METROLOGICAL EVALUATION OF CHARACTERIZATION METHODS APPLIED TO NUCLEAR FUELS

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Abstract. *In manufacturing the nuclear fuel, characterizations are performed in order to assure the minimization of harmful effects. The uranium dioxide is the most used substance as nuclear reactor fuel because of many advantages, such as: high stability even when it is in contact with water at high temperatures, high fusion point, and high capacity to retain fission products. Seveal methods are used for characterization of nuclear fuels, such as thermogravimetric analysis for the ratio O / U, penetration-immersion method, helium pycnometer and mercury porosimetry for the density and porosity, BET method for the specific surface, chemical analyses for relevant impurities, and the laser flash method for thermophysical properties. Specific tools are needed to control the diameter and the sphericity of the microspheres and the properties of the coating layers (thickness, density, and degree of anisotropy). Other methods can also give information, such as scanning and transmission electron microscopy, X-ray diffraction, microanalysis, and mass spectroscopy of secondary ions for chemical analysis. The accuracy of measurement and level of uncertainty of the resulting data are important. This work describes a general metrological characterization of some techniques applied to the characterization of nuclear fuel. Sources of measurement uncertainty were analyzed. The purpose is to summarize selected properties of UO_2 that have been studied by CDTN in a program of fuel development for Pressurized Water Reactors (PWR). The selected properties are crucial for thermalhydraulic codes to study basic design accidents. The thermal characterization (thermal diffusivity and thermal conductivity) and the penetration-immersion method (density and open porosity) of UO_2 samples were focused. The thermal characterization of UO_2 samples was determined by the laser flash method between room temperature and 448 K. The adaptive Monte Carlo Method was used to obtain the endpoints of the probabilistically symmetric 95 % coverage interval for estimate the thermophysical l properties. The results obtained were compared to the reference data. The maximum deviation of thermophysical properties is 11% from room temperature to 448 K.*

Keywords*: Characterization, nuclear fuel, thermophysical properties, uranium dioxide.*

1. INTRODUCTION

In manufacturing the nuclear fuel, the characterizations are performed in order to ensure that the fuel is according to its technical specifications in order to minimize the harmful effects of burning and temperature on the fuel. The specifications are established according to the understanding of the fuel behavior. For example, the measurement of open porosity of fuel pellets is necessary because this property affect the release of volatile fission products. It is fundamental the knowledge of several thermophysical properties such as density, thermal diffusivity and thermal

conductivity of the nuclear fuel. The evaluation of nuclear reactor performance under normal operation or accident conditions using accurate and reliable properties data is critical in the design and analysis of current and future reactors. For such analyses, accurate data are necessary. All measurements are subject to uncertainty and a measured value is meaningless without a quantitative statement of its quality in form of an uncertainty. The difficulties associated with accurate measurements are often due to combined effect of a variety of factors. This fact can be revealed by the differences among results discussed in several reports and workshops and represents a challenge to all thermophysicists, as these materials have an enormous economical importance. The objective of uncertainty evaluation is to quantify the extent and nature of the knowledge of the output quantity of the measurement model. Knowledge of the model input quantities is encoded by the assignment of probability density functions (PDFs) to those quantities. A main requirement is to ascribe to the output quantity a so-called coverage interval that contains a specified proportion, e.g., 95 %, of the distribution of values that could reasonably be attributed to that quantity. Although extensive research (Fink, 2000) has been done on nuclear material properties, the uncertainties have been determined from experimental uncertainties, scatter in the data, deviations of the data from recommended equations, differences between available equations in the literature, and estimates of errors arising from extrapolation beyond measurements. However, Fink (2000) does not report detailed information about the sources of uncertainties and the coverage interval. Therefore a revision on the experimental procedures and data, including recommendations of other experts will contribute to the nuclear fuels thermophysical properties databases, with special emphasis on the estimation of measurement uncertainties. This work describes a general metrological evaluation of UO_2 pellets characterization methods. The selected proprieties are density, thermal diffusivity and thermal conductivity.

