The use of unsteady method for determination of thermal conductivity of porous construction materials in real conditions

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Abstract. The possibility to determine the thermal conductivity of construction materials outside the laboratory conditions is useful for professional practice mainly for control and inspection activities on real existing buildings. The requirement to determine the thermal conductivity can be useful above all for different thermal insulation materials but for other materials as well, even for compact soils or rocks. This paper describes methods and instrument which can be used for these measurements, as well as the results of measurement of porous building materials. Measurements presented in this paper were carried out by the needle and surface sensor. Four different materials were selected for verification of technical parameters of Isomet 2104. Besides the thermal conductivity there were determined also thermal diffusivity and volume-specific heat capacity of materials. The carried out measurements confirmed the applicability of this device for practical measurements of thermal conductivity in real conditions. For porous materials, there were determined significant differences between the data presented by the manufacturer or in the literature and measured values, in some cases. Differences between the measured values of thermal diffusivity and volume-specific heat capacity of porous materials were always statistically significant. Authors tested different materials including thermal insulation based on agricultural products.

Key words: dynamic method, measurement, sensor, thermal properties.

INTRODUCTION

Thermal conductivity is an important parameter of thermal-technical properties of materials. It is used mainly in civil engineering. Its real value is a function several internal and external variables (density, moisture, temperature, porosity, chemical and mineralogical composition and phase composition, crystalline modification etc.). The attention is paid to the problems of insulation properties in different publications, e.g. (Muizniece et al., 2015; Kocova & Kic, 2016; Pleiksnis et al., 2016; Týbl & Kic, 2016; Valasek et al., 2016).

There are used steady and unsteady methods to determine the thermal conductivity of the material used. Stationary methods are characterized by steady state temperature of measured material sample. These methods determine steady heat flux through the material from the surface of a higher temperature to the surface of a lower temperature. Stationary methods are based on the use of sensors with special shape, e.g. plate method, cylinder method or ball method. These methods are suitable for laboratory conditions.

Unsteady methods are characterized by monitoring of dynamic development of temperatures. They do not require steady thermal state. They are based on monitoring of thermal wave transmission through the measured sample. Unsteady methods include a method of unsteady heat flux and heat pulse method (Hot Wire Method), laser flash method and other methods based on monitoring of heat pulse. Heat energy may be supplied in these methods either at the beginning of the measurement (impulse heat source) or continuously during the whole measurement (continuous heat source), or periodically by constant power (periodic heat source). The methods are applied mainly in laboratory instruments.

The construction of modern laboratory techniques emphasizes rapid measurement, ease manipulation and durability. Therefore some new methods based on dynamic unsteady method are useful and more popular. Examples might be the instrument Shotherm QTM (Showa Denko, Japan) THASYS, THESYS, MTN01 (HUKSEFLUX Thermal Sensors, Netherlands) and Isomet 2114 (Applied Precision, Ltd., Bratislava, Slovakia).

The instrument Isomet 2114 has suitable construction and properties which enable its use also in real buildings (control and revision measurements). Determination of thermal conductivity can be important even for compact soils and rocks. Determination of thermal conductivity of plasters in reconstruction of historical buildings is described in some publications, e.g. Cerny, R. et al. (2006).

The aim measurements described in this article is to verify the use of a dynamic method, applied in design and construction of the instrument Isomet 2104, for the measurement of thermal conductivity of porous thermal insulating materials (polystyrene, Thermo HANF® Premium Plus,). Results obtained from the measurements with the needle and surface sensors were compared.

Porous materials do not transfer the heat only by conduction. A significant part of the heat radiation is transmitted between the individual particles. Non-porous materials (e.g. metals) transfer the heat only by conduction. Problems of heat transfer within the porous heat insulating materials are published e.g. in Koru (2016) and Antonyová et al. (2016).

The instrument Isomet 2104 was also used according to the manufacturer's recommendation for determination of thermal diffusivity and volumetric heat capacity, also termed volume-specific heat capacity. Results of measurements of thermal conductivity of polystyrene published e.g. Tsutsumi (1997). The aim of this research was to compare the results of these measurements with data reported in the literature and confirm the applicability of this measuring instrument for this purpose.

