Simplified AC-Heated Probe Method[1]

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ABSTRACT

A new ac-heated probe method using two low-inertial probes (wire and strip of foil) is described for measurement of liquid thermal conductivity, thermal diffusivity, thermal effusivity and specific heat. The probes are immersed in the liquid and heated by an infrasonic-frequency, sine-wave current. Projections of the signal on the imaginary axis (quadrature parts) are registered at the same frequency as the voltage drive to the probes. With the use of new simple asymptotic formulas, it is possible to calculate directly the absolute values of the thermal properties of liquids. The thermal effusivity is determined using the signal from the foil probe and its area. The thermal conductivity is determined using the signal from the wire probe and its length. The measured value of the thermal effusivity can be used to calculate corrections to the thermal conductivity value. The thermal diffusivity and volumetric specific heat can then be easily calculated. Ways to eliminate free convection are proposed.

KEY WORDS: 3-omega method, heated strip; heated wire; liquids; periodic heating; specific heat; thermal conductivity; thermal diffusivity; thermal effusivity.

1. INTRODUCTION

The ac-heated wire method (periodic heating method, or 3-omega method) was originally described for measurement of thermal conductivity, thermal diffusivity, thermal effusivity and specific heat by Filippov [1]. Developments were reported in the dissertations of Nefedov [2], Kravchun [3], and Tleoubaev [4] at Moscow Lomonosov State University. The method is based on measured amplitude and phase of the tripled frequency signal appearing on the bridge's diagonal in one of arm of which a low-inertial probe of thin wire immersed into the liquid is connected [5-9] while the bridge is driven by a sine-wave current. The method has advantages relative to other methods, as follows:

(a) very thin probed layer of liquid that makes the method especially effective at high temperatures because the thermal conductivity is purely conductive, without a significant radiative contribution,

(b) high information content: four thermal properties are measured,

(c) compact and simple measurement cell, that requires very small quantities of liquid (a few cm^3) for measurements, and

(d) opportunity for total automatization of the measurement process with continuous scanning of the temperature and pressure of the sample.

Despite all these merits, the method is not widely used compared to the transient hot-wire method (see, for example, Ref.[10]).

Concerns, which limited the widespread use of the ac-heated wire, include:

(a) the method was usually operated in a relative mode with a toluene as reference fluid,

(b) misgivings that convective flows can result in acquisition of unreliable thermal properties values [11],

(c) absence of formulas for the direct calculation of thermal properties – the properties were obtained by iterations using cylindrical Kelvin functions, and

(d) complicated measurement procedure requiring both the amplitude and phase of the tripled frequency signal.

In the present work, ways to overcome all these limitations are described. The simplified procedure requires measurement only of the projection of the main frequency signal on the imaginary axis, the so-called quadrature signal, and uses simplified asymptotic formulas for data treatment. Recording the quadrature signal, which provides information about the thermal properties of probe and the liquid is much easier than recording the tripled frequency signal parameters. The quadrature signal is nearly not sensitive to the temperature drift of the cell, so temperature control does not need to be as accurate.

2. THEORY

2.1. Solution of the Differential Equation for Temperature Oscillations

When solving the problem for the complex field of temperature oscillations, the common thermal conductivity equation is reduced (for the complex alternating component of the temperature $\tilde{T}$) to the solution of the wave differential equation, being a special case of the Helmholtz equation [12,13]:

$$\Delta \tilde{T} - (2\, i\omega / a)\, \tilde{T} = 0 \qquad (1)$$

where Δ is the Laplace operator; i is the imaginary one; ω is the angular frequency of voltage that drives the probe; and a is the thermal diffusivity of the fluid surrounding the probe. This equation is a second order, elliptical differential equation [12,13].

