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A New Transient Two-Wire Method for Measuring the Thermal Diffusivity of Electrically Conducting and Highly Corrosive Liquids Using Small Samples

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Abstract The transient hot-wire (THW) technique is widely used for measurements of the thermal conductivity of most fluids, and some attempts have also been carried out for simultaneous measurements of the thermal diffusivity with the same hot wire. However, for some particular liquids like concentrated nitric acid solutions or similar nitric mixtures, for which the thermal properties are important for industrial or security applications, this technique may be difficult to use, because of possible technological incompatibilities between measurement probe materials and highly electrically conducting and corrosive liquids. Moreover, the possible highly energetic (explosive) character of these liquids requires minimum volume liquid samples and safety measurement devices and processes. It is the purpose of this paper to report on a modified THW technique (previously used for thermal-diffusivity measurements in soils), which is associated with a specific patented double-wire probe and is shown to be valid for direct thermal-diffusivity measurements in liquids. This method responds to the previous requirements and allows automatic and quasi-simultaneous thermal-conductivity and thermal-diffusivity measurements to be made safely on liquids compatible with the tantalum technology, with liquid sample volumes $< 2\,\mathrm{cm}^3$. Low uncertainties are found for the thermal-diffusivity data when relative measurements are carried out with reference liquids like water or toluene.

Keywords Corrosive electrically conducting liquid · Nitric acid · Small sample · Thermal diffusivity · Transient hot-wire technique · Two parallel tantalum wire probe

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1 Introduction

The transient hot-wire (THW) technique is now recognized as the primary method for accurate determination of the thermal conductivity λ of fluids, with also the possibility to obtain the thermal diffusivity a by using the same hot-wire experimental data [1–5]. These determinations of λ and a are achieved by using the experimental temperature response of a thin metallic wire (hot wire) immersed in the fluid and suddenly submitted to a constant heat pulse, with the assumption of an infinitely long line heat source and a purely conducting heat transfer regime around the wire. To approach the first assumption, a sufficiently long wire and large volume ($>30\,\text{cm}^3$) are generally used for experiments, with the possibility of very low ($<1\,\%$) uncertainties claimed for absolute λ measurements but with larger uncertainties associated with absolute a measurements (1.8 % in [2], 4 % in [3], 5 % in [5], (7 to 9) % in [1], 50 % in [4], ...), thus showing the specific difficulties of thermal-diffusivity determination in liquids by the classical THW technique.

A variant of the above THW method, using a sinusoidal alternating current for heat generation in the wire, has been developed and tested in water and glycerin, with the claimed advantage of smaller ($13\,\text{cm}^3$) liquid samples and uncertainties <19 % [6,7] for thermal-diffusivity measurements.

The aim of the present work is to develop and test an apparatus (with an original patented measuring cell described in a previous paper [8] for thermal-conductivity measurements) and a method specifically adapted to the thermal-diffusivity determination of very small samples ($<2\,\text{cm}^3$) of electrically conductive and highly corrosive liquids which may be, moreover, very energetic (with explosion risks during measurements). The original method proposed for the thermal-diffusivity measurement of such liquids (called *special* in this paper) is a modified THW method using a two-wire probe, according to a measurement principle previously developed in [9] for the thermal-diffusivity determination of soils; moreover, for applications to such *special* liquids, the tantalum technology has been chosen because tantalum metal has well known electrical and thermal properties [10,11] and a very high resistance to corrosion with most concentrated strong acids (nitric, sulfuric, hydrochloric, and hydrobromic,...) [12]. Moreover, tantalum wires are manufactured with small diameters ($\geq 25\,\mu\text{m}$) which can easily be reduced by a specific electrochemical treatment and coated by a tantalum pentoxide anodic film of controlled nanometer thickness [13–15], to ensure its electrical insulation.

