

First-principles distillation inference models for product quality prediction

Modeling approach involves a short-cut simulation of a column section

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Advanced process control (APC) makes money by maximizing a key process variable (usually throughput) and pushing plant equipment to its limits. At the same time, product quality targets are maintained. This is critical. As Fig. 1 illustrates, pushing equipment to its limits without controlling product qualities forces the operator into a counter-productive giveaway operation.

Tools on the market can perform maximization, but quality control is more problematic. Quality control must rely on real-time quality measurement, which is difficult. Traditionally APC relied on onstream analyzer measurements, but due to their measuring location, analyzers are too slow to be useful for synchronizing quality control with fast-acting constraint controllers. Also, issues of cost and reliability impede the use of analyzers in closed-loop control.

In recent years, industry has started to develop methods for treating the equipment itself as an “onstream analyzer,” passing process measurements through a mathematical inference model to produce a product quality estimate. The concept, illustrated in Fig. 2, is an inverse simulation problem. Simulation inputs feed and product properties and estimates unit conditions for manufacturing those products. Inference inputs unit conditions and estimates product properties.

A number of years ago, we came up with inferential models for distillation columns that are based on scientific

principles.¹ The modeling approach involves a short-cut simulation of a column section, typically a bottom half of the stripping section or a top half of the rectifying section (Fig. 3). Hence, the name of the model: general distillation short-cut (GDS).

GDS works by fitting a bottom (or top) column composition that would best agree with column measurements. That is fairly easy to accomplish with binary distillation. The novelty of GDS is that by looking at a number of tray temperatures it is equipped to address multicomponent distillation.

Table 1 is an example GDS model reading two temperatures to come up with three equations, permitting composition inference of a product containing three components. The model employs three first principles: bubble point, section separation “Colburn ratios” and mass balance. GDS relies on Colburn’s formulae for distillation column section performance,² describing the ratio between tray composition and bottom (or top) composition as a function of component volatility (K value), internal reflux and number of trays in the section. These formulae provide a way to interpret tray temperature measurements correctly, taking into account the effects of pressure, reflux and multi-component environment.

In terms of complexity, Table 1 equation coefficients

are obtained by nonlinear calculations, but the resulting equations are linear, allowing once-through calculations with no convergence issues. That makes GDS very suitable for online closed-loop work. Perhaps the most complex GDS calculation

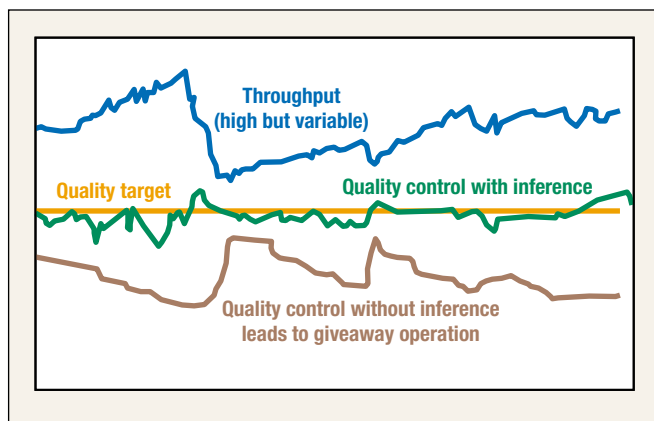


Fig. 1. Why apply inferential control?

Table 1. Example bottom section model

Three bottom composition unknowns:

Benzene, toluene, xylene (XB_1 , XB_2 , XB_3)

Three linear equations:

- Bottom bubble point equation:
 $\sum(KB_i \cdot XB_i) = 1$ $KB_i = YB_i/XB_i$ at bottom conditions
- Section separation equation (Colburn ratios):
 $\sum(R_i \cdot XB_i) = 1$ $R_i = Y7_i/XB_i = F(i, T_7, P, L/V)$
 T_7 = Tray 7 temperature
 $Y7_i$ = Tray 7 vapor composition
- Mass balance equation:
 $\sum(XB_i) = 1$

