Tracerco News

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Delivering Precision Diagnostics Worldwide.

Chemical Tracers Find Elusive Leak in Hydrotreater Feed/Effluent Exchangers

Co-Authored by Dave Ferguson, *Tracerco Business Development Manager – Tracers* and Troy Witherill, *Refinery Process Engineer*

High sulfur content in the effluent of a hydrotreater stream is a signal that the unit has a problem with either the reactor catalyst bed or the feed/effluent exchangers. Plant resources can often isolate the offending vessel by analyzing samples online or pressure testing exchangers offline. These approaches take time and can be expensive when they involve a shutdown to search for the problem. At times, conventional efforts fail to locate the offending vessel. An online technique using radioisotope tracers has been used for many years to leak test exchangers benefiting the plant by minimizing lost production costs of a search during a shutdown. But, every technique has its limit and some leaks are too small to measure using radioisotope technology. A new service using chemical tracers has been employed by Tracerco to find very small leaks.

Identify the Problem

A Mid-West Refinery in the U.S. operates a Naphtha Hydrotreater with a bank of four feed/effluent exchangers (Figure 1). The flow through the Hydrotreater varies from 5,000 to 10,000 bbls/day. The effluent from the reactor is cooled by the exchangers and then sent as feed to the Platforming Unit. Results of Platformer Feed sample analysis showed that sulfur levels, which had been between 0 to 2 ppm, were now fluctuating between 2 and 10 ppm. Figure 2, on page 2, shows a distinct trend to higher sulfur levels. Plant personnel had tried to determine which exchanger was leaking by sample analysis, but had been unsuccessful. They contacted Tracerco to gain assistance in identifying the leaking exchanger before taking an outage to fix the problem.

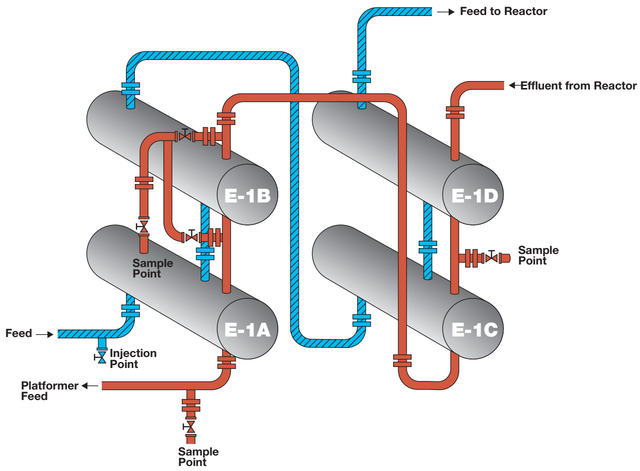

Figure 1 – *Four Feed/Effluent Exchangers in the Naphtha Hydrotreating Unit*

Leak Study Methods

Tracerco has a long history of performing online heat exchanger leak tests using radioactive materials and helium as tracers. The standard technique is to mount radiation detectors on the feed and effluent lines of each exchanger and inject a radioisotope tracer that is compatible with the feed stream. As the tracer flows through the exchangers with the higher-pressure feed stream, the detectors on the feed piping will respond to the passage of the tracer, producing bell-shaped Gaussian

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response curves on the computer. If one of the exchangers has a leak, the leak to the effluent side will be at the same concentration of tracer as its percentage in the feed. Though diluted by the effluent, the detectors on the effluent exit lines will respond, producing a response curve on the computer. By comparing the area under the leak detector response curve to the area under the feed detector response curve, the percentage leak can be approximated.

The limitation of this technique is in its sensitivity. This technique is the easiest and quickest way to perform an on-line leak test if 0.5 percent or more of the feed is leaking into the effluent. Sometimes, the sensitivity can be improved to 0.2 or even 0.1, but the more typical limit is 0.5 percent.