To determine the density and open porosity of the sintered pellets, it was used the xylol penetration-immersion method developed by the Kraftwerk Union research center UO_2 laboratory in Erlangen / Germany which was absorbed, transferred and implemented in the CDTN's nuclear fuel laboratories (Ferreira and Lopes, 2007). For the pellets thermal diffusivity measurement were utilized the flash laser method (Parker *et al.*, 1961, Ferreira *et a.l., 2002.*). This method has been considered by several Metrology National Institutes and other organizations as a standard method for the measurement of thermophysical properties of solids (Couto *et al*., 2003). Due its experimental obstacles the original flash method has been reviewed and some alternative and effective approaches are proposed (Grossi, 2008). A stochastic modeling has been developed and validated by Standard Samples. Inverse Heat Conduction Problems (IHCPs) solved by Finite Volumes technique were applied to the measurement process with real initial and boundary conditions (Grossi, 2008, Grossi, 2009). The GUM uncertainty framework (Guide to the Expression of Uncertainty) (JCGM, 2008a) was used to obtain the associated standard uncertainty with an estimate of the input quantities. The adaptive MCM-Monte Carlo Method (JCGM, 2008b) was used to obtain the endpoints of the probabilistically symmetric 95 % coverage interval for estimate of the output quantities and its uncertainties. Monte Carlo Method is a sampling technique that provides an implementation of the propagation of distributions: the process is undertaken numerically rather than analytically. The data of thermal diffusivity and thermal conductivity of UO_2 samples between room temperature and 448 K are presented and discussed.

2. EXPERIMENTAL

2.1. Preparation and pellets density measurements

It was examined in particular 3 samples of UO_2 pellets. Two pellets (2737 and 2738 samples) were taken from a same batch of UO_2 powder and manufactured by uniaxial pressing process under compactation pressures of 400 MPa and 500 MPa,. The samples were sintered in H_2 atmosphere at 1700 ° C for three hours. A third pellet (2839) was produced pressing kernels manufactured by the sol-gel process developed by Nukem/Germany that was absorbed, transferred and implemented at the CDTN's nuclear fuel laboratories (Ferreira *et al*, 2009). By this process, developed to the manufacture of nuclear fuel for High Temperature Gas Cooled Reactors (HTGCR), a uranium nitrate solution is transformed into spherical droplets that become hard in reaction with ammonium gas and are collected in an ammonium hydroxide solution. In the subsequent steps, the kernels are washed, dried, calcined, sintered and coated. In the present case the kernels were not sinterized, but were uniaxial pressed in pellets for PWR in the calcined state. The drying step was performed at 160 ^oC for 16 h and the calcination, at 800 ^oC for 3 h, followed by reduction at 650 ^oC for 4 h in hydrogen atmosphere, and by passivation under CO_2 atmosphere during the oven cooling down.

To measure the density and open porosity of the sintered pellets, it was used the xylol penetration-immersion method developed by the Kraftwerk Union research center UO_2 laboratory in Erlangen / Germany which was absorbed, transferred and implemented in the CDTN's nuclear fuel laboratories (Ferreira *et al*, 2007). The method permits to obtain the open porosity in absolute (% V – percentage of the pellet volume) and relative terms (% P – percentage of the total porosity that is open). The samples are initially placed in a tray and oven-dried by 120 °C for 2 h. After cooling in a desiccator under vacuum the samples are weighed to determine its masses with a calibrated analytical balance (resolution: 10^{-4}g). The tray with the samples is placed in a recipient in the interior of the desiccator where a vacuum in the range of (10^{-1} to 10^{-2})Torr is established and maintained for 2 h in order to extract the air from the samples open porosity. After this time, the recipient is flooded with xylol from a recipient coupled with the desiccator in order to allow xylol to penetrate in the samples open porosity. The recipient with the impregnated samples is removed from the

desiccator and 1 h is waited for temperature equalization with the environment and so that the xylol penetrates completely in the open porosity. The measurement of the sample in the analytical balance, gives by the difference between the impregnated sample mass and the mass measured after the drying, the xylol mass contained in the open porosity. The xylol excess present in the surface of sample must be removed with an absorbent paper before weighing. Otherwise it will be obtained a bigger value than the true value of open porosity. After knowing the xylol density, it is calculated the xylol volume corresponding to this mass, which is equal to the sample open porosity volume. After this measurement, the sample is measured immersed in xylol. It is obtained then, by the difference between the sample mass and the mass under buoyancy, the mass of the dislocated xylol volume. This volume is equal to sum of the material volume without pores and the closed porosity volume. The xylol density is determined before the measurement of the first sample, by the determination of the buoyancy in a steel sphere, whose mass and diameter are known with accuracy by measurements with calibrated micrometer (resolution: 10^{-3} mm) and calibrated analytical balance (resolution: 10^{-4} g).