MATERIALS AND METHODS

Isomet 2104 (Applied Precision, Ltd., Bratislava, Slovakia) is a portable instrument controlled by microprocessor, to which the manufacturer supplies exchangeable needle and surface sensors of various ranges. The calibration constants are stored in the sensors memory.

This instrument measures the thermal conductivity of compact and loose materials in a range of $0.015-2 \text{ W m}^{-1} \text{ K}^{-1}$. For the thermal conductivity in the range $0.015-0.050 \text{ W m}^{-1} \text{ K}^{-1}$ is permissible measurement error 5% of reading $+ 0.003 \text{ W m}^{-1} \text{ K}^{-1}$. There is also measured the temperature (measurement error of $\pm 1 \text{ °C}$) and specific heat capacity of materials (the measurement error of 15% of the value of $+ 1,000 \text{ J m}^{-3} \text{ K}^{-1}$). The measured values can be stored in the internal memory of the instrument. The content of memory is accessible via display. Data can be transferred via RS-232 interface to a PC. The instrument can be powered from AC or battery.

To determine the thermal conductivity of the material is used unsteady method. This compared to stationary methods significantly reduces the time of measurement. The device analyses the time dependence of thermal responses to impulses of heat flux supplied to the material. The heat flux creates scattered electric output from the resistor located in the sensor. The sensor has a thermally conductive connection with the analysed material. Temperature resistance is sensed by a semiconductor sensor.

Temperature variation as a function of time is tested in discrete points. The obtained points are interleaved by regression polynomials using the method 'least squares'. The coefficients of the regression polynomials are used to calculate the thermo-physical parameters using analytical formulas.

Air temperatures and relative humidity were measured by sensor FHA 646–21 including temperature sensor NTC type N with operative range from -30 to +100 °C with accuracy ± 0.1 °C, and air humidity by capacitive sensor with operative range from 5 to 98% with accuracy $\pm 2\%$ connected to the measuring instrument ALMEMO 2590-9 (Ahlborn GmbH, Germany).

There was used for the indirect measurement of material moisture the capacitive sensor FH A696–MF with operative range of mineral construction materials from 0 to 20% with accuracy 0.1%. The sensor was connected to the data logger ALMEMO 2690-8 (Ahlborn GmbH, Germany).

Measurements presented in this paper were carried out by needle and surface sensors. Four different materials were selected for verification of technical parameters of Isomet 2104.

The first material is expanded polystyrene foam (density 15 kg m⁻³) removed from an old building during the demolition. It is an inexpensive lightweight material of white colour used in constructions for decades because of its thermal insulating properties. There is a formula according to the CSN EN 13163+A for determination of thermal conductivity λ based on the density of this material presented in the Eq. (1).

$$\lambda = 0.02714 + 5.1743 \cdot 10^{-5} \cdot \rho + \frac{0.173606}{\rho} \tag{1}$$

where: λ – thermal conductivity, W m⁻¹ K⁻¹; ρ – material density, kg m⁻³.

Hejhálek (2012) gives the Eq. (2) describing the dependence of a thermal conductivity on temperature for expanded polystyrene (density of 15 kg m^{-3}).

$$\lambda = 0.0354 + 2.00 \cdot 10^{-4} \cdot t + 4.77 \cdot 10^{-7} \cdot t^2 \tag{2}$$

where: λ – thermal conductivity, W m⁻¹ K⁻¹; *t* – temperature of material, °C.

The second material is extruded polystyrene foam of blue colour, manufactured by the Dow France S.A.S. (France) under the trademark AGMA XL-X. The product is designed for heat insulation of agricultural buildings, especially for ceilings and sloping side walls. The material has a homogeneous cellular structure preventing water absorption; it is a barrier to water vapour. Manufacturer declares possibility to use the low pressure water for washing and possibility to use disinfectants.

The third material is Thermo HANF® Premium Plus, which is a natural insulation material manufactured from hemp by company Thermo Natur GmbH & Co. KG (Nördlingen, Germany). As a binder of hemp particles is used a corn-starch. This material can be used as good quality insulation.

