



## ESTIMATION OF THERMAL CONDUCTIVITY AND VOLUMETRIC HEAT CAPACITY IN TRANSIENT EXPERIMENTS

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***Abstract.** This paper deals with the development of an experimental setup for the estimation of the thermal conductivity and of the volumetric heat capacity of solids. It consists of a heater symmetrically assembled between two pieces of the specimen with unknown properties. Transient simulated temperature measurements taken in the specimen are used in the estimation procedure based on the Levenberg-Marquardt Method of minimization of the least-squares norm. An analysis of the sensitivity coefficients and the use of a D-optimum criterion permit the optimum design of the experiment.*

***Keywords:** Thermal Conductivity, Volumetric Heat Capacity, Parameter Estimation, Levenberg-Marquardt*

### 1. INTRODUCTION

The accurate knowledge of thermophysical properties is of importance for the correct prediction of the thermal behavior of bodies. Several experimental techniques have been developed in the past for the estimation of thermal conductivity and thermal diffusivity, by using steady-state as well as transient experiments. Such techniques include, among others, the guarded hot-plate method (ASTM, C177), the Flash method (Taylor and Maglic, 1984) and the hot-wire method (Blackwell, 1954). Transient techniques have the advantage of involving faster experiments than steady-state techniques.

More recently, the use of inverse analysis techniques of parameter estimation have been used for the identification of thermophysical properties, by utilizing minimization procedures involving transient measurements (Taktak et al, 1993, Dowding et al, 1995, 1996, Orlande et al, 1994, 1995, Guimarães et al, 1997, Mejias et al, 1999).

The main objective of this work is to revisit the analysis of Taktak et al (1993), in order to design an experiment for the simultaneous estimation of thermal conductivity and volumetric heat capacity of solids, by using transient temperature measurements taken in the solid. Three possible arrangements for the experimental setup are analyzed in this work. The

best arrangement, as well as experimental variables such as the sensor's location, heating and final times, are chosen by using the D-optimum criterion (Taktak et al, 1993, Ozisik and Orlande, 1999, Beck and Arnold, 1977). The Levenberg-Marquardt method (Beck and Arnold, 1977, Ozisik and Orlande, 1999) is used for the minimization of the least-squares norm. The accuracy of such a parameter estimation approach is verified by using transient simulated measurements containing random errors, as described next.

## 2. PHYSICAL PROBLEMS AND MATHEMATICAL FORMULATIONS

The physical problems considered here, involving the experimental apparatus to be used for the simultaneous estimation of thermal conductivity and volumetric heat capacity, consists of a heater symmetrically placed between two identical pieces of length  $L$ , of the solid with unknown properties. The heater is turned on for a period  $0 < t \leq t_h$ . Transient temperature measurements taken in the solid in the period  $0 < t \leq t_f$ , where  $t_h \leq t_f$ , are used for the estimation of the properties. Three possible experimental arrangements analyzed here for the setup are summarized in Table 1. The first two arrangements involve finite regions with boundary conditions of constant temperature and thermal insulation, respectively, at the surface not in contact with the heater (surface at  $x = L$ ). The last arrangement considers the length  $L$  of the solid to be so large that it can be treated as a semi-infinite region.

Table 1. Different arrangements examined for the experimental setup

Arrangement	Region	Boundary condition at $x = L$
Problem 1	Finite	Constant temperature
Problem 2	Finite	Thermal insulation
Problem 3	Semi-infinite	–

By taking into account the symmetry of the experimental apparatus, the mathematical formulations of the three different problems examined here can be given in dimensionless form as

$$c^* \frac{\partial \theta}{\partial \tau} = k^* \frac{\partial^2 \theta}{\partial X^2}, \quad \text{in } 0 < X < 1, \text{ for } \tau > 0 \quad (1.a)$$

$$k^* \frac{\partial \theta}{\partial X} = -u(\tau_h - \tau), \quad \text{at } X = 0, \text{ for } \tau > 0 \quad (1.b)$$

$$a \frac{\partial \theta}{\partial X} + b\theta = 0, \quad \text{at } X = 1, \text{ for } \tau > 0 \quad (1.c)$$

$$\theta = 0, \quad \text{in } 0 < X < 1, \text{ for } \tau = 0 \quad (1.d)$$

Where:

$$u(\tau_h - \tau) = \begin{cases} 1, & 0 < \tau \leq \tau_h \\ 0, & \tau > \tau_h \end{cases}$$

