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## **SYSTEM AND COMPONENT EXAMPLES: SYSTEMS THAT WORKED AND WHY THEY WORKED; SYSTEMS THAT DID NOT WORK AND WHY THEY DID NOT WORK**

This chapter is a series of anecdotes and examples that, collectively, could be referred to as “war stories.” Stories in which I have not personally participated are credited to their authors.

### **A CEMENT COMPANY IN IOWA**

The problem was an unacceptable high level of maintenance on a process analyzer sample-conditioning system. The plant was spending approximately 4 man-hours (2 men 2 hours each) per shift to maintain flow through the sample probe to the process analyzers that were critical for the process operation. The sample probe was sampling a hot (1000+°F/1832+°C), wet (there was an 80GPM water spray nozzle immediately upstream of the sample probe entry port), dirty (a cement kiln tumbles a fine limestone dust along with powdered additives to create the granular kiln product we call cement) sample gas from the end of an operating cement kiln.

The process analyzer sample probe would plug up with a concrete-like material approximately every 30 minutes, even though it had an electric heater around the probe head, blowback air connected to the probe head, and the heated probe head and blowback air valves were enclosed in an insulated probe protection enclosure. The maintenance personnel would open the probe protection enclosure, remove a threaded plug from the enclosure end of the probe assembly, insert a solid metal rod slightly smaller than the internal diameter of the probe, and proceed to hammer on the atmospheric end of the rod with a sledge hammer until it drove the concrete-like plug out of the process end of the probe and into the operating cement kiln.

The area where the probe was installed was inside a huge building enclosing the end of the rotating cement kiln (approximately 10 ft/3 m outside diameter) and up at the fourth level above the ground (approximately 60 ft/18 m above grade). Maintenance of the probe required climbing a 10 ft/3 m ladder up to a small platform beneath the end of the probe. The ambient temperature in the area of the platform was approximately 80 to 90°F/27 to 32°C in the Iowa wintertime and probably 140 to 150°F/60 to 65°C in the Iowa summertime.

The process analyzer sample probe had a blowback line installed into the probe with valves installed to sequence a sample flow shut-off to the process analyzer when blowback of the sample probe was taking place. The process analyzer sample-conditioning system was programmed for blowback to occur for 30 seconds every 10 minutes. This was designed to keep the probe clear for continuous process analyzer sample operation. The valve sequencing was designed to protect the process analyzer system’s integrally heat-traced and insulated process analyzer sample transfer line, and to keep the process analyzers at line’s end from experiencing the high-pressure blowback air if the kiln end of the process sample probe was plugged during blowback.

The source of high-pressure blowback air was the plant instrument air system that experienced occasional line blockage from condensate freezing in the line at Iowa winter temperatures of -20 to -30°F/-29 to -34°C. This plant instrument air system maintained a main system pressure of approximately 85 psig/586 kpag. The high-pressure blowback air was delivered to the process analyzer sample probe by approximately 80 ft/26 m of  $\frac{1}{4}$  in./6 mm copper transfer tubing. Whenever the high-

pressure blowback system was actuated, there would be an initial short rush of 80 psig/552 kpag heated air followed by a decreasing volume of increasingly cool air (which contained increasing amounts of moisture). This flow would continue at the decreased volume and pressure because of the constriction of the quarter-inch OD delivery tubing.

We assumed that the relatively cool low-volume, low-pressure blowback air was cooling further as it expanded through the blowback block valve into the relatively larger process analyzer sample probe due to Joule-Thompson effect cooling. We further assumed that this Joule-Thompson effect cooling was getting below the dew point of the instrument air source and contributing moisture to combine with the dust coming into the process analyzer sample probe from the operating cement kiln. This moisture, in addition to the moisture from the kiln cooling spray, was combining with the cement dust to form the concrete-like blockage that was forming approximately every 30 minutes inside of the process analyzer sample probe.

In the immediate vicinity of the process analyzer sample probe was a 48 in./1.2 m OD duct with a surface temperature between 600 and 800°F/315 to 425°C. As an initial trial for improving the maintenance interval of this process analyzer sample probe, we changed the instrument air supply line to the probe to  $\frac{3}{8}$  in./9 mm copper tubing, and we wrapped several turns of this tubing around the hot 48 in./1.2 m duct to maintain the air temperature above its dew point during the entire blowback operation. This improved the maintenance interval of the process analyzer sample probe by a perceptible amount.

Next we added two capacity tanks of approximately 0.06 cu ft/2 L each in a parallel configuration inside of the heated-and-insulated probe protection enclosure, and we changed the valve that admitted air to the process analyzer sample probe to a larger internal diameter valve with an electropneumatic operator for absolute fastest positive actuation of this valve. The prior valve in the service had been a simple electric solenoid valve with a poppet-and-seat as its flow path. We changed the blowback time duration and interval to blowing back 15 seconds every 10 minutes. Even though this process analyzer system was being utilized for kiln operation control, its primary purpose was as a CEMS (continuous emissions monitoring system) to meet USEPA reporting requirements and this required 7 minutes of good and steady data out of every 15 minute time period. Thus we could not blowback, as an example only, 10 seconds every 3 minutes. This frequent blowback interval would upset process analyzer sample flow and put an artifact on the output of each process analyzer signal, which would disrupt the 7 minute require-

ment of good and steady data. This improved the maintenance interval of the process analyzer sample probe to plugging only one time every 6 to 10 hours.