Smaller leaks can be found with radioisotope tracers by a sampling technique. The tracer material and injection procedure are exactly the same as before, but instead of mounting detectors on the feed and effluent lines, sample points on the effluent lines have to be established. Sample points generally consist of a sample cooler where hot effluent is condensed/cooled and collected in sample containers. The samples are counted with a very sensitive radiation detector over several minutes. Compared to the online tracer technique where the tracer material flashes past the detector in a few seconds, much smaller amounts of radiotracer can be measured in the samples through accumulation of counts over time. This technique has been used to find leaks as low as 100 ppm. Unfortunately, finding smaller leaks than this requires the injection of so much radiotracer that its disposal becomes an issue as well as the safety of the crew performing the leak test.

Helium has also been used as a tracer for finding small leaks online, but the leak size can not be quantified. An article about this technique is featured on page 3.

New Tracer Approach

To address the need to find leaks smaller than 100 ppm, Tracerco researched chemical tracer technologies. We identified a range of compounds, some of which are compatible with organic and some with aqueous streams, that are chemically and thermally stable, and can be readily detected in samples by specialized gas chromatography. These chemicals can be found in hydrocarbon samples at concentrations as low as 1 part per billion (ppb).

In the case of the U.S. Mid-West Refinery the four feed/effluent exchangers each had a sample point, which meant the tracers could be injected into a single injection point (Figure 1). Each exchanger was tested with a separate tracer. This allowed a two man crew to test the four exchangers. One tracer could have been used, but all four exchangers would have to have been sampled at the same time, requiring a five man crew. It was more cost-effective to use two people and four tracers.

Because the effluent stream contained hydrogen sulfide (H_2S), the decision was made to collect the samples in stainless steel sample cylinders instead of using a sample cooler. Hydrogen and H_2S would have been continuously released to the atmosphere while collecting the samples with a sample cooler. Ten sample cylinders were mounted to a manifold at the first sample point. As the tracer was injected, the first sample cylinder was opened and closed in 10 seconds. The other nine sample cylinders were opened and closed at 10 second intervals. The sample cylinders were then removed from the manifold and immersed in an ice bath to condense the naphtha and tracer. Each cylinder was depressurized through a tube containing activated charcoal and the naphtha in the cylinder was collected in plastic bottles.

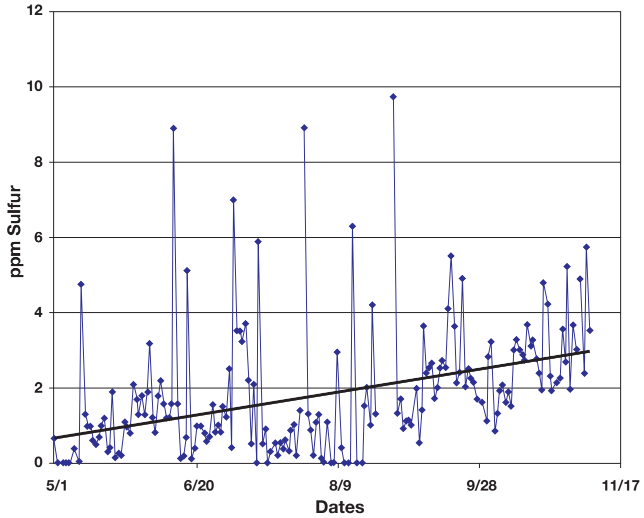

Figure 2 *Platformer Feed Sulfur Levels*

The activated charcoal tubes and the sample bottles were sent to the Tracerco lab for analysis. The results showed that three of the sets of samples were negative. The set of samples for the E-1B exchanger showed tracer in samples 5 through 10 (Figure 3).

Unfortunately, the tracer was slower coming through the exchangers than had been expected. The ninth sample had the highest reading and the tenth sample was the first sample on the trailing edge of the response curve. If another 10 samples had been caught, there would have been tracer in samples 11 through 13, at least, and probably in a few more as the trailing edge of a response curve is usually more drawn out than the leading edge.