The following dimensionless variables were defined:

$$X = \frac{x}{L}, \quad \tau = \frac{k_R}{\rho c_R L^2} t, \quad k^* = \frac{k}{k_R}, \quad c^* = \frac{c}{c_R}, \quad \theta = \frac{k_R}{q_0 L} (T - T_0) \quad (2.a-e)$$

where  $k_R$  and  $c_R$  are reference values for thermal conductivity and volumetric heat capacity, respectively,  $\rho$  is the density of the solid,  $q_0$  is the magnitude of the applied heat flux during the period  $0 < t \leq t_h$  and  $T_0$  is the constant temperature at the boundary  $x = L$ , which is also assumed to be the initial temperature in the region.

The dimensionless problem given by Eqs. (1) can be used for the formulation of **Problem 1**, involving a constant temperature boundary condition at  $X = 1$ , by making  $a = 0$  in Eq. (1.c). For the case of **Problem 2**, involving an insulated boundary at  $X = 1$ , we make  $b = 0$  in Eq. (1.c). For the case of **Problem 3**, where the region is considered as a semi-infinite medium, the boundary condition (1.c) does not appear in the formulation and the quantity  $L$  is taken as a reference length for the problem. Alternatively, we can say that  $\theta = 0$  as  $X \rightarrow \infty$ .

**Problems 1, 2 and 3**, as defined in Table 1 and formulated by Eqs. (1), are denoted as *Direct Problems* when the physical properties  $k^*$  and  $c^*$  are known. The objective of such direct problems is to determine the temperature field in the region. They can be solved analytically by using the Classical Integral Transform Technique (Ozisk, 1993), as presented below.

The analytical solution for **Problem 1** is given by:

$$\theta(X, \tau) = \sum_{m=1}^{\infty} 2 \cos(\lambda_m X) \left\{ \frac{1}{k^* \lambda_m^2} \left( 1 - e^{-\frac{k^*}{c^*} \lambda_m^2 \tau} \right) \right\} \quad \text{For } 0 < \tau \leq \tau_h, \quad (3.a)$$

and

$$\theta(X, \tau) = \sum_{m=1}^{\infty} 2 \cos(\lambda_m X) \left\{ \frac{1}{k^* \lambda_m^2} \left( e^{\frac{k^*}{c^*} \lambda_m^2 (\tau_h - \tau)} - e^{-\frac{k^*}{c^*} \lambda_m^2 \tau} \right) \right\} \quad \text{For } \tau > \tau_h, \quad (3.b)$$

Where:

$$\lambda_m = (2m - 1)\pi/2 \quad (4)$$

The solution for **Problem 2** is obtained as:

$$\theta(X, \tau) = \frac{\tau}{c^*} + \sum_{m=1}^{\infty} \frac{2}{k^* \lambda_m^2} \cos(\lambda_m X) \left( 1 - e^{-\frac{k^*}{c^*} \lambda_m^2 \tau} \right) \quad \text{For } 0 < \tau \leq \tau_h, \quad (5.a)$$

and as

$$\theta(X, \tau) = \frac{\tau_h}{c^*} + \sum_{m=1}^{\infty} \frac{2}{k^* \lambda_m^2} \cos(\lambda_m X) \left( e^{\frac{k^*}{c^*} \lambda_m^2 (\tau_h - \tau)} - e^{-\frac{k^*}{c^*} \lambda_m^2 \tau} \right) \quad \text{For } \tau > \tau_h, \quad (5.b)$$

