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Data Reconciliation of Streams with Low Concentrations of Sulphur Compounds in Distillation Operation

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Abstract

Traditionally data reconciliation of continuous processes operating in steady states is performed by constrained nonlinear optimization where the difference between measured and reconciled variables is minimized. In the present procedures weighting factors and statistical methods are applied to restrict the optimization problem. As the analyzed compositions and streams also have errors this approach might lead to misleading results. For example after reconciliation components may split into distillate and bottom streams against their true physical nature. Here a procedure is proposed to reconciliate information from distillation columns. In the procedure a column model is used to retain the real behavior of components more soundly than in traditional approaches. The procedure is discussed with the help of three industrial scale test cases.

Keywords data reconciliation; small concentration; distillation

1. Introduction

Data reconciliation of continuous processes operating in steady states is traditionally performed by constrained nonlinear optimization where the

difference between measured and reconciled variables is minimized. Small concentrations are dealt with by setting adequate weighting factors to the objective function. Another approach is to apply simple bounds on the components present in small concentrations, which is likely to produce gross errors in other reconciled variables [1], [2].

A method to reconcile small concentrations below their detection limit (and therefore unmeasureable) has been proposed by Dovi [3], [4]. In the method a probability distribution with a detection limit is combined to the reconciliation procedure. However, in our case, the small concentrations are measurable.

Generally the methods mentioned above are insufficient and might even lead to misleading results. For example in distillation operation reconciliation can create compounds in distillate or bottoms streams against their true physical volatility. The objective of this paper is to present a method to reconcile small concentrations particularly in a distillation operation to retain the real behavior of components better than in approaches using weighting factors or statistical distributions.

Three case studies are presented below to demonstrate these methods. A particular interest in this work was to apply the methods to streams containing small amount of sulphur (the last one of the three case studies) as the trend in Europe is to regulate the sulphur content in liquid traffic fuels (European Directives 98/70/EY and 2003/17/EC). At the same time the use of heavier crude oils that contain more sulphur increases. The process analysis and operation of these processes is challenging since the analysis of sulphur compounds is difficult. The sampling is problematic as the compositions are small and the samples easily evaporate. Obviously the safety issues have to be taken into account. Nevertheless good quality field data is important for successful operation of these processes. In practice incorrect and insufficient data has to be improved by for example removing the effect of random errors of the measurements i.e. the data has to be reconciled.

2. The reconciliation model

Our data reconciliation program is implemented as an integral part into the steady state flowsheet simulator FLOWBAT. It includes also the traditional methods described above. The weighting factors to the objective functions can be set according to user specifications. The software sets the material balance equations as well as the sum of mass/mole fractions as constraint equations. Upper and lower bounds on all variables can also be specified. The optimization method used is SQP.

In the new approach we take benefit the thermodynamic package of the simulator: The large data bank (over 5000 components) and estimation methods for the basic thermodynamic properties of pure compounds and an extensive collection of methods to calculate and estimate the thermodynamic properties of mixtures. From the library of unit models in this work are relevant the distillation models ranging from the equilibrium model to non-equilibrium models both efficiency based and rate based using the Maxwell-Stefan multicomponent mass transfer theory.

The main idea of the new approach is to simulate a distillation column first based on the measured feed. The calculated compositions are then compared to the measured product. If the deviation is small (say within 5%), the variable representing this measurement will be nearly fixed in the optimization procedure. The bounds on the variable will be set at +/- 5% of the measured value, while bounds on other variables will be set less rigorously. These limits were found to be adequate by trial and error using several test cases.

These restrictions on the variables change the feasible region of the optimization. The adjustments of the measurements obtained through data reconciliation are generally small, but placing rigorous bounds on all variables can make the optimization infeasible or cause difficulties in achieving convergence. Another new approach is to calculate the distribution ratios [5] of the components based on simulated distillate and bottoms composition.

3. Case studies

Three cases will be described below in which the distillation simulation is applied as a part of the data reconciliation procedure.

3.1. Dehexanizer

The distillation column considered is used for hexane removal [6], [7]. The feed 281413 kg/h is stabilized gasoline. The distillate contains C5-C6 hydrocarbons and the bottoms product is suitable feed for the reformation unit. The problem has 25 analyzed components.

In the first of the two procedures used, the reconciliation was performed by applying material balance constraints and the same relative upper and lower bounds 30% for all optimization variables.

