

CLAUS SULFUR RECOVERY TAIL GAS APPLYING 100 MILLION HOURS OF OPERATIONAL TIME TO THE NEXT GENERATION ANALYZER

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ABSTRACT

The measurement of H₂S/SO₂ in Claus sulfur recovery unit (SRU) tail gas has been adequately addressed by UV spectroscopy for over 40 years. Reliability of the analytical principle was established in the first generation of analyzers and in the second generation sample handling was improved to the point where automatic control if not universal is at least considered the norm. With a deep understanding of the process and a population of 1,100 second generation analyzers it was possible in the third generation to address failure modes external to the analyzer. Reliability is not limited to the analyzer / sample system; it extends to the process and contains elements of health and safety. Analyzer professionals are compelled to look beyond what we consider our "deliverables", to address abnormal operations and bad piping design. The paper combines extensive feedback from analyzer professionals and a survey of sulfur recovery operators to address the external failure modes. Looking back on 100 million hours of operational time and one year of field testing the third generation analyzer the paper discusses reliability as viewed by the real end use customer; Operations.

THE PROCESS, DEVELOPMENT OF THE TAIL GAS ANALYZER

Sulfur recovery is the unit operation of converting hydrogen sulfide (H₂S) to elemental sulfur and is an essential block in an integrated refinery complex or sour gas processing plant. Referred to as *the Modified Claus* process; in a sub stoichiometric first step 1/3rd of the H₂S undergoes combustion to SO₂ in a reaction furnace then passes through two or three catalytic converters to

produce elemental sulfur. The Modified Claus process is capable of ~97-98% recovery efficiency⁽¹⁾. The primary consideration for high efficiency is control of the (feed forward) air:gas ratio by trim (feedback) control using the H₂S/SO₂ tail gas analyzer.

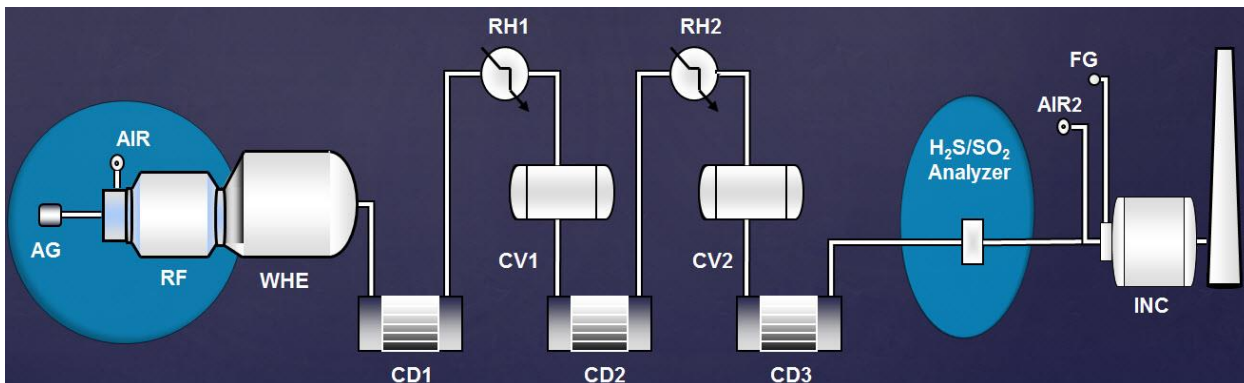


FIGURE 1. MODIFIED CLAUS SRU PROCESS & INSTRUMENT DRAWING

Sulfur is primarily used for the production of sulphuric acid which is the largest traded chemical commodity in the world. While sulfur does have a commodity value in most cases the driving force for process measurement and control is largely an environmental concern. The US EPA Clean Air Act of 1970 and the ground-breaking study by Alberta Environment on *The Capability of the Modified-Claus Process*⁽¹⁾ coincided with the first attempts to control the Modified-Claus process using an on-line analyzer and closed loop control.