Where:

$$\lambda_m = m\pi \quad (6)$$

The solution for **Problem 3** is given by:

$$\theta(X, \tau) = \frac{X}{k^*} \left[ \text{Erf} \left( \frac{X}{2} \sqrt{\frac{c^*}{\tau k^*}} \right) - 1 \right] + \frac{2}{k^*} e^{-\frac{X^2 c^*}{4\tau k^*}} \sqrt{\frac{\tau k^*}{\pi c^*}} \quad \text{For } 0 < \tau \leq \tau_h, \quad (7.a)$$

and

$$\theta(X, \tau) = \frac{X}{k^*} \left[ \operatorname{Erf} \left( \frac{X}{2} \sqrt{\frac{c^*}{\tau k^*}} \right) - \operatorname{Erf} \left( \frac{X}{2} \sqrt{\frac{c^*}{(\tau - \tau_h) k^*}} \right) \right] + \frac{2}{k^*} \left( e^{-\frac{X^2 c^*}{4\tau k^*}} \sqrt{\frac{\tau k^*}{\pi c^*}} - e^{-\frac{X^2 c^*}{4k^*(\tau - \tau_h)}} \sqrt{\frac{(\tau - \tau_h) k^*}{\pi c^*}} \right) \quad \text{For } \tau > \tau_h, \quad (7.b)$$

### 3. INVERSE PROBLEM

For the *Inverse Problem* considered here, the thermal conductivity  $k^*$  and the volumetric heat capacity  $c^*$  are regarded as unknown quantities. For the estimation of such properties, we consider available for the inverse analysis the transient readings  $Y_i$  taken at times  $t_i$ ,  $i = 1, \dots, I$  of one temperature sensor located in the solid with unknown properties. The unknown properties are estimated here through the minimization of the ordinary least-squares norm defined as

$$S(\mathbf{P}) = [\mathbf{Y} - \mathbf{T}(\mathbf{P})]^T [\mathbf{Y} - \mathbf{T}(\mathbf{P})] \quad (8.a)$$

Where:

$$[\mathbf{Y} - \mathbf{T}(\mathbf{P})]^T = [Y_1 - T_1(\mathbf{P}), Y_2 - T_2(\mathbf{P}), \dots, Y_I - T_I(\mathbf{P})] \quad (8.b)$$

$$\text{and } \mathbf{P} = [k^*, c^*] \quad (8.c)$$

The estimated temperatures  $T_i(\mathbf{P})$  are obtained from the solution of the direct problem, given by Eq. (3-7) by using estimated values for the unknown parameters.

We use in this paper the Levenberg-Marquardt Method (Beck and Arnold, 1977, Ozisik and Orlande, 1999) for the minimization of the objective function given by Eq. (8.a). The iterative procedure of such method is given by

$$\mathbf{P}^{k+1} = \mathbf{P}^k + (\mathbf{J}^T \mathbf{J} + \mu^k \mathbf{\Omega}^k)^{-1} \mathbf{J}^T [\mathbf{Y} - \mathbf{T}(\mathbf{P}^k)] \quad (9)$$

where  $\mu^k$  is the damping parameter and  $\mathbf{\Omega}^k$  is a diagonal matrix, which can be taken as the identity matrix or as the diagonal of  $\mathbf{J}^T \mathbf{J}$ . The *sensitivity matrix*  $\mathbf{J}$  is defined as

$$\mathbf{J}(\mathbf{P}) = \left[ \frac{\partial \mathbf{T}^T(\mathbf{P})}{\partial \mathbf{P}} \right]^T = \begin{bmatrix} \frac{\partial T_1}{\partial k^*} & \frac{\partial T_1}{\partial c^*} \\ \frac{\partial T_2}{\partial k^*} & \frac{\partial T_2}{\partial c^*} \\ \vdots & \vdots \\ \frac{\partial T_I}{\partial k^*} & \frac{\partial T_I}{\partial c^*} \end{bmatrix} \quad (10)$$

The elements of the sensitivity matrix are denoted as the *sensitivity coefficients*. They provide a measure of the sensitivity of the estimated (or measured) temperatures with respect to changes in the unknown parameters. Clearly, the solution of inverse problems involving

sensitivity coefficients with small magnitudes is extremely difficult, because the choice of very different values for the unknown parameters would result in basically the same value for the measured variables. Also, the columns of the sensitivity matrix are required to be linearly independent in order to have the matrix  $\mathbf{J}^T \mathbf{J}$  invertible, that is, the determinant of  $\mathbf{J}^T \mathbf{J}$  cannot be zero or even very small.