For this analysis, however, the conservative approach was applied and a short trailing edge was extrapolated. The average concentration for the samples was determined and the grams of chemical tracer that passed the sample point while the samples were being

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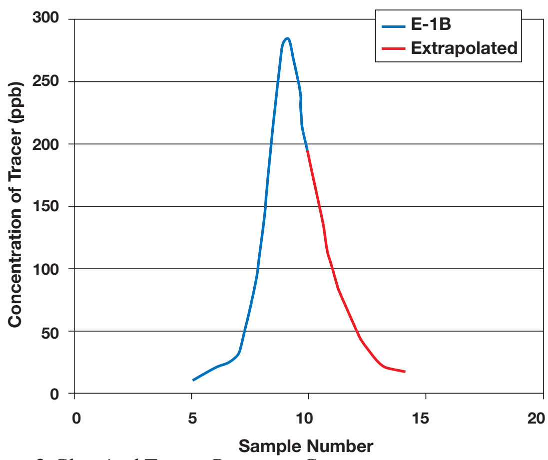

Figure 3 *Chemical Tracer Response Curve*

Online Helium Leak Test Results Assist Operations to Examine a Hydrotreater System for Off-Spec Product

Co-Authored by David Tidwell – *Sr. Project Technologist* and Rodney Thompson – *Principal Project Engineer, Pasadena TX, USA*

Online and offline helium leak detection is appropriate for a wide range of industrial applications because of its inherent ability to detect exceptionally small leaks. Helium's inert nature and small atomic size allows it to pass through the smallest of leaks and does not readily react with any other substance. It is non-toxic, non-hazardous and non-destructive.

The application of TRACERCO Diagnostics™ Helium Leak testing can assist in decreasing downtime, reduce maintenance/repair costs and assure process system integrity.

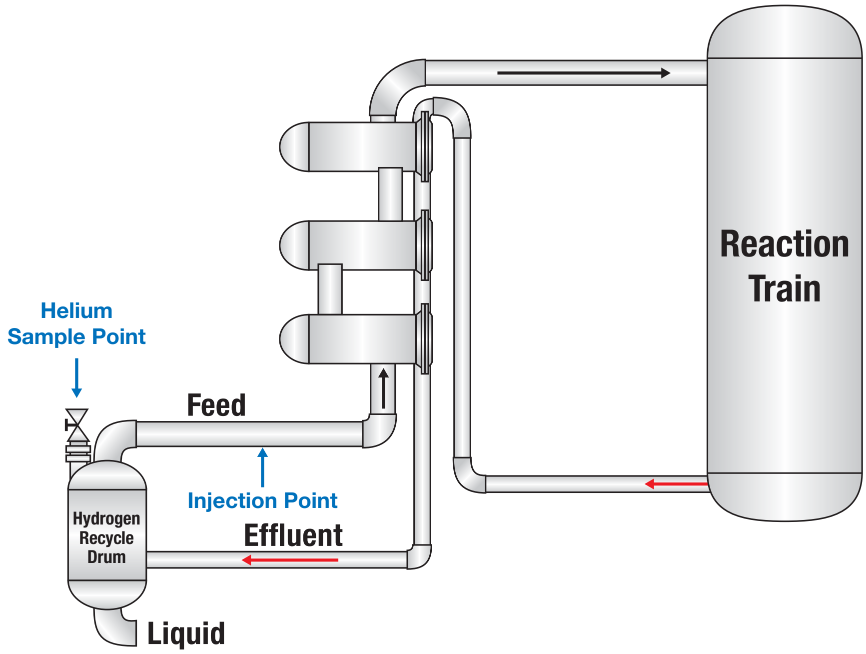

Figure 4 *An online helium leak test was performed on the Reactor Feed/Effluent Exchangers to confirm or deny the existence of a leak.*

Helium Case Study

A facility on the Gulf Coast of the USA was experiencing QA/QC difficulties with a Jet Fuel Thermal Oxidation Test on product from their hydrotreating unit. Cross contamination of the product stream with untreated feed was suspected to be causing the product to fail its quality test. There were three possible locations that could have been the source of the contamination including:

- Stripper Bottoms/Feed Exchangers
- Product Recycle Line
- Reactor Feed/Effluent Exchangers

Laboratory analysis of the product stream into and out of the stripper bottoms/feed exchangers indicated no leakage and so these were discounted as a source of the contamination. Additionally, the product recycle line was eliminated as a source of contamination as the pressure differential between the feed and product sides made it impossible for the untreated feed to flow into the product. The last possible source of contamination was the reactor feed/effluent exchangers. It was determined that a helium leak test could provide information on whether one or more of the exchangers within this system were leaking. The reactor feed/effluent exchanger system was studied to determine the best method of conducting a leak test. Due to the very high operating temperature and significant presence of hydrogen sulfide gas within the system a full risk assessment was carried out to determine the most appropriate injection and sample points. Injection could be carried out upstream of the exchangers into the feed line without any issues. It was determined that it would be more safe to sample from a point downstream of the exchangers using a sample point on the hydrogen recycle drum. Injection and sample points selected for the helium leak test are shown in Figure 4.

