



# A HYBRID RESPIROMETRIC METHOD FOR MORE RELIABLE ASSESSMENT OF ACTIVATED SLUDGE MODEL PARAMETER

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## ABSTRACT

A novel hybrid respirometric principle is proposed that is particularly suited to sludge and wastewater characterisation in the context of activated sludge process models. Advantages of two respirometric principles are combined and their disadvantages eliminated to increase measuring frequency and precision. Emphasis is put on decreasing the bias in parameter estimates that results from the use of unreliable sensor constants in the calculation of respiration rates. To this end checks for dissolved oxygen probes, aeration systems and pumps are built into the respirometer's operation. Checks are to be run while the respirometric batch experiment is conducted so that between-experiment variation is eliminated and within-experiment variation is minimised. It is also stressed that a combined sensor/process model should be used to estimate the process parameters rather than a sequential procedure in which the sensor constants are first used to calculate respiration rates, that are subsequently used for sludge and wastewater characterisation. Finally, three possible practical implementations of the new principle are discussed in relation to maximising parameter estimation accuracy. © 1998 Published by Elsevier Science Ltd. All rights reserved

## KEYWORDS

Activated sludge; error diagnosis; identification; mathematical modelling; respirometry; sensors.

## INTRODUCTION

Batch respirometric experiments are a valuable tool for the characterisation of wastewater and sludge properties within a modelling context (Henze *et al.*, 1995). To collect respiration rate data several respirometric measuring principles developed in the past are at the disposal of the modeller. These principles can be classified into a limited number of basic principles according to only two criteria: (1) the phase where oxygen is measured, gas or liquid; (2) the flow regime of either gas or liquid phase, flowing or static (Spanjers *et al.*, 1996). In all cases, the respiration rate is obtained from oxygen mass balances over these phases. When the oxygen concentration is measured in the liquid phase (dissolved oxygen, DO) only the mass balance over this phase needs to be considered. Recently, two of these liquid phase principles were successfully applied in assessing activated sludge model parameters (Spanjers and Vanrolleghem, 1995; Vanrolleghem *et al.*, 1995).

The first (flowing gas/static liquid) principle considered calculates the respiration rate from the DO mass balance in an aerated activated sludge reactor. To allow calculation of respiration rate, the oxygen mass transfer coefficient ( $K_{La}$ ) must be known.  $K_{La}$  is obtained from an estimation procedure that uses the temporary disturbance of the DO concentration after addition of substrate. Disadvantage of this principle is the low accuracy of the estimated  $K_{La}$  where the oxidation of slowly biodegradable material is still ongoing when the reaeration has started.

The other (no gas/flowing liquid) principle calculates the respiration rate from the DO mass balance over a non-aerated reactor. This principle requires knowledge of flow rate and reactor volume (to calculate retention time) and DO measurements at two different locations: at the inlet and at the outlet of the reactor. In order to obtain reliable DO measurements a flow switching technique that allows one to work with only a single DO-probe for both measurements has been advocated. This, however, limits the frequency at which the respiration rate data can be obtained, due to the time it takes for the DO-probe to reach equilibrium with the mixed liquor DO.

Characterisation of activated sludge and wastewater in the context of activated sludge process models requires respirometric measurements with both high accuracy and measuring frequency. The respirometric principles described above cannot meet these two requirements in all cases. Table 1 compares the two principles in terms of these properties. It is obvious that a respirometric technique combining their advantages and eliminating their disadvantages will offer excellent opportunities for activated sludge and wastewater characterisation.

Table 1. Comparison of two respirometric principles in view of applicability in experiments for estimating activated sludge process model parameters

	Principle used to assess respiration rate	
	DO in continuously aerated activated sludge	DO fall over closed flow-through cell
Advantage	high measuring frequency	accurate for complex substrates
Disadvantage	not accurate for complex substrates	low measuring frequency

The work described below concerns a new hybrid respirometric principle obtained by combining the two different respirometric principles mentioned above. The paper first describes the basic set-up of the new hybrid sensor and the mass balances on which the characterisation of the sludge and wastewater will be based. Next, the way the model parameters are to be assessed is presented and contrasted with the traditional approach for parameter estimation from respirometric data collected with the respirometric principles of Table 1. Next, the possibilities the new set-up provides for sensor calibration, error detection and diagnosis are given and the positive impact these have on parameter estimate accuracy is illustrated. Three implementations of the basic principle are subsequently discussed with respect to improving estimation accuracy. At this moment these sensors are under construction and practical evaluation will be reported shortly.