2.2. Thermal characterization by Flash Method

The flash method for measuring thermal diffusivity has been increasingly used since its introduction in 1961, by Parker *et al.* (1961). The laser flash apparatus (Fig. 1) is regularly used for measurements of the thermal diffusivity of solids at the Nuclear Technology Development Center - CDTN. It consists of a CO_2 laser (of 100 W total power) working at 10.6 μm wave length, an infrared thermometer, a vacuum pump and measuring control and data processing systems. An infrared thermometer measures the transient temperature, and the thermal radiance signal is digitized using a 16 bits A/D converter of an NI PCI-6052 data acquisition device. A LabView programming is used to acquire data. The sample (8 mm in diameter and about 2.5 mm in thickness) is placed in a vacuum furnace and isothermally heated. In order to avoid any transmission of the laser beam through the samples, a thin carbon layer was applied over the front surfaces. The rear surfaces of the samples were also coated to ensure an uniform emissivity. The system allows the irradiation of the front face of the sample obtaining a register of the rear face transient temperature of the sample. The apparatus is specially designed to operate under conditions imposed by the requirement to measure the thermal diffusivity of nuclear fuels. Measurements were carried out with constant laser pulse energies, which produced rear-surface temperature excursions from 1.9 K to 2 K.

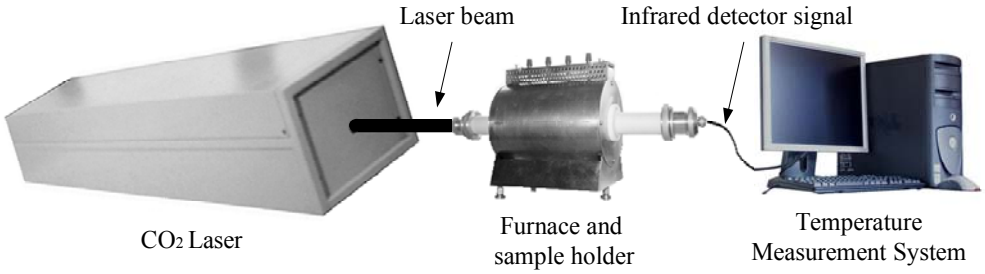

Figure1. Experimental Apparatus Thermophysical Properties Measurement Laboratory.

2.2.1. Mathematical model

When the material is uniformly stimulated on its whole front face, heat transfer within the material can be considered one-directional. The model is given by the solution of the one-dimensional heat transfer equation considering the real initial and boundary conditions of LMPT experimental apparatus, where a discoid specimen intensely heated on its frontal face by a short laser pulse (Migliorini *et al.*, 2009b). The computer algorithm was developed and implemented using the Compaq Visual FORTRAN platform. The direct numerical solutions to the thermal diffusion process in the sample are obtained by the Finite Volume Method through CONDUCT.for subroutine (Fig. 2). In the FLASH.for subroutine are implemented the Monte Carlo Method (MCM) and the solutions for Inverse Heat Conduction Problems. The ARRANGE.for subroutine establishes a range of values expected for all inputs parameters of the model based on its initial previous evaluation. The ADAPT.for subroutine implements all initial and boundary conditions. The generation of random draws from Uniform, Gaussian or Triangular distributions is done in the subroutine NORMAL.for. This methodology reduces the degree of model simplification, providing results with improved physical meaning and uncertainty reduction. Inverse Heat Conduction Problem solutions were obtained by application of a nonlinear programming algorithm, based on the Coordinate Descent Method and on the Golden Section Search Method. Thus, an objective function characterized by the deviation between the experimental temperature transients and the numerical solutions at sample rear-face is minimized. An optimal inverse solution for the problem is obtained. By using the optimal values evaluated for the input parameters of the model, the following outputs parameters and uncertainties are obtained as results: thermal diffusivity and thermal conductivity. In such way it is possible to

characterize the thermophysical properties of the analyzed material. The mathematical model was validated by using Inconel 600, BSC Pure Iron and Pyroceram 9606 standard samples.