General solutions of this equation are:

(1) planar waves in case of foil probe - linear combination of exponents:

$$\tilde{T} = A\,exp\{-ikx\} + B\,exp\{ikx\} \qquad (2)$$

where $k=(-2i\omega/a)^{1/2}$ is the complex wave number of the temperature oscillation; and x is the distance from a plane at the foil's center; and

(2) cylindrical waves in case of wire probe - linear combination of modified Bessel functions I_0 and K_0 or Kelvin functions $ber\kappa + ibei\kappa$ and $ker\kappa + i\,kei\kappa$:

$$\tilde{T} = A\,I_0(i^{1/2}\kappa) + B\,K_0(i^{1/2}\kappa) = A\,(ber\kappa + ibei\kappa) + B\,(ker\kappa + ikei\kappa) \qquad (3)$$

where κ is the dimensionless thermal similarity parameter for the field of the temperature oscillations, the analog of $Fo^{-1/2}$:

$$\kappa = r(2\omega/a)^{1/2} \qquad (4)$$

Fo is Fourier number, r is radius coordinate.

The first terms in these general solutions describe a wave whose amplitude grows with an increase of the argument (x or r), i.e., the approaching wave. Correspondingly, the second terms describe a wave whose amplitude diminishes with an increase of the argument, i.e., the departing wave.

To find the particular solution of a thermal problem, it is necessary to define the complex constants A and B substituting the following boundary conditions into these general solutions:

(i) probe's thermal balance - law of energy conservation;

(ii) Sommerfeld radiation condition for decay of the temperature wave at infinity [12];

(iii) equality of temperatures and thermal flows at the probe's surface, i.e., condition of ideal thermal contact; and

(iv) some additional conditions, if needed, for solving more difficult problems [9,14].

As a result, the complex alternating temperature of the probe $\tilde{T}$ ($Re\,\tilde{T} > 0$, $Im\,\tilde{T} < 0$) is related with its reduced dimensionless complex temperature $\tilde{\Theta}$ as follows:

$$\tilde{T} = [W/(4C_p\, m\omega)]\, \tilde{\Theta}\,(\kappa,\eta) \tag{5}$$

where W is the amplitude of electrical power in the probe; C_p is the specific heat of the probe material at constant pressure; m is the mass of the probe; and κ is the thermal similarity parameter - see Eq. (4), where its radius r is used in the case of the wire, and its half-thickness h is used in the case of the foil; η is the ratio of the volumetric specific heats of the probe material and its environment divided by 2:

$$\eta_w = C_{pw}\,\rho_w /(2C_p\rho) \tag{6}$$

$$\eta_f = C_{pf}\,\rho_f /(2C_p\rho) \tag{7}$$

where the subscripts w and f relate to the wire and foil, respectively.

For probes located in vacuum, κ is very small ~(10^{-4} to 10^{-5}), $\eta \rightarrow \infty$, $\kappa\eta \rightarrow \infty$, $\kappa^2\eta \rightarrow \infty$, and $\tilde{\Theta} \rightarrow -i$. Equation (5) can be used to obtain a calibration value of $(dR/dT)/(C_p m)$ and then to determine a value of $\tilde{\Theta}$ from the measured $\tilde{T}$ value [6-8].

For probes located in liquid, the following expressions for $\tilde{\Theta}$ were obtained:

(1) in case of infinitely extended foil:

$$\tilde{\Theta}_f\,(\kappa_f\, .\eta_f) = [\, i + i^{1/2} / (2\kappa_f\, \eta_f)]^{-1} \tag{8}$$

(2) in case of infinitely long wire:

$$\tilde{\Theta}_w\,(\kappa_w\,,\eta_w) = [\, i - (ker'\kappa_w + i\cdot kei'\kappa_w)/(ker\kappa_w + i\cdot kei\kappa_w)/(\kappa_w\eta_w)]^{-1} \tag{9}$$

where $ker\kappa$, $kei\kappa$, $ker'\kappa$, $kei'\kappa$ are Kelvin functions and their derivatives with respect to the thermal similarity parameter κ.

During derivation of these equations (which are valid for all values of κ and η) it was assumed that the temperature waves inside the probes are absent because their lengths

$$l^* = 2\pi[a/(2\omega)]^{1/2} \tag{10}$$

are much larger than the foil half-thickness h and the wire radius r since the metal has much larger thermal diffusivity than the liquid.