### THE HYBRID RESPIROMETRIC PRINCIPLE

Figure 1 schematises the hybrid principle. Two reactors, an aerated and a non-aerated one, are connected in such a way that activated sludge can be circulated continuously from one to the other. The dynamics of the DO concentration in both reactors can be described by the following mass balance:

$$\frac{dS_o}{dt} = K_{La} \cdot (S_o^* - S_o) + \frac{Q}{V} (S_o^{in} - S_o) - r_o \quad (1)$$

Obviously, in the non-aerated reactor  $K_{La}$  is zero and the first term on the right-hand side cancels. Also, all state variables and parameters ( $S_o$ ,  $K_{La}$ ,  $S_o^*$ ,  $Q$ ,  $V$ ,  $r_o$ ) correspond to the reactor under consideration and  $S_o^{in}$  relates to the other reactor. In general, respiration is due to two processes, growth and decay, typically modelled as:

$$r_o = \frac{(1 - Y_H)}{Y_H} r_{\text{growth}} + (1 - f_p) r_{\text{decay}} \tag{2}$$

$$r_{\text{growth}} = \mu_{\text{max}} \frac{S_S}{K_S + S_S} X_{\text{BH}} \tag{3}$$

$$r_{\text{decay}} = b_H X_{\text{BH}} \tag{4}$$

To allow respiration rate calculation, the substrate concentration  $S_S$  and the active biomass concentration  $X_{\text{BH}}$  must be known. Mass balancing:

$$\frac{dS_S}{dt} = \frac{Q}{V} (S_S^{\text{in}} - S_S) - \frac{r_{\text{growth}}}{Y_H} + r_{\text{hydrolysis}} \tag{5}$$

$$\frac{dX_{\text{BH}}}{dt} = \frac{Q}{V} (X_{\text{BH}}^{\text{in}} - X_{\text{BH}}) + r_{\text{growth}} - r_{\text{decay}} \tag{6}$$

provides this information. Note that no specification of the hydrolysis process, or nitrification and other processes are given for brevity. A typical batch respiration rate profile (respirogram) is depicted in Figure 2.

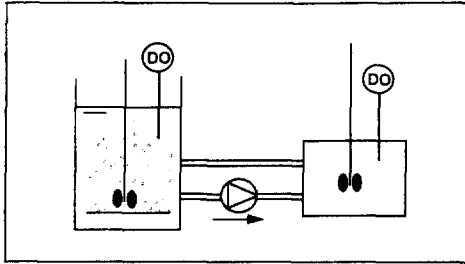


Figure 1. Schematic diagram of the hybrid respirometer consisting of an aerated and non-aerated reactor.

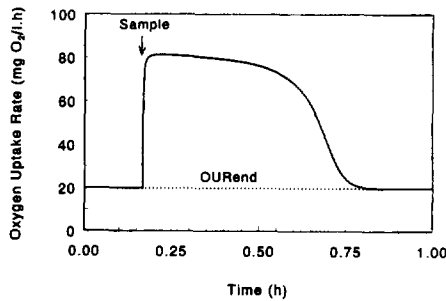


Figure 2. Typical respirogram obtained after pulse injection of a wastewater sample to endogenously respiring activated sludge.

In a batch experiment the sample is typically injected in the open aerated reactor only. This implies that the initial conditions in the two reactors are different, i.e.

$$\begin{aligned} S_S \text{ aerated reactor}(t=0) &= S_S \text{ sample } \sqrt{\text{sample}/\text{vaerated reactor}} \\ S_S \text{ non-aerated reactor}(t=0) &= 0 \end{aligned}$$

The mass balances, however, take this perfectly into account and allow one to describe the respiration rates and, hence, the resulting DO concentrations in both reactors (provided the reactors are ideally mixed).

From this mass balancing one can already observe that the set-up results in two mass balances where respiration rate occurs (for the DO balances in the aerated and non-aerated reactor, respectively). Therefore, when these two DO concentrations are measured as in the proposed set-up, one in fact obtains two independent measurements of the respiration rate. Obviously, the quality of the estimates of the sludge and wastewater characteristics obtained under these conditions will benefit from this, as discussed in detail below.