Eight to ten measurements were carried out for each experimental material. Measurements were carried out under laboratory conditions at 20-26 °C. Measurements simulating operating non-laboratory conditions were carried out in non-heated hall at 14–15 °C. If the dimensions allow, the sensor was placed at various locations of material sample. In case of material Thermo HANF® Premium Plus were available two samples.

The fourth material is a paper with a low volume of voids.

RESULTS AND DISCUSSION

The sample size and density of materials are given in Table 1.

Table 1. Dimension and density of materials according to the catalogue values given by producers and measured values

Material	Dimensions of material samples		Density of material, kg m ⁻³		
	Height, m	Width, m	Length, m	Catalogue	Measured
Expanded polystyrene	0.060	0.367	0.497	10–60	15
AGMATE XL-X	0.047	0.147	0.148	38	34.6
TERMO HANF Premium Plus	0.080	0.110	0.110	30–42	39.9
Paper	0.296	0.210	0.040	700-1,300	771

The Table 2 shows the values of thermal conductivity λ_m of polystyrene calculated according to the Eq. 1 (column A), thermal diffusivity *a*, volume-specific heat capacity *c*, measured by the needle probe (column B) and by the surface probe (column C). The Table 2 also presents material temperature t_m and moisture w_m and the temperature t_e and relative humidity φ_e of the ambient air determined together with measurement of thermal conductivity. The table also contains statistical indicators of measured values. The data are the mean values \pm SD. Different letters (a, b, c) in the superscript are the sign of high significant differences (ANOVA; Tukey HSD Test; $p \le 0.05$).

Table 2. Expanded polystyrene (density of material 15 kg m⁻³)

Parameter	А	В	С
$\lambda_m \pm SD$, W m ⁻¹ K ⁻¹	0.039 ^b	$0.0279 \pm 0.00035~^{\rm a}$	$0.0402 \pm 0.00051^{\ b}$
$c \pm SD$, J m ⁻³ K ⁻¹	1.905 10 ⁴ °	$1.4010^6\pm0.0^{a}$	$6.63 10^4 \pm 0.37 10^{4 \text{b}}$
$a \pm SD$, m ² s ⁻¹	2.05 10 ^{-6 c}	$2.00\ 10^{-8}\pm 6.62^{-24}\ a$	$6.08\ 10^{-7}\pm0.32^{-7}^{\mathrm{b}}$
$t_m \pm SD$, °C	$20.00\pm3.23~^{a}$	21.93 ± 3.74^{a}	$23.76\pm0.84^{\text{ a}}$
$w_m \pm SD, \%$		2.71 ± 0.25 a	2.71 ± 0.25 a
$t_e \pm SD$, °C		$21.44\pm3.67^{\text{ a}}$	$22.13\pm1.14^{\mathrm{a}}$
$\varphi_e \pm SD, \%$		39.31 ± 2.41 a	$28.40 \pm 1.74^{\; b}$

Fig. 1 shows values of thermal conductivity calculated according to the Eq. 2 (data A), measured using a needle probe (data B) and by the surface probe (C data).



Figure 1. Dependence of thermal conductivity λ_m of polystyrene on the material temperature t_e .

Table 3 presents values of thermal conductivity λ_m of material AGMATE XL-X declared by the manufacturer (column A), thermal diffusivity *a*, volume-specific heat capacity *c*, measured using a needle probe (column B) and by the surface probe (column C). The Table 3 also presents material temperature t_m and moisture w_m and the temperature t_e and relative humidity φ_e of the ambient air determined together with measurement of thermal conductivity. The table also contains statistical indicators of measured values. The data are the mean values \pm SD. Different letters (a, b, c) in the superscript are the sign of high significant differences (ANOVA; Tukey HSD Test; $p \leq 0.05$).