2 Theoretical Method

For an infinite line heat source, immersed in a fluid of infinite extent and uniform temperature T_0 with a power q per unit length initiated at time $t = 0$ and only dissipated in the liquid by conductive heat transfer, the instantaneous local temperature of the fluid at a radial distance r from the line source is given by [16]

$$\Delta T\,(r,t) = T\,(r,t) - T_0 = -\frac{q}{4\pi\lambda}\mathrm{Ei}\left(\frac{-r^2}{4at}\right) \tag{1}$$

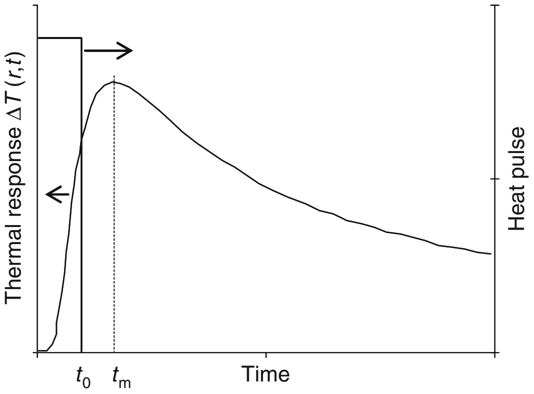


Fig. 1 Schematic of the temperature response $\Delta T\,(r, t)$

where Ei is the exponential integral function.

When the infinite line heat source is replaced by a constant heat pulse of duration t_0, the analytical solutions of the thermal response $\Delta T(r, t)$ can be expressed as [17,18]

$$\begin{aligned}&\Delta T\ (r, t) = 1 \text{ for } 0 \le t \le t_0\\ &\Delta T\ (r, t) = \tfrac{q}{4\pi\lambda}\left[\mathrm{Ei}\left(\tfrac{-r^2}{4a(t-t_0)}\right) - \mathrm{Ei}\left(\tfrac{-r^2}{4at}\right)\right] \text{ for } t > t_0\end{aligned} \tag{2}$$

Making use of the fact that the temperature response $\Delta T\ (r, t)$ (given by Eq. 2) at a distance r from the line source displays a maximum ΔT_m (Fig. 1) for $t = t_\mathrm{m}$ (corresponding to $\mathrm{d}\Delta T/\mathrm{d}t = 0$), the following expression is obtained [9]:

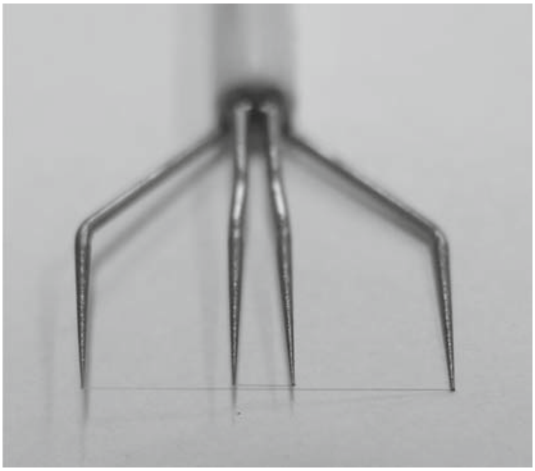
$$a = \frac{r^2}{4}\left[\frac{1/\left(t_\mathrm{m} - t_0\right) - 1/t_\mathrm{m}}{\ln\left(t_\mathrm{m}/\left(t_\mathrm{m} - t_0\right)\right)}\right] \tag{3}$$

Equation 3 can be used for the determination of the thermal diffusivity a by using only the knowledge of the distance r and of the two times t_0 and t_m (without need of any temperature value). Until now, Eq. 3 has only been used for thermal-diffusivity measurements in soils [9,17–23], without applications to liquids. This paper will demonstrate that, for appropriate q and t_0 values (to prevent free convection and radiative effects), Eq. 3 can also be used for thermal-diffusivity measurements in liquids.

3 Experimental Apparatus

The experimental apparatus is composed of an electrical measurement system (Sect. 3.3) associated with a stainless-steel vessel (Sect. 3.2) equipped with an independent tantalum two-wire probe (Sect. 3.1). The device consisting of the vessel and the probe is patented [24] and specifically developed for small ($<2\,\mathrm{cm}^3$) *special* liquid samples and has recently been described in [8] for thermal-conductivity measurements.

