Determination of Uncertainty of Thickness Measurement of Thin Layers in Manifacier's Method

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ABSTRACT

In this paper a procedure to determine the uncertainty of thickness measurement of thin layer is presented. The spectrophotometer method uses the spectrum of light transmission and some dependences between interference extremes and thickness of thin layer. The final result has been achieved by using a method recommended by Guide to the Expression of Uncertainty in Measurement. The standard uncertainty has been estimated by approach, called type B, which is based on manufacturer's specification of spectrophotometer, experience in reading of results on transmission spectrum and literature data.

Keywords: uncertainty, thin layer, correlated variables

1. INTRODUCTION

In investigations of propriety of thin layers the exact measurement of thickness is one of most important act. This parameter is necessary to estimate the equableness of settling and to calculate the surface resistance and consistently admissible power of examined element [2]. In this paper only transparent and conductive thin layers deposited on glass background are taken into consideration. In Fig. 1 the transmission and multiple reflection of light beam is showed.

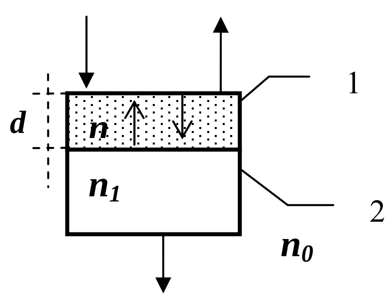

Fig. 1. Transmission and reflection of light across a single layer 1 of thickness d on transparent glass 2 with corresponding refractive indexes n, n_1 and n_0 (air).

Equation (1) is used to calculate the transmissivity T of thin layer when incidence angle is equal zero and absorption of background is negligible.

$$T(\lambda) = \frac{16\, n_0 n_1 n^2 \cdot \alpha}{C_1^2 + C_2^2 \alpha^2 + 2C_1 C_2 \alpha \cdot \cos(4\pi\, nd / \lambda)} \qquad (1)$$

where

$$C_1 = (n + n_0)(n_1 + n), \quad C_2 = (n - n_0)(n_1 - n)$$

and α is the absorptivity of thin layer.

When $\cos(4\pi\, nd / \lambda) = \pm 1$ then values of T are maximum or minimum. The envelopes of the family of extremes $T_{max}(\lambda)$ and $T_{min}(\lambda)$ are given by formulas (2)

$$T_{\max}(\lambda) = \frac{16\, n_0 n_1 n^2 \cdot \alpha}{(C_1 + C_2 \alpha)^2}\ , \quad T_{\min}(\lambda) = \frac{16\, n_0 n_1 n^2 \cdot \alpha}{(C_1 - C_2 \alpha)^2} \qquad (2)$$

The typical spectrum of light transmission $T(\lambda)$ with an envelope of extremes $T_{max}(\lambda)$ and $T_{min}(\lambda)$ is shown in Fig. 2.

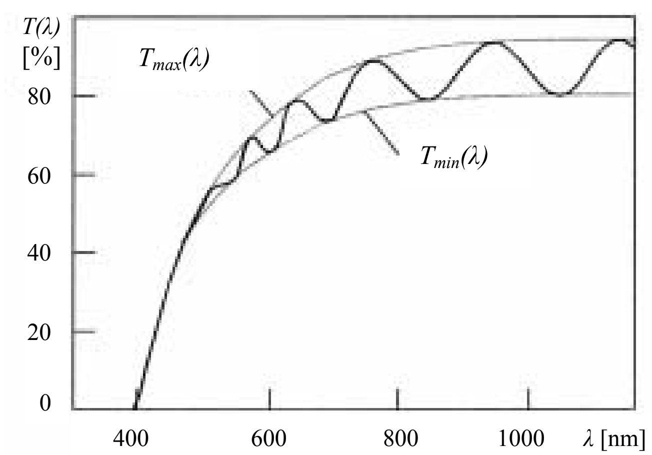

Fig. 2. The spectrum of light transmission with extremes $T_{max}(\lambda)$ and $T_{min}(\lambda)$