The first report of an on-line tail gas analyzer was a technical paper presented at the 1970 Analysis Division Symposium based on a gas chromatograph installed at the Dow Chemical Freeport TX (USA) facility⁽²⁾. Amoco Oil and DuPont Process Instruments published a paper at the 1972 Analysis Division Symposium on an Ultraviolet (UV) based tail gas analyzer installed at the Amoco Whiting IN (USA) refinery⁽³⁾. Shortly after this in 1974 Western Research participated in a pilot study using a prototype UV-based tail gas analyzer at the Shell Waterton AB (Canada) gas plant.

It was already noted in the 1972 paper that “the non-continuous response of a process GC is often inadequate to provide the close control”. Given the process dead time of 30 seconds (to 2 minutes or more at turndown) and the requirement of a continuous sampling subsequent developments by various manufacturers were all based on UV spectroscopy with extractive sampling techniques which remains the case today.

There is a collective experience from ~2,700 tail gas analyzers (DuPont, Western Research and the amalgamation of these two; AMETEK Process Instruments); five models; two full generations; and now moving into a 3rd generation tail gas analyzer. The 3rd generation analyzer is the evolution of the close coupled 880 NSL (no sample line) analyzer with 1,100 units in service and ~100 million hours of operational time.

It has been noted by experienced analyzer engineers that certain process analyzer applications are much more difficult, among these being crude tower feed/bottoms and Claus tail gas⁽⁴⁾. Further to this point it has been estimated that something less than half of all process analyzers are placed in control. Analyzers placed in control get a great deal of scrutiny from Operations. Failure due to adverse conditions and unexpected (abnormal) results are not differentiated from analyzer failure. Having a large population of analyzers dedicated to one process gave insight beyond just the analyzer. A critical mass of information was acquired to look beyond the “deliverables” to address failure modes that are external to the analyzer and sample handling.

TWO INDUSTRY SURVEYS

Looking back on 40 years of experience the 1st generation (~1972-1997) proved UV worked, the weak link was sample handling, steam jacketed pipe and a large volume of sample gas. The 2nd generation and the application of a packed reflux type demister improved sample handling reliability but closed loop control was still not universal with process problems blamed on the analyzer. For the 3rd generation the focus was on what could be addressed in the way of failure modes external to the analyzer. With the aid of two industry surveys, a close study of the 2nd generation analyzer was undertaken.

For feedback from hands on professionals a voice of customer (VOC) survey polled analyzer technicians for their views. The 2nd generation top of the pipe analyzer has a strong following and many said “change nothing” in relation to the sample handling and “don’t change the size” (meaning do not shrink it down and make the oven hard to access with gloves on). In terms of desired new features the list included double block from the process (safety), a single steam pressure, automatic flow control, over range measurement and improved digital communications. The paper will expand on these aspects in detail.

A second survey was conducted to obtain valuable end user experience in addition to analyzer professionals. Three distinct stakeholder groups in sulphur recovery were polled;

- Front end engineering design (FEED) and start up engineers
- SRU Operations
- End user analyzer specialists (engineers and technicians)

It was deemed important while measuring reliability not to restrict feedback to just the analyzer profession, recognizing that on many occasions Operations has a different opinion as to “the analyzer is working”. The survey was extended to Operations, start up engineers and process design specialists for their perspective. The spread in the response was suitable with all three individual groups representing between 20-40% of the responses. To the question of reliability ~90% rated tail gas analyzers “reliable most of the time / all of the time”. This response was gratifying based on a comparison question asked when conducting process training for Operators over the past 15 years; “Is the analyzer in control mode?” for which ~25% say “No”. The take away being that even when the analyzer is considered reliable Operations decides to use it in manual control.