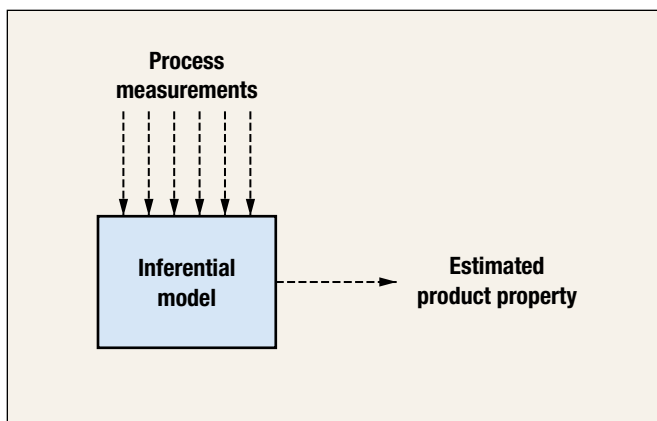


Fig. 2. The inferential control concept is an inverse simulation problem.

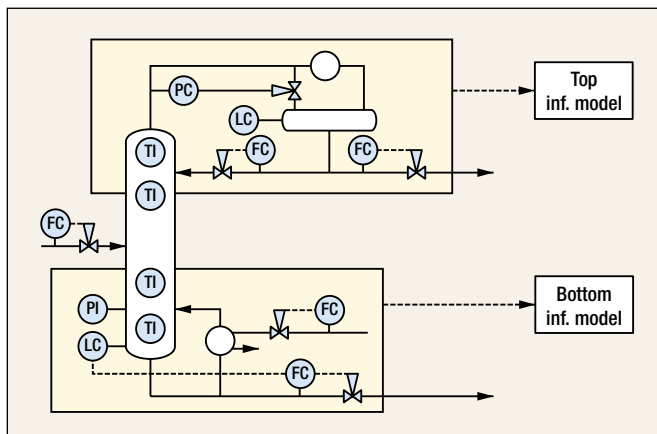


Fig. 3. The GDS approach involves a short-cut simulation of a column section.

involves heat balances to estimate internal reflux of column sections needed for calculating separation parameters.

Since the initial GDS publication,¹ many GDS inferences have been implemented. Last year we took stock and reported performance of these models.³ Experience indicates that where tray temperature measurements are available, and where we have reliable data for calculating internal reflux, the inference models are excellent. When measurement data are imprecise and we are forced to make assumptions, the models might drift over time—though they could still be useful for closed-loop control.

During presentation of our paper last year, two questions were asked:

1. “Empirical models are gaining popularity because they are inexpensive to set up, for example, as a spreadsheet regression model. Are there reasons why they should not be used for distillation processes?”

2. “GDS seems successful in dealing with common refinery columns, separating components to typical purities of 0.1 to 5%. What about high-purity columns, separating products to ppm levels? Would GDS successfully handle that problem?”

Back then, time allotted for discussion did not permit answering any of those questions, nor were there easy answers. Now this article attempts to answer both questions in the detail that they deserve. Regarding empirical versus scientific models, the article will provide arguments in favor of the latter. Regarding superfractionator inferences, we had the chance to work on a benzene, toluene, and xylene (BTX) separation unit where product specifications are around 200 ppm (Fig. 4). This article relays the benzene tower experience. Amazingly, it appears that whereas the GDS models were probably successful in predicting benzene column product contamination, the lab test per-

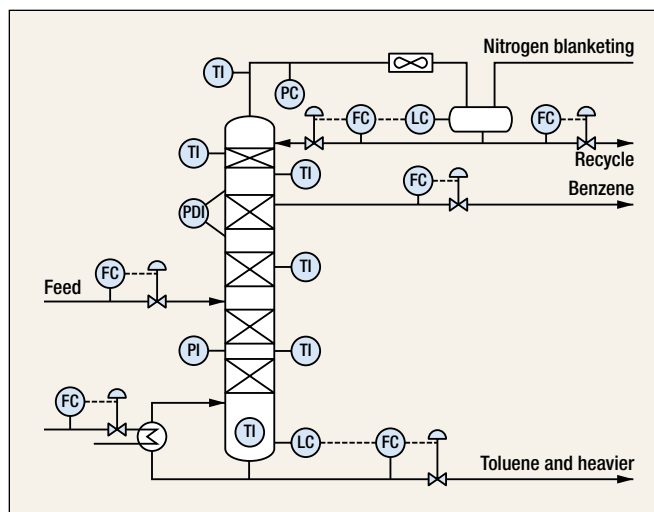


Fig. 4. The benzene column had tight product specifications.