In the second procedure, the simulation of the dehexanizer was performed first, after which the simulation results were compared to the measurements. If the deviation was less than 5%, the bounds of the respective variable were set at +/- 5% of the measured value. It is clear that the simulation predicts most

accurately the light components in the distillate and the heavy components in the bottoms. Nine compositions of 15 restricted ones were in the bottoms flow. For other variables, bounds were set as in the first case. The balance equations were met equally well in both cases. A considerably better minimum was achieved by a factor of two decades when applying the more rigorous constraints for some variables. Obviously the minimum found by the optimization procedure is affected by the chosen optimization parameters. A trial and error approach was required to find the solutions.

3.2. MTBE column

The second case study consisted of MTBE separation column in which the non-reacted isobutene and methanol are removed from the MTBE after MTBE reactors [7]. Sixteen components were used in the data reconciliation. The feed flow was 42718 kg/h.

The same two procedures as above were applied. In the first procedure, without rigorously constrained variables, the optimization failed to obtain a solution, i.e. at the set level of tolerance the material balance equations were not met. The rigorously constrained procedure guided by the distillation model did converge successfully.

3.3. A gasoline fractionator

The third case considered is a gasoline fractionator [8]. Two components containing sulphur (hydrogen sulfide, ethyl-mercaptan) were present in the column in small concentrations. From the feed flow 102179.5 kg/h 15 components were analyzed properly and reconciled as in the previous cases. The remaining components were described as pseudocomponents. In the simulation (as in reality), two feed flows are mixed to form one feed flow to the column.

In order to guide the reconciliation of the small concentrations, for which the adjustments are considerable, it was attempted to constrain these variables more rigorously than other variables like in the examples above. This seemed to harm the whole optimization and produced large adjustments in many variables. Therefore, another procedure was used. In this approach the split ratio of the feed and distillate was added as an additional constraint for the small concentrations:

$$C_i = \frac{m_{i,sim}}{F_{tot,sim,i}} - \frac{m_{i,meas}}{F_{tot,meas,i}} \qquad (1)$$

where

C_i constraint of component i
$m_{i,sim}$ mass flow of component i in the distillate according to the simulation
$m_{i,meas}$ mass flow of component i in the distillate according to the measurements
$F_{tot,sim,}$ total feed (mass flow) of component i specified in the simulation
$F_{tot,meas,i}$ total feed (mass flow) of component i according to measurements

The additional constraint equation (1) was imposed on the two sulphur components. The allowed deviation of the split ratio was set at 40%. When applying more rigorous bounds, convergence was difficult. As seen in Table 1 the simulated split ratio and the measured split ratio are not in full agreement. However, the split ratios are closer to the simulated results. Thus in the method more confidence is put into the simulation than the analysis of the compositions. This is a justified approach if a good knowledge of the system VLE is available.

Table 1. Split ratio of the sulphur components.

Component	measured	simulated	reconciled
H2S	0.38	1.00	0.77
EtSH	1.10	0.73	0.99

4. Discussion & summary

The distillation model was found to be a helpful tool to guide the data reconciliation procedure. In cases when the distillation model agrees well with the measurements, the feasible region can be reduced, which in turn provides a fast method for achieving an acceptable minimum for the adjustments to the measured values of the process variables. The optimization result is influenced by the limits set for variables whose measured values are zero. A higher accepted reconciled value for these variables generally produces a lower value of the objective function. On the other hand, the value should not rise above the detection limit of the measurement.

Commonly the simulated values of the lightest and heaviest compounds, which hardly show any distribution in the separation, were in good agreement with the measurements. Some distributing components showed considerable deviations from the measured values. This might partly be due to the product rate specification, which was set according to the measured values of the product rates. The separation is also affected by the efficiency specified for the model calculation, in this work an estimated efficiency of 75 % was used in all cases.

For sulphur compounds in small concentrations, for which the adjustments are considerable, it was found out that the approach using split ratios to guide the reconciliation was more successful than limiting the concentrations by simulations.

To further develop the concept of using a distillation model in the data reconciliation procedure, it is suggested to use an iterative approach. The idea would be to set the feed and one product flow as well as the composition of all flows as optimization variables; this would eliminate the problem of deviations between the calculated and the measured unspecified flow rate in the distillation model. The distillation model would then be used as a "set of constraining equations" for the optimization to find the minimum adjustments for the measured values. It might be beneficial to consider the use of one of the methods using the non-equilibrium approach (efficiency based or rate based) for more accurate simulation of especially the distributing components.

The method can easily be applied also with other unit operations. The key issue is to use physical and chemical knowledge, instead of just relying on purely statistical methods.

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