Next we added a local air compressor with a 20 gal/~80 L capacity tank that operated at 125 psig/862 kpag. We now set the blowback duration and interval to 10 seconds every 8 minutes. We maintained the duct heating of the blowback air transfer line. When this high-pressure air would discharge from the heated capacity tanks through the large-port, fast-acting valve, it would sound as if a shotgun had discharged in the area near the process analyzer probe protection box. The explosive air discharge would dislodge material from the inside of the process analyzer sample probe, and the process analyzer sample flow disruption was of such short duration that the process analyzer output signal would settle down within less than 60 seconds to give the required 7 minutes of good and steady data every 15 minute time period. Over what has now become many years of operation, the maintenance requirement of this process analyzer sample probe continues to be less than 30 minutes of maintenance required per month of process analyzer probe operation.

#### **TEFLON TUBING COLLECTS WATER BRUCE KEEN, KEEN SYSTEMS**

Teflon tubing is actually very porous to many gases and vapors. Air and water vapor readily permeate through it. A long Teflon tubing run in one customer's laboratory was observed to have liquid water in low points of the line where it ran through an air-conditioned laboratory. The laboratory was located in an area known for its warm and moist climate. The subject tubing run was connected to a compressed gas cylinder that was located in a room that did not have any air-conditioning or humidity control. The analyzer was located in the laboratory that was air-conditioned. The tubing was looped over some equipment, creating low spots in the long tubing run. The low spots were full of water and the sample was bubbling through it. The hot moist air in the outer room had permeated into the Teflon tubing. In the air-conditioned laboratory, the warm moist air had condensed inside of the Teflon tubing and liquid water had formed.

#### **MOLECULAR DISAPPEARANCE IN SOUTH CAROLINA BRUCE KEEN, KEEN SYSTEMS**

An air monitor, located in South Carolina, had 15 sample points. The sample line lengths ranged from 150

to 700ft/45.7 to 213.4m. An aspirator was used to pull the sample through the system.

The analyzer cycle time was 60 seconds. The sample transport lines were  $\frac{1}{4}$ in./6.35mm OD  $\times$  .035in./0.79mm WT type 316 stainless steel tubing. The lines were not heated or insulated, just bare tubing. The sample transport time was 60 seconds for 700ft/213.4m of  $\frac{1}{4}$ in./6.35mm tubing. All sample lines had a sample point inlet filter to keep out ambient dust.

The customer wanted to see 97% of the calibration standard value in three cycles or less. The calibration standard cylinder was attached to the analyzer using 3ft/0.9m of stainless steel tubing. An initial measurement was made. The calibration cylinder was moved to a sample point 350ft/106.7m away and readings were taken. It took over 17 minutes to get the readings above 90% of the calibration cylinder standard concentration.

Several possibilities were discussed. The flow rates and lag times were recalculated. It was decided to try an experiment. A 350ft/106.7m length of black Nylon tubing, coiled up in box, was placed next to the analyzer. It was connected to the calibration standard. Several readings were taken with the analyzer. A reading of over 90% was observed in one minute and a reading of 97% was observed within two minutes. The maximum reading was 98.7%. Heating the tubing improved the level of the first reading by several percent. Since the tubing was in a cardboard box, it was easy to shine a heat lamp into the box, which raised the overall temperature.

It was requested that the stainless steel tubing be flushed out with a solvent to make sure that it was clean. The contractor said it wasn't necessary because he had used brand new tubing and had taken very good care of it until installation. It was finally decided to flush the 350ft/106.7m length of tubing with acetone. The contractor fabricated a tank. Bottled nitrogen was used to pressurize the tank and force the acetone through the tubing. The tubing was flushed with 2gal/7.75L of acetone, and then dried with bottled nitrogen for two hours.

After this treatment, the calibration standard gas flow gave an analysis value of over 90% within 30 seconds. Since the analyzer cycle time was 60 seconds, the calibration standard flow was started 30 seconds after the stream switching selection initiated flow for this stream.

The customer was happy and wanted to consider the problem solved. The customer was informed that heat tracing the lines would further improve the response and prevent freezing of the lines in winter. Since the analyzer system was only sampling air, the customer thought it was impossible for the sample lines to freeze in the winter. The customer was reminded that rain, fog,

and humidity could form condensate at low ambient temperatures, and these would all cause problems if the lines were not heat traced. Finally, the project engineer agreed that the conditions described could be a problem, but management thought it was ridiculous to waste money on heat tracing these sample lines.

### **FREEZE THAT STUFF IN SOUTH CAROLINA BRUCE KEEN, KEEN SYSTEMS**

A call was received from the project engineer at the South Carolina plant in late November. One of the outside lines to the analyzer building had frozen the night before. They had been experiencing water problems in their rotameters but decided to just live with it.

The sample came from a building that was at 75°F/24°C and had a humidity of over 60%. Carpet yarn was the end product and the high humidity kept the static down. The sample line ran overhead, across a parking lot, and then to the analyzer shelter. The line had several low spots in it that were filled with condensate. When the temperature went below the freezing mark, the condensate in the sample line simply froze. When finally the lines actually froze, management decided that heat tracing would probably be necessary.

