impurity. The temperature dependence of the thermal conductivity measurements is expressed by a simple polonomial for each gas in Table III.

4. DISCUSSION

4.1. Comparison with Literature Values

To compare the measured values with values given in the literature we use the polynomials in Table III. We extrapolate the temperature range in both directions by up to 40 K. Most of the older publications do not indicate which "calorie" was used as the unit of heat. Nor was the purity of the gases specified in many cases. Both omissions can result in systematic differences.

Table II. Experimental Thermal Conductivities in $\mathrm{mW \cdot m^{-1} \cdot K^{-1}}$ at Different Temperatures ϑ (°C)

		ϑ (°C)								
Gas	Purity (%)	30	40	70	100	110	130	150	160	190
H_2	99.999		193.5	206.9	220.0		232.75		244.9	256.8
He	99.995		159.8	270.15	180.2		190.0		199.8	209.0
Ne	99.99	49.6		53.8		58.0		61.95		65.8
CH_4	99.95	34.6		40.5		46.8		53.6		60.8
N_2	99.999	26.1		28.8		31.4		34.0		36.5
Air	dry	26.2		29.0		31.7		34.4		36.9
Ar	99.995	17.8		19.7		21.6		23.3		25.0
Kr	99.99	9.5		10.6		11.7		12.8		13.8

4.1.1. Hydrogen (Fig. 3)

Values from correlations [9, 10] giving weighted means from different works are smaller than the values stated here by at least 3%. The deviations increase with increasing temperature to about −5%. This observation supports a systematic deviation as a result of the hydrogen being contaminated by air. The good agreement with older data at 0°C (cf. Ref. 11, −1.1%; Ref. 12, −0.3%) is explained by the very slight contamination at the low temperature.

4.1.2. Helium (Fig. 4)

Values from correlations [5, 9, 10] are, in general, smaller than the values measured by up to −5%. More recently, correlated data [32] show

Table III. Polynomials of $\lambda(\mathrm{mW \cdot m^{-1} \cdot K^{-1}})$ in Terms of ϑ(°C); Maximum Deviation Between Polynomial and Measurements in Parentheses

H_2	$\lambda(\vartheta) = 174.885 + 0.474\vartheta - 2.242 \times 10^{-4}\vartheta^2$	(0.03%)
He	$\lambda(\vartheta) = 145.647 + 0.359\vartheta - 1.339 \times 10^{-4}\vartheta^2$	(0.06%)
Ne	$\lambda(\vartheta) = 46.301 + 0.111\vartheta - 4.241 \times 10^{-5}\vartheta^2$	(0.08%)
CH_4	$\lambda(\vartheta) = 30.488 + 0.134\vartheta + 1.359 \times 10^{-4}\vartheta^2$	(0.07%)
N_2	$\lambda(\vartheta) = 23.997 + 0.706 \times 10^{-1}\vartheta - 2.536 \times 10^{-5}\vartheta^2$	(0.06%)
Air	$\lambda(\vartheta) = 24.095 + 0.716 \times 10^{-1}\vartheta - 2.143 \times 10^{-5}\vartheta^2$	(0.01%)
Ar	$\lambda(\vartheta) = 16.313 + 0.509 \times 10^{-1}\vartheta - 2.598 \times 10^{-5}\vartheta^2$	(0.08%)
Kr	$\lambda(\vartheta) = 8.647 + 0.294 \times 10^{-1}\vartheta - 1.344 \times 10^{-5}\vartheta^2$	(0.01%)

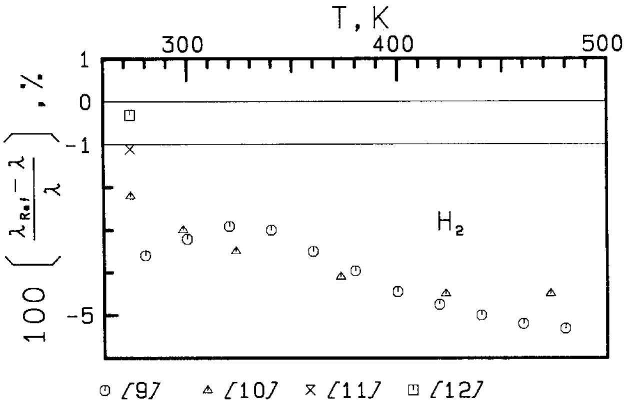

Fig. 3. Comparison of the experimental thermal conductivities with values λ_{Ref} given in the literature for H_2.