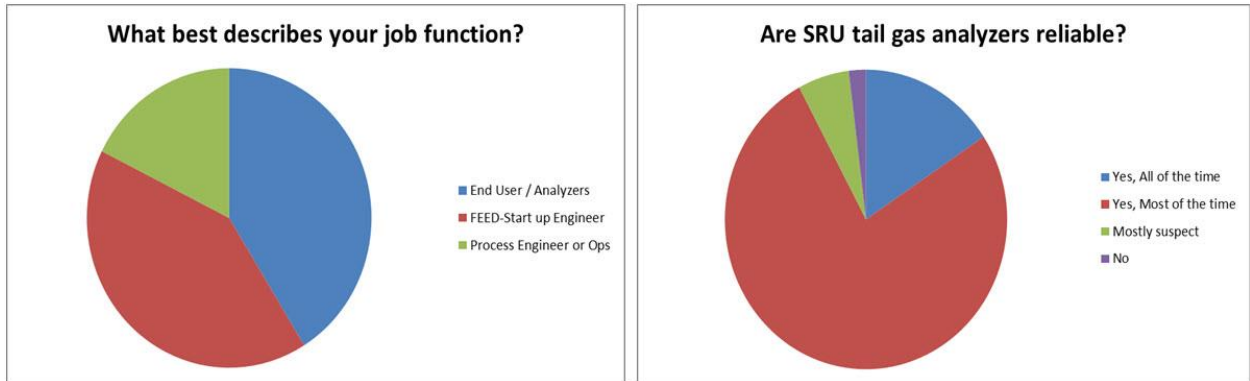


FIGURE 2. SRU INDUSTRY SURVEY

ADDRESSING EXTERNAL MODES OF FAILURE, PREDICTING ADVERSE PROCESS CONDITIONS

Risk can be managed and reduced by understanding the potential failure modes of an analyzer, its sample conditioning system and the process. The first step in the process is to define all of the critical conditions needed to make a valid measurement. The second step is to determine which diagnostic methods can be used to evaluate each of the conditions. Diagnostic methods require the collection or generation of data which is then evaluated against the conditional requirements and to provide either a direct or indirect indication of the potential failure modes. There are three types of diagnostics that are implemented in the analyzer. They are observational, model-based, and functional. These three diagnostic types are differentiated by the means through which the evaluated data is collected. This data is used to predict failure and manage reliability aspects internal to the analyzer / sample handling system and was covered in a previous paper ⁽⁵⁾.

The following expands on the methods applied to predicting and reacting to failure modes which are external to the analyzer and sample handling system. Having a long history in sulfur recovery there was strong confidence to enter into the process world and address problems that were previously beyond our control. Carefully considering feedback from the process stakeholders' attention was placed on the three main external failure modes; one is process based, one is utility based and one is environment based.

1. Failure Mode (Process): Entrained liquid sulfur (sulfur fog, sulfur mist)

SRUs are designed to operate at a certain capacity and refinery SRUs in particular go through a wide range of turn down according to crude slate. When a SRU goes below half load a phenomena occurs where the over cooling of the gas stream causes sulfur to form a fog, small sub-micron drops of sulfur that defy agglomeration in the process demister pads ⁽⁶⁾. The fog can then collect on the analyzer optics. Further complicating this is the attendant increase in process pressure (from ~1 to ~2 psig) causing unintended increase in the flow rate of the analyzer, drawing up more entrained sulfur.



FIGURE 3. SULFUR ENTRAINMENT – FOG AND MIST

The remedy is to adjust the sample flow rate relative to the process pressure. Simply measuring the flow is not sufficient and placing a flow sensor in direct contact with SRU process gas is not good practice. The 3rd generation analyzer monitors the pressure differential between the process and the sample aspiration and automatically adjusts the flow rate relative to the process pressure during the zero cycle (every 90 minutes). During start up, shut down, turn down, plugged rundowns and any time there is entrained sulfur leaving the final condenser the flow rate can be minimized to reduce sulfur mist take up.

2. Failure Mode (Utilities): Poor quality steam, bad steam traps

Steam traps are known to commonly fail, at any given time 20 to 30% of steam traps are defunct. The process connection point for a tail gas analyzer depends must be maintained at 300F (sulfur freezes at 246F). To maintain the heat integrity a custom steam jacketed ball valve is used. The problem of wet steam or a plugged steam trap is not usually a sudden failure. Most often it is intermittent it can come and go with wet weather or from night to day or result in “choking” which gives the appearance of slow response. The remedy is an embedded RTD in the process connection flange which provides an alarm. This advanced warning is unequivocal it indicates compromised heat integrity, calling attention to and eliminates a problem that can be the cause of intermittent failure.

