

## Dynamic evaluation of heat flow reaction calorimeters under reflux conditions<sup>1</sup>

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### Abstract

An alternative method to obtain the product of the overall heat transfer coefficient by the transfer area ( $U \cdot A$ ) in heat flow reaction calorimetry under reflux conditions is exposed. The new method uses the dynamics of the system during non-steady states to obtain  $U \cdot A$  and the effective value of the heat capacity flow ( $\dot{m}_c \cdot c_{p,c}$ ) of the cooling fluid in the condenser. The validation of this method, its statistical evaluation and its limitations are extensively treated using pure solvents and water at their boiling point. The dynamic method gives excellent estimates of  $U \cdot A$ , being less dependent on stirring speed and geometry of the reactor cover than the pseudo-dynamic method. The probability of obtaining an error smaller than 10% is higher than 95%, except for small volumes and very high stirring speeds.

*Keywords:* Dynamics; Heat flow reaction calorimetry; Overall heat transfer coefficient; Reflux

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### 1. Introduction

Heat flow calorimetry has been extensively used to study processes under reflux conditions. Several methods have been proposed to determine the overall heat transfer coefficient, which is required to complete the heat balance of the instrument [1]. In this sense, the method of Nomen et al. [2,3] has special relevance [4,5]. On the other hand, Wiss et al. proposed a procedure to design safe reactions, predicting the behaviour of a reactor at the boiling point [6]. However, there remain two problems to be solved. The first one is the influence of noise, which is inherent to boiling and condensation in small systems and introduces a subjective factor in the definition of baselines. The

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second one is the lack of calorimetric information during the interval of time in which reflux is established. In this sense, when a reaction begins below the boiling point of the mixture and the temperature rises up to boiling, there is a change in the main calorimetric signal, which switch from  $(T_r - T_j)$  to  $\Delta T_c$ . It could be said that the instrument acts now as a hybrid heat flow–balance calorimeter. When reflux starts, the produced vapour has to reach the condenser surface in order to establish the signal. To reach the condenser, it has to heat up all the cold walls of reactor and connections. As a consequence, a delay time is always observed, though an externally heated cover were employed for the reactor.

The goal of this paper is to expose a method to calibrate the product of the overall heat transfer coefficient by the transfer area ( $U \cdot A$ ) under reflux conditions that is based on the dynamic evaluation of the heat balance. This method should avoid to trace baselines, reducing the arbitrariness that it implies. The influence of operating conditions in order to determine the limits and the accuracy of the method is studied. In addition, a method to eliminate or reduce the loss of calorimetric information when reflux is established is exposed.

## 2. Theory

### 2.1. Pseudo-dynamic evaluation of the effective heat capacity flow of cooling fluid through the condenser

Under reflux, the heat balance of the reactor can be written as:

$$\dot{Q}_r + \dot{Q}_c = U \cdot A \cdot (T_r - T_j) + \dot{m}_c \cdot c_{p_c} \cdot \Delta T_c + \dot{Q}_{dos} + \dot{Q}_{loss} \quad (1)$$

If no reaction, other transformations nor dosing take place, Eq. 1 is reduced to:

$$\dot{Q}_c = U \cdot A \cdot (T_r - T_j) + \dot{m}_c \cdot c_{p_c} \cdot \Delta T_c + \dot{Q}_{loss} \quad (2)$$

The effective heat capacity flow of cooling fluid through the condenser,  $\dot{m}_c \cdot c_{p_c}$ , is necessary to complete the heat balance. This parameter can be obtained from an accurate measure of the flow of cooling fluid through the condenser and a good estimation of its specific heat, or determined using a connection of the calibration heater as it is suggested by Nomen et al. [2,3] According to the experience of our group, the first method gives significant errors. The second procedure requires extreme accuracy in tracing baselines, but usually affords better results.

If it is assumed that  $\dot{Q}_{loss}$ ,  $\dot{m}_c \cdot c_{p_c}$ ,  $U \cdot A$ ,  $T_r$  and  $T_j$  are essentially the same before and during the connection of the calibration heater, and after switching it off, Eq. (2) can be written as a linear expression:

$$\Delta T_c = a_0 + a_1 \cdot \dot{Q}_c \quad (3)$$

where:

$$a_0 = \frac{1}{\dot{m}_c \cdot c_{p_c}} \cdot (U \cdot A \cdot (T_r - T_j) - \dot{Q}_{loss}) \quad (4)$$