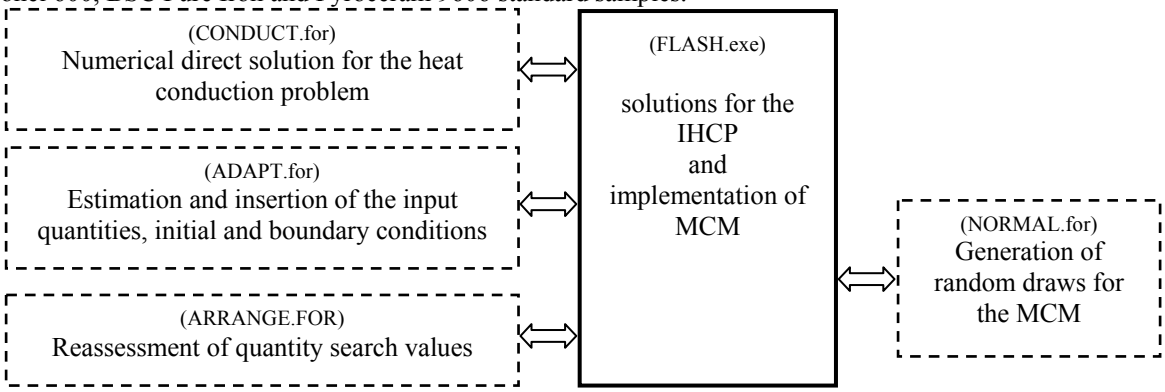

Figure. 2 Schematic diagram of the stochastic model structure: main software and subroutines.

2.3. Uncertainty of measurement

Uncertainty evaluation requires a careful analysis of the measurement process. The use of the Ishikawa method allows identification of all possible sources of uncertainty. The primary input quantities of the density are xylol density, mass and diameter of sphere. So, its uncertainty is function of these input quantities. Its variations with temperature can be obtained from the thermal expansion data. The sources of uncertainties in the measurement of thermal diffusivity are associated with the specimen itself, temperature measurement, time measurement, non-uniform heating of the sample and heat losses (Migliorini *et al.*, 2009a; Camarano, 2010). The thermal conductivity is function of thermal diffusivity, specific heat and density values. Then, its uncertainty is function of these inputs quantities.

2.3.1. Uncertainties associated with the density

Density value is affected by several factors in the measurement process, including the analytical balance, environmental conditions, procedure used to obtain the measurement results, operator's technique and skill of making weightings, stability of weighing equipment, uncertainties of the standard used, ability to determine the density of the air being displaced, ability to determine the density or volume of the standard and item being weighed, and buoyancy effect due to displacement of air on the volume of the standard and the volume of the item being weighed. Variation in the measure due to repeatability of the total measurement process is described using the standard deviation equation. This analysis assumes a normal distribution curve.

The uncertainty in the sintered density ρ results from the combination of the uncertainty of the xylol density (ρ_{xylol}) and uncertainty of difference (ΔM) between the impregnated mass specimen (M_{impr}) and the mass measured (M) after the drying expressed by

$$u^2(\rho_{\text{xylol}}) = \left(\frac{\partial \rho}{\partial \rho_{\text{l}}}\right)^2 \cdot u^2(\rho_{\text{l}}) + \left(\frac{\partial \rho}{\partial x}\right)^2 \cdot u^2(\Delta M). \tag{1}$$

The uncertainty of the xylol density (ρ_{xylol}) results from the combination of the uncertainty of diameter of the sphere and the uncertainty of difference between mass of the sphere and the mass of the sphere under buoyancy. The uncertainty of mass (M) or the impregnated mass specimen (M_{impr}) results from the uncertainty due to repeatability of measurements $u_{\text{R}}(M)$, calibration $u_{\text{c}}(M)$, resolution $u_{\text{r}}(M)$, the drift of the analytical balance, $u_{\text{D}}(M)$ temperature effects and air buoyancy effects $u_{\text{ep}}(M)$, linearity and eccentricity of analytical balance $u_{\text{L}}(M)$, given by

$$u^2(M) = u_{\text{R}}^2(M) + u_{\text{c}}^2(M) + u_{\text{r}}^2(M) + u_{\text{D}}^2(M) + u_{\text{ep}}^2(M) + u_{\text{L}}^2(M). \tag{2}$$

The uncertainty of diameter results from the combination of the uncertainty due to repeatability of measurements $u_{\text{r}}(d)$, calibration $u(d)$, resolution u_{r}, drift of the micrometer $u(C_{\text{dm}})$, flatness and parallelism $u(C_{\text{pl}})$:

$$u^2(d) = u_{\text{r}}^2(d) + u^2(d) + u_{\text{r}}^2 + u^2(C_{\text{dm}}) + (C_{\text{pl}}). \tag{3}$$

The uncertainty on ΔM is function of the uncertainty of $M_{\text{impr.}}$ and M measurements. Considering these input quantities correlated, the expression given for the combined standard uncertainty of ΔM is*:*

$$u(\Delta M) = u(M_{\text{impr.}}) + u(M). \tag{4}$$

The uncertainty of density was estimated in 1 % for the entire temperature range.