2.2. Thermal Effusivity Absolute Values by Foil Probe

To separate the real and imaginary parts, Eq.(8) can be re-written as

$$\tilde{\Theta}_f(\kappa_f\,\eta_f) = [2^{1/2}\kappa_f\,\eta_f - i\cdot\kappa_f\,\eta_f(\kappa_f\,\eta_f + 2^{1/2})] \,/\, [1 + 2\cdot 2^{1/2}\kappa_f\,\eta_f + 4(\kappa_f\,\eta_f)^2] \tag{11}$$

Using Eq.(5) it is possible to determine the absolute value of the thermal effusivity ε through measurement of the imaginary component of the signal from the foil probe (as it was done previously through measurement of the amplitude of the tripled frequency signal [1-3])

$$\varepsilon = W \,/\, [4(2\omega)^{1/2} S_f \, Im\,\tilde{\Theta}_f\,](1+\delta_f) \tag{12}$$

$$\delta_f \cong -\,4(\kappa_f\,\eta_f)^2 \tag{13}$$

where S_f is the area of the foil probe. The magnitude of the correction δ_f is very small (thickness of foil $2h$ is about 1 to 3 μm, so κ_f ~0 01 to 0.03, η_f ~1, $\delta_f < 0.36\%$) and it can be neglected, but it may also be taken into account, if desired:

$$\kappa_f\,\eta_f \cong -\,[2\cdot 2^{1/2}\, Im\,\tilde{\Theta}_f\; C_{pf}\;\; m_f\,\omega]/W \tag{14}$$

To ensure that the foil probe was a source of flat temperature waves, its width (~1 to 2 mm) should be many times longer than the length of the temperature wave (~0.01 to 0.05 mm), calculated using Eq.(10).

2.3. Approximate Formulas to Obtain Thermal Properties Using the Wire Probe

New simple formulas for direct calculation of absolute thermal properties values can be obtained using the second order asymptotic formulas for the Kelvin functions of Eq.(9):

$$ker\kappa = -ln(\kappa\gamma/2) + (\pi/16)\kappa^2 + O(\kappa^4); \quad (15)$$

$$kei\kappa = -\pi/4 + [1-ln(\kappa\gamma/2)]\kappa^2/4 + O(\kappa^4); \quad (16)$$

$$ker'\kappa = -1/\kappa + (\pi/8)\kappa + O(\kappa^3); \quad (17)$$

$$kei'\kappa = \kappa/4 - ln(\kappa\gamma/2)\kappa/2 + O(\kappa^3); \quad (18)$$

(γ is the Euler's constant, which is equal to 1.781072418...)

Substituting these approximate expressions into Eq.(9), the reduced dimensionless complex temperature $\tilde{\Theta}$ of the wire probe is obtained:

$$\tilde{\Theta}_w(\kappa_w,\eta_w) \cong -\kappa^2_w\eta_w ln(\kappa_w\gamma/2)(1+\delta_1)/(1+\delta_3) - i(\pi/4)\kappa^2_w\eta_w(1+\delta_2)/(1+\delta_3) \quad (19)$$

and consequently, substituting Eq.(5), following expressions are found for direct calculation of the liquid properties using the wire probe:

$$\lambda \cong -W/(16 L_w Im\tilde{T}_w)(1+\delta_2)/(1+\delta_3); \quad (20)$$

$$a \cong r^2_w\gamma^2\omega/2\, exp\{-(\pi/2)(Re\tilde{T}_w/Im\tilde{T}_w)(1+\delta_2)/(1+\delta_1)\}; \quad (21)$$

where δ_1, δ_2, δ_3 are small corrections:

$$\delta_1 = -(\pi/8)\kappa^2_w/ln(\kappa_w\gamma/2) + O(\kappa^4_w); \quad (22)$$

$$\delta_2 = (4/\pi)\kappa^2_w\eta_w\{[ln^2(\kappa_w\gamma/2)+\pi^2/16](1-0.5/\eta_w)+ln(\kappa_w\gamma/2)/2/\eta_w-0.25/\eta_w\}+O(\kappa^4_w); \quad (23)$$

$$\delta_3 = (\pi/2)\kappa^2_w\eta_w(1-0.5/\eta_w) + O(\kappa^4_w); \quad (24)$$

These corrections δ_1, δ_2, δ_3, do not exceed a few percent at $\kappa < 0.3$ ($0.5<\eta<1.3$), and can be calculated using the following expressions found from the approximate formulas of the first order:

$$\kappa^2{}_w\eta_w \cong -16C_{p\,w}m_w\omega\, Im\ \tilde{T}_w/(\pi W) \tag{25}$$

$$ln(\kappa_w\gamma/2) \cong (\pi/4)(Re\,\tilde{T}_w/\,Im\,\tilde{T}_w) \tag{26}$$

$$\kappa^2{}_w \cong (4/\gamma^2)\, exp\{(\pi/2)(Re\,\tilde{T}_w/\,Im\,\tilde{T}_w)\} \tag{27}$$

$$\eta_w = \kappa^2{}_w\eta_w/\,\kappa^2{}_w \tag{28}$$

As evident from Eq. (20), to obtain the absolute value of the thermal conductivity it is sufficient to measure the imaginary component of the wire probe signal. Knowledge of the probe length L_w is required. To obtain the thermal diffusivity absolute value from Eq.(21), it is sufficient to measure the phase (or the ratio of the real and imaginary components) of the wire's temperature oscillations and the frequency ω. Knowledge of the wire radius r_w is required. Also the values of applied power W and of derivative of the wire resistance with respect to temperature dR/dT are required.

The asymptotic expressions of Eqs.(15-27) are valid if one uses sufficiently thin wires and relatively low frequencies for the drive voltage so κ_w is not bigger than ~0.3. For example, for a wire of 12.7 μm diameter, at frequency of 5 Hz and typical value of thermal diffusivity of $\sim 9 \cdot 10^{-8}$ $m^2 \cdot s^{-1}$ (toluene at normal conditions), $\kappa_w = 0.168$.

Errors of values $Im\,\tilde{\Theta}$ and $Im\,\tilde{\Theta}/Re\,\tilde{\Theta}$, calculated with the approximate Eqs.(19), (22), (23), and (24) (see Fig.1) do not exceed 0.2 % at $\kappa_w < 0.3$ (calculations were made for values of η_w from 0.6 to 1.2, typical for liquids at normal conditions with a platinum probe).

Accurate measurements of $Re\tilde{\Theta}$, (and correspondingly of the ratio $Im\tilde{\Theta}/Re\tilde{\Theta}$) are possible only at the *tripled* frequency, because the value of $Re\tilde{\Theta}$ at the *main* frequency is sensitive to the bridge balance (which depends on the cell's temperature drift). In the following section, it will be shown how to measure a group of four thermal properties without measuring the tripled frequency signal parameters.

2.4. Use of Two Probes to Measure Four Thermal Properties

Using values of $Im\tilde{\Theta}$ from the wire and foil probes at the main frequency ω, the absolute values of four thermal properties can be determined using following procedure:

(i) obtain thermal effusivity ε from measured value of $Im\tilde{\Theta}_f$ of foil probe by Eq.(12);

(ii) obtain approximate values of thermal conductivity λ and product $\kappa^2_w\eta_w$ from measured value of $Im\tilde{\Theta}_w$ of wire probe by Eq.(20) without corrections and Eq.(25);

(iii) obtain approximate value of thermal diffusivity by the formula:

$$a=\lambda^2/\varepsilon^2 \quad (29)$$

and then approximate value of parameter κ_w by Eq.(4) (value of the wire radius r_w is approximately known);

(iv) obtain approximate value of parameter η_w by the formula:

$$\eta_w=(\kappa^2{}_w\eta_w \cdot a)/(r^2{}_w \cdot 2\omega) \quad (30)$$

(v) obtain values of corrections δ_2 and δ_3 by Eqs.(23) and (24);

(vi) obtain the exact value of thermal conductivity λ by Eq(20);

(vii) finally, obtain exact values of two other thermal properties - thermal diffusivity a on Eq. (29) and volumetric specific heat $C_p\rho$ by the formula:

$$C_p\rho=\varepsilon^2/\lambda \quad (31)$$

Another more convenient way to get the exact value of the thermal conductivity λ (instead of step vi) is through the use of the graph of Fig.2, where correction to the approximate thermal conductivity value versus parameter κ_w is plotted for various values of parameter η_w. Since the correction is small (it does not exceed few percent) the accuracy of this graph is adequate.