$$a_1 = \frac{1}{\dot{m}_c \cdot c_{p_c}} \quad (5)$$

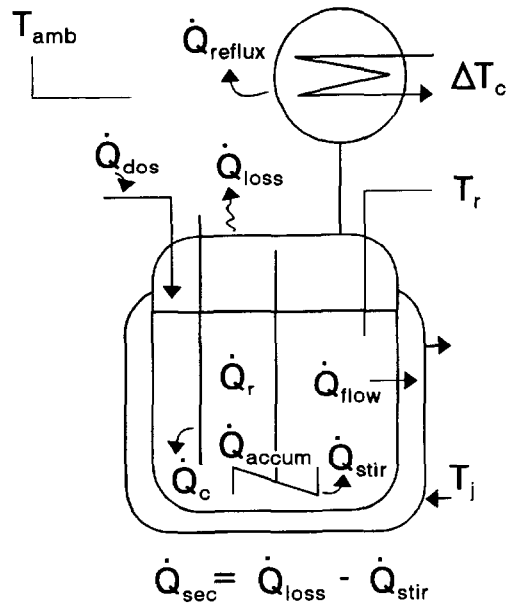


Fig. 1. Terms of the heat balance equation.

Obviously, only two values of  $\dot{Q}_c$  are available, and averages obtained before and after switching on the calibration heater, or before and after switching it off can be used to obtain the desired effective heat capacity flow. According to this method, the baseline is implicit in the calculations and the problems of defining its level could be avoided.

A rough estimation of  $U \cdot A$  can also be obtained if it is assumed that the heat flow through the reactor wall is much larger than the heat losses. Then,  $\dot{Q}_{\text{loss}}$  could be neglected in Eq. (4), giving:

$$U \cdot A \cong \frac{a_0}{a_1} \cdot \frac{1}{(T_r - T_j)_{\text{av}}} \quad (6)$$

The use of this last procedure should be restricted to cases in which heat losses were limited, like when an externally heated reactor cover is used.

## 2.2. Dynamic evaluation of the overall heat transfer coefficient

When, according to the method suggested by Nomen et al. [2,3], a change in the jacket temperature in absence of any transformation ( $\dot{Q}_r = 0$ ,  $\dot{Q}_{\text{dos}} = 0$  and  $\dot{Q}_c = 0$ ) is applied to evaluate  $U \cdot A$ ,  $\dot{Q}_{\text{loss}}$  can be considered constant and the difference between two states is given by:

$$U \cdot A \cdot \Delta(T_j - T_r) = \dot{m}_c \cdot c_{p_c} \cdot \Delta(\Delta T_c) \quad (7)$$

Deriving this equation it is obtained:

$$U \cdot A \cdot (\dot{T}_j - \dot{T}_r) = \dot{m}_c \cdot c_{p_c} \cdot \dot{\Delta T}_c \quad (8)$$

If no composition or pressure changes take place, under reflux conditions,  $T_r$  is essentially constant. Then  $\dot{T}_r \cong 0$ :

$$U \cdot A \cdot \dot{T}_j = \dot{m}_c \cdot c_{p_c} \cdot \dot{\Delta T}_c \quad (9)$$

Eq. (9) establishes a linear relation between the derivatives of  $T_j$  and  $\Delta T_c$ . Thus  $U \cdot A$  could be obtained through a simple regression if the effective value of  $\dot{m}_c \cdot c_{p_c}$  were known. The method of Nomen et al [2,3] uses a step change in  $T_j$  to evaluate  $U \cdot A$ . Due to the fast response of the temperature controller of the heat flow calorimeter, a step change produces only few points during the transition between steady states in order to perform the derivation operation and the above mentioned regression. The noise or fluctuation in the  $\Delta T_c$  signal – which is inherent to the discrete phenomenon of boiling and cannot be suppressed – would make the dynamic procedure not suitable for the desired evaluation.

However, if a ramp change in  $T_j$  were performed, as many points as required could be obtained, and a derivative filter could be applied. A second order Savitzky and Golay derivative filter [7,8] is used in the present work. Figure 2 shows a typical response in  $\Delta T_c$  obtained when a ramp in  $T_j$  is achieved.

Moreover, it can be expected that the  $\Delta T_c$  signal were delayed from the  $T_j$  change. The main cause of this delay is obviously the time of residence of the cooling fluid in the condenser, which is a function of its flow rate and the volume of the glass coil. To cope with this problem, a numerical displacement of data could be applied until the best regression of Eq. (9) were obtained.