3. Failure Mode (Environment): Solar gain, latent heat, internal heat release

The 2nd generation tail gas analyzer was the first to be close-coupled to the pipe. Placing the entire analyzer on the pipe eliminated the need for a shelter but also exposed the analyzer to solar gain and latent process heat. On a SRU the analyzer location is, as often or not is temperature compromised and engineering procurement Contractors (EPCs) rarely take care to add a sunshield to the analyzer.

While the existing analyzer is rated to 122°F (50°C) there is empirical evidence in the form of premature component failures that the internal temperature was far beyond the 50°C rating. Solar gain is the principal cause and in some cases latent process heat but also the internal heat release from the oven was a contributing factor.

The effect of temperature on analyzer electronics was very well covered in a paper from ISA-AD 2012 precisely quantifying how the life of electronic semiconductor circuits is affected adversely by temperature as indicated by the Arrhenius Equation. The equation predicts that for approximately each 10°C increase in operating temperature mean electronic component operating life is reduced by 50% ⁽⁷⁾.

One of the objectives for the 3rd generation analyzer was to increase the temperature rating to 60°C without the aid of any external cooling device (Vortec cooler, Peltier device, air conditioning). Given that the oven must be capable of 315°F (~160°C) operation it was somewhat in question if the heat release from the sample side onto the electronics side could be mitigated while also overcoming solar gain. A highly effective thermal insulating barrier was developed early in the design phase as well as a custom extruded aluminum convection heater replacing the off shelf oven heater. Advanced thermal management algorithms resulted in a net reduction of the internal temperature of the electronics enclosure by 15°C (27°F). The outcome is an industry first 60°C specification for ambient temperature and a 75% improvement in electronics life⁽⁷⁾. The analyzer is suitable for installation in hot climates with a sunshade being recommended.

EVOLUTIONARY IMPROVEMENTS

Digital communications and the HMI (human machine interface) were obviously due for updating, benefiting from improvements in electronics over the intervening eighteen years. For the sample handling and photometry great care was taken not to compromise the elements that worked well, avoid change for the sake of change. The following describes both categories.

IMPROVING ON WHAT ALREADY WORKED WELL

Photometer: For this application the measurement of H₂S/SO₂ does not require a high degree of precision or accuracy. Control is a simple binary action and too much can be made of comparing different methods of UV spectroscopy. The final control element is typically a butter fly valve sized to control 10% of the process air in the form as a trim air control loop which is manipulated its full range by the tail gas analyzer ⁽⁸⁾. In steady state full load conditions the trim air (“air demand”) is moving inside a peak to peak band of +/- 1% which results in a +/- 10% valve movement. Because of process dead time the peak to peak period is ~30 seconds (at full load) and the controller proportional gain is set low. The result is the time domain resolution of a photo-diode UV analyzer is 10x the resolution of the valve ⁽⁹⁾. Add to this that the control function is binary (“add air, cut air”, it never dwells at set point). The result is accuracy is not paramount; simplicity, support, reliability are the prime requirements.

The photometer for the 3rd generation retains the 2nd generation Xenon flash lamp and improves upon the photometer validation algorithms. The light from the sample cell is divided into four separate channels using partially reflective and partially transmitting beam splitters. The UV photometer uses four separate detectors each measuring a different narrow range of UV wavelengths. The intensity information from each of the four detectors is used to calculate the H₂S and SO₂ concentrations using a factory established calibration. The temperature of the photometer is carefully controlled to optimize the accuracy and precision of the reported concentrations. During the zero and span cycle an automatic multi-point photometric span calibration is performed on each of the four channels. The source pulse sequencing is automatically varied to create an intensity calibration for each detector channel. It is uniquely capable of automatic intensity calibration but calibration with a (manual) removable filter was retained as customer feedback indicated this was a widely accepted method for Operations to be assured the analyzer is working.

The dynamic range was improved to the extent the analyzer can measure 100% over range while maintaining linearity specifications. This is especially useful during an upset giving Operations a window beyond the normal air control parameter. Tail Gas is one of those applications where the instrument data sheet stays slavishly attached to the standard range, not acknowledging the ability of a next generation analyzer to provide this valuable information. It is up to the analyzer profession to communicate improvements such as over-range to front end engineering (FEED) designers and instrument engineers who stay rooted to past history and dated data sheets.