### **MOLECULAR CREATION IN SOUTH CAROLINA BRUCE KEEN, KEEN SYSTEMS**

The calibration standard was 7ppm. The analyzer would read 7ppm when the calibration bottle was connected directly to the analyzer. When the calibration standard was connected to the end of the 350ft/106.7m sample line, the initial readings would be observed as high as 35ppm. That was 7 times what was being introduced from the calibration cylinder. How can you get out more than you are putting in? Was the problem in the analyzer, in the sample transfer line, or in the calibration standard?

The molecules that were being analyzed had a tendency to hang up on the walls of the sample transport tubing. They would build up until they reached an equilibrium state. The analysis would be fine as long as there weren't any upsets in sample transfer line temperature, pressure, or flow rate.

When the customer tried to do a calibration from the end of the 350ft/106.7m sample transport line, this would cause a sample transfer line flow upset. This flow upset would cause some of the sample transfer line equilibrium molecules to come loose from the walls of the tubing and flow downstream to the analyzer. When

the calibration standard was allowed run for 30 minutes, the analysis would eventually come down to the expected amount. The transport tubing had once again reached an equilibrium state.

Heat-tracing the sample transfer lines would have helped this system. As the sample transfer line temperature is raised, the molecules become more active and less likely to adhere to the walls of the sample tubing. Heat-tracing also tends to remove condensate and other trace contamination problems. The more contamination of any type on the walls of the tubing, the more problems the system will have with equilibrium-based upsets. High flow rates will also help to minimize equilibrium-based upsets.

Many sampling systems and transport lines must operate for a period of time before they reach equilibrium. Some are sensitive enough that they must be heated to work properly.

#### **PROBLEMS WITH DIESEL FUEL BRUCE KEEN, KEEN SYSTEMS**

The analyzer was an NIR unit. It measures the properties of the fuel by shining a specific frequency of light through the sample in a flow cell. The optical receiver, on the other side of the light transmitter, sends the light signal back to the analyzer/computer for analysis. The sample flow cell was located near the process sample point, about 300ft/91.4m from the analyzer. The flow cell was mounted on the sampling system panel.

Diesel fuel has two major sampling problems. The first problem is wax. Whenever the temperature falls below a certain point, the wax forms and plugs up the elements. The second problem is water. All diesel fuel has water in it. Diesel fuel, at some process sample points in a refinery, may have a very high concentration of water. Sometimes the water is dissolved in the diesel fuel; sometimes the concentration is high enough that free water is present. Diesel fuel goes through a series of dryers before it goes to the storage tanks prior to shipment.

The sampling system was a typical medium- to high-pressure liquid system with armored rotameters and Swirlklean filters. The filter elements tended to plug when the temperature was below 40°F/5°C. The Teflon, hydrophobic filter elements were to help eliminate water from the sample when the temperature was low. They did not work with the cold wax buildup.

The NIR analyzer system had a large-bore flow cell. The filters were only to remove the water, not to totally clean the sample stream. If the water were to reach the diesel fuel's cloud point, the NIR analyzer could not perform the analysis. The light beam would

hit the water droplets in the cloudy diesel fuel and the light would be dispersed rather than transmitted through the sample.

The lab determined that the cloud point at times was as high as 100°F/38°C during process upset. The analyzer was installed to help solve process problems. After a series of trial-and-error experiments, the final solution was to remove all filters completely. The sample was heated to above its cloud point by using 50ft/15.2m of integrally steam-traced and insulated tubing. The final sample temperature was determined by controlling the pressure of the heating steam and the flow rate of the process sample. Since the flow cell was totally enclosed in an insulated box, the interior of the box was heated up to near the incoming process sample temperature. The heated sample cell solved the wax problem as well as the cloud problem (no need to remove the water in the process sample).

This procedure won't work on every analyzer, but it is worth remembering for sample systems where it is practical.

#### **BACK FLUSH FILTER SYSTEM BRUCE KEEN, KEEN SYSTEMS**

The sampling system was at a diesel fuel blending station. There were seven pipelines from different suppliers feeding the station. The streams were different quality levels of diesel fuel. The customer was having problems with the system plugging. The filters were being changed several times a day. The customer had been working on the problem for several weeks, so the system had several modifications and additions since it had left the original system integrator.

The system now had a very large process fuel filter, several smaller process parallel fuel filters, a Parker (Balston) sample filter, Swirlklean sample filters, and A+ Genie liquid-liquid sample separators. There had been several different people from different companies working on possible solutions. Each had tried something, which was added to the system and left in place. The system was in South America and the low ambient temperature was still above the wax plugging point. It was decided that wax was not the problem.

The filter elements from the Swirlklean filters were found to be black and slightly caked with particulate. The large process fuel filter, the parallel process filters, and the Swirlklean filters were all in series. That meant 100% of the sample went through the elements of the process fuel filter and the process parallel filters. A bypass was added to the base of the large fuel filter. That would allow the purge of the process pipe feeding the system and reduce the startup lag time without putting



such a load on the process and sample filter elements. Now 80% of the sample bypasses the filter elements and only 20% passes through to be filtered.

The next thing was to replace the Swirlclean filters with parallel filter housings from United Filtration Systems, using five layer screen elements. The United Filtration Systems element is a cylinder made up of concentric screens. The filter housing is set up as a bypass filter. The major amount of sample bypasses through the center of the concentric screens element. This element was chosen because it can be backflushed. It can withstand up to 300psid differential pressure across the element in either direction.