Following equations (3) are used to count the refraction index n

$$n = \left[N + (N^2 - n_0^2 \cdot n_1^2)^{1/2}\right]^{1/2}$$
$$N = \frac{n_0^2 + n_1^2}{2} + 2n_0 n_1 \frac{T_{\max}(\lambda) - T_{\min}(\lambda)}{T_{\max}(\lambda) \cdot T_{\min}(\lambda)} \qquad (3)$$

Refractive index can be defined on the base of knowledge of T_{max}, T_{min}, n_0 and n_1 values (see Fig. 3) for the same wavelength λ [3,4]. Thickness of layer, through which the beam of light crosses can be appointed from relationship (4)

$$d = \frac{M \cdot \lambda_1 \cdot \lambda_2}{2 \cdot [n(\lambda_1) \cdot \lambda_2 - n(\lambda_2) \cdot \lambda_1]} \qquad (4)$$

where

M – number of oscillations between two chosen extremes,
$\lambda_1, \lambda_2, n(\lambda_1), n(\lambda_2)$ - wavelengths (read from curve of light transmission) and corresponding to them refractive indexes.

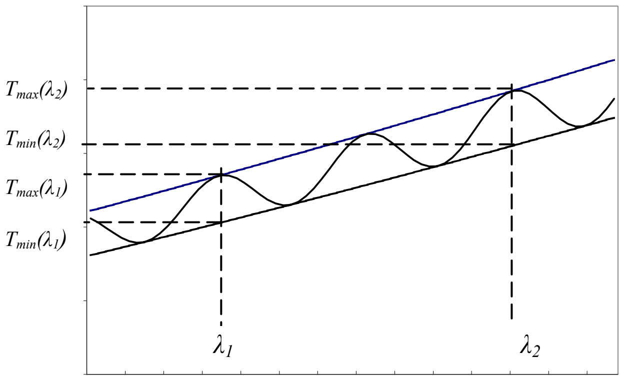

Fig. 3. The way of determine values on the base of the light transmission spectrum received from spectrophotometer.

2. STANDARD UNCERTAINTY

As an assumption it takes that in this equation mentioned above only two factors T_{max} and T_{min} are accompanied by their corresponding uncertainty. In equation (4) only the number of M is taken as faultless. The standard uncertainty *u(d)* will be estimated by approach, called type B which is based on manufacturer's specification of spectrophotometer, experience in reading of results on transmission spectrum and literature data [1]. It gives that change of values λ_1, λ_2 is significant for indexes $n(\lambda_1), n(\lambda_2)$ so it will be necessary to estimate and consider their correlation (if necessary). The measurement equation is as following

$$d = f(x_1, x_2, x_3, x_4) = f(\lambda_1, n(\lambda_1), \lambda_2, n(\lambda_2)) \qquad (5)$$

and respective combined standard uncertainty $u_c(\hat{d})$ is calculated from

$$u_c^2(\hat{d}) = \sum_{i=1}^{4} (\frac{\partial f}{\partial x_i})^2 \cdot u^2(\hat{x}_i) + 2\sum_{i=1}^{3} \sum_{j=i+1}^{4} \frac{\partial f}{\partial x_i} \cdot \frac{\partial f}{\partial x_j} \cdot u(\hat{x}_i, \hat{x}_j) \qquad (6)$$

where

$\hat{x}_i$ – estimate of input value x_i (in this case there are: $\hat{\lambda}_1, \hat{\lambda}_2, \hat{n}(\lambda_1), \hat{n}(\lambda_2)$), $u(\hat{x}_i)$ - contribution as an uncertainty in $\hat{d}$ arising from the uncertainty in x_i, $u(\hat{x}_i, \hat{x}_j)$ - covariance between x_i ,x_j (only two pairs λ_1-$n(\lambda_1)$ and λ_2-$n(\lambda_2)$ are expected to be correlated), $\frac{\partial f}{\partial x_i}$ - sensitivity coefficient (describes how the value of *f* varies with changes in the parameters x_i , x_j , etc.).