Fig. 2 Two-wire probe



3.1 Tantalum Two-Wire Probe

The active part of the measuring probe (Fig. 2) is composed of two parallel tantalum wires (lengths, respectively, about 15 mm and 2 mm), with each wire spot-welded on two 1 mm diameter tantalum prongs partially embedded in a 4 mm diameter ceramic rod. The longer wire (diameter of 25 μm) or "hot wire" is used as a line heat source supplied by a constant current of intensity I during a time t_0 (heat pulse of duration t_0) while the shorter wire (diameter 5 μm) or "cold wire" is used as a resistance thermometric sensor (supplied by a constant measurement current of intensity i), with negligible internal Joule heating. The 7.5 ratio between the wire lengths ensures negligible "hot-wire" end effects on the thermal response of the short wire.

The tantalum elements (wires and prongs), which have to be immersed in *special* liquids, are electrically insulated in situ (after spot-welding of the wires on the prongs) with a coating layer of tantalum pentoxide (Ta_2O_5), according to a procedure described by Alloush et al. [13]. This coating layer, with a thickness of about 70 nm, is formed by anodization and has proved to be strongly adherent and electrically efficient throughout a series of temperature cycles, with temperatures up to 370 K, for thermal-conductivity measurements in water and toluene [14].

3.2 Measuring Cell

The measuring cell, which has already been described in [8,24], is composed of a stainless-steel vessel of 2 cm^3 volume with the tantalum two-wire probe mounted on the top with a Teflon gasket. The cell is designed to work over the 0.1 MPa to 1 MPa pressure range and the 0 °C to 80 °C temperature range with the vessel immersed in a liquid thermostatic bath at a controlled temperature. The cell is also designed to allow easy filling and removal of the liquid in the vessel which includes, for this purpose, two specific liquid ports.

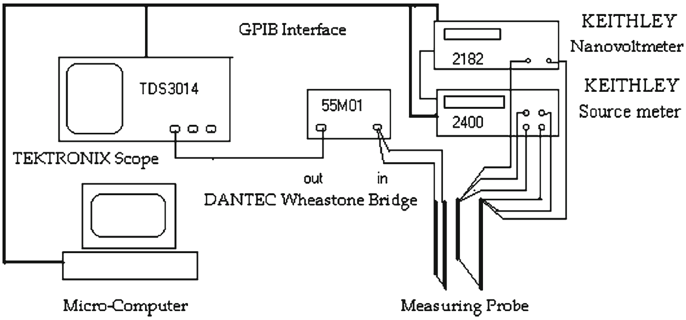


Fig. 3 Block diagram of the measuring system

3.3 Measurement System

A block diagram of the electrical measuring system is shown in Fig. 3. The whole experimental system is composed of the measuring probe, a source meter (Keithley 2400), a nanovoltmeter (Keithley 2182), a Wheatstone bridge (Dantec 55M01), a scope (Tektronix TDS 3014) and a microcomputer which links and controls the different elements.

The shorter wire is connected to one arm of the Wheatstone bridge, the voltage of which gives a direct measurement of the wire resistance and thus of the wire (and liquid) temperature. The scope is used to record the signal in order to transfer it in the microcomputer. The source meter (Keithley 2400) can supply and measure voltage or current very precisely by means of a four-wire technique. The precision of the source current in the working range (0 mA to 100 mA) is 0.034 % of the reading + offset. Because of the limited measurement capabilities of the meter, it was necessary to add a nanovoltmeter (Keithley 2182) to ensure better precision. On the (0–100) mV range, the resolution of this meter is 10 nV and the uncertainty over 1 year is better than 30 ppm of reading. The speed of acquisition of the source meter and of the nanovoltmeter can vary, depending on the choice of the integration time NPLC (number of power line cycles). To provide the best signal-to-noise ratio, the NPLC value is often chosen corresponding to a 20 ms sampling period and typically to an acquisition frequency of 10 Hz. The two meters are connected to a microcomputer via a GPIB interface. A LabView® program (National Instruments), specifically written for this application, controls the instruments and records the data.