#### 4. STATISTICAL ANALYSIS AND OPTIMUM EXPERIMENTS

After the minimization of the objective function given by Eq. (8.a), a statistical analysis can be performed in order to obtain confidence intervals and a confidence region for the estimated parameters. Confidence intervals at the 99% confidence level are obtained as (Beck and Arnold, 1977, Ozisik and Orlande, 1999):

$$\hat{P}_j - 2.576\sigma_{\hat{P}_j} \leq P_j \leq \hat{P}_j + 2.576\sigma_{\hat{P}_j} \quad (11.a)$$

where  $\hat{\mathbf{P}}$  are the values estimated for the unknown parameters.

The confidence region at the 99% confidence level is given by

$$(\hat{\mathbf{P}} - \mathbf{P})^T \mathbf{V}^{-1} (\hat{\mathbf{P}} - \mathbf{P}) \leq 9.21 \quad (11.b)$$

where  $\mathbf{V}$  is the covariance matrix of the estimated parameters given by

$$\mathbf{V} = (\mathbf{J}^T \mathbf{J})^{-1} \sigma^2 \quad (12)$$

An analysis of Eq. (11.b) reveals that some measure of the matrix  $\mathbf{V}^{-1}$  needs to be maximized in order to minimize the hypervolume of the confidence region and, as a result, obtain minimum variance estimates. Since the covariance matrix is given by Eq. (12), we can choose to maximize the determinant of the matrix  $\mathbf{J}^T \mathbf{J}$  in the so called D-optimum criterion (Beck and Arnold, 1977, Taktak et al, 1993, Dowding et al, 1995, 1996, Mejias et al, 1999, Ozisik and Orlande, 1999). By taking into account the maximum temperature in the region,  $\theta_{max}$ , and by assuming available for the analysis a large but fixed number of transient measurements, the elements of the matrix  $\mathbf{J}^T \mathbf{J}$  can be re-written as

$$[\mathbf{F}_{m,n}] = \frac{1}{\tau_f} \int_{\tau=0}^{\tau_f} \left( \frac{\partial \theta}{\partial P_m} \right) \left( \frac{\partial \theta}{\partial P_n} \right) \frac{1}{\theta_{max}^2} d\tau \quad (13)$$

where the subscripts  $m$  and  $n$  refer to the matrix row and column, respectively.

We use the criterion of maximum determinant of  $\mathbf{F}$ , the elements of which are given by Eq. (13), to choose the best experimental arrangement among those examined here that are summarized in Table 1. After selecting the experimental arrangement, we can use the same criterion to choose experimental variables, such as the location of the sensor, heating time and final time, as described next.

#### 5. RESULTS AND DISCUSSIONS

We present in Fig. 1.a-c the transient variation of the dimensionless sensitivity coefficients with respect to the unknown parameters for different measurement positions, for **Problems 1, 2** and **3**, respectively.

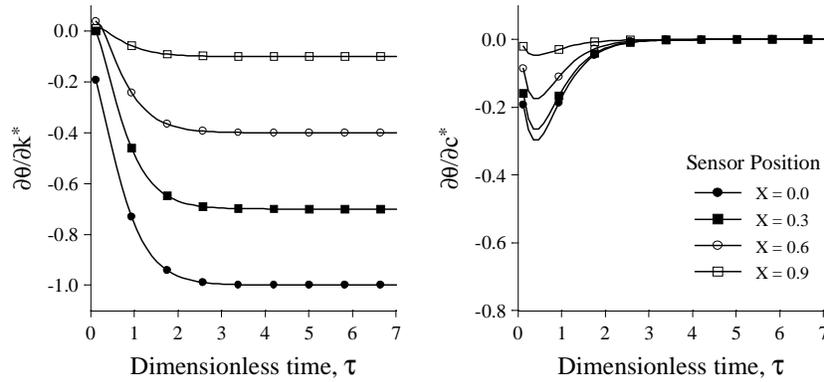


Figure 1.a – Sensitivity coefficients for **Problem 1**.