The covariance $u(\hat{x}_i, \hat{x}_j)$ can be replaced by product

$$u(\hat{x}_i, \hat{x}_j) = u(\hat{x}_i) \cdot u(\hat{x}_j) \cdot r(\hat{x}_i, \hat{x}_j) \qquad (7)$$

where $r(\hat{x}_i, \hat{x}_j)$ is the correlation coefficient and it may be estimated by experiment as

$$r(\hat{x}_i, \hat{x}_j) = \frac{u(\hat{x}_i) \cdot \Delta\hat{x}_i}{u(\hat{x}_j) \cdot \Delta\hat{x}_j} \qquad (8)$$

when a change $\Delta\hat{x}_i$ of the variable $\hat{x}_i$ causes the change $\Delta\hat{x}_j$ of variable $\hat{x}_j$.

3. DEGREE OF CORRELATION

Before we will accede to calculations the standard uncertainty it is very important to qualify the value of correlation coefficients between the variables λ_1-$n(\lambda_1)$ and λ_2-$n(\lambda_2)$. If they are negligible than the formula (6) will contain only the first component. Because we look for values of refractive index for select values of wavelengths, so in accordance from(9) one should qualify standard uncertainties $u(\hat{\lambda})$ and $u(\hat{n})$ and estimate change of value $\Delta\hat{n}$ caused with change of wavelength of about $\Delta\hat{\lambda}$. Then the correlation coefficient is

$$r(\hat{\lambda}\ , \hat{n}) = \frac{u(\hat{\lambda}) \cdot \Delta\hat{n}}{u(\hat{n}) \cdot \Delta\hat{\lambda}} \qquad (9)$$

Accuracy of spectrophotometer is 1% (done by manufacturer) and the curve of light transmission is drawn on plotting paper with millimeter scale, so the uncertainty $u(\hat{\lambda})$ of wavelength λ is

$$u(\hat{\lambda}) = \sqrt{D^2 / 3}\ ,\ D = 0{,}01 \cdot \hat{\lambda} + 0{,}5nm \qquad (10)$$

One accepted the rectangular distribution of variable λ (there are no grounds for believing that a small error is more likely than a large one) and value 0,5nm corresponds to 0,3mm accuracy on plotting area. Estimation of uncertainty $u(\hat{n})$ is more complicated. To simplify it one assume exact values of n_0 and n_1 (n_0=1 n_1=1,52) so from equations (3) we have

$$u^2(\hat{n}) = \left(\frac{\partial n}{\partial N}\right)^2 \cdot u^2(\hat{N}) \qquad (11)$$

$$u^2(\hat{N}) = (2n_0 n_1)^2 \left[\left(\frac{\partial N}{\partial T_{\max}}\right)^2 u^2(\hat{T}_{\max}) + \left(\frac{\partial N}{\partial T_{\min}}\right)^2 u^2(\hat{T}_{\min}) \right] \qquad (11a)$$

$$\frac{\partial N}{\partial T_{\max}} = \frac{1}{\hat{T}_{\max}^2} \qquad \frac{\partial N}{\partial T_{\min}} = \frac{-1}{\hat{T}_{\min}^2} \qquad (12)$$

$$u^2(\hat{T}_{max}) = D_{Tx}^2 / 3 \qquad u^2(\hat{T}_{min}) = D_{Tn}^2 / 3 \tag{13}$$

$$D_{Tx} = 0{,}01 \cdot \hat{T}_{max} + 0{,}002 \quad D_{Tn} = 0{,}01 \cdot \hat{T}_{min} + 0{,}002 \tag{14}$$

Also with the rectangular distribution of variables T_{max}, T_{min} and the same accuracy of transmission axis (value 0.002 corresponds to 0,3mm on plotting area).
Because the relate n=f(N) is composite function that the derivative form is the following (Eq.15)

$$\frac{\partial n}{\partial N} = \frac{N - \sqrt{N^2 - n_0^2 n_1^2}}{2\sqrt{N^2 - n_0^2 n_1^2} \cdot \sqrt{N + \sqrt{N^2 - n_0^2 n_1^2}}} \tag{15}$$

3.1 Results of calculation

To estimate the thickness of layer on the base of Eq. 4 two wavelengths and suitable values of T_{max} and T_{min} have been chosen from curve of light transmission and according to Eq.10-15 other parameters are calculated (see Table 1).