4 Experimental Results

4.1 General Considerations

An experimental determination of the thermal diffusivity a of a given liquid from Eq. 3 requires the measurement of three parameters: r (distance between parallel wires), t_0 (imposed duration of the heat pulse), and t_m (measured time between the maximum temperature rise of the “cold wire” and the outset of the heat pulse). So, the final uncertainty for the a determination is dependent on the uncertainties associated to measurements of these three parameters, but also of departures between the experimental and ideal physical models of heat transfer from the heated wire.

A theoretical analysis of potential sources of errors associated with the measurement method, proposed in this paper for liquids, has previously been developed by Kuitenberg et al. [18] for applications to thermal-diffusivity measurements in soils. In their study, these authors successively analyze the model errors and the measurement errors associated with the a determination, with the following conclusions:

- the model errors (caused by finite probe length and cylindrical probe shape) “cannot be treated separately” but “appear to be approximately the sum of the errors caused by the finite length (1.4 % to 0.6 %) and cylindrical shape (0.6 % to 0.5 %)”
- the measurement errors (caused by uncertainties relative to r, t_0, and t_m) are evaluated from Eq. 3 according to the following first-order approximation:

$$\frac{\Delta a}{a} \approx \frac{\Delta r}{r} + \varepsilon \frac{\Delta t_{\mathrm{m}}}{t_{\mathrm{m}}} - (1+\varepsilon)\,\frac{\Delta t_0}{t_0} \tag{4}$$

where ε is defined by

$$\varepsilon = \frac{t_{\mathrm{m}}}{r^2/4a} - \frac{t_{\mathrm{m}}}{t_{\mathrm{m}} - t_0} - 1 \tag{5}$$

and with the final conclusion that “accuracy in a will be limited by the accuracy of both r and t_{m}”.

This analysis by Kuitenberg et al. [18], as a matter of fact, demonstrates the impossibility of a general quantitative theoretical evaluation of the actual error associated with the a determination, for absolute measurements. This impossibility is mainly due to the interferences between model errors and measurement errors (example: what choice for the r value if the wire diameters are d_1 and d_2 or if the two wires are not perfectly parallel to each other during the measurements) and also between $\Delta r/r$ and $\Delta t_{\mathrm{m}}/t_{\mathrm{m}}$ (an increase in wire spacing, for example, would reduce the relative error $\Delta r/r$ but would also decrease the temperature maximum ΔT_{m} and increase t_{m} and, thus, modify the relative error $\Delta t_{\mathrm{m}}/t_{\mathrm{m}}$).

In conclusion, the best way to limit and estimate the uncertainties relative to the a determination is to use comparative measurements with reference liquids (with known thermal-diffusivity values). This procedure minimizes most coupled model and measurement errors associated with the probe configuration; thus, the final error is mainly due to uncertainty in the t_{m} determination.

4.2 Preliminary Results

As mentioned above (Sect. 3), it is necessary to ensure that the heat transfer between the “hot wire” and the liquid is only conductive. For this purpose, a short t_0 value ($t_0 = 10$ ms) of the heat pulse duration (hot wire) has first been chosen (for all tested

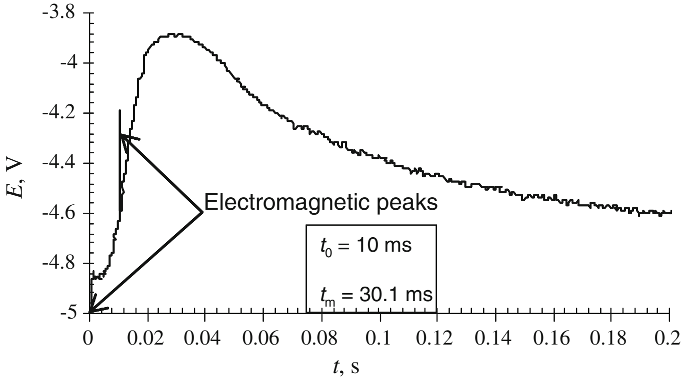


Fig. 4 Typical transient response of the cold wire in water at 20 °C (corresponding thermal-diffusivity value: $a = 1.24 \times 10^{-7}\,\mathrm{m^2 \cdot s^{-1}}$)