values up to 1% higher, possibly because of allowing for the more precise viscosity measurements in the correlation procedure to create a consistent set of data. Two hot-wire results for temperatures near 300 K [34, 35] show an excellent agreement. Within about 1%, the conductivities tabulated in Ref. 13 are in agreement with those measured here; the same is

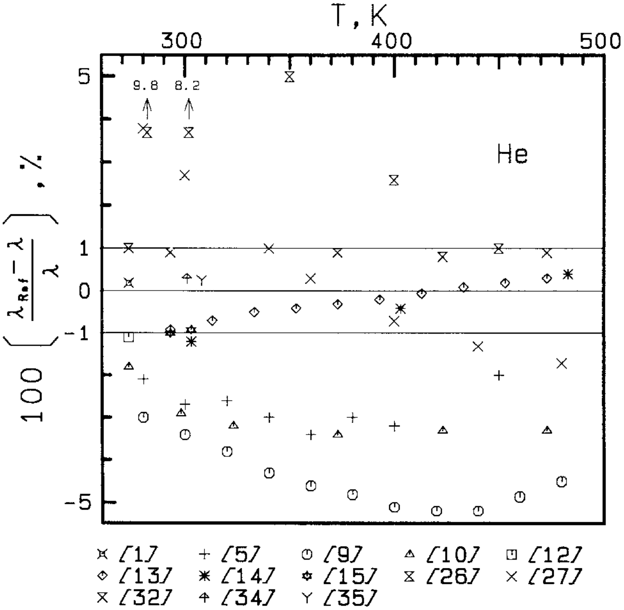

Fig. 4. Comparison of the experimental thermal conductivities with values λ_{Ref} given in the literature for He.

true for the measurements given in Refs. 1, 12, 14, and 15. For helium, too, contamination by air seems to be the reason for the systematic negative deviations of some correlations. The measurement results given in Refs. 26 and 27, however, show a completely different behavior. At ambient temperature, they exceed by far the values measured here and decrease below these values with increasing temperatures. This phenomenon suggests an unknown systematic uncertainty.

According to Ref. 23, the difference between measured conductivities and values calculated according to the kinetic theory of gases is about −4% (between 400 and 500 K). With the values stated here, this difference would be reduced to about −2%.

4.1.3. Neon (Fig. 5)

The thermal conductivity of neon is only about twice that of air. Therefore, the contamination effect is not as severe as with H_2 and He. Up to 360 K, there is very good agreement (0.5%) with the data of Refs. 5 and 9. At higher temperatures the values given in Ref. 5 are higher by up to 1%, while those given in Ref. 9 are smaller by up to 2%. Between 298 and 473 *K*, the values specified in Ref. 10 are smaller than those measured here by 1 to 1.5%. Compared with more recently correlated data [32] which are higher by about 1%, irrespective of temperature, the measurements are in good agreement regarding the uncertainties. Two values measured by means of the hot-wire method [34, 35] are even closer to our results. The thermal conductivity measured by Weber in 1917 at 0°C (mentioned in Ref. 12) is lower by 1.5%, whereas his most recent value determined in 1927 differs by only 0.2% [12].

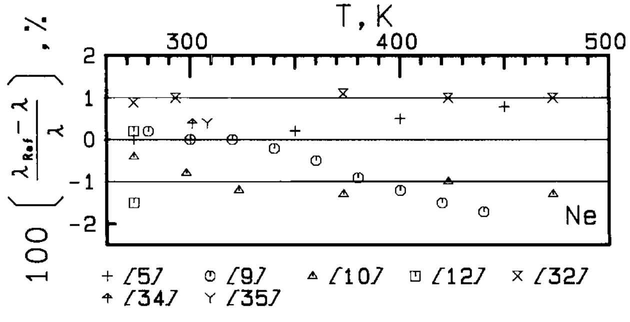

Fig. 5. Comparison of the experimental thermal conductivities with values λ_{Ref} given in the literature for Ne.

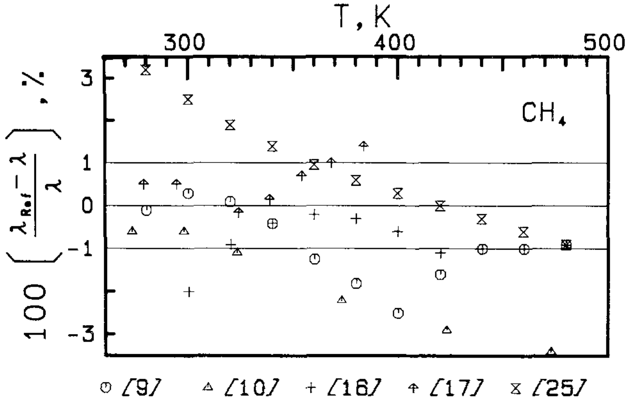

Fig. 6. Comparison of the experimental thermal conductivities with values λ_{Ref} given in the literature for CH_4.

