

## Special Situations Call for Extraordinary Measures

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Tracerco is often challenged by a customer's plight: Can a gamma scan of a column over 7.5m in diameter or a column with a wall thickness greater than 50mm generate results adequate to assist with diagnosing a problem? Will a gamma scan work on a liquid full column, like a liquid-liquid extractor? Typically when a column is greater than 7.5m diameter, has greater than a 50mm wall thickness, or is liquid full Tracerco takes a slightly different approach to the scanning technique to produce the best results.

A TRACERCO Diagnostics™ Tower Scan uses a small, gamma ray emitting radioisotope and a sensitive detector external to the column. The detector measures the intensity of radiation passing through the process equipment providing a profile of the relative density of the internal process which can be used to understand many aspects of the hydraulic operation of the equipment under investigation.

The following studies present examples of diagnostic results where a column's wall thickness or diameter exceeded the norm. It should be noted that due to safety reasons involved with the handling and transporting of radiation sources, there are practical limits to the size of equipment that can be successfully scanned.

### Case 1. Base Liquid Level Detected In Tower with 178 mm Thick Walls

An 2.4m diameter and 178mm wall thickness tower was experiencing high  $\Delta P$  at increased rates and flooding or foaming was expected. Tracerco was contacted to perform TRACERCO Diagnostics™ Tower Scans to deter-

mine if flooding was occurring and to determine if any other problem was present. Due to the extreme wall thickness a source with higher energy was selected in combination with an ultrasensitive detector, built specifically and exclusively by Tracerco for challenging projects. Two scans were performed at different operating conditions. (Figure 1) The first scan (blue scanline) indicated all the trays (1 – 15) were in place, holding aerated material and were operating with what appeared to be heavy entrainment. Trays 1 – 9 indicated more froth/aeration density (higher degree of entrainment) between the trays compared to Trays 10 – 15. Furthermore, there was no indication of a bottoms level identified in the base of the tower, while control room instrumentation indicated a 50% base liquid level.

A second scan was performed (red scanline) at reduced vapour rates. Under these conditions there was a slight reduction in froth height on the trays and a decrease in entrainment between the tray decks. However, a base liquid level was detected approximately 356 – 406mm below the top level tap, at a level of 70% (control room instrument indicated 100% level).

While the scans confirmed the tower was operating with high entrainment which could be the cause of the increased  $\Delta P$ , a more significant finding was the results that indicated the base level instrumentation was not reading correctly.

### Benefit

Presented with the challenge of a thick-walled vessel Tracerco successfully planned and executed TRACERCO

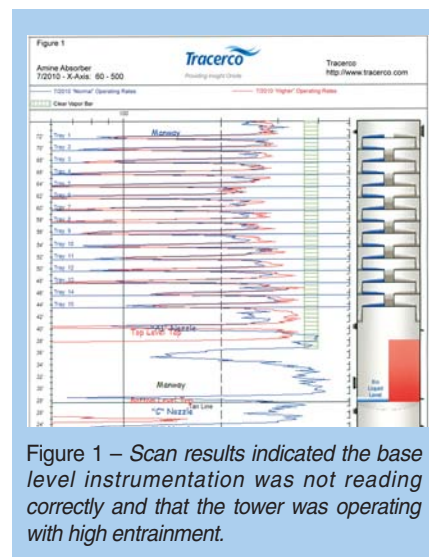


Figure 1 – Scan results indicated the base level instrumentation was not reading correctly and that the tower was operating with high entrainment.

Diagnostics™ Tower Scans using a high-energy source. The scans allowed diagnosis of the operating condition of this tower allowing the customer to avoid a needless shutdown and to learn of an associated operating problem regarding the liquid level.

### Case 2. TRACERCO Diagnostics™ ThruVision Detect Maldistribution In A 7.9m Diameter Vacuum Tower

The top section of a Lube Vacuum tower at a North American refinery was not fractionating efficiently, lowering the yield and quality of the top side stream product. This efficiency loss was attributed to liquid mal-distribution likely from corrosion products plugging the gravity flow distributor above the packed bed. The customer had already discovered corrosion products plugging the distributor filter.