liquids). Second, a low enough measurement current intensity ($i \cong 5\,\mathrm{mA}$) (cold wire) has been chosen for all tested liquids to ensure sufficient resolution and negligible heating. Finally, according to the tested liquid, a convenient choice of the current intensity I (typically: 200 mA to 500 mA) in the 25 µm diameter hot wire has to be made to ensure both a good determination of the time t_m obtained with the cold wire and no convective effects around the hot wire. This last condition can be easily verified, for a chosen I value, using the experimental voltage response $E(\ln(t))$ of the hot wire, which must be linear (as for a λ determination according to the classical THW method [8]) for $t \leq t_0$ to ensure the necessary conductive heat transfer regime; for most liquids, this measurement time for λ determination is greater than 1 s [8], which is clearly larger than the above t_0 value of the heat pulse.

Figures 4–6 present, for three different liquids (water, toluene, and Galden®) the transient voltage response $E(t)$ (representative of the temperature responses $T(t)$ of the "cold wire") to a constant heat pulse of duration $t_0 = 10\,\mathrm{ms}$ (supplied by the 25 μm diameter hot wire).

On these figures, it can be observed that each response $E(t)$ of the "cold-wire" shows two electromagnetic peaks corresponding to the beginning and end of the heat pulse, and thus allows control of the time parameter t_0 given by the Keithley 2400 source meter.

The thermal-diffusivity values indicated on the corresponding captions are calculated from Eq. 3 with $r = 110\,\mu\mathrm{m}$ (distance between the axes of the two wires measured with a light microscope) and the corresponding t_m values (obtained by using a polynomial fit of the response $E(t)$ in the neighborhood of the top of the curve, with $\mathrm{d}E/\mathrm{d}t = 0$).

Water and toluene are commonly used as primary standards for reference liquids [2,5,14,25–28], while Galden® is a dielectric perfluorinated liquid with a lower thermal diffusivity. A comparison between measured and mean reference results concerning the thermal-diffusivity values, for the same liquids at 20 °C, is given in Table 1.

Taking into account the previous analysis relative to the uncertainties of the a determination (Sect. 4.1), if the water is chosen as a reference ($a_0 = 1.42 \times 10^{-7}\,\mathrm{m^2 \cdot s^{-1}}$),

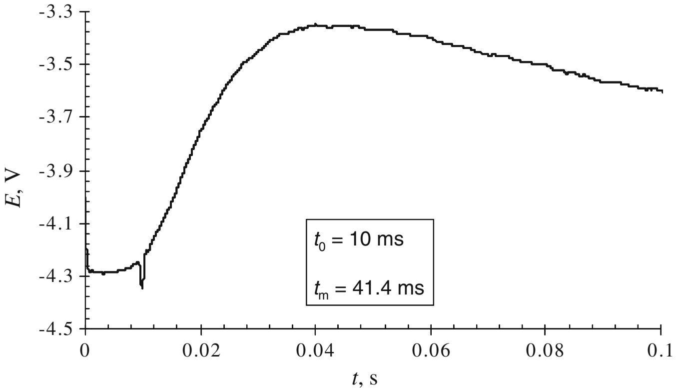


Fig. 5 Typical transient response of the cold wire in toluene at 20 °C (corresponding thermal-diffusivity value: $a = 8.41 \times 10^{-8}\,\mathrm{m^2 \cdot s^{-1}}$)