Small tanks were added between the filter elements and the analyzer. They could accumulate some of the filtered sample, so it could be backflushed across the screen element every 15 minutes.

Basically the sample flows through filter A for 15 minutes, with part of the filtered sample going into accumulator tank A. After 15 minutes the valves switch, and the sample flows through filter B and accumulator tank B. Filter A is backflushed with the sample in accumulator tank A. The valves switch every 15 minutes as long as the analyzer is in operation.

The system has been working for an extended period of time and has greatly reduced the filter plugging problems. The customer is satisfied with the results.

## **NAPHTHA CRACKING FURNACE**

### **BRUCE KEEN, KEEN SYSTEMS**

The typical Naphtha Cracking Furnace sampling system has an ONIX Systems "Py-Gas" or Analytical Specialties brand probe at the initial process sample point. This probe is a miniature self-contained distillation tower. It condenses the heavy components back into the process while letting the light components go overhead. The probe has a temperature controller on the overhead component stream that is adjusted to a set point that will give the desired overhead/bottom separation.

The system is useful for analyzing the ratio between different components. If a  $C_3/C_4$  ratio is used, the set point must be above the dew point of all of the  $C_4$  at process pressure. The ratio is a fairly accurate measurement. Measuring for an actual percentage is not practical, since there is no way of knowing how much of the sample was condensed back into the process. The amount returned to process changes and cannot be relied upon to give a percent, or volume, analysis. The ratio is the only usable data that can be obtained.

The sample lines from the process sample probe need to be heat-traced to prevent sample condensation. The entire sampling system must also be heated. Liquid drainers, below coalescing filters, are typically found before both the bypass rotameters and the sample line to the analyzer. If the "distillation" probe fails, you don't want to send liquid sample to your analyzer. Also, small amounts of condensate always seem to find their way overhead in these systems. The liquid drainers are also necessary to remove any liquids that might flood the "distillation" probe and go on to the analyzer.

The following account is from a trip made during the time frame of the Mideast oil embargo. Many of the normal feed stocks were not available, and the plants were using whatever feedstock they could find. The subject sample system had been in operation for several years at a refinery along the U.S. Gulf Coast. The customer had requested a site visit to observe the problems the process sample-conditioning system was having. After this observation, possible solutions were to be recommended.

The customer indicated where the plant was using steam to clean out the process sample transfer lines to the sampling system. The accumulation of spent sample on the ground and against the instrument house looked like chocolate cake batter. It had accumulated in a mass that was several feet across and piled up about a foot deep, where each sample line was routinely blown clean. The accumulation was from many weeks of steaming out the lines. The customer was asked how long they had been required to steam out the process sample transfer lines. The answer was ever since they had to switch to the new and heavier process feedstocks. Some of the feeds actually were heavy enough to plug process control valves.

We looked at the "distillation" probes. Most of them were no longer exercising temperature control of their overhead streams. The temperature controllers had failed, and they had not been repaired. The condensing section of the "distillation" probe wasn't condensing out the heavy sample components and was not allowing them to drip back into the process. These heavy components were getting down into the process sample transfer lines. Since these process sample transfer lines were meant to handle a vapor sample, not a liquid sample, the lines did not have a continuous downward slope all the way from the sample point to the instrument house. The steam tracing was helping to "cook" these heavy components that collected in the low places in the process sample transfer lines. These lines had to be steamed out every week. Many times the lines were plugged before the week ended and the analysis was lost for that furnace.

A “distillation” probe must have several inherent conditions present in order to operate efficiently:

- A hot sample with a high water content at the inlet.
- A cold top section to condense the liquids back into the process.
- A temperature controller to keep the top section at the high-temperature set point.
- A low sample flow rate so the condenser section will be able to handle the cooling load.
- Enough contact area in the lower and middle sections, from internal tower packing materials to efficiently condense the heavy hydrocarbons, allowing them to drip back into the process line.

(For a better understanding of “distillation” probes, contact one of the probe suppliers mentioned at the beginning of this vignette.)

The first recommendation after the initial observation phase was to repair all of the temperature controllers and set them back to their original temperature control points.

The second recommendation was to insulate the top of the “distillation” probe and the temperature control box. This would improve the efficiency of the cooling section.

The third recommendation was to introduce steam at the base section of the probe. By increasing the amount of water available to condense, the lower section of the probe will stay cleaner longer. That should allow more condensate to drip back into the process line. This water condenses before most of the heavy oils will condense. This water coats the metal surfaces, so the heavy oils won't stick. This will allow the heavy oils to drip back into the process as planned.

The last recommendation was to reduce the temperature of the process sample transfer line heat-tracing so that it would be less likely to cook the heavy oils that managed to pass into the overhead stream from the “distillation” probe.

All of the above-mentioned recommendations helped the operation of this process sample-conditioning system. The main fix was the end of the oil embargo and the return to lighter feeds to the process. However, if you have a problem with liquids in a cracking furnace sample conditioning system, check the operation of the temperature controllers as an initial observation.

### **HYDROFLUORIC ACID (HF) BRUCE KEEN, KEEN SYSTEMS**

The System Integration contractor sent two technicians to the field to bring back six sampling systems that had been in process evaluation service for a few months. The

customer was through running some process evaluation tests and the technicians were to salvage the systems for the System Integrator.

The systems had been in use for a period of three to six months. The sample was Butane Recycle and several similar streams. All of the streams contained trace to 1% HF as a process carryover (contaminant). Normal HF carryover levels were expected to be less than 0.1% for most of the streams.