To quantify the location and magnitude of liquid mal-distribution, diagnostic

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# Gamma Scans

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techniques including a TRACERCO Diagnostics™ Tower Scan and TRACERCO Diagnostics™ ThruVision Scan were employed. The tower scan results indicated that the two beds were in place with characteristics of severe liquid mal-distribution (Figure 2). The top bed appeared to exhibit excessive liquid on the south side accompanied by liquid deficiency on the west side of the tower. Bed 2 appeared to be holding an excessive amount of liquid in the top section of packing and exhibited severe liquid mal-distribution. The liquid mal-distribution appeared to become less severe lower in the bed below the section of packing with excessive liquid accumulation. A close look at the collected data for the chimney tray between the beds indicated that liquid was overflowing the risers on the south and east scans. Liquid appeared to be level with the top of the

risers on the north scan and 50 to 76mm below the top of the risers on the west scan. The high liquid level was caused by reduced draw rate from the collector.

With the draw rate increased to prevent the collector from overflowing, a TRACERCO Diagnostics™ ThruVision Scan was performed at the elevation noted in Figure 2 in order to view in greater detail the pattern of liquid mal-distribution.

The data in Figure 3 represents the TRACERCO Diagnostics™ ThruVision Scan results performed on Bed 2 of packing located below the VGO Draw. Two relative dry spots were identified – one in the northeast quadrant and the second on the west side of the tower.

With knowledge of the liquid mal-distribution patterns refinery staff developed an extraordinary online fix. The fix involved a pump-back reflux circuit off the top pump-around that was designed and installed in four months.

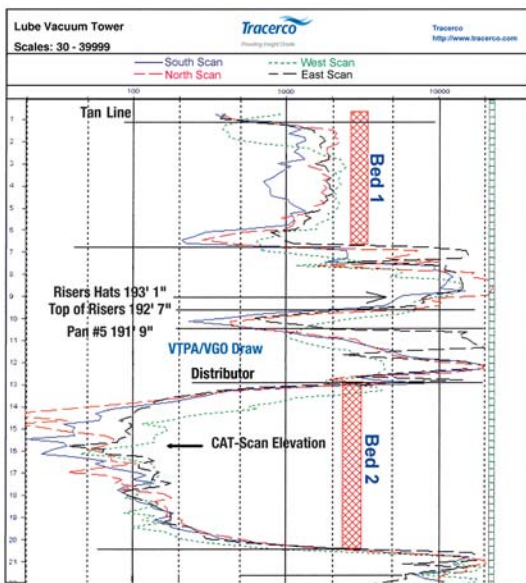


Figure 2 – TRACERCO Diagnostics™ Tower Scan data indicated that both beds were in place with characteristics of severe liquid maldistribution.

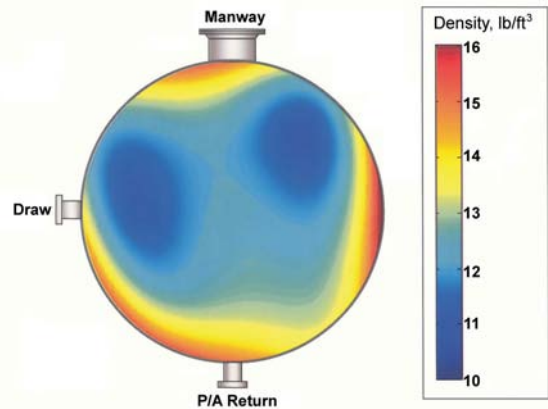


Figure 3 – TRACERCO Diagnostics™ ThruVision Scan color contour plot results illustrate where two dry spots were identified in a lube vacuum tower.

### Benefit

The TRACERCO Diagnostics™ ThruVision Scan was used to determine the orientation of the new liquid distribution system focusing on the low liquid density areas of the tower cross-section. After start-up of the new circuit 65-70% of the lost top side stream yield was recovered with on specification product; exceeding expectations from the fix.