### 2.3. Suppression of the lack of calorimetric information when reflux is being established

If the heat generation by stirring can be neglected, no dosing, reactions nor connections of the calibration heater are present, and a linear relation between the heat

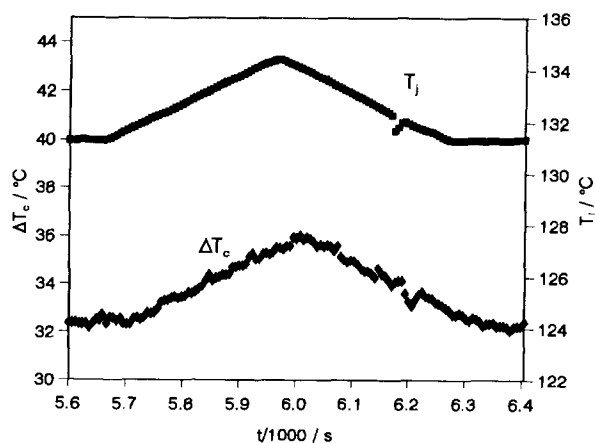


Fig. 2. Response of  $\Delta T_c$  to a double ramp on  $T_j$  during a dynamic calibration.

losses of the calorimeter and room temperature is assumed [9], the heat balance of the reactor when it is heated up to reflux can be written as:

$$O = U \cdot A \cdot (T_r - T_j) + \dot{m}_c \cdot c_{pc} \cdot \Delta T_c + C_{tot} \cdot \dot{T}_r + \alpha \cdot (T_r - T_{amb}) \quad (10)$$

Eq. (10) takes no consideration of the dynamic behaviour of the reactor wall during non steady states, as a consequence, it can be only accepted for very slow temperature ramps. Nevertheless, at the beginning of boiling, vapour has to heat up a big amount of wall's material, and it is very difficult to estimate the amount of mass of wall to be heated and its limit temperatures. This is the main uncertainty that makes quite impossible the complete remotion of the lack of signal before a stable reflux.

However, when a externally heated reactor cover is used and the condenser is directly attached to it, the amount of wall to be heated is a minimum, and only the introduction of a simple delay time in the  $\Delta T_c$  data used to fit Eq. (10) could be enough to reduce the incidence of the lack of signal. Once again, the best regression of Eq. (10) can be used as a criterion to determine such a delay time.

### 3. Experimental

All experiments have been carried out in a Mettler-Toledo RC1<sup>®</sup> reaction calorimeter, with a 2 L glass reactor (AP01).

Most of the experiments were performed with 1 L and 1.7 L of pure acetone, ethanol, di-chloromethane and water, heatable metal-teflon cover, conventional laboratory coil condenser directly attached to the cover, and anchor stirrer.

One experiment was carried out with 1.7 L of water with a non-insulated non-heated glass cover without insulation and the complete reflux-distillation kit of Mettler-Toledo (Fig. 3).

Other experiment was carried out with 1.7 L of water using a gas injection turbine.

The behaviour of the proposed methods has been studied at different stirring speeds (50, 150 and 250 rpm for anchor stirrer and 500, 700 and 800 rpm for gas injection turbine) and different flows of cooling fluid (0.25 and 0.40 L·min<sup>-1</sup> of water).

Reflux conditions were achieved working on adiabatic mode with a constant difference between jacket and reactor temperatures proportional to the boiling point of the solvent (6°C for acetone, 18°C for ethanol, 3°C for di-chloromethane and 31°C for water) [6]. Temperature control is switched to adiabatic mode when the temperature of the solvent is 5°C below its normal boiling point.

### 4. Results and discussion

In order to avoid excessive numerical information, a statistical evaluation is presented in Figs. 3 to 7 and 11. In each figure, every point corresponds to one experimental condition (for example, 1 L of water in AP01 with metal-teflon cover, anchor stirrer at 150 rpm, and a normal condenser directly attached to the cover with 0.25 mL of water as a cooling system). In all figures, acetone, ethanol and di-chloromethane are indicated

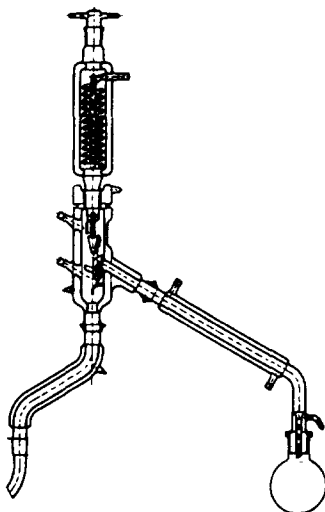


Fig. 3. Reflux-distillation kit (courtesy of Mettler-Toledo).

as organic solvents, and water is mentioned separately because the behaviour of water is completely different to the rest of the substances used in the experiments.