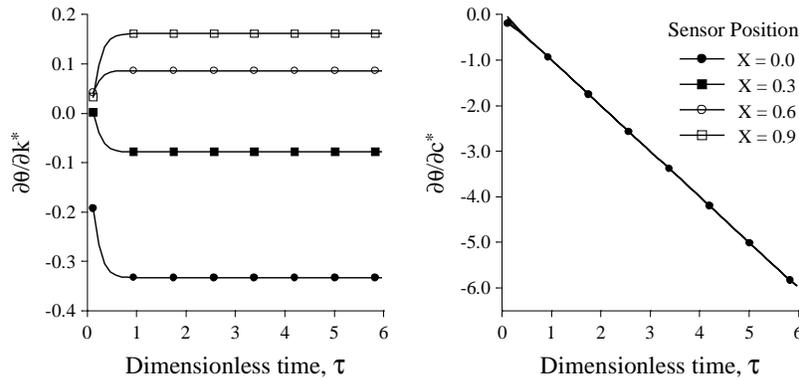


Figure 1.b – Sensitivity coefficients for **Problem 2**.

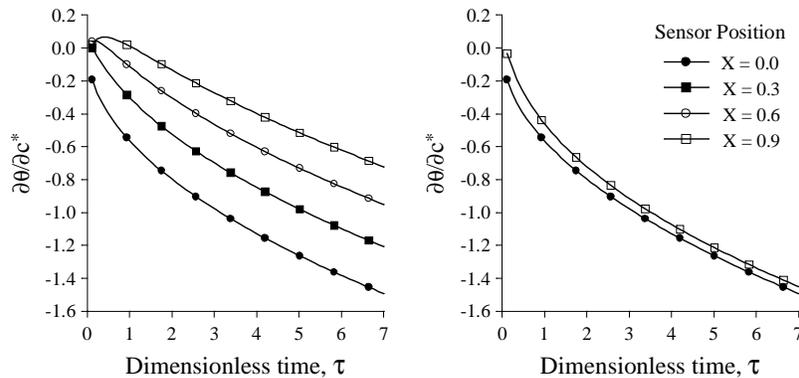


Figure 1.c – Sensitivity coefficients for **Problem 3**.

Figures 1.a,b reveals that the sensitivity coefficients are linearly independent for **Problems 1** and **2**, where the solid with unknown properties is treated as a finite region. For such Problems, the sensitivity coefficients attain larger magnitudes for measurements taken closer to the heated boundary at  $X = 0$ , except for the sensitivity coefficients with respect to  $c^*$  for **Problem 2**, which vary very little with the measurement position. Therefore, the conditions for the simultaneous estimation of  $k^*$  and  $c^*$ , by using the measurements of a single sensor taken either at the boundary  $X = 0$  or in a location near it, are good for **Problems 1** and **2**. The location of the sensor at the boundary is preferred because it results in non-intrusive measurements.

On the other hand, Fig. 1.c shows that, for each measurement location, the sensitivity coefficients tend to be linearly dependent for **Problem 3**, involving a semi-infinite region. In fact, the sensitivity coefficients with respect to  $k^*$  and  $c^*$  are identical for measurements taken at  $X = 0$ . As a result, the simultaneous estimation of  $k^*$  and  $c^*$ , by using the experimental arrangement of **Problem 3**, is not possible if only non-intrusive measurements of a single sensor are used in the analysis.

The foregoing analysis of the sensitivity coefficients reveals that the experimental arrangements involving finite regions, such as in **Problems 1** and **2**, should be preferred over the arrangement of **Problem 3**, which considers the solid as a semi-infinite region, for the simultaneous estimation of  $k^*$  and  $c^*$ . The arrangements of **Problems 1** and **2** permit the use of a single non-intrusive sensor for the estimation of such quantities, which is impossible for the case of **Problem 3**.