In general, if only the wire probe is used for thermal conductivity measurements, then handbook data are adequate to calculate the parameter η_w by Eq.(6) and parameter κ_w can be found using Eq. (25) along with Fig.2 to determine the correction for λ.

3. INFLUENCE OF FREE CONVECTIVE FLOWS

Using ac-heated probes, free convective flows can occur because of the probe's constant overheating relative to the cell walls. The thermal boundary layer thickness δ for free convection can be estimated [15] (if the probe is vertical):

$$\delta \simeq [(4\nu^2\xi)/(g\,\beta'\,T)]^{1/4} \tag{32}$$

where ν is the kinematic viscosity of the liquid, ξ is the distance from the lower end of the probe, g = 9.8 m·s^{-2}, β' is the factor of liquid's thermal expansion, and T is the probe's overheating.

The layer thickness δ for toluene at 473K with an overheat T=4K is about 0.1mm at ξ = 0.032 mm and increases as $\xi^{1/4}$. At the same time, the length of the temperature wave is about 0.01-0.05 mm, and since the temperature wave is strongly attenuated (over the length of wave in $exp\{-2\pi\}$~500), only the motionless boundary layer of liquid adjacent to the probes surface is probed during the measurements.

As a further check a corresponding thermal problem was analytically solved, setting the temperature oscillations $\tilde{T}$ to zero at some distance δr from the wire surface (i.e., due to the flow of liquid parallel to the probe surface). Computations based on this model showed that when the length of the temperature wave is less than δr (thickness of boundary layer of liquid), then the flow of liquid does not influence to wire's temperature oscillations $\tilde{T}_w$.

The most simple and reliable way to minimize the influence of convective flows is reduction of the cell diameter. In small diameter cells, convective flows are practically absent, which guarantees obtaining of reliable values of the properties. As shown experimentally in Ref.[16], slow flows do not influence the probe's temperature oscillations at Reynolds numbers up to 150, which corresponded to the flow velocities up to a few $cm \cdot s^{-1}$ (at a frequency of 23 Hz).

Based on these calculations and our experience, the optimum design of the cell is a 12-20 μm platinum diameter wire, centered along the axis of a tube of 3 to 5 mm ID. Tension is maintained using a small spring made of nickel 100 μm diameter wire to compensate the probe's thermal expansion.

Initial attempts to measure thermal properties of reference liquids -toluene and carbon tetrachloride - using intentioned wire probes almost always resulted in inaccurate thermal property values. The amplitude of lateral oscillations in the middle of the probe without the spring is equal to:

$$L_w(\alpha' \mid \tilde{T} \mid /2)^{1/2} \qquad (33)$$

where α' is the coefficient of thermal expansion (for platinum, α' ~$9x10^{-6}K^{-1}$). For an untensioned probe this movement can exceed the wire diameter ($2r_w$ ~ 12 to 20 μm) and is not permissible.

5. CONCLUSIONS

A new simplified ac-heated probe method is developed with direct calculation formulas for measurements of thermal conductivity, thermal effusivity, thermal diffusivity and volumetric specific heat of liquids. This method uses two low-inertial probes - wire and foil - and is based on the quadrature signal at frequency of the drive voltage. Installation based on the method can be easily assembled with a commercial vector generator and lock-in microvoltmeter. Preliminary test measurements of thermal conductivity of toluene and carbon tetrachloride at ambient temperature using a tungsten wire probe gave satisfactory ~2-3% agreement with previous results.

This method and formulas also could be used in a new intelligent transducers for non-interrupted monitoring of thermal properties in chemical engineering processes.