To evaluate the influence of stirring speed, reactor filling, flow of cooling fluid in the condenser, and physico-chemical properties of the reactor contents, on the determination of the heat transfer coefficient and the specific heat, the information is split in many figures and the experimental results are compared with values obtained by the standard RC1 calibrations for the same conditions.

#### 4.1. Determination of the effective value of $\dot{m}_c \cdot c_{Pc}$

The determination of  $\dot{m}_c \cdot c_{Pc}$  has been performed using the above described method (Eq. (3)) and its results compared to those from the integral method. Fig. 4 shows the relative discrepancy between both evaluations with respect to the stirring speed. In many cases this difference overcomes the error that could be accepted as suitable (10%). The special transport properties of vapour of water produce abnormal results for this compound. However, it is difficult to verify which of the methods gives the more reliable value. Moreover, it has to be noted that the result produced by the pseudo-dynamic method is always more similar to that obtained by a direct measure of the flow of coolant than that obtained by the integral method.

The first observation is that values from the pseudo-dynamic method are lower than those from the integral one. The most dramatic case is water, for which the level of noise in  $\Delta T_c$  is the highest observed, diffculting the definition of baselines for the integral method. It can be also noticed in Fig. 4 that the relative difference between both values slightly decreases when stirring speed grows up. This observation is explained by the

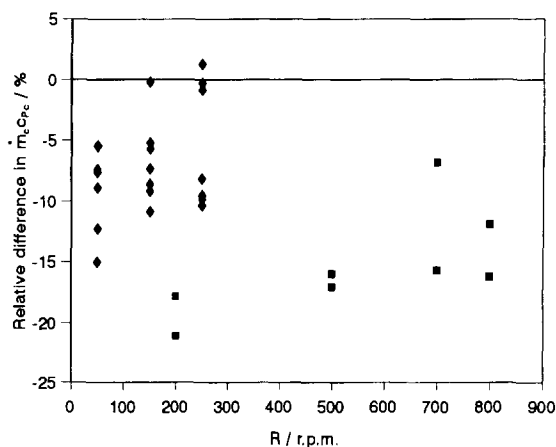


Fig. 4. Relative difference between  $\dot{m}_c \cdot c_{p_c}$  calculations performed by integral and pseudo-dynamic methods as a function of the stirring speed: ◆, pure acetone, ethanol, di-chloromethane; ■, water.

reduction of noise in  $\Delta T_c$  signal when high stirring speeds are used, and the size of bubbles is essentially reduced. In this case, the uncertainty in tracing the baseline for the integral method is cut back, and results from both methods would become a little more equivalent.

For a constant flow of cooling fluid,  $\dot{m}_c$ , values of  $\dot{m}_c \cdot c_{p_c}$  obtained by any method should be essentially equal. However, for low stirring speeds (50 rpm. and anchor stirrer), lower values of  $\dot{m}_c \cdot c_{p_c}$  are obtained than when high stirring speeds are used. This effect could be due to the evaporation of the condensed liquid falling from the condenser, taking place the contact between it and the hot non-wetted wall over the agitated liquid. This effect, known as vapour amplification [10], would be higher at low stirring speeds, because the non-wetted surface area is larger. On the other hand, when high stirring rates are used, not only the amount of wetted wall increases, but also the fluctuations that are due to boiling are reduced. In this case, the vapour amplification is minimized and base line tracing for the integral method is easier, and both methods should produce equivalent results.

Fig. 5 shows the discrepancy between both methods with respect to the volume of liquid. It seems evident that better results are produced when 1 L of solvent or water are used. For such a volume, the wetted wall is always smaller than the jacket wall. For higher volumes (1.7 L), and as a normal consequence of the vortex induced by stirring, the wetted surface can be larger than the jacket one, producing an abnormal behaviour of the  $U \cdot A$  term of the heat balance assumed by the pseudo-dynamic method.

Table 1 shows a summary of the probability that the relative difference between the calculated values of  $\dot{m}_c \cdot c_{p_c}$  from integral and pseudo-dynamic methods were lower than 10%, assuming a normal distribution of discrepancies.