The systems were taken out of service and flushed with a neutralizing solution most probably consisting of dilute aqueous sodium hydroxide or dilute aqueous calcium hydroxide solution. Then they were left open to evaporate the flush liquid. After three months of field evaporation, technicians arrived to take the systems back to the System Integrator's facility.

Technicians were wearing gloves (HF is toxic when absorbed through the skin) to handle all components of these sample-conditioning systems. All of the  $\frac{1}{4}$  in./6.35 mm and  $\frac{3}{8}$  in./9.52 mm Monel process sample transfer lines attached to these sample systems could be crumbled without using tools. All of the liquid bypass filters, hand valves, and compression fittings were corroded beyond use. Everything had to be disposed of at a facility that specialized in handling HF-contaminated material.

Several phone calls were made to ask why the systems were so corroded, since Monel is shown to be compatible with HF in most corrosion tables. The answer given was that oxygen and water both accelerate the corrosion rate. Water does not increase corrosion to the extent that air does.

All of the systems designed since by the System Integrator have had Hastelloy C components. There has been no known failure from HF corrosion since using Hastelloy C components. Monel may be acceptable for noncritical parts if you can ensure that air will never be able to get to the system. Most such problems would be expected to occur during plant turnaround or during routine maintenance. It doesn't take much corrosion to give a metal edge a rough surface. The first time you turn a ball valve, the seal will be scratched and it will never seal again. The same for metal-to-metal seats. Minor corrosion will keep the stem tip from sealing against the seat. From this point on you won't be able to isolate the system from the process to work on it.

It is strongly recommend to use Hastelloy C instead of Monel for HF service.

### **HF, GLASS, AND INSTRUMENT SHELTERS BRUCE KEEN, KEEN SYSTEMS**

A request was received to go to the field to review a process analyzer sample-conditioning system that had

failed and to recommend what would be required to put it back on-line. The process sample was butane recycle and the HF level was expected to be around 0.1% during normal process operation.

The sampling system was designed to select a liquid sample, vaporize it, and neutralize the HF out of the vapor that is created. The neutralization system was a set of internally-packed series/parallel cylinders. Cylinders 1A and 1B were in series but this pair was in parallel with cylinders 2A and 2B, which were also in series. When 1A was used up, both 1A and 1B were to be changed out. 2A and 2B were to be used while 1A and 1B were being replaced. This was supposed to give twice the required amount of neutralizer and ensure that HF would not get through.

There were glass tube rotameters installed immediately downstream of the neutralizer. The glass tubes had been internally eaten away. There was a glass powder refluxing in the sample flow at the bottom of the rotameter housings. That was all that was left of the glass flow tubes.

The instrument shelter was a galvanized steel building. It had a corner missing. The hole was about 2ft along each of the south and west walls and about 1 in. high. The HF residual in the butane sample had eaten the building corner away. The internal parts of the analyzer were corroded, but most of them were replaceable. The sampling system had failed quickly enough that the analyzer internals were not totally destroyed.

The process operators were asked what they thought the HF acid level had been when the system failed. The consensus of opinion was that the HF level was probably 20%. This was probably an overestimate on the amount of HF acid, but levels well above 1% can routinely be expected during process upset operating conditions.

The neutralizer was designed to handle 0.1% continuous HF level in the butane process sample. The neutralizer would survive 1% HF for only a few hours. Many of the materials that will neutralize HF will create water as a by-product during the neutralization process. As the neutralization material gets wet, the area for HF contact is reduced, and the sample begins to channel through the system rather than flowing through for full-area contact.

Materials that rely on large surface area, such as activated alumina, also have a surface problem. Activated alumina has much less of a water problem than many of the other neutralizing materials, but it depends on the HF finding its way into the internal structure of the alumina material. Activated alumina is readily available in different-sized spheres, and all of them are easy to pour into the neutralization cylinders. It has been cited that a  $\frac{1}{2}$ in. diameter sphere of alumina has a square yard

of surface area internal to the sphere due to its porous structure. That sounds impressive but it does illustrate the problem. If the HF can't penetrate to the internal alumina structure, the amount of surface area decreases dramatically. When the HF level increases, there is so much activity on the surface of the alumina spheres that the HF can't penetrate to the internal surfaces. The neutralizer goes into overload very quickly, and anything down stream will get a sample with very high levels of HF acid.

Recommendations that were made:

- Send the system back to the factory to be completely rebuilt.
- Make everything out of Hastelloy C that is in the sample flow path.
- Eliminate the rotameters and use gauges, protected by Hastelloy C gauge protectors across a restrictor, as a flow indicator.
- Hire a System Integrator that is familiar with HF, and let the integrator provide a process sample-conditioning system for you if you are considering building a system where HF may be present.

If that isn't possible, consider the following:

- Don't use neutralizers except as an absolute last resort.
- Make the sample-wetted parts of the analyzer and sampling system out of HF acid-resistant materials.
- Anything that can't be made out of acid-resistant material should be considered disposable and made to be replaced routinely.
- Teflon is very permeable to HF. It is considered a very poor choice for anything in a system with HF. A totally-supported valve packing might survive, but Teflon tubing and fittings are totally unacceptable.
- Reconsider hiring an experienced System Integrator to supply a process sample-conditioning system for your project.