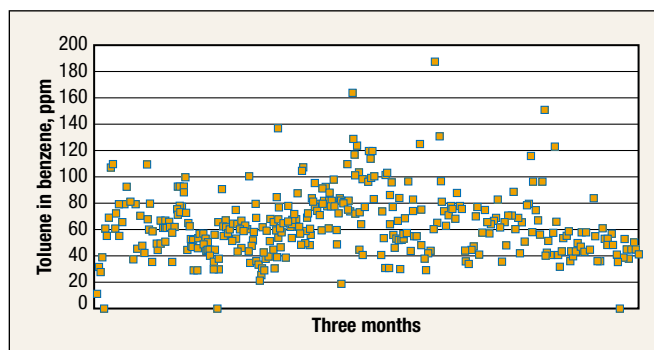


Fig. 5. Three of the data show a value of zero ppm, a level that is simply not feasible for such a column.

cision was insufficient for validating the models.

Why not empirical models? This section explains the difficulties of applying empirical regression to infer distillation column product purities. There is no need to discriminate between neural net versus simple regression models, because both rely on Gaussian statistics, and the empirical modeling issues are all related to Gaussian theory limitations. We are not generally against statistical models, especially for equipment where first-principles knowledge does not exist. But in distillation, there are several strikes against empirical models.

Regression requires independent inputs. As shown in Fig. 2, inference models input flow, temperature and pressure measurements, and output estimated product properties. Gaussian theory requires that all inputs be independent; however, that is not feasible. Temperatures, pressures and flows are related in several ways: mass balances, heat balances and equilibrium equations. Ignoring these relations makes the modeling process theoretically incorrect. A more correct statistical approach would involve Bayesian theory, which takes into account a priori knowledge of dependence among input variables. Of course that a priori knowledge brings us back to the first principles of distillation.

Savvy empirical models practitioners have tried to circumvent the problem of dependent input variables. For example, in a distillation column where typically all temperatures trend up and down together, people have tried taking one temperature as the key input, while expressing the other temperatures as differences from that key value. That is a step in the right direction. But in the end, it is impractical to consider all of the a priori relations.

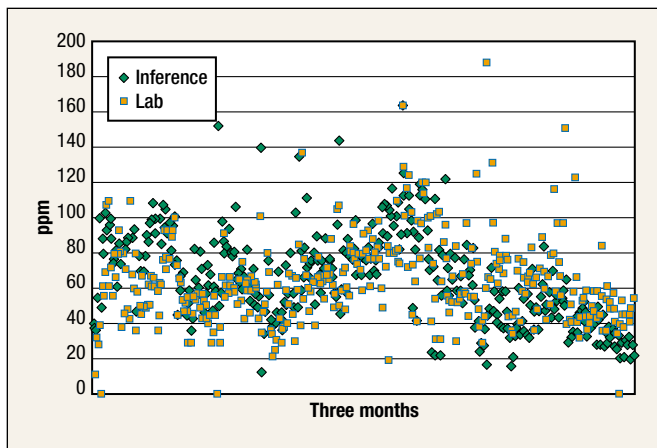


Fig. 6. This model was calibrated by adjusting only one parameter: tray efficiency.

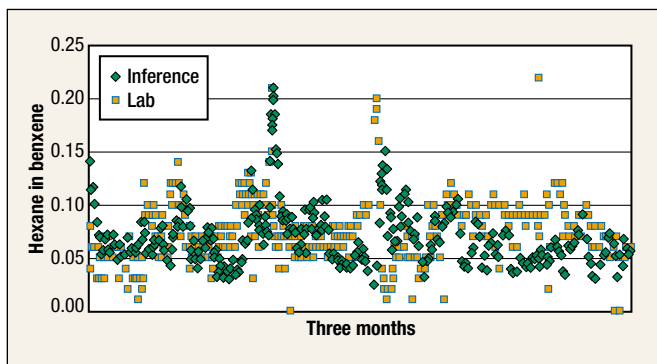


Fig. 7. These data were used to calibrate the model, i.e., determine the number of theoretical trays in the section

What then happens to models that use dependent variables? They have incorrect coefficients and, hence, would drift upon changes in process conditions.