Figure 2 shows the transient variation of the determinant of  $\mathbf{F}$  for the case of **Problem 1**, for different measurement locations, by considering  $\tau_h = \tau_f$ . As expected from the analysis of the sensitivity coefficients, measurements taken at the boundary  $X = 0$  provide more useful information for the simultaneous estimation of  $k^*$  and  $c^*$  and, as a result, the values of  $\det(\mathbf{F})$  are larger for this position.

Figure 3 illustrates the effects on  $\det(\mathbf{F})$  of considering a heating time,  $\tau_h$ , smaller than the final experimental time,  $\tau_f$ . This figure shows that  $\det(\mathbf{F})$  undergoes a sudden increase when the heater is turned off and, generally, the maximum value of  $\det(\mathbf{F})$  for  $\tau_h < \tau_f$  is larger than for  $\tau_h = \tau_f$ . Such is the case because the sensitivity coefficients tend towards linear independence when the heating is stopped. The maximum value of  $\det(\mathbf{F})$  is reached with heating and final times of approximately 2.5 and 3.3, respectively. Note that a curve joining the peaks for  $\tau_h = 2$  and 2.5 is rather flat, showing that any value in this range will be very close to the optimum heating time. On the other hand, the behavior of  $\det(\mathbf{F})$  is very sensitive to the choice of the final time  $\tau_f$ . Note that  $\det(\mathbf{F})$  decreases very fast after its maximum value is reached. Therefore, the analysis of Fig. 2 and 3 reveals that the optimum experiment for **Problem 1**, by considering one single measurement in the analysis, involves the sensor located at  $X = 0$ , with heating time chosen in the interval  $2 \leq \tau_h \leq 2.5$  and with final time given approximately by  $\tau_h + 0.8$ .

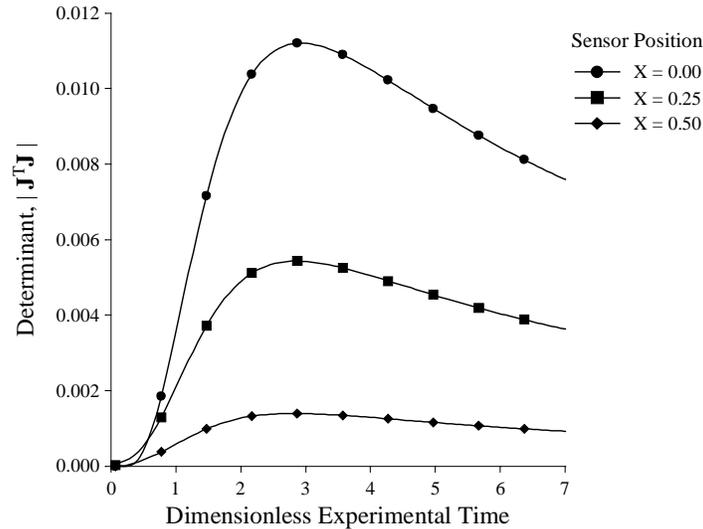


Figure 2 – Determinant for **Problem 1** for different measurement locations with  $\tau_f = \tau_h$ .

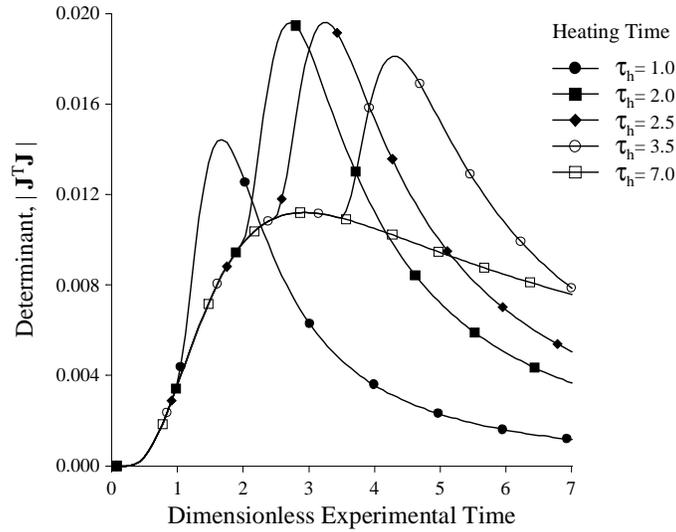


Figure 3 – Determinant for **Problem 1** for different heating times at  $X = 0$ .