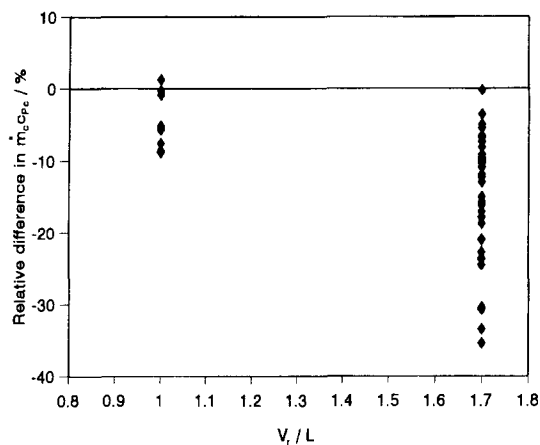


Fig. 5. Relative difference between  $\dot{m}_c \cdot c_{pc}$  calculations performed by integral and pseudo-dynamic methods as a function of the reaction volume.

Table 1

Probability of that the relative difference in  $\dot{m}_c \cdot c_{pc}$  calculations performed by integral and pseudo-dynamic methods were lower than 10%

Operating conditions	$e$ (%)	$\sigma$ (%)	$\Pr(e \leq 10\%)$ (%)
R = 50 rpm	-8.89	3.30	63.3
R = 150 rpm	-6.75	3.23	84.4
R = 250 rpm	-5.41	4.80	83.1
$\dot{m}_c = 0.25 \text{ L} \cdot \text{min}^{-1}$	-7.65	3.95	72.6
$\dot{m}_c = 0.40 \text{ L} \cdot \text{min}^{-1}$	-6.17	4.15	82.1

#### 4.2. Pseudo-dynamic evaluation of $U \cdot A$

Applying Eq. (6) it is possible to try to obtain rough estimations of  $U \cdot A$  using only a single connection of  $\dot{Q}_c$ . Fig. 6 shows the error of this procedure as a function of the stirring speed. Only a very small interdependence between these two variables would be noted. Although in most of the cases relative errors are lower than 10%, it could be noticed that the error at 50 rpm is a little higher than for other stirring speeds, as it is observed in experiences below reflux.

An increase in the error of  $U \cdot A$  calculations is observed when the lid is not insulated. In this case, heat losses and heat flow are put together into the baseline of the signal. Since it is not possible to isolate both values in the dynamic evaluation,  $U \cdot A$  calculation is affected by these heat losses.

Table 2 summarizes the statistical analysis of the method as the probability of obtaining errors lower than 10%. It can be observed that when the non-insulated non-heatable glass cover is used  $U \cdot A$  calculations with the calibration heater under



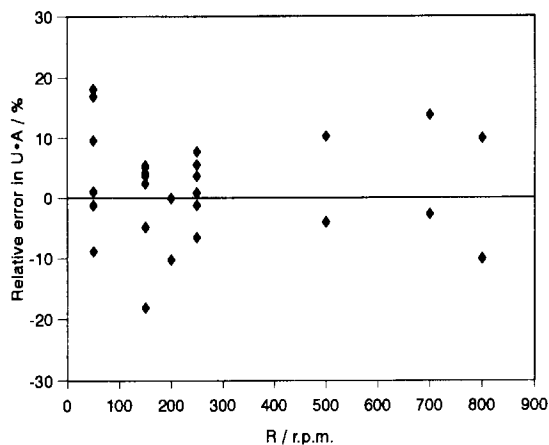


Fig. 6. Relative error between  $U \cdot A$  calculations performed by integral and pseudo-dynamic methods as a function of stirring speed.

Table 2

Probability of that the relative difference in  $U \cdot A$  calculations performed by integral and pseudo-dynamic methods were lower than 10%.

Reactor cover	Operating conditions	$\bar{e}$ (%)	$\sigma$ (%)	$\Pr(e \leq 10\%)$ (%)
Metal-Teflon <sup>®</sup>	R = 50 rpm	-8.89	3.30	63.3
	R = 150 rpm	-6.75	3.23	84.4
	R = 250 rpm	-5.41	4.80	83.1
	mean value	-7.65	3.95	72.6
Glass	mean value	-6.17	4.15	82.1

reflux conditions are not reliable. On the other hand,  $U \cdot A$  calculations are favoured by higher stirring speeds.

#### 4.3. Dynamic evaluation of $U \cdot A$

To analyze the accuracy of the  $U \cdot A$  calibration method performing ramps in  $T_j$ , some experiments changing the most influencing variables are carried out. Fig. 7 shows the effect of the stirring speed.

It can be observed that stirring speed is not as important as it is in the  $\dot{Q}_c$  calibration. This fact could be explained by the formation of bubbles near to the wall, the best agitated area [11], instead of at the calibration heater, which is placed in the worst homogenized zone of the reactor.