A forty five second concentrated slug of helium gas was injected into the feed cycle of the exchangers. The recycle gas off the top of the recycle gas knock out drum was monitored using a portable helium mass spectrometer unit to detect the presence of helium. This involved the purging of a small amount of the off gas across the path of an analyzer probe connected to the mass spectrometer. It was determined from a previous test carried out under very similar conditions that if a leak were present, a helium response would be detected in approximately four minutes. System cycle time was calculated to be around ten minutes. No helium response was detected in the time window expected for a leak. A helium response was detected after eleven minutes. This response was determined to be the recycle gas and not from a leak. It was concluded that no leak was present within the exchanger system.

The lack of a helium response indicating that there was no leak within the reactor feed/effluent exchanger system provided the facility with a confirmation that all three potential leak points within the hydrotreating unit were not the source of the off spec jet product. This allowed the team to concentrate their efforts on looking at other possible upstream and downstream processes to determine the cause of their problem.

TRACERCO Diagnostics™ Scans Monitor Fouling using Comparative Scan Results over Several Months

By Lowell Pless – *Business Development Mgr. – Sealed Source Technologies, Pasadena TX, USA*

In some processes a common problem that towers experience over a period of time is fouling of trays and packing with solid deposits. Fouling can cause conditions such as liquid maldistribution in packed columns or premature entrainment/flooding in trayed columns resulting in a loss of efficiency and an increase in operating pressure drop. Fouling may result from foreign material entering a column or from polymerization or decomposition occurring inside the column. The fouling may start under normal conditions or begin to build due to abnormal operating conditions.

Many operators implement a routine maintenance program where the column is periodically scanned to monitor the progress of the worsening condition over time. This type of program typically begins with a baseline scan of the column. This is especially important with a condition like fouling since the scan results usually show the problems being caused as a result of the fouling rather than detecting the fouling directly.

Baseline scans provide a valuable reference that can be used to identify and monitor patterns in column performance. A baseline scan establishes a reference point, a historical standard of column performance. The baseline scan should be performed when the column is clean, known to be in good mechanical shape, and operating with no known problems. A baseline scan of a distillation column eliminates uncertainty on future column scans, and enhances the accuracy and sensitivity of all future scans. Subsequent scans will reveal areas that are fouled or plugged in the early stages of formation and may allow operational changes to slow or halt the fouling process; allow correlations to be drawn between operating conditions and the rate of fouling; or measure the rate of deterioration caused by the fouling to better prepare needed corrective action.

Case Study

Two years ago a US Chemical Plant had replaced some packing in a tower that periodically experienced high pressure drop due to fouling. The customer contacted Tracerco to conduct a baseline 2 x 2 Grid Scan first to determine if the startup had proceeded without incident so that no liquid maldistribution or other problems were present, and second to document the hydraulic operation of the column for future reference particularly with regard to being able to monitor for fouling.