#### **IMPROVED OPERATION OF A PY-GAS SAMPLER DAVID JONES, PREMCOR-LIMA, OH**

The process block valves below each of the two Py-Gas (trademark name) samplers on the catalytic reformer regeneration process were continually plugging. As a result the catalyst regeneration procedure had to be run

without indication of O<sub>2</sub> levels in the reformer during the regeneration process. This is both inefficient and dangerous.

This failure came about as the temperature-sensing capillary corroded away within a few weeks after the installation of the samplers. The materials of construction for the Py-Gas sampler body (Hastelloy C) were chosen correctly for this hot and wet service (moisture saturated at 300°F/149°C and at 300 psig/2068 kpag).

The cooling of the process stream at the Py-Gas sampler mount valve/process block valve generated copious amounts of condensate with a high concentration of hydrochloric acid. The entrained hydrochloric acid was what corroded the temperature-sensing capillary located inside the body of the sampler. The other consequences of no temperature control were that less liquid was generated, more of the particulate in the streams precipitated out in the pre-filter section and was not washed away because of insufficient condensate to properly flush the pre-filter section of the sampler. The amount of condensate that was generated was only enough to evaporate on the process block valve below the sampler and to block the valve opening completely in less than a week of service.

The first problem solved was the choking of the valve. The bottom section of the Py-Gas sampler was replaced during a shutdown with a larger diameter pre-filter and a bigger block valve. This meant it would at least run longer before it blocked in the event that the other proposed remedies for the temperature-controlled section of the sampler did not work.

The other proposed remedies involved buying a new temperature controller and modifying the temperature-sensing portion of the sampler by adding a piece of tubing to the exhaust portion and placing the temperature-sensing capillary in this exhaust stream. It was assumed that the temperature of this exhaust stream would be directly proportional to the temperature of the process sample gas inside the cooling section of the sampler.

Prior to these repairs, it had been typical to have to isolate the Py-Gas sampler every 10 to 14 days to rod out the block valve, steam clean the pre-filter section, and then put it back into service. After these repairs we monitored the system closely for performance. With a few adjustments to the temperature controller setting to solve overcooling/freezing problems, the sampler worked significantly better than before. We next pulled the Py-Gas sampler apart 18 months later during a unit shutdown just to check the internal condition. It was found to be very clean with no sign of impending blockage.

### **FURNACE O<sub>2</sub> ANALYZER** **DAVID JONES, PREMCOR-LIMA, OH**

The extractive-type zirconium-oxide-sensor O<sub>2</sub> analyzer installed on the crude vacuum furnace came with stainless steel eductors. The typical length of service before failure of the eductor was about 4 to 5 days. This was totally unacceptable as the furnace had six analyzers installed. This meant that a technician might have to climb up the furnace almost every day to remove, clean, or replace an eductor. The cost of the eductors being around \$250 each was also unacceptable as they could often only be cleaned two or three times before being discarded due to corrosion because of the sulfur in the fuel gas stream. One must appreciate that even a few ppm of sulfur compounds in the refinery fuel gas with a high constant flow rate and the precipitation problem caused the blockage and corrosion within the short time period noted.

One fix tried was heating the motive air going to the eductor in an attempt to reduce the tendency of the sulfur compounds to precipitate. No significant improvement was noticed from this trial.

Searching for alternative methods of sample stream motivation resulted in an eductor made entirely of Teflon! These Teflon eductors cost only \$100 apiece, so regardless of anything else, they were going to effect a small financial savings. The results were much better than expected.

The analyzers' maintenance interval went to as much as three months between eductor failures. The actual realized savings were very large, since both the cost of maintenance went down and the savings on fuel gas increased due to more economical and consistent furnace operation utilizing the now-reliable oxygen-trim signal.

Teflon eductors are now used on all extractive zirconium O<sub>2</sub> systems. It pays off in reduced maintenance alone when one considers the number of extractive zirconium O<sub>2</sub> analyzers in use at a typical refinery.

### **CONTROL OF PRESSURE AND** **FLOW ON A CERTIFIED EMISSION** **MONITOR SYSTEM (CEMS)** **DAVID JONES, PREMCOR-LIMA, OH**

The applications are an O<sub>2</sub> analyzer and an SO<sub>2</sub> analyzer, both of which are an integral part of a Certified Emissions Monitoring System (CEMS), part of the EPA-mandated emissions monitoring system on a Klaus sulfur recovery unit. The incinerator stack on this unit runs at a design high level of 1% SO<sub>2</sub> or 10,000 ppm.



The SO<sub>2</sub> analyzer is sensitive to pressure, as are all UV absorbance measurement principle instruments. With this in mind, we considered that the sample had to be filtered before presentation to the analyzer. The pressure drop across the filter was expected to slowly increase as the filter element loaded in service. The constant pressure at the analyzer sample cell was considered resolved by putting a pump in ahead of the analyzer to enable some form of pressure control.

This would have worked well were it not for the fact that the sample had to be presented to the analyzer at almost 300°F/149°C and without the benefit of being dried. This caused major problems with the pump's longevity in service. Even though it had a heated-head and Teflon-lined pumping chamber, it failed frequently.

The following redesign was implemented nine years ago, and the system worked well until this past year with all of its original components, apart from sample cell replacements. About 60% of the components were replaced recently because of damage from their relocation with the addition of a new analyzer.