It seems that highest errors and deviations in  $U \cdot A$  correspond to the highest stirring speeds, but most of them come from experiments performed with only 1 L of volume

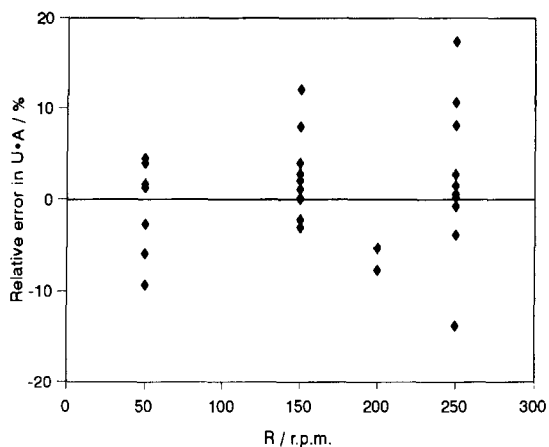


Fig. 7. Relative error between  $U \cdot A$  calculations performed by integral ( $T_j$  ramps) and pseudo-dynamic methods as a function of stirring speed.

(Fig. 8). It is obvious that high stirring speeds produce bigger vortices and variations in the wall wetted surface, which are more significant when small volumes are used.

Table 3 summarizes the statistical analysis of the dynamic evaluation method as the probability of obtaining errors lower than 10%. Except for very high stirring speeds, errors are very small.

#### 4.4 Delay time of a calibration in dynamic conditions of $T_j$

The delay time of measures under reflux conditions is directly related with the time of residence of the cooling fluid in the condenser, which corresponds to the time that it

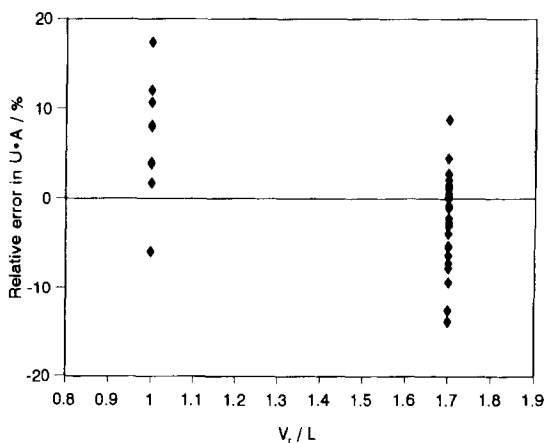


Fig. 8. Relative error between  $U \cdot A$  calculations performed by integral ( $T_j$  ramps) and pseudo-dynamic methods as a function of the reaction volume.

Table 3

Probability of that the relative difference between  $U \cdot A$  calculations performed by integral and pseudo-dynamic methods were lower than 10%. Calibration with  $T_j$  ramps.

Operating conditions	e (%)	$\sigma$ (%)	Pr( $e \leq 10\%$ ) (%)
All experiments	0.13	6.68	86.5
R = 50 rpm	-0.93	4.81	95.9
R = 150 rpm	2.49	4.37	95.5
R = 250 rpm	2.28	8.04	76.9
mean vlaue	-6.17	4.15	82.1

needs to reach the outlet temperature sensor of the condenser. Fig. 9 confirms the expected functionality between the delay time and the mass flow of cooling fluid in the condenser.

#### 4.5. Suppression of the lack of calorimetric information when reflux has being stabilished

In this case the delay time is related to the amount of energy that has to be supplied by vapour to heat up all the elements in the path from the liquid surface to the condenser surface. As it can be seen in Fig. 10, this fact produces a lack of information during the change of the main calorimetric signals. To mitigate this problem, a simple numerical displacement of the  $\Delta T_c$  signal to adjust it with the other signals of the heat balance equation is proposed. Fig. 10 also shows the result of such a operation.

In order to verify the goodness of the method, several experiments were carried out using pure solvents. In all of them, it is observed an acceptable reduction of the transient reflux as well as the power of the dynamic method to calibrate the system. The

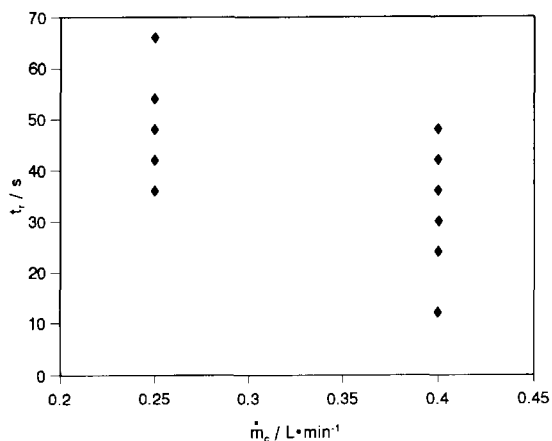


Fig. 9. Delay time of the system under reflux conditions as a function of the flow rate of the cooling liquid,  $\dot{m}_c$ , measured with a flowmeter.