The baseline scan results are shown in Figure 5. For purposes

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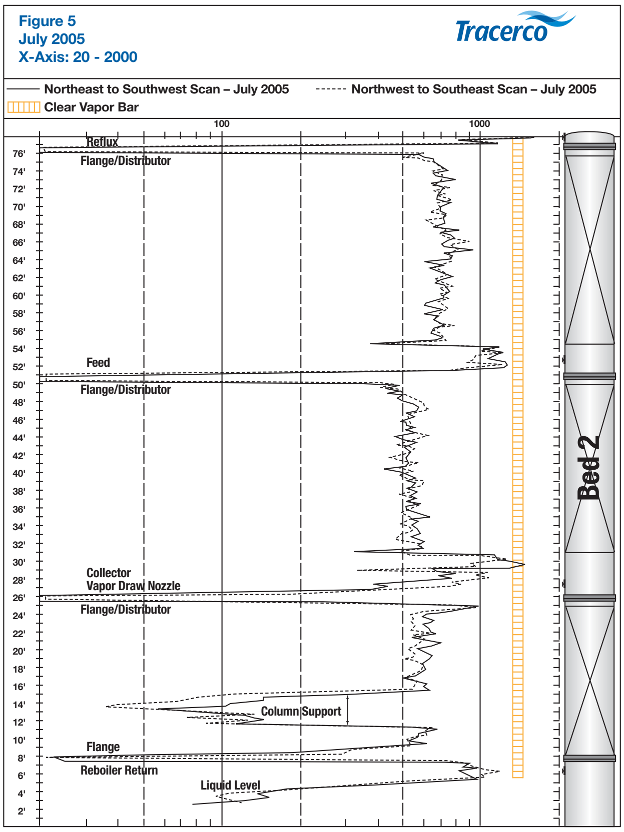

Figure 5 *July 2005 baseline scan results indicated all three beds of packing were in place and exhibiting good liquid/vapor distribution. For the purposes of demonstration only two of the four scanlines are shown.*

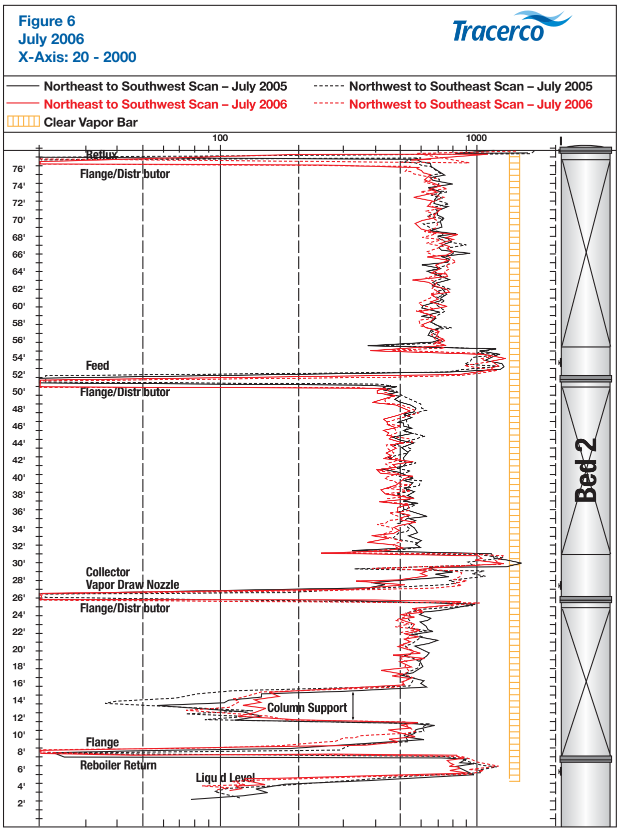

Figure 6 *July 2006 another scan was performed to check the columns condition.*

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collected was calculated. When this result was divided by the total grams of chemical tracer that was injected, the resulting percentage leak was revealed. The results indicated that the rate of leakage of the feed into the effluent was 0.0044 percent or 44 ppm at the time of the test.

At a convenient time for the refinery, the unit was brought down and the E-1B exchanger was pressure tested with water. One tube had an obvious leak and four other tubes had small leaks. The tubes were plugged and the unit was brought back online. The sulfur levels returned to the 0 to 2 ppm level.

Conclusion

The plant personnel were able to avoid lost production from a longer shutdown and the maintenance costs of opening and hydrotesting the other three exchangers. This project demonstrated the capabilities of the new approach to leak testing exchangers. Ultra Low Sulfur Diesel Hydrotreaters operate at such low sulfur specifications that very small leaks in the feed/effluent exchangers can result in off-specification effluent. With the new chemical tracers that are now available, leak rates as low as 5 ppm can be detected.

Fouling

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of demonstration only two of the four scanlines of the Grid Scan are shown.

All three beds of packing were in place and at their proper elevations. Beds 1 and 2 exhibited good liquid/vapor distribution. Bed 3 was mostly obscured by a huge metal band used to support the column in its structure. Even so the scan showed some slight liquid maldistribution in the top of the bed. This was the only area of the column that exhibited any cause for concern.