4.1.4. Methane (Fig. 6)

The values tabulated in Ref. 9, 10, and 16 are smaller than the values measured here by up to 3%. For Ref. 17 there is good agreement within 0.5% up to 350 K. The data given in Ref. 25 are higher near room temperature (ca. 3%) and decrease with increasing temperatures to a value about 1% smaller than our results at 480 K.

4.1.5. Nitrogen (Fig. 7)

The data of Refs. 5, 9, 10, 18, and 19 lie within a range of scatter of 1%. The values of Ref. 20 are above this range. The calculated values given in Ref. 19 and the correlated data of Ref. 33 are systematically smaller than those measured here by about 0.5 to 1%.

4.1.6. Air (Fig. 8)

The values stated in Ref. 9 are systematically higher than those given here by up to 0.8%. As the temperature increases the values of Ref. 10 deviate by up to 0.8%, as do the values given in Ref. 21. Recently, recommended data based on critically evaluated experimental material [30, 31] show somewhat higher values (up to 1.3%). Regarding the uncertainties both in Refs. 30 and 31 and here, a good agreement with Refs. 9, 30, and 31 may be claimed.

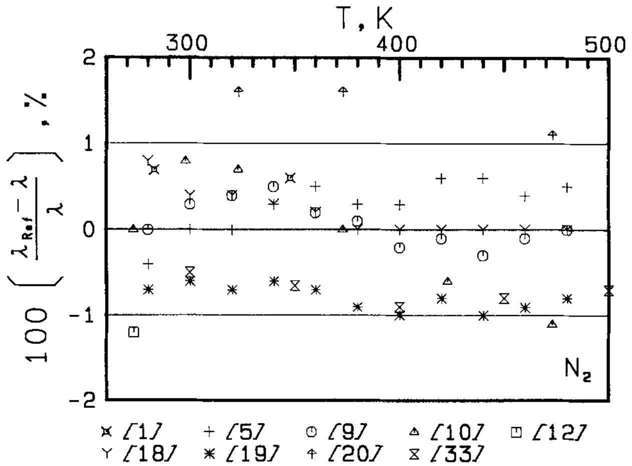

Fig. 7. Comparison of the experimental thermal conductivities with values λ_{Ref} given in the literature for N_2.

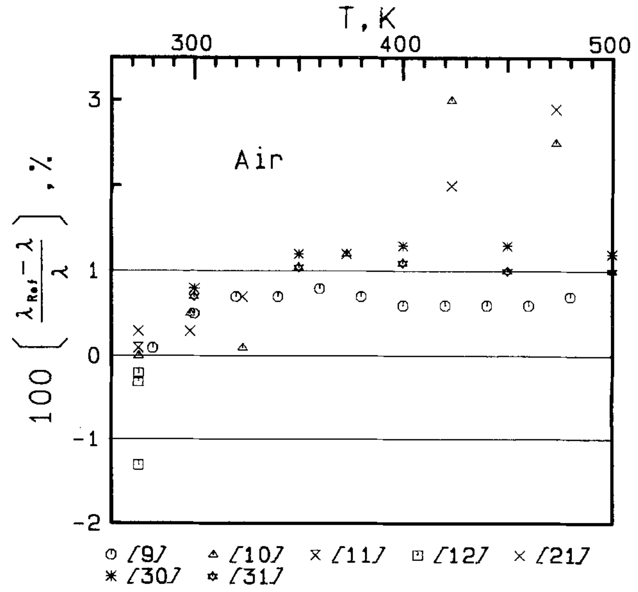

Fig. 8. Comparison of the experimental thermal conductivities with values λ_{Ref} given in the literature for air.

4.1.7. Argon (Fig. 9)

Due to contamination by desorbed air, the thermal conductivity of argon is expected to increase a little (cf. Fig. 2). All tabulated values [5, 9, 10, 18, 32, 33] are in fact systematically higher than the values measured here. There is very good agreement (within 0.5%) with the values given in Refs. 9 and 18. Smaller measurements are found in Refs. 12 and 14. Two hot-wire results near 300 K [34, 35] show a small deviation of about 0.6%.