Table 1.

$\hat{\lambda}_1$ nm	D_1 nm	$u(\hat{\lambda}_1)$ nm	$\hat{T}_{max1}$	$\hat{T}_{min1}$	$\hat{N}$	$\frac{\partial n_{\lambda 1}}{\partial N}$	$u(\hat{n}_{\lambda 1}$
453,0	5,0	2,887	0,725	0,685	1,900	0,191	8,5E-3

$$\Delta\hat{\lambda}_1 = 7{,}5nm \qquad \Delta\hat{n}_{\lambda 1} = 0{,}003$$

$$r(\hat{\lambda}_1, \hat{n}_{\lambda 1}) = \frac{u(\hat{\lambda}_1) \cdot \Delta\hat{n}_{\lambda 1}}{u(\hat{n}_{\lambda 1}) \cdot \Delta\hat{\lambda}_1} = \frac{2{,}887 \cdot 0{,}003}{8{,}5E\text{–}3 \cdot 7{,}5} = 0{,}135$$

$\hat{\lambda}_2$ nm	D_2 nm	$u(\hat{\lambda}_2)$ nm	$\hat{T}_{max2}$	$\hat{T}_{min2}$	$\hat{N}$	$\frac{\partial n_{\lambda 2}}{\partial N}$	$u(\hat{n}_{\lambda 2}$
695,0	7,4	4,041	0,795	0,740	1,939	0,172	6,6E-3

$$\Delta\hat{\lambda}_2 = 7{,}5nm \qquad \Delta\hat{n}_{\lambda 2} = 0{,}001$$

$$r(\hat{\lambda}_2, \hat{n}_{\lambda 2}) = \frac{u(\hat{\lambda}_2) \cdot \Delta\hat{n}_{\lambda 2}}{u(\hat{n}_{\lambda 2}) \cdot \Delta\hat{\lambda}_2} = \frac{4{,}041 \cdot 0{,}001}{6{,}6E\text{–}3 \cdot 7{,}5} = 0{,}082$$

Results of calculation shows that the value of correlation coefficient is rather low and there is a very probable that the dependency between accuracy of determine wavelengths and refractive index can be neglect. One assume that in the equation (6) all input quantities are independent of one another.

4. COMBINED UNCERTAINTY

In equation (4) all input quantities (except for M) obtained in a measurement procedure, even partial results (like $n(\lambda_1)$ and $n(\lambda_2)$), are liable to an uncertainty. It becomes necessary to estimate and combine uncertainties of the input results.
Because the true value of input variable x_i is unknown so it may be replaced by its estimate $\hat{x}_i$.

Then the equation (6) is replaced by

$$u_c^2(\hat{d}) = \sum_{i=1}^{4} G_i^2 \cdot u^2(\hat{x}_i) = \\ = G_1^2 u^2(\hat{\lambda}_1) + G_2^2 u^2(\hat{n}(\hat{\lambda}_1)) + G_3^2 u^2(\hat{\lambda}_2) + G_4^2 u^2(\hat{n}(\hat{\lambda}_2)) \tag{16}$$

where the sensitivity coefficient G_i is evaluated as relevant partial differential showed in Eq. 17

$$G_1 = \frac{\partial f}{\partial \hat{\lambda}_1} = \frac{M}{2} \frac{\hat{\lambda}_2^2 \cdot \hat{n}(\lambda_1)}{[\hat{n}(\lambda_1) \cdot \hat{\lambda}_2 - \hat{n}(\lambda_2) \cdot \hat{\lambda}_1]^2}$$