A dP cell of 0 to 100inWC range was installed, uti-

lizing a tee-fitting, into the  $\frac{1}{4}$ in. Teflon process sample line where it exited the analyzer. The output of the dP cell went to a Moore pressure controller with a setpoint of negative 20inWC. The output of this pressure controller provided the control signal to position a pneumatically-operated needle valve, which allowed ambient air to bleed into the sample line at the eductor and thus provided motive power to the process sample. This air bleed system controlled the pressure at the outlet of the analyzer to within  $\pm 1$ inWC.

Immediately prior to the eductor, another line was teed off to the O<sub>2</sub> analyzer. This line went first to a pump that had a pressure controller in parallel with it. This allowed the flow to the O<sub>2</sub> analyzer to be controlled, since the O<sub>2</sub> analyzer was sensitive to flow. At this point the sample had been cooled and dried utilizing a refrigerant dryer, so the previously mentioned pump problems were not a factor.

Over the nine years the system has been in operation, it has always passed the USEPA annual RATA test within the 20% error allowance of the regulations.

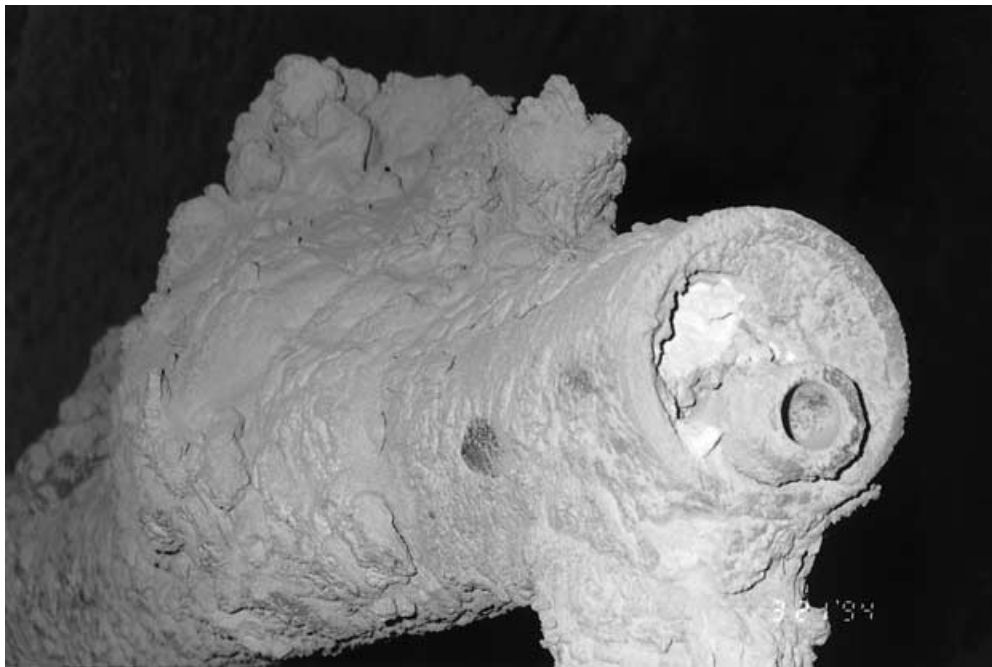
450 SYSTEM AND COMPONENT EXAMPLES



**Figure 12.1** Typical cement production facility (note rotary kiln behind right-hand stack).



**Figure 12.2** Typical cement kiln interior (approximately 8 ft inner diameter—process analyzer sample probe is supported behind left kiln wall).



**Figure 12.3** Close-up of process analyzer sample probe (supported end). Pipe surrounding sample probe tube is approximately 4 in. outside diameter; probe sample tube is approximately 1 in. outside diameter.

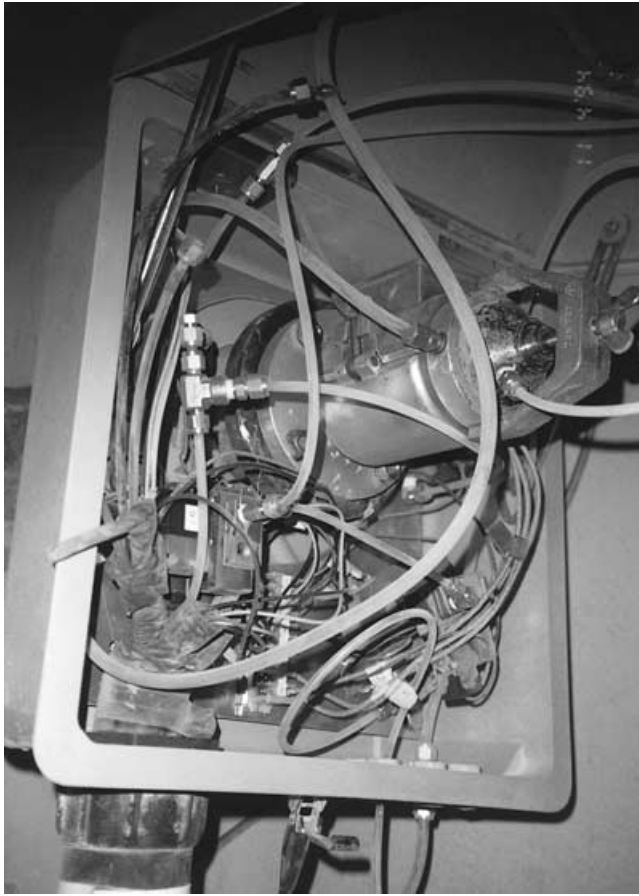


**Figure 12.4** Rear view of supported process analyzer sample probe showing process material build up (residual) and sample probe kiln header box penetration.