### Case 3. Liquid-Liquid Extractor Applications

One category of special applications is liquid-liquid extractors. Besides the fact that liquid-liquid extractors are not “typical” distillation or fractionation unit operations, what makes them a special application for a TRACERCO Diagnostics™ Tower Scan? The radiation source and detector system that is used in scanning measures density changes of the process materials inside the tower. With a typical fractionation tower there is a large density difference between the vapour and liquid phases. Thus there is a large density range between clear vapour and clear liquid to detect process or operating conditions existing in a typical fractionation tower –

entrainment, foaming, flooding, internal damage, etc. But liquid-liquid extractors are liquid full, with no large vapour-liquid density difference. Meaningful scan results from a liquid-liquid tower rely on the density difference between the two liquid phases being significant enough to distinguish as some extraction processes involve two liquids that have comparable densities.

So what will a TRACERCO Diagnostics™ Tower Scan of a liquid-liquid extractor tower show? Since we do not have the large vapour-liquid density difference to work with, the information learned from a scan of a liquid-liquid extractor is less extensive than for a “typical” fractionation tower but not any less important. As the following examples show, scans of liquid-liquid extractors can detect problems when the two liquid phases are not properly mixing, preventing the tower from doing its proper extraction operation.

Figure 4 shows a liquid-liquid absorber tower with trays where amine is the heavy liquid phase and LPG is the light liquid phase. This absorber was designed for the trays to hold a level of amine

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# Gamma Scans

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(heavy phase) on the trays and for the LPG (light phase) to bubble up through the amine. On the top trays of this absorber (Trays 1 – 6) there was a dense mass sitting on the tray decks. Trays 1 – 6 were holding approximately 250mm of amine. The remainder of the tray spacing was filled with light density phase LPG.

In this particular absorber there was a relatively large density difference between the amine (sp. gr. 1.0 – 1.1) and the LPG (0.5 – 0.6). The scan profile of trays operating normally closely resembles what would be seen from a typical liquid-vapour fractionation tower. The big difference is in the magnitude of radiation passing through the column. Note the radiation (horizontal X-axis) scale, which was 1 – 800 in Figure 4.

In a typical fractionation tower the maximum counts would be in the high 1,000's due to the much lower density of the vapour phase.

Note the change in the scan profile at Tray 7. Tray 7 has a level of dense phase liquid measuring approximately 965mm, much higher than the trays above. In addition Trays 8, 9, and 10 did not appear to have any appreciable level of dense phase liquid. The scan results showed that the amine or heavy phase liquid cannot flow past Tray 7 most likely due to a restriction in the downcomer from Tray 7. Trays 8 – 10 actually were “starved” of amine so only the light phase liquid LPG was seen on these trays. The customer is now aware of the type of operating bottleneck they have and can formulate an action plan to remedy the situation.

Figure 5 shows the scan results from an extraction tower with packed beds. If the

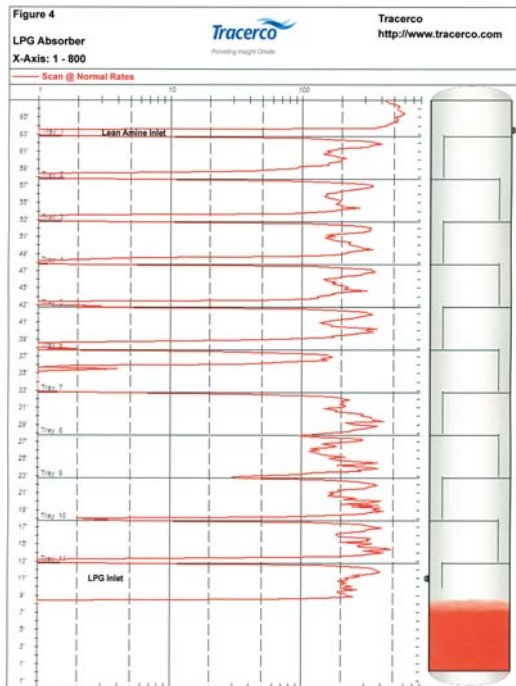


Figure 4 – Illustrates a scan plot of a trayed extraction tower where amine is the heavy liquid phase and LPG is the light liquid phase. A build-up of the heavy phase on Tray 7 showed a liquid restriction where the amine had trouble flowing past Tray 7.