2.3.2. Uncertainties associated with the thermal diffusivity

The sources of uncertainty concerning to the sample are due to its geometrical quality and its chemical and optical properties. The thickness of the sample at a temperature (L) is calculated from the thickness measured at room temperature (L_0) corrected by a term to taking into account the expansion of the sample (Δ_L). Mathematical expression of the thickness of the sample is written as

$$L = L_0 + \Delta_{\text{L}}. \tag{5}$$

The uncertainty in the thickness results from the combination of the uncertainty of measurement thickness (L_0) and the uncertainty in the correction due to expansion of the sample (Δ_L):

$$u_c^2(L) = u^2(L_0) + u^2(\Delta_{\text{L}}) + 2u(L_0, \Delta L). \tag{6}$$

The sample thickness is measured with certified micrometer. The uncertainty of thickness results from the uncertainty due to repeatability of measurements, resolution, calibration, flatness, parallelism, drift of micrometer and the correction of thermal expansion. The uncertainty is calculated based on the uncertainty of the thermal expansion of solid UO_2 for a typical expansion coefficient of 9.78 x 10^{-6} K^{-1} (Fink, 2000) and a 3 K uncertainty of temperature due to temperature gradients. Uncertainty on linear expansion coefficient from room temperature to 448 K is assumed to be 0.05 x 10^{-6} K^{-1}.The uncertainty in thermal diffusivity resulting from thickness measurement is estimated to be less than 0.1 %.

The uncertainty factors related to the sample temperature correspond to the experimental conditions especially induced by the furnace temperature, the nature of the atmosphere and all other sources associated with the radiation thermometer. The uncertainty of radiation thermometer results from the uncertainty due to resolution, calibration, drift, time constant of thermometer and other factors as effective emissivity of the sample, stability and the homogeneity of the furnace temperature. Drift in radiation thermometers between calibrations arises from changes in the optical components, in the radiation detector and in the signal processing system. Non-linearity is caused by the non-ideal performance of the detector and electronics. In our measurement s the opposite face temperature increase is always kept below 3 ºC. The non-linearity effect of the IR thermometer is assumed to be negligibly for small temperature changes (smaller than 10 ºC). The sample is coated on both sides with carbon film to improve and keep controlled the sample emissivity and absortivity. Based on these characteristics, it was assumed that the uncertainty in the temperature $u(T)$ results from the combination of the uncertainties due to repeatability of measurements $u_r(T)$, the calibration $u(C)$, the resolution $u(C_R)$, the drift of thermometer $u(C_d)$ and the emissivity $u(C_\varepsilon)$:

$$u^2(T) = u_{\text{r}}^2 + u^2(C) + u^2(C_{\text{R}}) + u^2(C_{\text{d}}) + u^2(C_{\varepsilon}). \tag{7}$$

The uncertainty in temperature sample was estimated in 0.2 %.

The uncertainty on the time measurement results from the combination of the uncertainties due to measurement instruments and data acquisition board. The manufacturer states the following characteristics of the used data acquisition board:

- signal resolution of 16 bit (1 in 65 536 or 0.002 %);
- timing resolution 50 ns and
- timing accuracy 0.1μs.

Based on these characteristics, the uncertainty due to digital data acquisition board was evaluated in 0.01 %. The sampling frequency was set to 1 kHz. The time origin measurement error was evaluated in 1 ms. The error assign to the sample frequency is lower than 0.2 %. Therefore, the uncertainty in thermal diffusivity resulting from time scale was evaluated in 1.5 %.

The sample heating uniformity is directly related to the uniformity of the laser beam. The uniformity of a laser may change from shot to shot and it is also dependent on the energy level of the laser beam. A 3 % uncertainty due to the effect of non-uniform heating was assumed in this uncertainty budget (Cezairliyan, 1994).

The contribution of the heat losses is expressed by an overall heat transfer coefficient. Computational simulations were used to estimate the influence of the heat losses on the thermal diffusivity. In all cases, the influence of heat losses on the thermal diffusivity accuracy is estimated in 2 % (Grossi, 2008).

In order to check the accuracy of the thermal diffusivity measurement system we used an inconel 600 standard (Blumm and Lindemann, 2009). The maximum deviation of all mean values determined by the LMPT (Laboratório de Medição de Propriedades Termofísicas) from the certified value is of 2 %, which is within the mutual uncertainty of the values.