### PARAMETER ESTIMATION FROM RESPIROMETRIC DATA

Traditionally, parameter estimation from respirometric data starts with the calculation of respiration rates from the raw DO data using mass balances (1). Subsequently, a mathematical model, (2)-(4), that describes the biological conversions and their relation to the oxygen uptake rates is fitted to these data, producing the desired parameter values. In the sequel this procedure is critically evaluated with special emphasis on error propagation.

#### Calculation of respiration rate

For each respirometric principle methods have been developed to generate respiration rate data from the raw (gas or liquid phase) oxygen measurements. These methods make use of mass balances like (1). For the two liquid phase principles mentioned before, the respiration rates are calculated in the following way.

In the first principle the second term on the right hand side of (1) vanishes and respiration rates follow from:

$$\text{OUR} = -\frac{dS_o}{dt} + K_L a \cdot (S_o^* - S_o) \quad (7)$$

It is obvious that values for  $K_L a$  and  $S_o^*$  are required. Methods were developed to estimate these from either separate experiments (with the danger of between-experiment variation) or from the ultimate phase of the batch experiment itself (Vanrollegheem *et al.*, 1991). In addition, values must be given to the time derivative of the DO-data,  $dS_o/dt$ . Typically, these derivatives are obtained by simply backward differencing the DO-values:

$$\frac{dS_o(t)}{dt} = \frac{(S_o(t) - S_o(t - \Delta t))}{\Delta t} \quad (8)$$

This approximation may be quite rough (especially for low measuring frequency, i.e. large  $\Delta t$ ) and substantial deviations from the real respiration rate may be induced as a result. More advanced calculation of the slopes is therefore advised, e.g. by using filtering methods (Lindberg & Carlsson, 1996).

Respiration rates can also be obtained from DO data collected following the second respirometric principle mentioned above (no gas, flowing liquid). Here the first term of (1) vanishes and respiration rates are calculated from:

$$\text{OUR} = -\frac{dS_o}{dt} + \frac{Q}{V}(S_o^{\text{in}} - S_o) \quad (9)$$

Here the dilution rate  $Q/V$  must be known and is mostly obtained from a pump flow rate calibration prior to the experiments. Again, the rate of change of the DO concentration in the closed reactor must be determined. Here too, typically, backward differencing is adopted.

#### Effect of DO probe dynamics

In the calculation of respiration rates it is often assumed that an ideal oxygen probe is used, i.e. one that reacts infinitely fast on a change in DO concentration. While this assumption may be reasonable for activated sludge monitoring, it is often not allowed for batch experiments where the changes in respiration

rate are purposefully made fast to obtain maximum information on the biokinetics. The time constant of the probes is then no longer negligible compared to the process time constants and, consequently, the calculated respiration rates may deviate from the real respiration rates. To deal with this, the oxygen probe dynamics should be taken into account before equations (7-9) are used for calculation of the respiration rate. Using the generally accepted first order model describing the dynamics of DO probes (Lee & Tsao, 1979)

$$\frac{dS_o^{\text{probe}}}{dt} = \frac{(S_o - S_o^{\text{probe}})}{\tau_{\text{probe}}} \quad (10)$$

one can calculate the true DO concentration in the reactors by using the probe output and, for instance, a backward difference approximation of the change of this probe output:

$$S_o = S_o^{\text{probe}} + \tau_{\text{probe}} \frac{dS_o^{\text{probe}}}{dt} \quad (11)$$

The DO probe time constant  $\tau_{\text{probe}}$  is typically obtained from a probe calibration test using a step change in the oxygen concentration (Vanrolleghem *et al.*, 1991), but may as well be obtained during the batch experiment itself provided sensor set-up is properly done (Spanjers & Olsson, 1992).

### Errors in calculated respiration rates

Summarising, to calculate the respiration rates following either of the respirometric principles, one uses two approximations of time derivatives (equations 8 and 11), whose errors accumulate. In addition, device constants (flow, aeration efficiency) are required whose errors also end up in the calculated respiration rate. The values of these device constants are typically obtained from previous sensor calibration experiments and not from the batch experiment that is bound to be used for parameter estimation. This may also create errors due to slight differences in sensor constants between batch experiments and sensor calibrations.