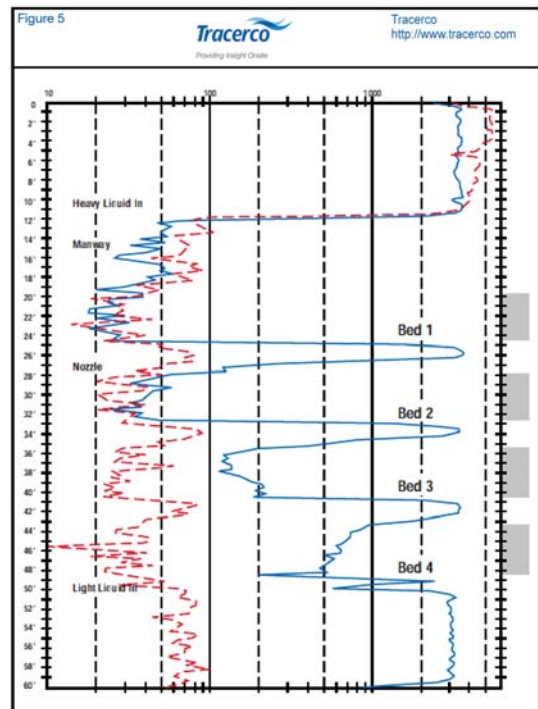


Figure 5 – An illustration showing the scan results from an extraction tower with packed beds. The blue-solid scanline illustrates scan results of the tower with operating problems. The red-dashed scanline indicates the process density being uniform throughout the tower.

two liquid phases were well mixed then the scan profile would be nearly a straight line due to the process density being uniform throughout the tower. The red-dashed scanline curve in Figure 5 shows this. The blue-solid scanline shows what the extractor was in good operating condition due to all the liquid. However when the operation was bad, the pockets of light liquid trapped under each bed in the scan clearly identified where the beds were. It is ironic but a scan of a well operating liquid-liquid extractor usually reveals less information than a scan of an extractor with problems. First, pockets of lower density material were noticeable below each bed, showing that the light liquid phase was not able to flow through the packed beds. Second, Beds 3 and 4 were much lighter in density than Beds 1 and 2. Thus the tower was not well mixed but holding the light liquid phase in the bottom and keeping the heavy liquid phase in the top. This situation was due to the bottom of each bed of packing having a layer of fouling. The scan results showing good mixing were obtained after the extractor tower had been cleaned and returned to service (red-dashed scanline).

One indirect result from the scan when the tower operation was bad was being able to determine that the bed placement was correct. Normally it would be very difficult to see where the beds are located from a scan when the extractor was in good operating condition due to all the liquid. However when the operation was bad, the pockets of light liquid trapped under each bed in the scan clearly identified where the beds were. It is ironic but a scan of a well operating liquid-liquid extractor usually reveals less information than a scan of an extractor with problems.

If you are considering scanning a tower with a large diameter, thick walls or a liquid-liquid extractor and you think that you might not receive adequate results please contact us to discuss your tower. There may be options to consider to help diagnose any problems present.

# Empirical Data for Validation of Reactor Design

By Dave Ferguson, Business Development Manager – Tracer Applications, Pasadena, Texas USA

Even though formulas abound and experience is plentiful for successful reactor design there is still a degree of uncertainty present. Engineers and chemists programme computers, wear out calculators, and scratch their heads until they often have to make an educated guess. However there are ways to gather empirical data to validate reactor design using tracer technology.

## Steps to a Successful Reactor Design

The first step is to find all the published information available about the reaction that is going to occur, including temperature, pressure, pH of components and products, etc. From this information, some calculations and presumptions can be made as to the size, materials of construction, heat transfer to expect, etc.

The second step most projects go through is to build a pilot plant. In the pilot plant, various conditions can be trialed to determine the optimum conditions, degree of mixing, component ratios, etc.

The third step is to extrapolate the theory and the empirical data for scale-up of the pilot plant into a production unit.

The fourth step is to build and start up the production unit. Early in the life of the production reactor, the performance is measured and evaluated. Unfortunately, the performance is sometimes less than expected.

The fifth step of a successful reactor design, therefore, is to understand why it is not operating at optimal performance and plan modifications to improve performance.