Figure 4 shows the transient variations of  $\det(\mathbf{F})$  for different heating times, by considering in the analysis the experimental arrangement of **Problem 2** with a single sensor located at  $X = 0$ . The optimum heating time for **Problem 2** can be chosen in the interval  $0.6 \leq \tau_h \leq 0.8$ , with final time taken as  $\tau_h + 1.2$ . A comparison of Fig. 3 and 4 reveals that the use of the constant temperature boundary condition, as in **Problem 1**, can provide more accurate results for the estimated parameters than the use of the thermal insulation boundary condition, such as in **Problem 2**. This is the case because larger values of  $\det(\mathbf{F})$  are obtained with the experimental arrangement of **Problem 1**. We note that the constant temperature boundary condition is easier to be implemented for low thermal conductivity solids. This can be accomplished by putting a block of metal with very high thermal conductivity into contact with the solid of unknown properties. The temperature gradient in the metal block will be very small at the interface with the solid, thus approximating the constant temperature boundary condition. On the other hand, the thermal insulation boundary condition is easier to be implemented than the constant temperature boundary condition for solids of high thermal conductivity.

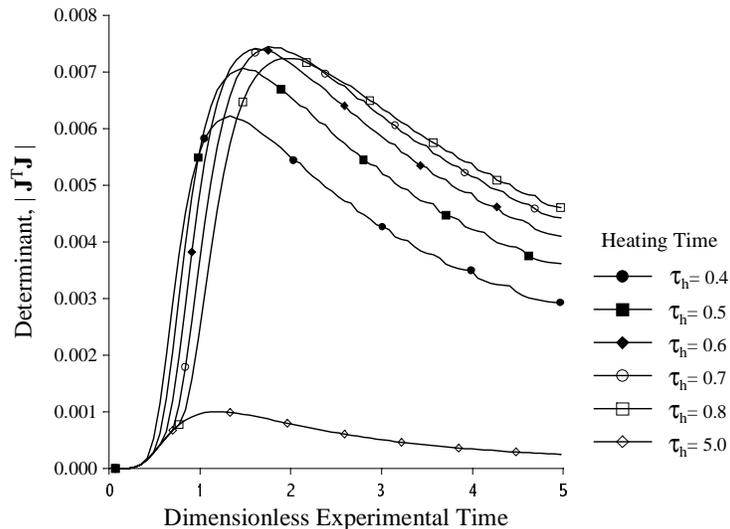


Figure 4 – Determinant for **Problem 2** for different heating times with  $X = 0$ .

It is worthwhile noting that, as expected from the linear-dependence of the sensitivity coefficients, the determinant of the matrix  $\mathbf{F}$  was null for the case of **Problem 3**, with a single sensor located at  $X = 0$ .

After choosing the experimental variables to be used on the two different arrangements of **Problems 1** and **2**, let us now address the accuracy of the estimated parameters obtained with the present estimation procedure based on the Levenberg-Marquardt method. The subroutine DBCLSJ of the IMSL, based on such method, was used in order to obtain the results shown below.

Tables 2 and 3 show the results obtained by using different initial guesses for the estimation of the unknown parameters  $k^*$  and  $c^*$ , with simulated transient measurements of a single sensor located at  $X = 0$ , in the experimental arrangements of **Problems 1** and **2**, respectively. The standard deviations and the 99% confidence intervals for the estimated parameters are also included in these tables.