Empirical models require large volumes of lab data. Statistical regression requires hundreds of laboratory data and that poses a problem. A fair percentage of daily lab data are biased and reliable process data are obtainable only by test-runs. During a test-run, the unit is kept at steady state for several hours. Then samples are drawn by careful procedure—in properly sealed sample bottles—taken to the lab without delay and tested immediately in the presence of the process engineer.

There is no hope that the quantity of lab data needed for regression would come from high-quality test runs, and empirical model developers would have to rely on *imprecise* everyday lab data. Imprecise because it permits occasional spike contamination, sampling during process changes, inappropriate sampling procedures and long delays between sampling and testing. Fig. 5 is an example of the spread in daily lab data taken on our benzene column. The lab results for toluene contamination vary between 0 to 200 ppm, but we will show later that the process itself was well behaved during that time, with product impurity actually varying between 50 and 100 ppm. We conclude that the standard deviation of error in this set of lab data is about 30 ppm, and furthermore, about 5% of the data are completely wrong. That assessment is supported by the fact that three of the data show a value of 0 ppm, a level that is simply not feasible for such a column.

Can this lab data still provide the basis for a useful model? That might be possible, if the error is normally distributed without bias. Model developers often assume that those lab data that do not fit their correlation are wrong. They label those data “outliers”, removing them from the original set, thus obtaining

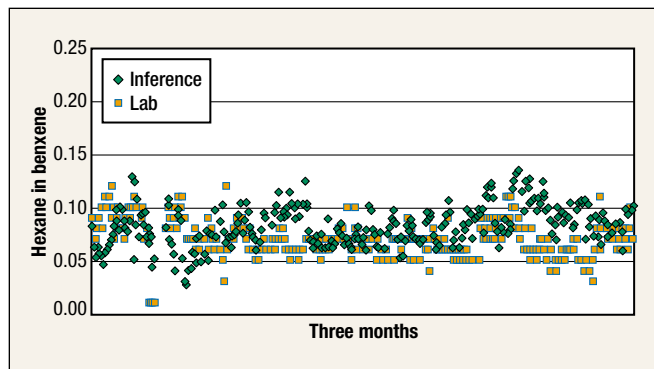


Fig. 8. Selecting hexane as the main aliphatic contaminant leads to model imperfections.

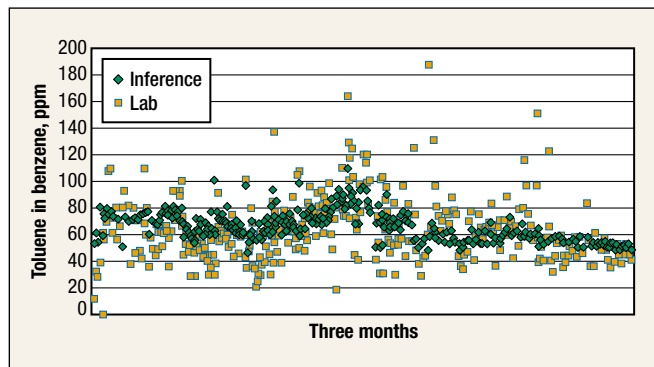


Fig. 9. While this model may not show much improvement over Fig. 6, it is a better model in a number of respects.

excellent fit, only to find out later that regression fitting does not guarantee future prediction. How many outliers are acceptable? Eliminating just a few points from a large set of random data could entirely alter the regression formulae. Can we possibly delete over 1% of data from a set and still call this data reliable? What about 5%?

No, is the logical answer. Data should be deleted from a set only with evidence that the point in question is erroneous. In that respect, first-principles models provide a mechanism for identifying erroneous data. Developing first-principles models is done without regard to the lab data and often before lab data are collected. Trending the uncalibrated model against lab data provides a powerful tool. While the uncalibrated inference model may not agree with the lab, the two should surely trend together. Fig. 6 shows a trend of an inference model versus the lab data of Fig. 5. This model was calibrated by adjusting only one parameter—tray efficiency—and only for the purpose of bringing the lab data and model to the same scale. Perhaps one does not yet believe the model to the extent of throwing away all of the many outliers, but we can certainly label as suspect those lab results that have gone much up or down without a process reason for exhibiting such behavior.