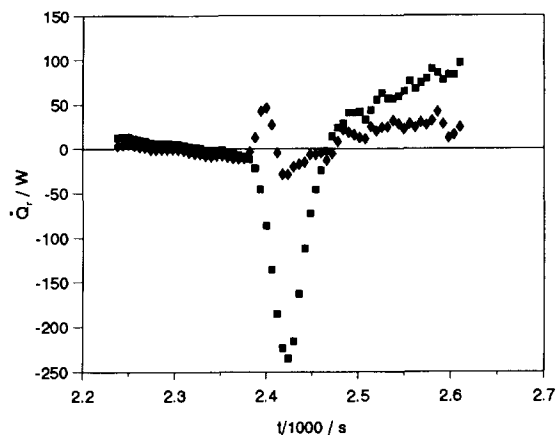


Fig. 10.  $\dot{Q}_r$  calculated with experimental and numerical displaced  $\Delta T_c$  data:  $\blacklozenge$ , experimental data;  $\blacksquare$ , corrected signal.

total elimination of the transient reflux has been quite impossible. This fact could be due to the sampling period of the instrument that impose a delay time, which must be an integer multiple of the sampling interval.

As it can be expected, delay times are essentially a function of the experimental conditions ( $\dot{Q}_{\text{flow}}$ ), and geometry of the reflux system. Fig. 11 compares the estimated delay times using the heatable cover with a directly attached condenser for different pure solvents, and using the standard glass cover with the complete reflux-distillation kit for the cases of water and ethanol. For the first configuration, delay times between

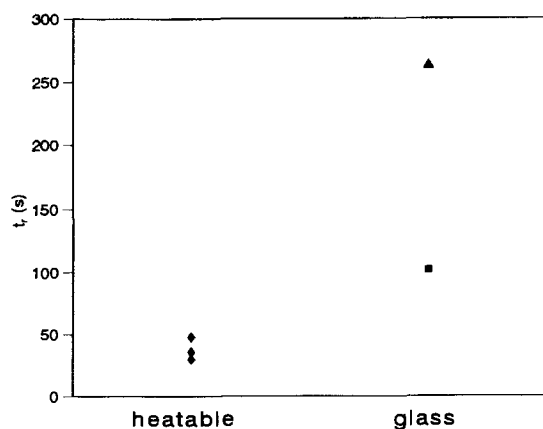


Fig. 11. Delay time in reflux transient for experiments carried out with the condenser attached in a metal-  
teflon cover and with the complete reflux-distillation device:  $\blacklozenge$ , pure acetone, ethanol, di-chloromethane;  $\blacksquare$ ,  
water;  $\blacktriangle$ , ethanol.

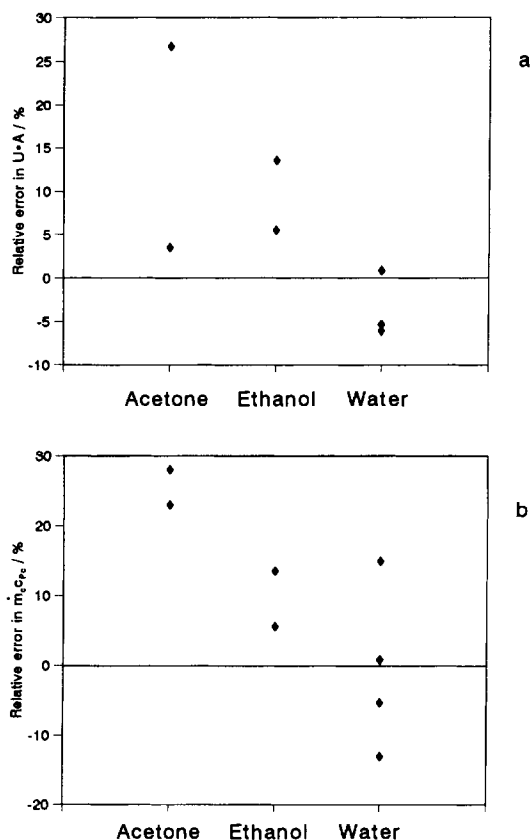


Fig. 12. Relative difference in calorimetric parameters calculated with the proposed method as a function of the solvent: a Relative error in  $U \cdot A$ ; b, Relative error in  $c_p$ .