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LIST OF SYMBOLS

a thermal diffusivity $a=\lambda/C_p\rho$

$ber\kappa$, $bei\kappa$, $ber'\kappa$, $bei'\kappa$ Kelvin functions and their derivatives

C_p specific heat at constant pressure

$C_p\rho$ volumetric specific heat

g acceleration of gravity

h half-thickness of the foil probe

I_0, I_1 modified Bessel functions

K_0, K_1 modified Bessel functions

$ker\kappa$, $kei\kappa$, $ker'\kappa$, $kei'\kappa$ Kelvin functions and their derivatives

L length of probe

l^* length of temperature wave $l^* = 2\pi[a/(2\omega)]^{1/2}$

m mass of probe

r radius

S foil probe area

T probe overheat

$\tilde{T}$ complex alternating component of the temperature

W amplitude of power supply in the probe

x distance from plane of foil center

GREEK SYMBOLS

α' temperature factor of probe linear extension

β' temperature factor of liquid volume expansion

γ Euler's constant γ=1.7810724...

Δ Laplace operator

δr temperature boundary layer thickness

$\delta_1, \delta_2, \delta_3, \delta_f$ small dimensionless corrections

ε thermal effusivity, $\varepsilon = (\lambda C_p \rho)^{1/2}$

η ratio of volumetric specific heats of probe material and liquid divided by 2:

$\eta_w = C_{pw}\rho_w / (C_p\rho)$, $\eta_f = C_{pf}\,\rho_f / (C_p\rho)$.

κ thermal similarity parameter: $r_w(2\omega/a)^{1/2}$ for the wire and $h_f\,(2\omega/a)^{1/2}$ for the foil

λ thermal conductivity

ν kinematic viscosity

ρ density

$\tilde{\Theta}$ reduced dimensionless complex temperature of the probe

ω circular frequency of voltage

SUBSCRIPTS

f refers to foil probe

w refers to wire probe

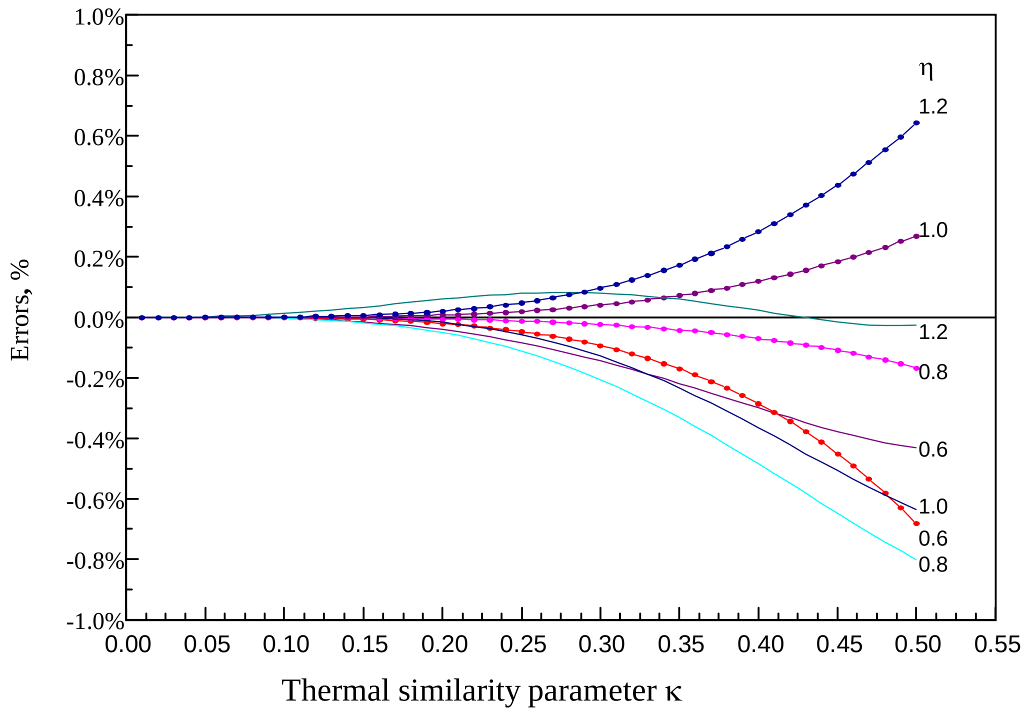

Fig.1. Errors of values of $\mathrm{Im}\tilde{\Theta}$ (smooth lines) and $Im\tilde{\Theta}/Re\tilde{\Theta}$ (rough lines) calculated using the approximate Eqs.(19,22-24) relative to the exact expression Eq.(9) vs. κ_w for some typical values of η_w.