4.1.8. Krypton (Fig. 10)

For Krypton there is a rather clear systematic deviation from the correlated values given in Refs. 10, 32, and 33. Irrespective of the temperature, these values are higher by 1 to 1.5%; desorbed air is suspected to be the reason. The hot-wire results of Refs. 34 and 35 are somewhat higher as well. The values given in Ref. 9 decrease with increasing temperatures; the data given in Ref. 22, too, show this tendency. Very large deviations are exhibited by the measurements from Ref. 21. With a specified uncertainty of 1%, however, the values are smaller by about 6% at 273 K and by about 13% at 473 K.

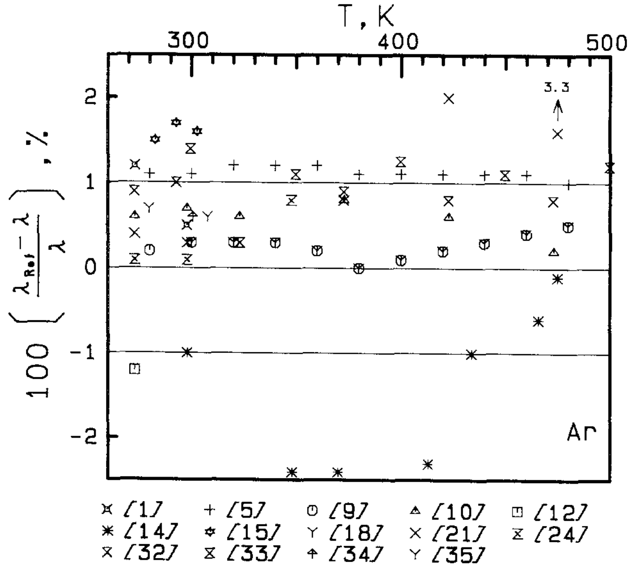

Fig. 9. Comparison of the experimental thermal conductivities with values λ_{Ref} given in the literature for Ar.

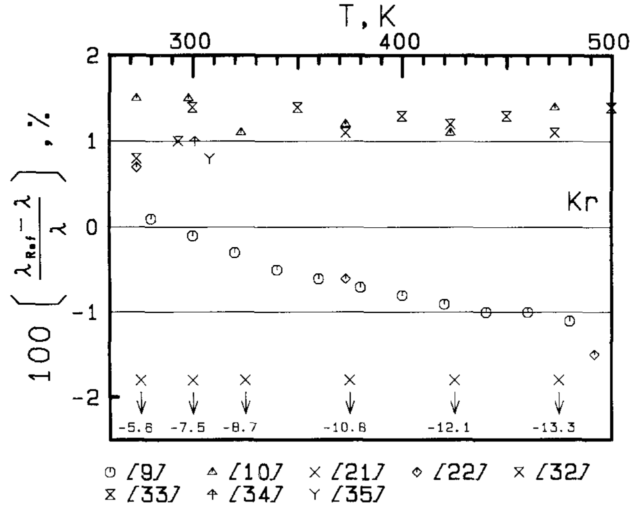

Fig. 10. Comparison of the experimental thermal conductivities with values λ_{Ref} given in the literature for Kr.

4.2. Comparison with Results Obtained According to the Kinetic Theory of Gases

According to the kinetic theory of gases, the ratio $Eu = \lambda/\eta c_v$ (Eucken factor) for monoatomic gases should be very close to 2.5 (η, viscosity; c_v, specific heat capacity at constant volume). Carefully evaluated Eu values [34, 35] based on hot-wire conductivity data and selected values of viscosity show a deviation of at most 0.25% for all noble gases at a distinct temperature (near 300 K). Here, we have to take into consideration a temperature interval of 200 K. Among the sources for viscosities and conductivities there are correlations which have been calculated in such a way that a thermodynamically consistent set of data is created, i.e., looking at those references a correct Eu value is self-evident. With the values determined here for λ, the correlated viscosity values given in Refs. 28 and 32 and the directly measured viscosities of Ref. 36, together with $c_v = 3/2(R/M)$ (gas constant $R = 8.3144\ \mathrm{J \cdot mol^{-1} \cdot K^{-1}}$; M; mass related to the amount of substance), we find a temperature dependence of Eu represented in Figs. 11 and 12. With increasing temperatures, the value for helium decreases rapidly to 2.50 (viscosities from Ref. 28). With the viscosities given in Ref. 36, Eu decreases from values higher to values lower than 2.5. Values based on Ref. 32 are systematically smaller by 0.6%. For neon, the Eu values based on Ref. 28 are systematically higher than 2.5; for Refs. 32 and 36 there is a similar behavior as in the case of helium. With Ref. 28 the