**Figure 12.5** View of process analyzer sample probe showing kiln header box exterior wall penetration detail and sample probe protection box detail. Integrally heat-traced-and-insulated process sample transfer line exits sample probe protection box from bottom of box.





**Figure 12.6** View of interior tubing and wiring surrounding sample probe heated filter. Integrally heat-traced-and-insulated process sample transfer line exits sample probe protection box from lower-left bottom of box.



**Figure 12.7** Sample probe protection box (interior view) with sample probe heated filter (center). Integrally heat-traced-and-insulated process sample transfer line exits sample probe protection box from lower-left bottom of box.



**Figure 12.8** Kiln interior showing water injection cooling lance in lower right (below probe support).



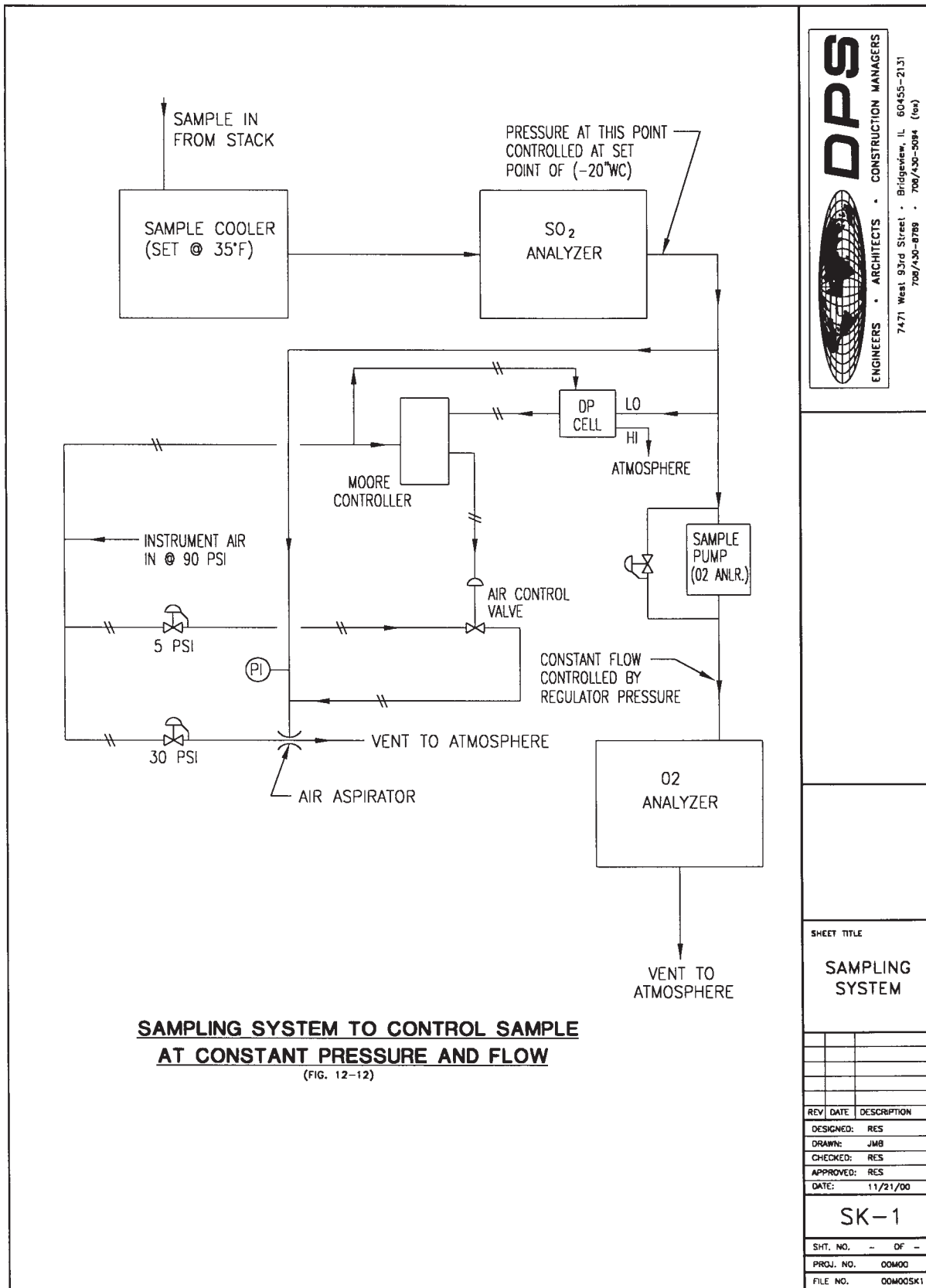
**Figure 12.9** Close-up view of spray nozzles on end of water injection cooling lance.



**Figure 12.10** Kiln header box with water injection cooling lances (2) (center of photo with water supply hoses attached).



**Figure 12.11** Kiln header box access door (open) showing internal support structure and water injection cooling lance (internal connections).



**Figure 12.12** Application schematic for analyzer sample delivery at constant pressure and constant flow rate. Reprinted, with permission, from Premcor Inc.