## Information Gathering

One of the most common questions asked about reactor performance at both the pilot plant and production stages is “What is the residence time and distribution of the components in the reactor?” With all the instrumentation that is available to monitor the reactor, this question is surprisingly difficult to answer. This question is followed by another, “What techniques are available that can be used to gather information about an on-line reactor so the design can be improved?”

The answer is that tracers can be used to gather this information. Radioactive tracer can often be injected and its movement and timing can be determined using external detectors. However, this technique does not work in every situation. In those cases, a radioactive or chemical tracer can be injected and samples can be collected from the outlet stream for analysis.

If the reactor is supposed to operate in some regime that is near plug flow, then a detector on the inlet and one on the outlet will produce responses similar to those shown in Figure 6. Using the Method of Moments analysis, the Mean Residence Time (MRT) and the Inverse Peclet Number can be calculated.

If the reactor is supposed to be operated in a reasonably well mixed regime, then an inlet and an outlet detector will produce responses similar to those shown in Figure 7 and the Method of Moments analysis can be used to calculate the MRT and the Stirred Tank Equivalent (STE).

## First Case Study

One company operates a gas phase reactor with two

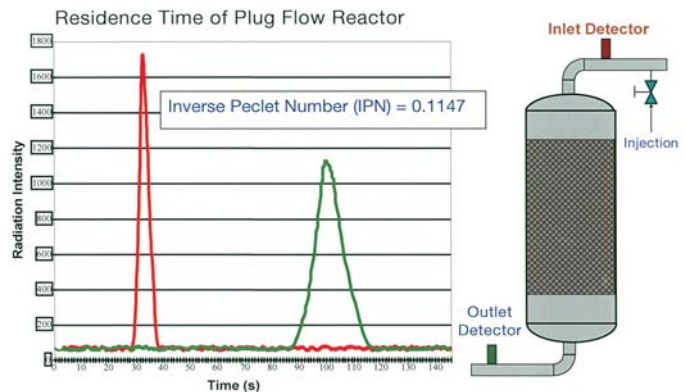


Figure 6 – Illustration of a reactor that is supposed to operate in some regime that is near plug flow. Detector placement on the inlet and one on the outlet will produce responses similar to the above illustration.

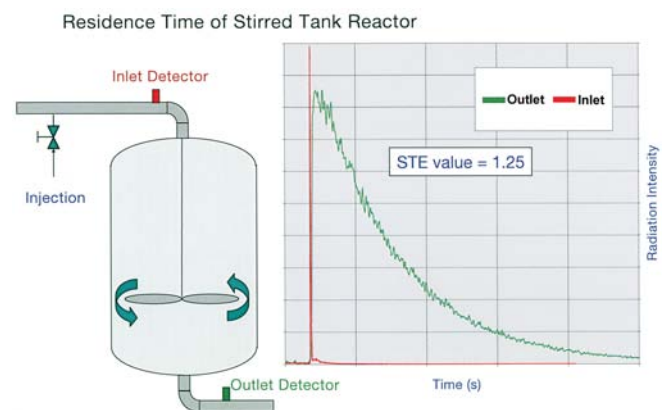


Figure 7 – Response data from a reactor operating in a reasonably well mixed regime with an inlet and outlet detector placement.

fixed catalyst beds. Upon startup, mal-distribution was suspected to be the cause of lower than expected conversion. Four rings of six detectors were positioned at the elevations shown in Figure 8.

The first set of results (Figure 9) show the responses of the detectors in the top ring of the upper bed. It is obvious that the responses are not all of the same height or width. Using the Method of Moments analysis, the areas under each response curve were calculated. The areas under all six responses were summed and then each

area was divided by the summed area to calculate a percentage.

The radar plot in Figure 9 shows that almost 20 percent of the flow was measured near each of the NE and E detectors, about 17 percent at the NW and SE detectors, and 11 and 8 percent at the SW and W detectors respectively. Since each detector should have seen 16.7 percent, a total of 40 percent to the NE and E and only 20 percent to the SW and W, indicated serious mal-distribution was present.