To reduce such between-experiment errors, one should try to create experimental conditions during the batch experiments that allow deduction of the device constants while the experiment is running. Such methods have already been developed for the probe constant (Spanjers & Olsson, 1992) and the  $K_{La}$ -value (Vanrolleghem *et al.*, 1991). Below, it will be shown that the operating procedure of the hybrid respirometer was purposefully designed to accommodate estimation of all the necessary sensor constants while the experiments run. In this way, no between-experiment errors occur and the within-experiment errors originating from changes in device constants can be minimised.

### Parameter estimation from respirometric data

In contrast to the numerous applications of respiration rate data as such (Spanjers *et al.*, 1996), the aim of respirometry pursued here is to deduce values for activated sludge model parameters. Hence, one should consider a respirometer no longer as a sensor producing respiration rates, but as a device that provides raw data that are related to the respiration of organisms. Therefore it allows one to estimate the model parameters describing the conversions that result in oxygen uptake. This, however, does not necessarily mean that respiration rate has to be calculated explicitly. Below it will be illustrated that it is better to fit an overall model (describing both the sensor's operation and the biological processes) on the raw data (DO) rather than to calculate some derived variable ( $r_o$ ) first (using some error-prone approximations) and then fit a reduced model (only incorporating the biological conversions) to these derived data. Indeed, when fixing sensor constants such as  $K_{La}$  or  $Q/V$  to a wrong value, an apparently reliable respirogram will be obtained, but calculated respiration rates will be proportionally affected by these erroneous sensor constants (see eqs 7 and 9, respectively). Hence, errors will propagate to, for example, estimates of the maximum growth rate,  $\mu_{\text{max}}$ , because (by combining (2), (3), (7) and (9) for excess substrate, i.e.  $S_s \gg K_s$ ) one obtains:

$$\mu_{\text{max}} = Y_H \frac{K_{La}(S_o^* - S_o) - \frac{dS_o}{dt} - (1 - f_p)r_{\text{decay}}}{(1 - Y_H)X_{\text{BH}}} \quad \text{or} \quad \mu_{\text{max}} = Y_H \frac{Q}{V} \frac{(S_o^{\text{in}} - S_o) - \frac{dS_o}{dt} - (1 - f_p)r_{\text{decay}}}{(1 - Y_H)X_{\text{BH}}} \quad (12)$$

from which it is quite clear how errors in  $K_{La}$  (or  $Q/V$ ) affect the estimates of  $\mu_{\text{max}}$ .

To substantiate this, reference is made to the procedure used for the (flowing gas/static liquid) respirometric principle in which  $K_La$  is estimated first and the respirogram is subsequently calculated (Vanrollegheem *et al.*, 1991). First, a subset of the DO data of a respirometric experiment is taken and  $K_La$  is obtained from the best fit of a first order reaeration model. Subsequently, respiration rates are calculated for the  $K_La$  value found. Finally, a biodegradation model, (2)-(4), is fitted to the respiration rates to find best estimates for, for instance,  $\mu_{\max}$ . Hence, a data analysis procedure is followed in which estimates of the overall parameter set  $\{K_La, \mu_{\max}\}$  are obtained sequentially. However, the appropriateness of the sequentially estimated parameters should be evaluated by using their values in the overall model that describes both the sensor and biological dynamics. This model simulates the original DO dataset obtained in the respirometric experiment. It is a fact that the fit of this description using the *sequential parameter estimates* (first  $K_La$ , then  $\mu_{\max}$ ) can at most (but not likely) be as good as the fit of a description that uses a parameter set obtained from a *simultaneous estimation* of the  $\{K_La, \mu_{\max}\}$  parameter set on the complete DO data (without intermediate calculation of OUR data). Hence, the latter parameter set is the more appropriate as it best describes the original dataset.

Another reason why it is advisable to use an overall sensor/process model to assess model parameters instead of first calculating respiration rates and applying a reduced (bioprocess only) model to these, is the following. For certain respirometric set-ups it may be quite complicated to explicitly calculate the respiration rates from the raw DO data as the respiration rate is only implicitly involved in the DO dynamics occurring in the respirometer. Moreover, as the biological model, e.g. (2)-(6), has to be written down anyway to interpret the biological response, adding the sensor model may only require a minor effort and the parameter estimates that are pursued can be obtained at once. The only advantage for the user in calculating respiration rates explicitly is the possibility to graphically represent these (viewing a respirogram is illustrative!) and more easily come up with a biological interpretation of the phenomena (Coen *et al.*, 1997).