One year later a scan was performed to check on the integrity of the column internals. The column was still operating well with no problems but it was decided to check the packing density to be sure there was no indication of fouling. The results of this scan are shown in Figure 6, on page 4, along with the baseline profile obtained earlier. As can be seen in Figure 6 the results showed the column was in the same good condition and was operating the same as when the column was scanned previously – no sign of any fouling.

In July 2007 the column was scanned again to check the column's condition. A small increase in ΔP had been observed and operations staff were curious to see if any telltale signs of fouling would be observable from the scan results. Figure 7 shows the scan results compared to the baseline data. The results appeared simi-

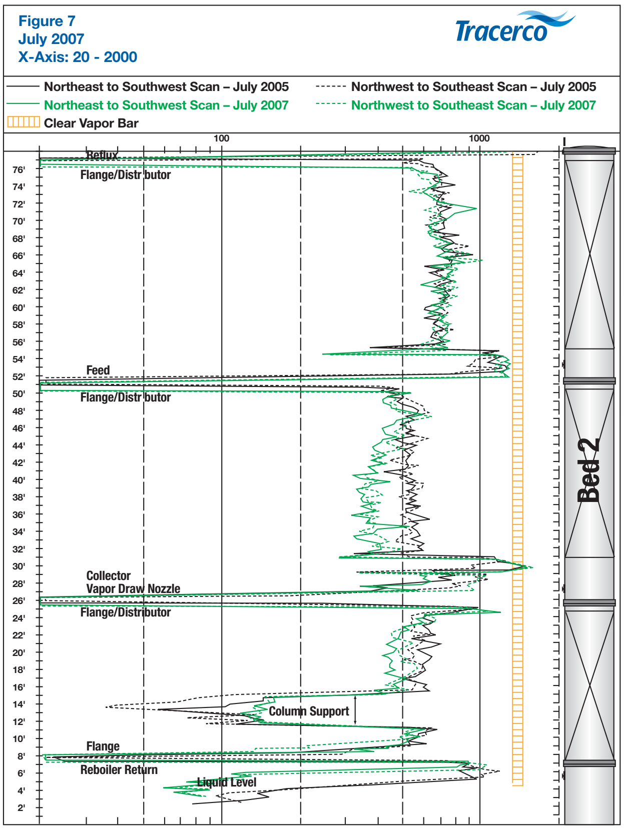

Figure 7 *Illustrates an overlay of the July 2005 and July 2007 scan results. The results appeared similar to the baseline; except Bed 2 had a slight increase in density towards the bottom of the bed.*

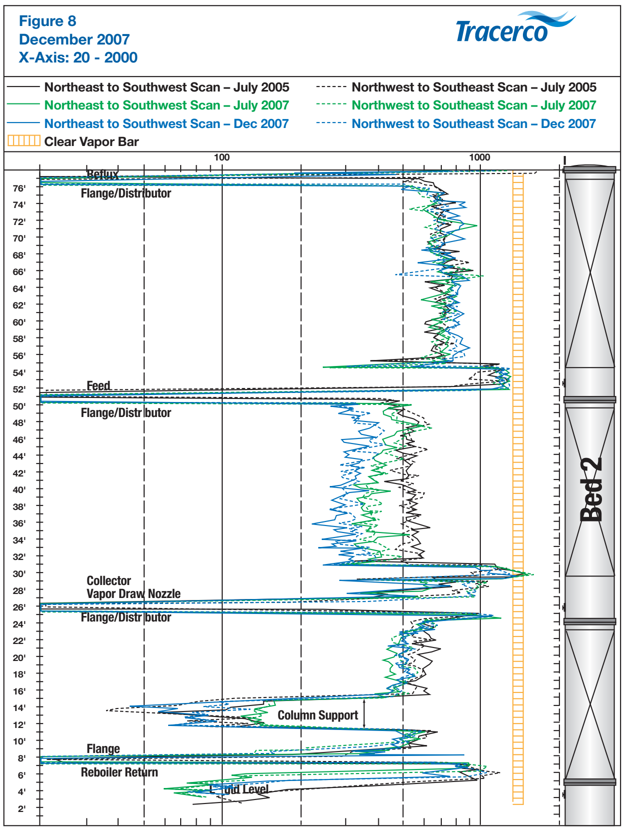

Figure 8 *A few months later the column's operation was pronouncedly worse. When compared to the previous scans the density of Bed 2 had increased.*

Fouling

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lar to the baseline; except Bed 2 had a slight increase in density towards the bottom of the bed. This would be an early indication of liquid holdup due to fouling.