30 and 48 seconds are measured with a very small dispersion. However, for the second, the physico-chemical properties (boiling point, vapour pressure, latent heat, ...) of the solvent seem to have a strong influence.

This observation is parallel to that the dispersion evaluating  $U \cdot A$  under reflux by any method is higher when the complete reflux-distillation kit is used. As a consequence, to obtain reliable results in reflux experiments, it could be strongly advised to avoid using unnecessary connections between the reactor lid and the condenser, and to mitigate heat losses using a heatable reactor cover.

Eq. (10) also gives an estimation of  $U \cdot A$  and  $c_p$ , during the ramp that leads the system to the stationary reflux. Obviously, it is not possible to estimate the heat losses during the change of temperature, limiting the scope of validity of this method. Figs. 12(a) and (b) show the relative errors of the results of such a evaluation.

In all of the cases, discrepancies between values obtained by integral and dynamic methods decrease when higher boiling point solvents are used. However, the dispersion of results is also bigger in this case.

## 5. Conclusions

The pseudo-dynamic method ( $\dot{Q}_c$  step) gives excellent results of  $\dot{m}_c \cdot c_{p,c}$ . Low stirring speeds should be avoided due to the vapour amplification, that produces underestimated  $\dot{m}_c \cdot c_{p,c}$  values. On the other hand, high stirring speeds can favour the reduction of the size of bubbles, causing a more homogeneous boiling, and giving more reliable  $\dot{m}_c \cdot c_{p,c}$  estimations. High values of  $\dot{m}_c$  slightly decrease the discrepancies of estimates from pseudo-dynamic and integral methods. However, the pseudo-dynamic method should be applied carefully to obtain  $U \cdot A$  because the strong influence of stirring and geometry of the reactor cover, which is consequence of the assumptions of the model.

The dynamic method (ramp in  $T_i$ ) gives excellent estimates of  $U \cdot A$ , being less dependent on stirring speed and geometry of the reactor cover than the above method. The probability of obtaining an error smaller than 10% is higher than 95%, except for small volumes and very high stirring speeds that can reduce this probability to 76% in the case of an anchor stirrer without baffles. The best results with this configuration are obtained with a stirring speed of 150 rpm.

Both methods avoid the tracing of baselines, mitigating the effect of noise.

To reduce the loss of calorimetric information when reflux is being established, a simple numerical displacement of  $\Delta T_c$  signal gives good results when a heatable cover is used. Delay times are in the range of 30 to 48 s.

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**Symbols**

$A$	Heat exchange area/m <sup>2</sup>
$c_{Pc}$	Specific heat of the cooling liquid/J kg <sup>-1</sup> K <sup>-1</sup>
$c_{Pr}$	Specific heat of the reaction mixture/J kg <sup>-1</sup> K <sup>-1</sup>
$C_{tot}$	Heat capacity of the reaction system/J K <sup>-1</sup>
$e$	Relative difference/%
$\dot{m}_c$	Flow rate of the cooling liquid/kg s <sup>-1</sup>
$Pr$	Probability/%
$\dot{Q}_c$	Calibration power/W
$\dot{Q}_{dos}$	Power required to put the dosed reactant at the same temperature as $T_r$ /W
$\dot{Q}_{flow}$	Heat flow through the wall of the reactor/W
$\dot{Q}_r$	Reaction power / W.
$\dot{Q}_{reflux}$	Heat flow in the condenser/W
$\dot{Q}_{loss}$	Heat losses/W
$R$	Stirring speed/rpm
$T_{amb}$	Room temperature/K or °C
$T_j$	Jacket temperature/K or °C
$\dot{T}_j$	Derivative of reactor temperature/K s <sup>-1</sup>
$T_r$	Reactor temperature/K or °C
$\dot{T}_r$	Derivative of jacket temperature/K s <sup>-1</sup>
$t$	Time/s
$t_r$	Delay time/s
$U$	Overall heat transfer coefficient/W m <sup>-2</sup> ·K <sup>-1</sup>
$V_r$	Volume of the reaction mass/L

*Greek symbols*

$\alpha$	Power losses coefficient/W K <sup>-1</sup>
$\sigma$	Standard deviation/%
$\Delta T_c$	Difference between inlet and outlet temperature of the condenser liquid/K
$\dot{\Delta T}_c$	Derivative of $\Delta T_c$ /K s <sup>-1</sup>
$\Delta T$	Mean value of the $T_r - T_j$ /K