3. RESULTS

Table 1 summarized the parameters used to fabricate the pellets and the characterizations results. The uranium dioxide theoretical density (TD) was considered to be 10.96 $g \cdot cm^{-3}$.

Table 1. Manufacturing parameter and percent of theoretical density

Sample	Compactation Pressure Mpa	Green Density $g \cdot cm^{-3}$	Sintered Density $g \cdot cm^{-3}$	Percent of Theoretical Density %	Open porosity	
					% *V*	% *P*
2839	300	-	9.90	90.3	6.80	70.6
2737	400	5.89	10.22	93.2	0.95	14.0
2738	500	6.09	10.40	95.0	0.75	14.7

Table 2 to 4 shows the thermal diffusivity and thermal conductivity of UO_2 as function of temperature for all samples. All thermal diffusivity values presented here result from the average of five successive measurements carried out on repeatability conditions and determined by original model of Parker *et al.* (1961). The results show a similar trend as experimental data reported by Lucuta *et al.* (1995). The maximum difference from the values proposed by Lucuta *et al*. (1995) is 11 %. The thermal conductivity was determined by the model proposed by Grossi (2008). The thermal conductivity of the pellet 2839 is lower than the other because its lower density. The values are different from the values presented by Fink (2000) but the uncertainty recommended for thermal conductivity is equal to 10 %. So, in this case we need further experiments. By comparison, probabilistically and symmetrically, a 95% coverage interval for thermal diffusivity and thermal conductivity for all samples, obtained by the Monte Carlo Method, is on the order of 4.6 % to 13.7 %. The application of MCM for M = 50 trials had been found suitable in practice because of computational time. Although the use of a small value of M is inevitably less reliable than that of a large value, we assigned to characterize the knowledge for outputs quantities a Gaussian PDF (probability density function) because of the small value used for M. So, this uncertainty can be considered to be for a worst-case test situation.

Table 2. Thermal physical properties of Uranium dioxide sample P2839

Temperature / K	Thermal diffusivity / $mm^2 \cdot s^{-1}$		Thermal conductivity / $W \cdot m^{-1} \cdot K^{-1}$	
	Mean Value	95 % Coverage Interval	Mean Value	95 % Coverage Interval
298	2.44	0.18	6.43	0.30

Table 3. Thermal physical properties of Uranium dioxide sample 2737

Temperature / K	Thermal diffusivity / $mm^2 \cdot s^{-1}$		Thermal conductivity / $W \cdot m^{-1} \cdot K^{-1}$	
	Mean Value	95 % Coverage Interval	Mean Value	95 % Coverage Interval
304	3.01	0.22	6.80	0.60
328	2.90	0.32	6.09	0.60
377	2.69	0.24	5.70	0.50
448	2.38	0.22	5.50	0.50

Table 4. Thermal physical properties of Uranium dioxide sample 2738

Temperature / K	Thermal diffusivity / $mm^2 \cdot s^{-1}$		Thermal conductivity / $W \cdot m^{-1} \cdot K^{-1}$	
	Mean Value	95 % Coverage Interval	Mean Value	95 % Coverage Interval
298	3.42	0.31	7.05	0.54
325	3.19	0.36	6.86	0.38
375	2.93	0.40	6.16	0.54
448	2.54	0.35	5.50	0.59

3. CONCLUSION

Safety analyses are required by the national regulatory bodies to prove that the UO_2 fuel can be burned safely in the reactors. In these analyses is necessary the known of thermophysical properties that are used in thermal hydraulic codes to study design basis accidents. Available open literature on thermophysical properties of UO_2 fuels has been reviewed and from analysis of these data, the uncertainties recommended for equations were determined from scatter in data and deviations of data but without information about the sources of uncertainties. This study presents a metrological evaluation of characterization method, through a series of tests performed on LMPT/CDTN specimens of UO_2. The results of density, thermal diffusivity and thermal conductivity were presented, along with a detailed analysis of the sources of uncertainties. The analyses showed that the relative uncertainty value for density is 1 %, ranging from 7.3 % up to 13.7 % for thermal diffusivity and 4.6 % up to 11.0 % for thermal conductivity, within 95 % confidence level. These values are the same order of magnitude of that recommended uncertainties published by Fink (2000). However these results appear to be adequate new measurements are needed and a more detailed investigation becomes necessary.

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