Table 3. AGMATE XL-X (density of material 34.6 kg m⁻³)

Parameter	А	В	С
$\lambda_m \pm SD$, W m ⁻¹ K ⁻¹	0.029 ^{a, b}	$0.0274 \pm 0.00026~^{a}$	$0.0365\pm 0.0005~^{\rm b}$
$c \pm SD$, J m ⁻³ K ⁻¹	7.13 10 ⁴ °	$1.4010^6\pm0.0^{\mathrm{a}}$	$9.54 10^4 \pm 0.41 10^{4 b}$
$a \pm SD$, m ² s ⁻¹	4.07 10 ^{-7 c}	$1.97 10^{\text{-8}} \pm 4.08^{\text{-10 a}}$	$3.83\ 10^{-7}\pm 1.22^{-8}^{\mathrm{b}}$
$t_m \pm SD$, °C		$20.63\pm4.86^{\text{ a}}$	$23.55\pm1.14^{\mathrm{a}}$
$w_m \pm SD, \%$		undetectable	undetectable
$t_e \pm SD$, °C		$20.46\pm5.16~^{\rm a}$	20.70 ± 2.84 a
$\varphi_e \pm SD, \%$		36.37 ± 42.91 a	27.70 ± 2.14^{b}

Table 4 presents values of thermal conductivity λ_m of material Thermo HANF® Premium Plus declared by the manufacturer (column A), thermal diffusivity *a*, volumespecific heat capacity *c*, measured using a needle probe (column B1 and B2). Columns B1 and B2 are values of two different samples. Surface probe with its weight caused a deformation of the material; therefore the measurement was not carried out. The Table 4 also presents material temperature t_m and moisture w_m and the temperature t_e and relative humidity φ_e of the ambient air determined together with measurement of thermal conductivity. The table also contains statistical indicators of measured values. The data are the mean values \pm SD. Different letters (a, b) in the superscript are the sign of high significant differences (ANOVA; Tukey HSD Test; $p \le 0.05$).

Parameter	А	B1	B2
$\lambda_m \pm SD$, W m ⁻¹ K ⁻¹	0.040 ^a	$0.041\pm0.001^{\text{a}}$	0.041 ± 0.001^{a}
$c \pm SD$, J m ⁻³ K ⁻¹	6.4 10 ^{4 b}	$1.410^6\pm0.0^{\mathrm{a}}$	$1.410^6\pm 0.0^{a}$
$a \pm SD$, m ² s ⁻¹	6.25 10 ^{-7 b}	$3.0\ 10^{-8}\pm 0.0\ ^{a}$	$2.98 10^{-8} \pm 4.33^{-10} a$
$t_m \pm SD, ^{\circ}C$		20.63 ± 4.86 a	23.55 ± 1.14 a
$w_m \pm SD, \%$		$2.12\pm0.268^{\mathtt{a}}$	2.22 ± 0.308^{a}
$t_e \pm SD$, °C		21.6 ± 4.85 ^a	21.7 ± 5.24 ^a
$\varphi_e \pm SD, \%$		$31.6\pm0.18~^{a}$	31.6 ± 0.23 a

Table 4. Thermo HANF® Premium Plus (density of material 39.9 kg m⁻³)

Table 5 shows values of thermal conductivity λ_m , thermal diffusivity *a*, volumespecific heat capacity *c* of the paper material with a density of 771 kg m⁻³ according to Ražnjević (1984) (column A) and the values measured using the surface probe (column B). There are presented in the Table 5 also material temperature t_m and moisture w_m , and the temperature t_e and relative humidity φ_e of the ambient air determined in the measurement of thermal conductivity. The tables also contain statistical indicators of measured values. The data are the mean values \pm SD. Different letters (a, b) in the superscript are the sign of high significant differences (ANOVA; Tukey HSD Test; $p \le 0.05$).