A few months later the column's operation was pronouncedly worse. The column was experiencing poorer separation and an elevated ΔP. Fouling and/or flooding was suspected. The column was scanned in the same manner as the previous scans and the results are presented in Figure 8 with the scan results compared to the baseline data and the previous scan conducted in July 2007. When compared to the previous scans the density of Bed 2 had increased. This shift in density was very likely due to increased fouling and liquid hold-up.

One question you might pose is, "Wouldn't these results have been obvious if this most recent scan had been the first scan ever done on this column?" The answer is, yes and no. It is correct that the difference in density between Bed 2 and Bed 1 would have been obvious but without historical data on the beds' relative densities we may not have been able to confidently come to the conclusion that Bed 2 was fouled.

Why not? For one thing the density profile through Bed 2 was consistent. Typically when a bed of packing has fouling there is a dense area where liquid is held up due to the fouling restriction causing a density gradient through the bed. Figure 9 shows this typical kind of result. Second the liquid distribution in this column was not too bad. Usually with fouling, the fouling pattern itself is not uniform so there is usually obvious liquid maldistribution as can also be seen in Figure 9.

Had this been a first-time scan we should ask the question, "Is the liquid load on both Beds 1 and 2 the same?" If the liquid loads are the same and the customer confidently knows this (and the packing material is also the same) then making the conclusion of fouling is not such a "stretch" but, many times this information is not known. Or perhaps there is an operator who remembers that back a few years ago when the column was opened during a turnaround Bed 2 was fouled. However, with the perspective of having the baseline data and the monitoring scans, there is very little doubt about the conclusion of fouling in Bed 2.

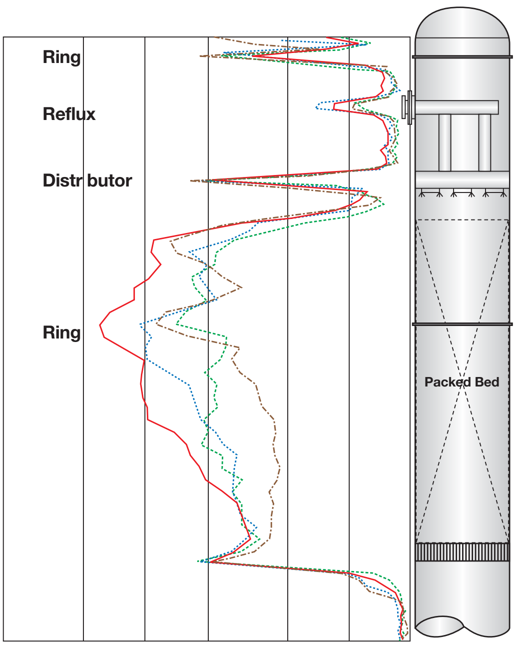

Figure 9 *Typically when a bed of packing has fouling there is a dense area where liquid is held up due to the fouling restriction causing a density gradient through the bed.*

Conclusion

The practice of establishing baseline data enhances the quality of all future scan interpretations. With baseline results to refer to, our skilled professionals can detect very subtle changes in a process column with either trays or packing. A baseline scan is most effective when the column has been entered recently, and the condition of internal hardware has been documented in the form of mechanical inspection reports and photographs. The value of baseline scans is further enhanced when the material balance and operating details are documented in conjunction with the scan results. Additional information, such as simulations, the performance of auxiliary equipment, and the ratings on trays, packing, or distributors may also be useful at a later date. Baseline scans can also be used to evaluate the effects of tower revamps, and to document the start-up of a column.

For situations where you have a fouling process, or other type of deteriorating condition, a monitoring program could save you from a process upset and a lot of misery. By monitoring the progress of the deterioration you can plan for remedial action before the condition gets "out-of-hand".

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Aberdeen, UK
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