Against the use of a combined sensor/process model and simultaneous estimation of sensor and bioprocess characteristics speak the problems to be expected by the increased number of parameters to be estimated at once. This may cause problems to the numerical optimisation algorithms and may substantially increase calculation times (Vanrollegheem & Keesman, 1996). However, careful use of the algorithms and proper choice of the initial estimates of the parameters can alleviate such problems. For instance, one can use the sequential estimation procedure (including, for instance, the explicit calculation of respiration rates) to obtain good initial estimates of the parameters that are used by an overall estimation step with the full model.

#### POSSIBLE IMPLEMENTATIONS OF THE HYBRID RESPIROMETRIC PRINCIPLE

With the combined sensor/process model, one can make predictions of the DO concentrations and compare these with the experimental findings. Model parameters are adjusted by an algorithm until predictions and data correspond best, resulting in their estimates. The quality of these estimates depends, however, on the quantity and quality of the raw data. Below, three possible implementations will be given that aim to achieve this. Moreover, the reliability of the measurement set-up is increased by introducing checks into the operation of the hybrid respirometer that help to detect and where possible correct changes in sensor characteristics, such as flow and mass transfer rates, DO-probe fouling and drift. In this way within- and between-experiment errors can be minimised.

##### Implementation 1

The first implementation (Fig. 3) can be obtained pragmatically by connecting a respirometer working according to the first principle (gas flow/no liquid flow) *in casu* a RODTOX device (Kelma bvba, Belgium) and a respirometer of the second principle (no gas flow/liquid flow) *in casu* a RA-1000 device (Manotherm, The Netherlands).

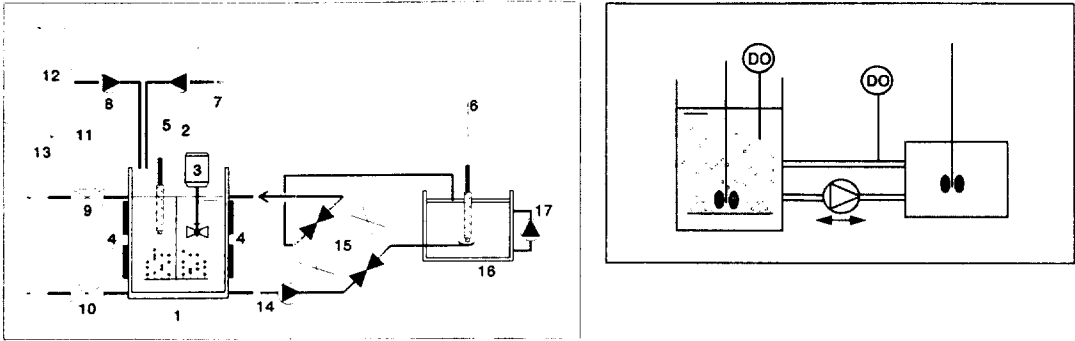


Figure 3. (left) Implementation 1 of the hybrid respirometric principle by combining a RODTOX (item 1) and a RA-1000 device (item 16). (right) Schematic diagram of implementations 1 and 2.

The operation of the non-aerated respirometer is based on an intermittent change of the flow direction with a fixed period (typically 1 minute) so that the DO-probe (item 6 in Fig. 3) alternatively measures the DO concentration of the sludge entering the closed reactor and that of the sludge leaving the reactor (Spanjers & Olsson, 1992). The other probe (item 5) measures the DO concentration in the aerated reactor and is not affected by the changing flow direction through the other reactor. When a sample of wastewater is introduced in the open reactor the two probes will record a dynamic DO profile until all biodegradable substrate is oxidized. Raw data obtained from this implementation look like those in Figure 4.

With DO probe (item 5) the DO concentration can continuously be measured in the aerated mixed liquor (DO<sub>in</sub> in Fig. 4). So far, the DO data obtained from the probe installed at the closed respiration chamber are less frequently collected because the DO value for this probe is only reported by the respirometer when equilibrium is reached after the step change it subsides by periodic flow redirection (see circles in Fig. 4).