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# Reactor Design

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The second graph (Figure 10) shows the results of the ring of detectors at the bottom of the lower bed. The detector responses were more similar to each other, but there was still a 58 to 42 percent flow bias to the east side of the reactor. The Mean Residence Time (MRT) was measured at 36.8 seconds from the upper bed top ring to the lower bed bottom ring and the overall MRT was 53.4 seconds.

The engineering staff operating this reactor took the opportunity at the next scheduled shutdown to change the inlet distributor and the redistributor between the beds. When the reactor was restarted, the performance improved.

## Second Case Study

A North American plant that converts corn into ethanol for blending with gasoline wanted to determine the Mean Residence Time (MRT) of the fluid through one of their fermentation tanks. The tank was 30.5m in diameter and had a liquid depth of about 8.5m, holding 6.1 million liters of water, corn, and sugar (Figure 11). One of the primary objectives was to determine if short circuiting of the feed was occurring.

Due to the size of the tank, this project would have taken an excessive amount of radioisotope tracer. Instead, 1000kg of salt was dissolved in a 3785 liters portable tank with a mixer, the salt water was pumped into the fermentation tank, and then samples were taken of the tank effluent every 10 minutes for over 10 hours. The samples were sent to a nuclear research

reactor for Neutron Activation Analysis. Each of the samples was briefly exposed to neutron radiation from the nuclear reactor which made the sodium atoms in the samples radioactive. The samples were then counted with a gamma spectrophotometer to determine the concentration of sodium in each sample.

The Method of Moments modelling for non-ideal flow was used for the calculation of the MRT and Stirred Tank Equivalent (STE). Figure 12 shows the test results.

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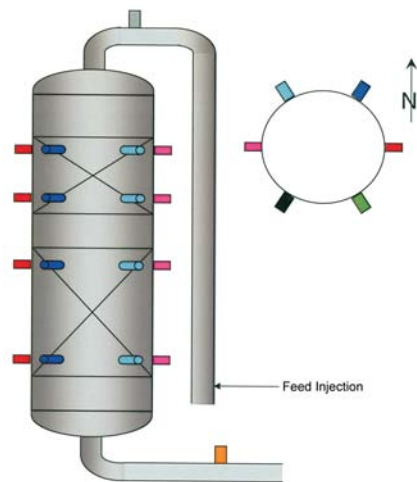


Figure 8 – Four rings of six detectors positioned at elevations show to produce results illustrated in Figures 9 and 10.

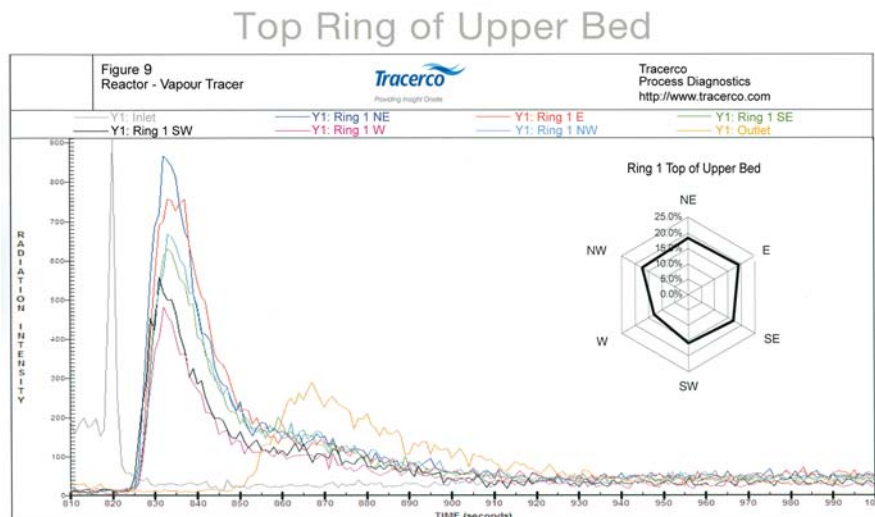


Figure 9 – The first set of results using the Method of Moments analysis indicated serious mal-distribution was present.