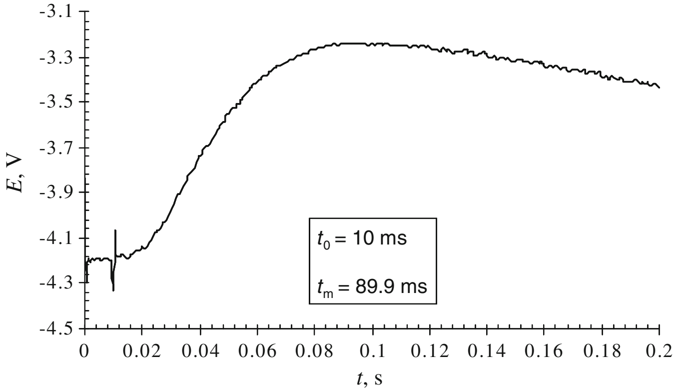


Fig. 6 Typical transient response of the cold wire in Galden® at 20 °C (corresponding thermal-diffusivity value: $a = 3.57 \times 10^{-8}\,\mathrm{m^2 \cdot s^{-1}}$)

Table 1 Reference and measured (absolute) a values

	Reference value ($\mathrm{m^2 \cdot s^{-1}}$)	Measured value ($\mathrm{m^2 \cdot s^{-1}}$) (absolute measurement)	Relative difference (%) (versus reference)
Water	1.42×10^{-7} [26,27]	1.24×10^{-7}	−12.7
Toluene	9.16×10^{-8} [2,28]	8.41×10^{-8}	−8.2
Galden®	3.99×10^{-8} [29]	3.57×10^{-8}	−10.5

an equivalent distance $r^* = 117.8\,\mu\mathrm{m}$ is obtained with Eq. 3 and the corresponding experimental time values: $t_0 = 10\,\mathrm{ms}$ and $t_m = 30.1\,\mathrm{ms}$. Using this r^* as the actual probe characteristic, the thermal-diffusivity values for toluene and Galden® become, respectively, $a = 9.65 \times 10^{-8}\,\mathrm{m^2 \cdot s^{-1}}$ and $a = 4.10 \times 10^{-8}\,\mathrm{m^2 \cdot s^{-1}}$.

Table 2 Reference and measured (relative) a values

	Reference value ($m^2 \cdot s^{-1}$)	Measured value ($m^2 \cdot s^{-1}$) (relative measurement)	Relative difference (%) (versus reference)
Water	1.42×10^{-7} [26,27]	1.35×10^{-7} (using r^{**})	−4.9
Toluene	9.16×10^{-8} [2,28]	9.65×10^{-8} (using r^{*})	+5.1
Galden®	3.99×10^{-8} [29]	4.10×10^{-8} (using r^{*})	+2.7
		3.89×10^{-8} (using r^{**})	−2.5

In the same way, the choice of toluene as a reference liquid ($a_0 = 9.16 \times 10^{-8}\,m^2 \cdot s^{-1}$) gives a new distance $r^{**} = 114.8\,\mu m$ (with a relative difference $<3\,\%$ versus the previous r^* value) and corresponding thermal-diffusivity values $a = 1.35 \times 10^{-7}\,m^2 \cdot s^{-1}$ for water and $a = 3.89 \times 10^{-8}\,m^2 \cdot s^{-1}$ for Galden®.

These results, summarized in Table 2, show that, using water or toluene as references, introduce relative differences between measured and reference thermal-diffusivities less than about 5 %, a maximum difference to be compared with claimed uncertainties of 1.8 % in [2], 4 % in [3], 5 % in [5], (7 to 9) % in [1], 50 % in [4],... for thermal-diffusivity measurements by researchers using other methods and larger liquid samples.

In conclusion, these preliminary experiments demonstrate the feasibility of the proposed method for thermal-diffusivity measurement in liquids, with a low uncertainty for the a determination when a convenient reference liquid is used. As observed in Sect. 4.1, for a given probe (and then for a given distance between the two parallel wires), this uncertainty is mainly dependent on the $\Delta t_m / t_m$ value, which, itself, is slightly dependent on the a value and consequently on the liquid.