Empirical models must identify a large number of coefficients. Scientific models incorporate model gains inherently, and the calibration procedure amounts to adjusting one or two parameters. Examples are tray efficiency and weight in a weighted average formula. The effect of signal-to-noise ratio on the calibration procedure is minimal.

Empirical models, on the other hand, must identify at least one gain coefficient associated with every measurement. That is a problem because normal day-to-day operation may not provide enough movement in the data to give meaningful information. Large enough process moves endanger product purity and cannot often be permitted. When data movement is too

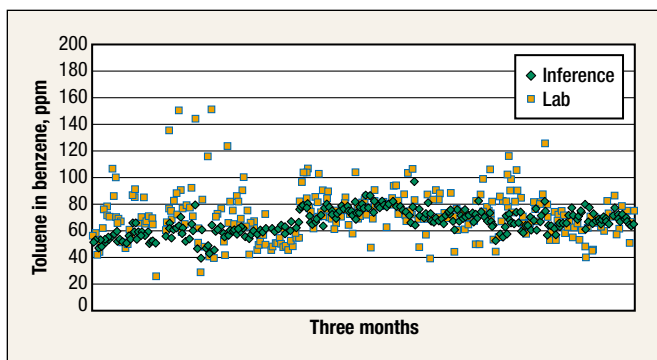


Fig. 10. This trend shows the model does not deteriorate over time.

small, the regression would simply model noise patterns.

First-principles models provide the means for checking instrument errors. The sister problem of erroneous lab data is erroneous instrument data. Instrument errors occur due to poor calibration, partial plugging of orifice meters, improper installation, incorrect meter range and, finally, also computer interface errors. Inference models developed from erroneous instrument data would necessarily be weak. Eventually instrument problems are identified and corrected. What would happen to a regression model then? It would have to be redeveloped, of course, but from what data? The old data sets with erroneous readings cannot be used. Assuming lab tests are carried out daily, creating a new set of hundreds of lab data would take many months, during which time the APC would be off.

We conclude that before starting inferential model development it would be prudent to survey all input measurements and identify all problems. The best way to accomplish that is via employing first-principles knowledge—testing the readings against mass balance, heat balance and equilibrium equations. If those readings cannot be reconciled against basic thermodynamic laws, they cannot be correct. There is an obvious conflict here because people choose the empirical approach to avoid the more time-consuming first-principles models, only to find out that the use of first principles is unavoidable, if one desires to identify erroneous data sets.

There is no replacement for process engineering. And what if the measurement set is inadequate? Suppose a key measurement is missing or is in the wrong location? To obtain a good model, the measurement set ought to “have the inferential information in it.” A first-principles modeler would identify an insufficient set of inputs at the outset by a simple sensitivity study. The modeling effort would then be halted until the missing measurement is installed. The empirical modeler would go through model development and the problem would only be found at the time of model validation. At that time, he or she would have to employ first principles to analyze the problem, wait for the measurement to be installed and then wait months until a quantity of lab data is available.

Ability to survive process modifications. During unit turnarounds, distillation columns are often modified by replacing trays, cleaning condensers or reboilers, etc. Any inferential model would need to be recalibrated upon column equipment modification. First-principles models might require equation coefficient changes, but empirical models would be turned off for several months until a meaningful set of lab data is accumulated and the model redeveloped from scratch.

Benzene column. Having experienced success in developing first-principles inferential models for “run of the mill” refinery applications,³ we next tried the more challenging high-

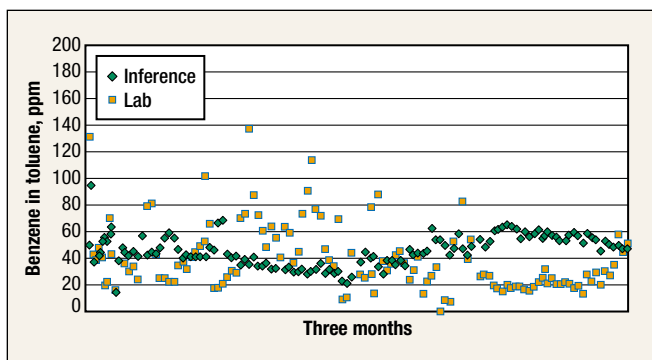


Fig. 11. An initial attempt was made to calibrate a stripping section GDS model by adjusting the number of trays.

performance benzene-toluene separation column shown in Fig. 4. This is a high reflux column with many separation stages packed in five beds, separating products down to ppm level. The top bed is a “pickling” section, removing light boiling aliphatics from benzene. Benzene is drawn as a sidestream below the section whereas the top distillate, containing most of the C₆ aliphatics, is circulated back to an upstream aromatics extractor. The desire is to minimize this costly recycle subject to a 0.25% maximum aliphatics in benzene target. Presently the operation is rather conservative, keeping the level of aliphatics in benzene to about 0.1%.