 Table 5. Paper (density of material 771 kg m⁻³)

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Parameter	Α	В
$\lambda_m \pm SD$, W m ⁻¹ K ⁻¹	0.151 ^a	0.160 ± 0.0013 ^b
$c \pm SD$, J m ⁻³ K ⁻¹	1.03 10 ^{6 a}	$7.4\ 10^5\pm 0.09\ 10^{5b}$
$a \pm SD$, m ² s ⁻¹	1.46 10 ^{-7 a}	$2.15 10^{\text{-7}} \pm 0.01 10^{\text{-7}} ^{\text{b}}$
$t_m \pm SD$, °C		24.27 ± 1.27
$w_m \pm SD$, %		9.53 ± 0.23
$t_e \pm SD$, °C		23.8 ± 1.55
$\varphi_e \pm SD, \%$		31.2 ± 1.41

By measurement of thermal conductivity λ_m of polystyrene were confirmed statistically non-significant differences between the values calculated according to the Eq. 1 and the values measured by surface probe. The values measured by needle probe are statistically different by from the values calculated according to the Eq. 1. Results of the measurement of volume-specific heat capacity *c* and thermal diffusivity *a*, determined by the needle and surface probes are different from each other, as well as they are different from the results presented in the literature, e.g. Yucel (2013). Differences of measurements of conditions were non-statistically significant; there was different only the relative humidity of air (Table 2).

The values of thermal conductivity λ_m obtained by measurement using surface probe are statistically consistent with the values calculated according to the Eq. 2 (see Fig. 1). The values of thermal conductivity λ_m measured by needle probe are statistically different by from the values calculated according to the Eq. 2. However, the growth of thermal conductivity λ_m of the material with increasing temperature t_m confirmed both measurements (see Fig. 1). The growth of thermal conductivity with increased temperature of a material corresponds with the trends reported in the literature, e.g. EPS CR Association or TZBinfo.

The values of thermal conductivity λ_m of material AGMATE XL-X obtained by measurement using needle probe are statistically consistent with the values declared by the producer. The values of thermal conductivity λ_m measured by the surface probe are statistically different from the value indicated by manufacturer.

Results of the measurement of volume-specific heat capacity c and thermal diffusivity a, determined by the needle and surface probes are different from each other, as well as they are different from the values presented by the producer. Operating conditions of the measurements were statistically consistent (Table 3). The minimal growth of thermal conductivity with temperature in the case of the material AGMATE XL-X confirms resistance of extruded polystyrene to the temperature fluctuations.

The values of thermal conductivity λ_m of both samples B1 and B2 of material Thermo HANF® Premium Plus obtained by measurement using the needle probe are statistically consistent with the values declared by the producer. The surface probe cannot be used for this material because it causes deformation of material samples. Results of the measurement of volume-specific heat capacity *c* and thermal diffusivity *a*, determined by the needle and surface probes are different are different from the values presented by the producer. Operating conditions of the measurements were statistically consistent (Table 4).

Due to unsatisfactory results in the detection of volume-specific heat capacity c and thermal diffusivity a (of previous porous materials) determined by the instrument Isomet 2104, there were carried out the control measurements of materials with a low volume of voids. The suitable material is a paper with density 771 kg m⁻³.

The statistically significant consistence of thermal conductivity λ_m of paper measured by the surface probe with the values presented by Ražnjević (1984) was not confirmed. Results of the measurement of the volume-specific heat capacity *c* and thermal diffusivity *a* also cannot be considered as statistically consistent with the literature data. However, it can be stated that from all measured values *c* and *a* presented in Tables 2–5, the determined values of paper are approaching the values reported in the literature.

CONCLUSIONS

The measurement confirmed the suitability of the tested instrument Isomet 2104 based on the non-stationary method of measurement for the rapid determination of thermal conductivity λ_m of porous materials, but there were also found statistical differences between the measured values and values reported in the literature or declared by the manufacturers.

The measurements did not confirmed the suitability of the instrument Isomet 2104 to measure the volume-specific heat capacity c and thermal diffusivity a. The results of measurement are not statistically consistent with the values presented in the literature or declared by the manufacturer of tested materials.

There is not confirmed the consistence of results obtained by the measurements with needle and surface probes. The suitability of the instrument Isomet 2104 for the measurement of the volume-specific heat capacity c and thermal diffusivity a of homogeneous materials should be tested in the future.

The instrument is able to register the changes of thermal conductivity with temperature changes of materials. This feature can be suitably used in the preparation of laboratory exercises for university students of engineering study.

Portability and short duration of the tests make it ideal for the use outside the laboratory. It will be useful in real conditions for inspection and audit activities on already completed buildings.

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