It must be obvious, however, that the DO data of probe (item 6) could in fact be collected continuously (oscillating line in Fig. 4) and could be used to improve the quality of the parameter estimation. However, to achieve this improvement, the dynamics of the DO probe subject to the intermittent step changes in the actual DO concentration should be modelled. Spanjers and Olsson (1992) have shown that this can be achieved. The new approach suggested here for this first implementation is that a first order probe model would be included in the combined sensor/process model used for description of the DO data. The time constant of this probe can then be included in the set of parameters to be estimated and can therefore be used directly to detect, for instance, probe fouling. Note that the probe installed in the aerated reactor cannot be checked in this way.

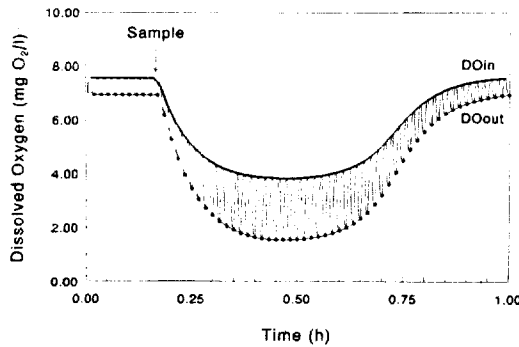


Figure 4. DO data obtained from the two DO probes in implementation 1 of the hybrid respirometer.

The fact that two DO probes are used that at regular times (typically every minute) measure the same DO concentration, allows one to check one probe with the other and detect any deviation between them. In this way an alarm on probe failure can be generated. If, in addition, one of the probes, most appropriately the one installed in the aerated reactor, is also equipped with an air check (Watts & Garber, 1993), the error can be corrected for by re-calibration. During this air check the probe is contacted with air, so that a saturation value should be reported if the probe is still calibrated properly. This air check also allows one to evaluate this probe's time constant and, hence, its condition. Air checking could, for instance, be performed at the end of each batch experiment.

Because there are intrinsically two independent measures of the oxygen consumption rate (mass balances (1) and (9)) and only one of the two DO data series of this hybrid respirometric set-up depends on the mass transfer efficiency, it is possible to estimate  $K_L a$  reliably. Moreover, the accuracy of this estimate will be considerably higher than the one obtained from the procedure that estimates its value from the reaeration part of a DO profile obtained in a respirometer operating according to the (flowing gas/static liquid) principle.

### Implementation 2

In the second implementation, for which reference is also made to Figure 3, the flow direction is not altered intermittently with a fixed, short period of about 1 minute, but flow redirection is only initiated after a respirometric experiment has finished. The reasoning behind this is that it is not to be expected that checking the probe's condition is necessary every minute as can be done in implementation 1, but that it is sufficient to perform this check after each respirogram is collected. Hence, in implementation 2 one can collect two continuous DO datasets without disturbance from the intermittent flow redirection. This results in typical datasets as given in Figure 5 (DO dynamics after flow redirection are not included, they correspond to one up/downcycle seen in Figure 4). With such data sets more reliable parameter estimation can be done compared to implementation 1 except for the case where sudden changes in probe time constants occur. This seems, however, quite unlikely and would, moreover, be detected when the experiment is finished. Again, only the probe installed at the respiration chamber can be checked with this set-up, the other probe being compared to it when the experiment finishes. An air check can be useful to find out the latter's time constant and allows re-calibration of both probes if necessary.

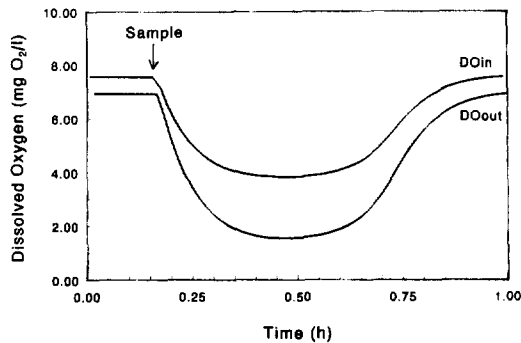


Figure 5. DO data obtained from the two DO probes in implementation 2 of the hybrid respirometer.