The next two beds form a rectifying section between the feed and benzene draw, and their task is to separate toluene from benzene, leaving a pure benzene sidestream. The lower two beds below the feed point form a stripping section, stripping benzene out of toluene to keep the bottoms essentially free of benzene. Toluene and xylene in the bottom stream are separated in a downstream column, not addressed by this article. Targets for toluene in benzene, as well as benzene in toluene, are 200 ppm, whereas actual operation applies a very high reflux, enough to keep both impurities at about 80 ppm. There is an incentive to cut reboiler heat duty and control product qualities more precisely to avoid giveaways.

Besides energy savings, there is also an operating flexibility issue. The benzene tower is difficult to run because of the tight dual specifications of toluene in benzene and benzene in toluene.

Operators make substantial effort to keep the aromatics reformer as well as extractor running steady without any change of conditions, and in so doing operating flexibility is lost. There is money to be gained by changing reformer and extractor conditions to match the economics of the day.

Pickling section model. The pickling model works by first inferring top composition, and then applying the Colburn ratio formulae to estimate the aliphatics contamination of benzene. Ideally, distillate composition inference would be obtained at overhead accumulator conditions, where the effect of a lighter component is the most pronounced. However, accumulator pressure control is accomplished by inert gas, which obscures the partial pressure of hydrocarbons, thus eliminating use of an accumulator thermodynamic equilibrium equation.

The next best option is to write an equilibrium equation between benzene and aliphatics at the top of the column. This raises two questions. First, what exactly are the aliphatics? Second, is the inert gas soluble in the reflux, thus affecting the top partial pressure? Regarding the former question, the model conservatively assumes normal hexane. Normal hexane, being the highest boiling aliphatic component, would give the high-

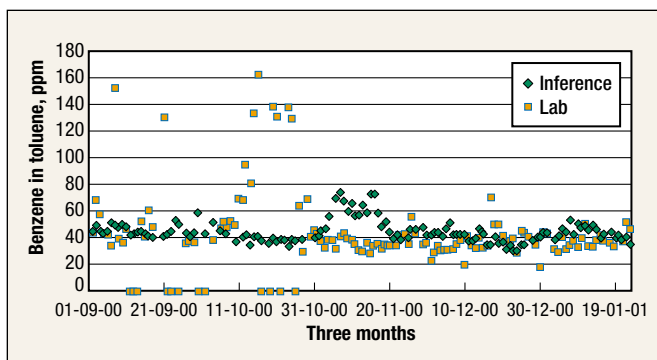


Fig. 12. The test data exhibit a better fit than the calibration data.

est aliphatic contamination estimate. As for inert gas, the model assumes no solubility of the gas in reflux, though that requirement is not rigid, and the solubility equation could be added if needed to improve model accuracy.

Fig. 7 shows a three-month trend of aliphatics in benzene contamination. These data were used to calibrate the model, i.e., determine the number of theoretical trays in the section. Fig. 8 shows model performance three months later without additional calibration. While the lab data contains improbable points, we must assume that most of the data are correct. It would appear that selecting hexane as the main aliphatic contaminant leads to model imperfections. The two trends do follow each other though occasionally they deviate. Considering model imperfections, our goal of minimizing recycle can only be partially met. Model inaccuracies prevent us from aiming at the 0.25% contamination limit, but they do permit an operating target of 0.15%, which would cut recycle by 33%.

Rectifying section model. Our rectifying model relies first on calculating the toluene content at a pivot point about two-thirds down the rectifying section, where Fig. 4 shows a temperature point. Once that composition is known, the model goes on to estimate the side-draw toluene contamination from a Colburn relation between the pivot and side-draw points.