The heating and final times for each of the problems were chosen based on the optimization procedure discussed above. They were taken as  $\tau_h = 2.2$  and  $\tau_f = 3.0$  for **Problem 1** and as  $\tau_h = 0.7$  and  $\tau_f = 1.75$  for **Problem 2**. During the experiment we assumed available 100 transient measurements for the inverse analysis. The simulated measured data were obtained from the solution of the associated direct problems, by using prescribed values of  $k^* = c^* = 1$  for the parameters. The measurements obtained in this manner are considered as errorless. In order to simulate actual measured data containing errors, we added a random term to such errorless measurements. This term was normally distributed with zero mean and constant standard deviation. The results shown in Tables 2 and 3 were obtained with measurements of standard deviation  $\sigma = 10^{-2} \theta_{\max}$ , where  $\theta_{\max}$  is the maximum measured temperature.

We note in Tables 2 and 3 that very accurate estimates were obtained for the unknown parameters, for both arrangements of **Problem 1** and **Problem 2**. Also, the estimated parameters were quite insensitive to the initial guesses used for the Levenberg-Marquardt method and convergence was achieved even with initial guesses of 4 orders of magnitude smaller than the exact parameters.

It is interesting to notice in Tables 2 and 3 that, as expected from the analysis of the determinant of  $\mathbf{F}$ , more accurate estimates were obtained with the arrangement of **Problem 1**. The average areas of the combined rectangular 99% confidence regions, obtained by multiplying the confidence intervals of the two parameters, were  $1.9 \times 10^{-4}$  for **Problem 1** and  $3.8 \times 10^{-4}$  for **Problem 2**.

Table 2. Results obtained for **Problem 1**

	Initial Guess	Estimated Parameters	Standard Deviation	99% Confidence Intervals
$k^*$	$10^{-1}$	1.00167	$1.41626 \times 10^{-3}$	(0.99803 ; 1.00532)
$c^*$	$10^{-1}$	1.00047	$5.14971 \times 10^{-3}$	(0.98721 ; 1.01374)
$k^*$	$10^{-2}$	1.00105	$1.39631 \times 10^{-3}$	(0.99745 ; 1.00465)
$c^*$	$10^{-2}$	1.00628	$4.58322 \times 10^{-3}$	(0.99448 ; 1.01809)
$k^*$	$10^{-3}$	0.99927	$1.40741 \times 10^{-3}$	(0.99564 ; 1.00289)
$c^*$	$10^{-3}$	0.99285	$5.10408 \times 10^{-3}$	(0.97970 ; 1.00599)
$k^*$	$10^{-4}$	1.00263	$1.42076 \times 10^{-3}$	(0.99897 ; 1.00629)
$c^*$	$10^{-4}$	1.00598	$5.17790 \times 10^{-3}$	(0.99264 ; 1.01932)

Table 3. Results obtained for **Problem 2**

	Initial Guess	Estimated Parameters	Standard Deviation	99% Confidence Intervals
k*	10 <sup>-1</sup>	0.99791	6.61547 x 10 <sup>-3</sup>	(0.98087 ; 1.01495)
c*	10 <sup>-1</sup>	0.99771	2.09646 x 10 <sup>-3</sup>	(0.99235 ; 1.00315)
k*	10 <sup>-2</sup>	1.00455	6.69145 x 10 <sup>-3</sup>	(0.98731 ; 1.02179)
c*	10 <sup>-2</sup>	0.99808	2.09533 x 10 <sup>-3</sup>	(0.99268 ; 1.00348)
k*	10 <sup>-3</sup>	1.00551	6.69893 x 10 <sup>-3</sup>	(0.98826 ; 1.02278)
c*	10 <sup>-3</sup>	0.99627	2.08664 x 10 <sup>-3</sup>	(0.99089 ; 1.00164)
k*	10 <sup>-4</sup>	1.00886	6.73990 x 10 <sup>-3</sup>	(0.99150 ; 1.02622)
c*	10 <sup>-4</sup>	0.99776	2.09218 x 10 <sup>-3</sup>	(0.99237 ; 1.00315)

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