### Implementation 3

A scheme of the third implementation is presented in Figure 6. In this hybrid set-up the DO probe in the aerated reactor is moved to the other connecting line between the respiration chamber and the aerated reactor. In this way this second probe, normally measuring DO in the aerated reactor, can also be subjected to a step change in DO and its probe constants can be evaluated without an air check system. As in implementation 2, DO probe checking is only performed at the end of the experiment by reversing the flow direction between the two reactors. Ideally, the two DO probes would then equilibrate to the values given by



the other probe prior to the step change. If not, one or other of the probes has drifted and a re-calibration should be made.

An additional sensor characteristic is the pump flow rate that may be subject to drift. A small modification of the pump reversal conducted for probe checking allows pump checking at the same time. Provided the flow in one direction is significantly different from the flow in the other direction, the DO drop over the respiration chamber will change after flow redirection. The dynamics of this DO drop are described by a first order model with time constant equal to the retention time in the respiration chamber. Hence, the DO data allow one to estimate the flow rates of the pump (respiration chamber volume divided by estimated retention time).

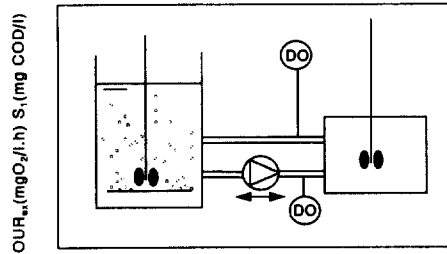


Figure 6. Scheme of hybrid implementation 3.

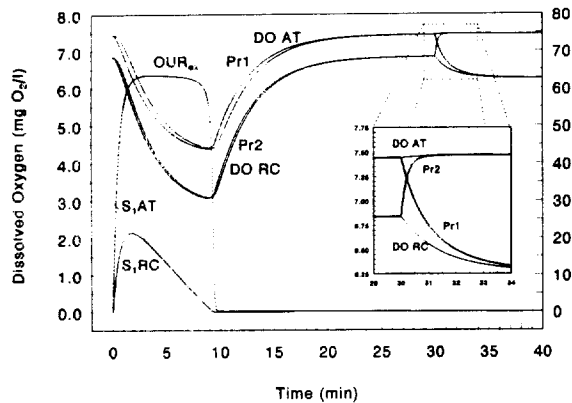


Figure 7. DO values and probe output data collected in a hybrid respirometer of implementation 3. Concentrations of substrate  $S_1$  in the aerated reactor (AT) and the closed reactor (RC) are given together with respiration rates.

In Figure 7 a simulation of the operation of such implementation of the hybrid respirometric method is given. First, the substrate  $S_1$  is distributed over both reactors with a time constant equal to the retention time in the respiration chamber. Substrate degradation starts immediately, albeit with a typical biological start-up phenomenon with a time constant of 0.5 to 2 minutes (Coen & Vanrolleghem, 1997). Substrate degradation induces a stoichiometrically related exogenous oxygen uptake ( $OUR_{ex}$ ). For the simulated experimental conditions the  $OUR_{ex}$  in both reactors is very similar, despite the initial difference in substrate concentrations in both reactors. This can easily be explained by the biological start-up phenomenon that gives sufficient time to build up the substrate concentration in the respiration chamber above the quite low saturation concentration  $K_s$ . The DO concentrations in the aerated reactor and the respiration chamber drop and the probes measuring these two DO levels follow. Probe 1 had a 0.5 min time constant, probe 2 being somewhat faster ( $\tau_{probe} = 0.2$  min). One indeed observes that the DO in the aerated reactor was followed slower than the DO in the respiration chamber. When the substrate is removed, oxygen levels increase again to their initial endogenous levels. At the end the pump is reversed with a flow rate half the original one (after 30 min. Fig. 7). Obviously, due to the longer retention time, the oxygen drop over the respiration chamber

due to the endogenous respiration increases by a factor two. As explained above the gradual increase in the DO difference allows one to estimate the pump flow rate. Also, both probe dynamics can be estimated from their response on the changes they are subjected to. Probe 2 is subjected to a simple step change as the DO concentration in the aerated reactor remains nearly constant. Probe 1 on the other hand is subject to a dynamically changing DO concentration. Hence, a somewhat more complicated analysis is required, but this is easily done using the sensor/process model that describes all sensor dynamics. Overall the DO data obtained in this implementation contain sufficient information to estimate all sensor and biological model parameters with much improved accuracy.

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