Obtaining a pair of temperature—pressure measurements at the pivot point was difficult. There is no pressure measurement at the pivot point, and we are forced to extrapolate from pressure drop across the first rectifying bed. There is also no pressure point at the side-draw location, but fortunately, knowledge that the side-draw contains essentially pure benzene permits calculating side-draw pressure from side-draw temperature. That helps with pressure extrapolation, making calculating toluene content at the pivot point possible.

At that pivot point there are only two components: benzene and toluene. Once the pair of temperature—pressure measurements is established, the composition can be calculated. To be precise there is also a small amount of hexane, but the hexane partial pressure is so small that its influence on the calculation is negligible. Following toluene calculation at the pivot point, the model estimates toluene contamination in benzene using a Colburn ratio between toluene at the pivot point versus side-draw point.

As discussed previously, Fig. 6 shows a preliminary calibration of the model against lab data by adjusting the equivalent number of theoretical trays in the section. We see that at the beginning of the three-month calibration period, the model reads generally above the lab; toward the end of the period, the model reads lower than the lab. The calibrated number of trays for the section was 7.8, which appears too low. Typically such signs indicate that one or more of the input readings are biased,

and we came to the conclusion that probably the representative rectifying section temperature was reading too low.

Fig. 9 shows a recalibrated model against lab data for the three-month period. A bias of 1.2°C was applied to the temperature reading and the number of section trays increased to 10. While this Fig. 9 model may not show much improvement over Fig. 6, it is a better model in a number of respects:

- Number of rectifying section trays is more believable
- Model reading matches the main cluster of lab results better throughout the period
- Pivot point composition is more stable.

Fig. 10 shows trends of model against later lab data without further calibration, confirming that the model does not deteriorate over time.

What can explain the success of this model? Until now it has been known that distillation inference models at ppm levels are notoriously unreliable. The key to this success is our ability to calculate the pivot point composition with reasonable accuracy. In our case, toluene concentration at the pivot point is about 5%. Considering that the model applies extrapolation to calculate pivot point pressure, and the temperature is biased, the likely pivot point toluene content precision would be about $\pm 1\%$, or 20% imprecision. The Colburn ratio is about 1,000, and one can argue that uncertainty in the number of theoretical stages, i.e., how tray efficiency changes with operation, introduces another error of about $\pm 20\%$. Taking into account both errors, the expected prediction repeatability is about $\pm 30\%$ (square root of the sum of error squares is 28%), probably the best that has been achieved for high-performance fractionators. In fact, this repeatability is better than the lab data that was used to calibrate tray efficiency and temperature bias.

Stripping section model. As is well known, applying two temperature-based inference models in closed loop, one for rectifying, the other for stripping, is problematic. The rectifying model determines how much benzene is to be drawn, the rest

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being C_{7+} to be removed with the bottoms. The stripping model prescribes how much material is to be evaporated from the bottom and how much would be permitted to flow down to the bottom as liquid. Thus, both models take actions that dictate the bottoms flow. Small disagreements are inevitable and would cause competition between the two models and closed-loop instabilities. To avoid model competition, we intended to control the column to a bottom model that would rely not on column temperatures but rather on predictions of the rectifying model and stripper L/V . Such a model would not compete against the rectifying model.

But how would one create essentially a regression model from the imprecise lab data? We resorted to first developing a GDS model for the stripper, then regressing a nontemperature-based bottom model from readings of the GDS models rather than lab data. However, the GDS attempts were not successful. Fig. 11 shows our initial attempt to calibrate a stripping section GDS model by adjusting the number of trays. We concluded that one or more of the inputs are erroneous in an unpredictable way, had to abandon the attempt at a GDS model and decided to simply control the reboiler to a constant section L/V . In the meantime, a three-month test was in progress, and all models were tested for ability to fit an unknown set without further calibration.

Afterwards, we were faced with Fig. 12, trending the benzene in toluene combination model against lab results. It points to an unusual phenomenon: the test data exhibit a better fit than calibration data. This has forced us to reconsider our previous conclusion about the input data being erroneous. It appears that the benzene in toluene lab data were substantially unreliable. The bottom GDS model itself, though there is no yardstick to validate it, is possibly not bad. However, at this stage there is no decision about whether to use a bottom model or a simpler L/V control variable. ■

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