$$G_2 = \frac{\partial f}{\partial \hat{\lambda}_2} = \frac{M}{2} \frac{-\hat{\lambda}_1^2 \cdot \hat{n}(\lambda_2)}{[\hat{n}(\lambda_1) \cdot \hat{\lambda}_2 - \hat{n}(\lambda_2) \cdot \hat{\lambda}_1]^2} \tag{17}$$

$$G_3 = \frac{\partial f}{\partial \hat{n}(\lambda_1)} = \frac{M}{2} \frac{-\hat{\lambda}_1 \cdot \hat{\lambda}_2^2}{[\hat{n}(\lambda_1) \cdot \hat{\lambda}_2 - \hat{n}(\lambda_2) \cdot \hat{\lambda}_1]^2}$$

$$G_4 = \frac{\partial f}{\partial \hat{n}(\lambda_2)} = \frac{M}{2} \frac{\hat{\lambda}_1^2 \cdot \hat{\lambda}_2}{[\hat{n}(\lambda_1) \cdot \hat{\lambda}_2 - \hat{n}(\lambda_2) \cdot \hat{\lambda}_1]^2}$$

and uncertainties of $\hat{\lambda}_1, \hat{\lambda}_2$ are following

$$u(\hat{\lambda}_1) = \sqrt{D_1^{\,2} / 3} \quad u(\lambda_2) = \sqrt{D_2^2 / 3} \tag{18}$$

where

$$D_1 = 0{,}01 \cdot \hat{\lambda}_1 + 0{,}5nm \quad D_2 = 0{,}01 \cdot \lambda_2 + 0{,}5nm \tag{19}$$

Uncertainties of refractive indexes are calculated on the base of equations (11)-(14).
Table 2 contains the values of two wavelengths from the spectrum (Fig.2), corresponding to them refractive indexes, sensitivity coefficients and calculated value of combined uncertainty. The number of extremes on this curve between this chosen points is equal M=4. The respective values of uncertainties ("uncertainty budget") of input quantities are in Table 1.

Table 2.

$\hat{\lambda}_1$ nm	$\hat{n}(\hat{\lambda}_1)$	$\hat{\lambda}_2$ nm	$\hat{n}(\hat{\lambda}_2)$	G_1	G_2	G_3 $\cdot 10^{-9}$	G_4 $\cdot 10^{-9}$
453,0	1,744	695,0	1,773	10,054	-4,342	-2611	1702

$$u_c^2(\hat{d}) = (842{,}5 + 307{,}9 + 492{,}7 + 126{,}2) \cdot 10^{-18}$$

$$\hat{d} = 1540\,nm, \qquad u_c(\hat{d}) = 42{,}06 \cdot 10^{-9} \approx 42nm$$

Analysis of components of combined uncertainty (Table 1 and Table 2) shows that the individual uncertainties are near of the same magnitude.

5. EXPANDED UNCERTAINTY

The final stage is to multiply the combined uncertainty by the chosen coverage factor k in order to obtain an expanded uncertainty. The expanded uncertainty is required to provider an interval which may be expected to encompass a large fraction of the distribution of values which could reasonably be attributed to the measurand (in this case the thickness of thin layer). For most purposes it is recommended that k is set to 2.

$$U(\hat{d}) = 2 \cdot u_c(\hat{d}) = 84\,nm \tag{20}$$

The relative expanded uncertainty is equal

$$U_{rel}(\hat{d}) = \frac{U(\hat{d})}{\hat{d}} = 0{,}055 = 5{,}5\% \qquad (21)$$

The final result can be presented in following form

$$d=(1540 \pm 84)\ nm \qquad (22)$$

for confidence factor $p=0,95$.

6. SUMMARY

Measurement of thickness of thin layer has been achieved by using spectrophotometer method. This approach uses a simplified method described by Manifacier [3,4]. In this presented case the estimation of thickness is base on transmission light curve and the accuracy of measurement depends very strong on accuracy and quality of applied spectrophotometer. There are not repeated observations so only estimation based on other results as manufacturer's data and experience of scientist is applied. This approach is called type B.

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