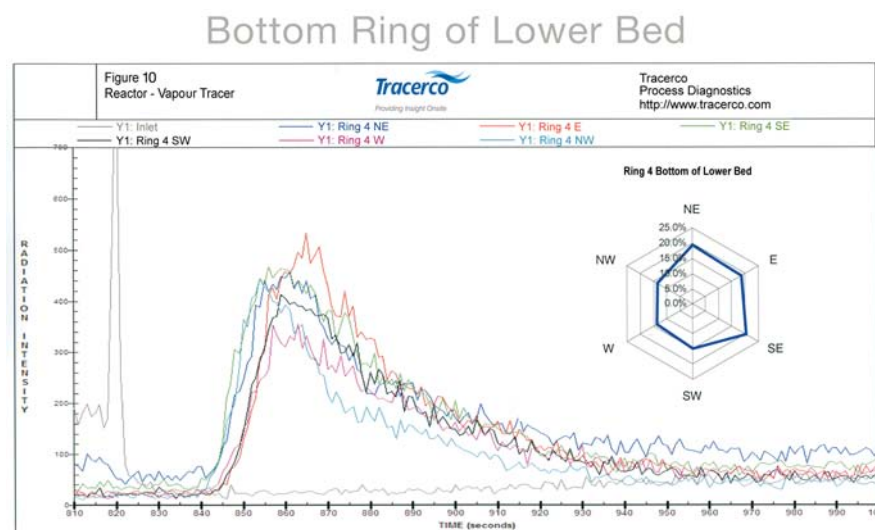


Figure 10 – Results of the ring of detectors at the bottom of the lower bed indicated there was still a 58 - 42% flow bias to the east side of the reactor.

## Reactor Design

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An STE of 2.25 indicated that complete mixing would not occur unless there were at least three tanks. Short circuiting of the raw feed through the vessel had also been a concern, however the study found no evidence to support this.

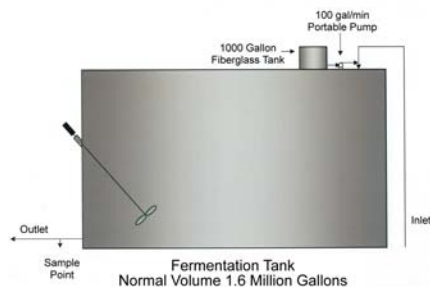


Figure 11 – A North American plant requested Tracerco to determine the Mean Residence Time (MRT) of fluid through one of their fermentation tanks. One of the primary objectives was to determine if short circuiting of the feed was occurring.

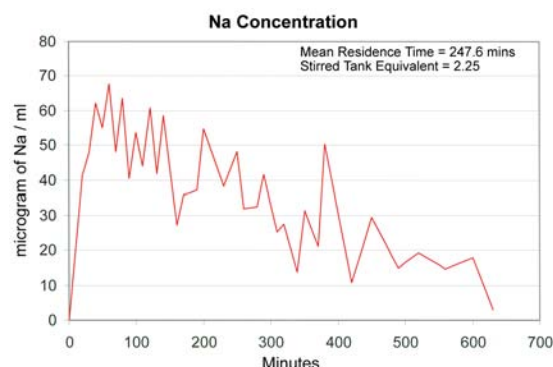


Figure 12 – The Method of Moments modelling for non-ideal flow was used for the calculation of the MRT and Stirred Tank Equivalent (STE).

### Conclusion

Some aspects of reactor performance are difficult to measure. Tracer technology can be used in pilot plant and full scale facilities to gather critical information needed for optimal reactor design.

## Tracerco Distillation Forum Available

Tracerco is often contacted by experts that are consulting or working on large Distillation or FCCU projects to provide field data for troubleshooting and optimisation. Tracerco uses this vast array of knowledge to feature case study articles at industry conferences, webinars, and on-site technical presentations for our customers. Our Team of Distillation, Heat Exchanger and FCC specialists have a

huge amount of practical experience along with the hundreds of job case studies to refer to for identifying process problems. This knowledge base provides our customers information of specific applications and techniques used worldwide to trouble-shoot and solve their process problems.

Tracerco has conducted multiple distillation forums describing and providing

examples of varied techniques useful for trouble-shooting and diagnosing hydraulic conditions within process equipment. Anyone who plays a role in process troubleshooting and optimisation is welcome to contact Tracerco to discuss scheduling a technical forum for your plant-site.



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