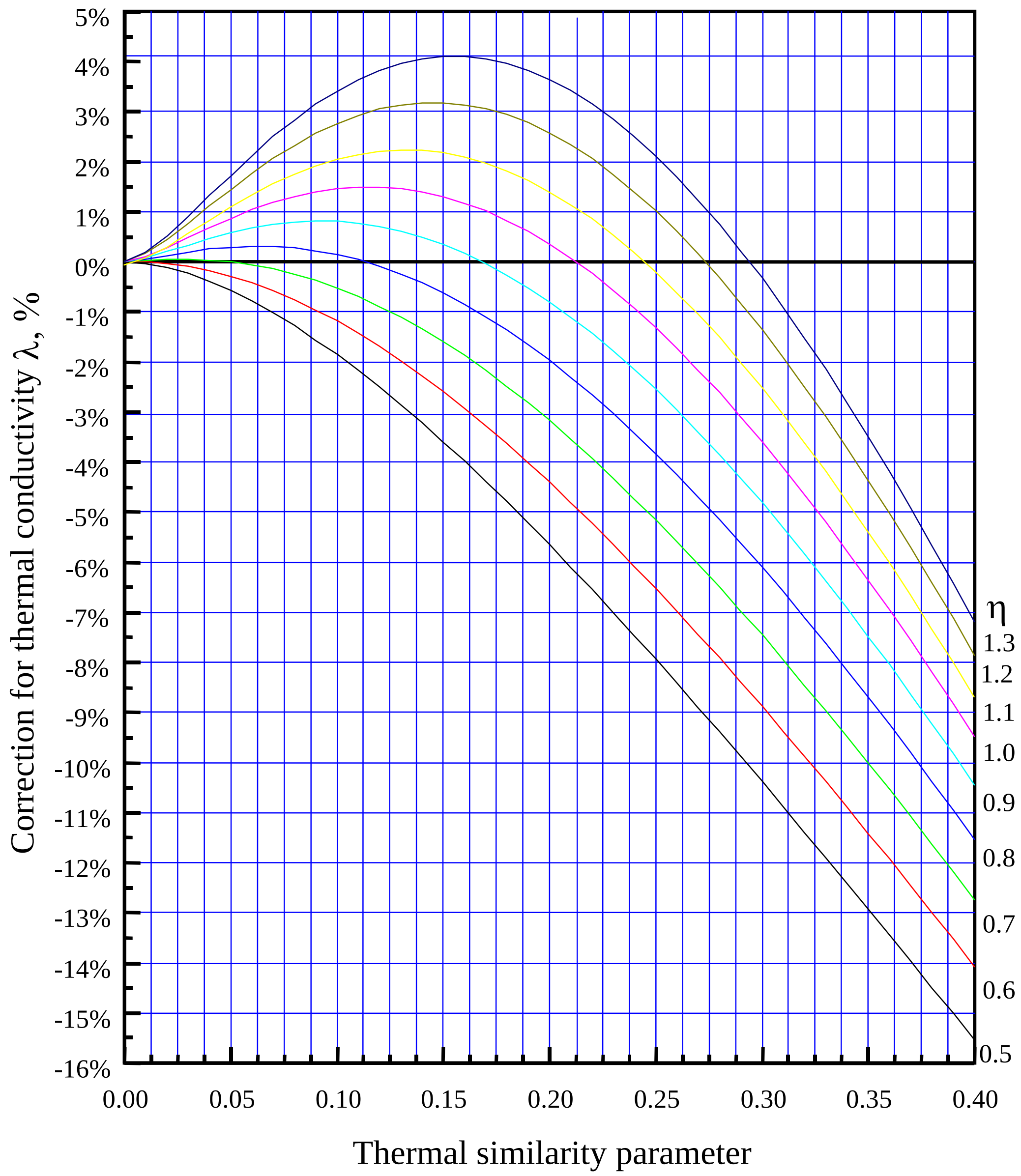

Fig. 2. Correction for thermal conductivity calculated by exact Eqs.(5) and (9) vs. κ_w for some typical values of η_w.