4.3 Measurements in a Nitric Acid Solution (90 % Concentration)

Nitric acid is very commonly used in industrial chemistry and for specific (propulsive, explosive, etc.) energetic applications; a good knowledge of its thermal properties such as its thermal diffusivity is useful.

Following the preliminary results presented in Sect. 4.2 for water, toluene, and Galden®, Fig. 7 shows the transient voltage response $E(t)$ given by the same two-wire tantalum probe immersed in a nitric acid solution (90 % concentration in volume) at 20 °C.

Using the corresponding value $t_m = 39.0\,ms$ (determined by a polynomial curve fit) and the equivalent wire distances ($r^* = 117.8\,\mu m$ and $r^{**} = 114.8\,\mu m$) previously calculated with the reference liquids (water and toluene), the following measured thermal-diffusivity values are obtained: $a_1 = 10.35 \times 10^{-8}\,m^2 \cdot s^{-1}$ (water reference) and $a_2 = 9.88 \times 10^{-8}\,m^2 \cdot s^{-1}$ (toluene reference), corresponding to a relative difference of $<5\,\%$.

These values a_1 and a_2 are in good agreement (relative difference $<8\,\%$) with the corresponding value ($a = 10.67 \times 10^{-8}\,m^2 \cdot s^{-1}$) obtained by using the thermal conductivity, the heat capacity, and the density values given (for the same concentra-

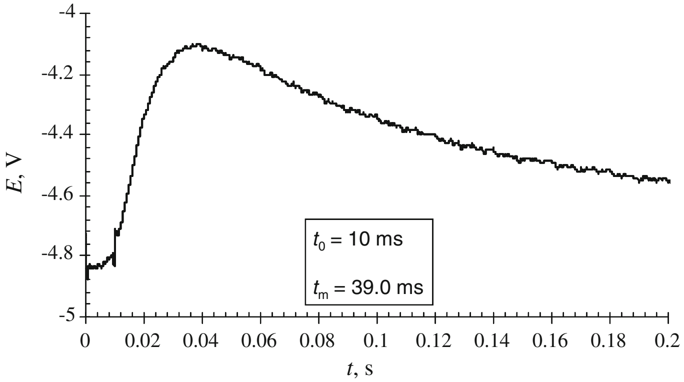


Fig. 7 Typical transient response of the cold wire in a nitric acid solution (90 % concentration) at 20 °C (corresponding thermal-diffusivity value: $a = 9.36 \times 10^{-8}\,\mathrm{m^2 \cdot s^{-1}}$)

tion and temperature conditions) by a calculation software based on the German VDI Waermeatlas [30]. No other thermal-diffusivity data obtained from direct measurements have been found in the literature for such concentrated nitric acid solutions.

5 Conclusion

The proposed two-wire THW method (originally developed and tested for thermal-diffusivity measurements in soils) presented in this paper has proved to be also suitable for measuring the thermal diffusivity of liquids (in particular *special* liquids), with liquid samples smaller than 2 $\mathrm{cm^3}$. Using relative measurements, with reference liquids like water or toluene (with known thermal-diffusivity values), maximum uncertainties of a few percent (depending on the tested liquid and the probe) have been realized; these uncertainties are comparable to the lowest ones obtained with the classical THW technique using larger liquid samples [1–7].

The experimental procedure for thermal-diffusivity measurement only requires determination of the response of the thermal sensor to the short (10 ms) heat pulse supplied by the "hot wire" and time parameter t_m corresponding to the top of this response. Moreover, the same patented [24] apparatus (two-wire probe and cell) can be used for thermal-conductivity measurements by the classical one-wire THW method [8], allowing successive but quasi-simultaneous measurements of λ and a, with complete control of the experimental procedure by LabView.

As demonstrated for concentrated nitric acid solutions, this direct method of thermal-diffusivity measurement, associated with the two-tantalum-wire probe, is also applicable to all liquids compatible with the tantalum probe technology.

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