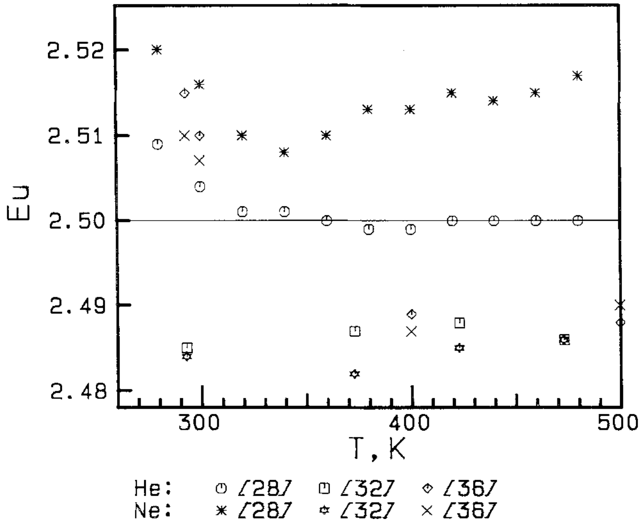

Fig. 11. Temperature dependence of $\mathrm{Eu} = \lambda\eta^{-1}c_v^{-1}$ for He and Ne.

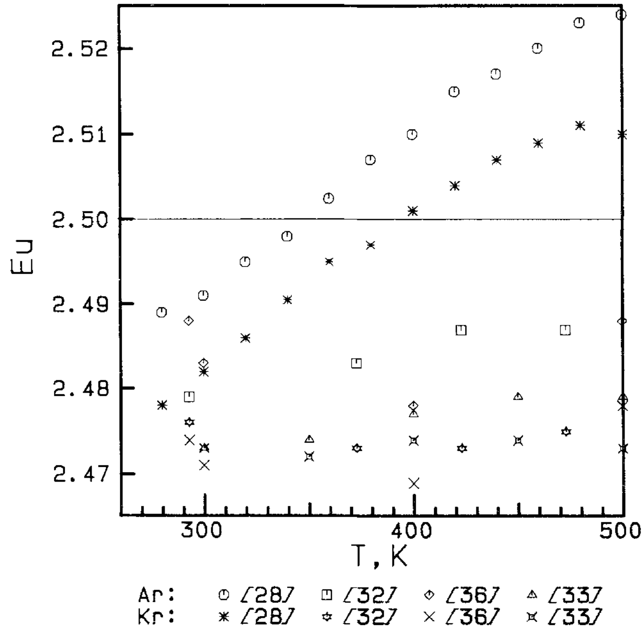

Fig. 12. Temperature dependence of $\mathrm{Eu} = \lambda\eta^{-1}c_v^{-1}$ for Ar and Kr.

values for argon and krypton increase monotonically with temperature; this increase is, however, less than half the increase stated in Ref. 29. There the largest difference between the Eu values (between helium and krypton at 0°C) is twice (about 5%) that stated here (about 2%, between neon and krypton at 0°C). In Ref. 29, this difference between argon and krypton is attributed to a dependence upon the molecular weight. Here, the uncertainty of the thermal conductivities and viscosities leads, however, to a systematic uncertainty for Eu of more than 1%, so the relative position of the Eu values for the different gases (Fig. 12) cannot be decided. It is thus not possible to check whether there is a dependence upon the molecular weight or not, but the results for noble gases at about 300 K, mentioned above, indicate that there is no dependence at all.

The Eu values of argon and krypton based on Refs. 32, 33, 36 and are, irrespective of temperature, systematically smaller than 2.5 by up to 1.2%.

The selected viscosities at 27.5 and 35°C cited in Refs. 34 and 35, respectively (which had been used to calculate Eu from the hot-wire data of conductivities), together with our conductivities yield an agreement within 0.1% for He and Ne, whereas there is a systematic deviation of −0.4% for Ar and −0.7% for Kr. A more recent investigation [37] supposes too high experimental viscosities in some of the sources used; this would account for the negative deviation mentioned.

In summary, it can be said that our Eu values for monoatomic gases which have been calculated with various viscosity values lie within a range of scatter of about 1% around $f = 2.50$. Regarding the uncertainties and considering the 200 K temperature interval, the compliance with the theoretically demanded value is good, although the origin of systematic deviations with regard to some tabulated data sets is not fully obvious.

For the diatomic gases nitrogen and hydrogen the following can be stated: in the range between 280 and 500 K, Eu increases for nitrogen from 1.95 to 1.97 and decreases for hydrogen from 2.07 to 2.04 [with the values of $c_v(T)$ given in Ref. 16].

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