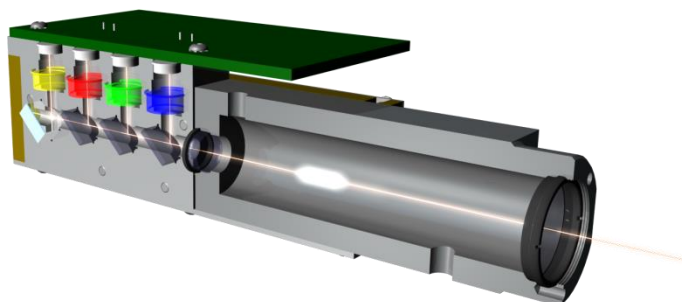


FIGURE 4. PHOTOMETER

Demister: What was well proven in the 2nd generation design was the veracity of using demisting pads to coalesce sulfur drops. Straight gravity drainage using an unpacked column (“cold finger” probe) works well enough when there is minimal entrained sulfur or when the drops are large and easily agglomerate. Adverse process conditions (plugged run down, failure of the process demister pad, sulfur fog at turndown) require a packed column and reflux action. The sulfur demister borrows heavily from the experience of dynamic reflux (pyrolysis) gas sampling. The subject has been extensively covered at previous symposia with reference made to the ability to prevent clouding of optical windows⁽¹⁰⁾. The hallmarks of a reflux sampler are surface area, vertical mounting and being close coupled but external to the process⁽¹¹⁾. One of the 3rd generation improvements borrowed from the reflux sampler was mounting the demister pads on

a shaft with retention rings ⁽¹¹⁾ which in the case of the tail gas demister keeps the mesh pads in firmly in place during the hot condensate steam blow back for ammonia salts.

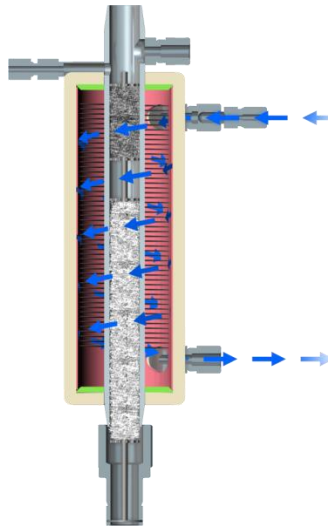


FIGURE 5. DEMISTER

Incomplete combustion of ammonia in the SRU process reacts with sulfur compounds to form ammonia salts, which can plug the demister. These salts are not removed by air blow-back but are water soluble. Hot condensate steam blow-back provides a steam propelled blast of hot water to dissolve any salts that have accumulated within the demister. An example of what these salts look like in the process and propensity to cause pressure drop is shown in the following figure.



FIGURE 6. AMMONIA SALTS ON CONDENSER TUBE SHEET

The analyzer is also equipped with anti-clogging hot air blowback feature that is automatically initiated if plugging is detected by the smart diagnostics. The 3rd generation demister retains the double demister pads (316 SS top and PTFE bottom). A change was made to connect up with vacuum compression o-ring (VCO) fittings in place of tube fittings making it easy to access and requiring minutes to disassemble. While not yet common in the process world VCO fittings have a place as they allow technicians easy access components inside a confined space like an oven.

NEW IMPROVEMENTS

Digital Interface: In our industry fifteen years is a typical life span of an analyzer while graphical interface and digital communications goes through several incarnations in the same period. The challenge is to stay up to date while at the same time finding commonality across a range of products for human engineering as well as cost of manufacturing and ongoing support. The next generation tail gas analyzer followed closely behind the 5th generation WDG oxygen analyzers and the same local HMI was implemented. AMEvision is an icon driven graphical interface with a color display for local communication with the analyzer. It provides screens showing trending functions, predictive maintenance indicators, analog output verification, and time stamped alarm/event log.

Remote communication is via a standard PC web browser enabled interface (no software need be installed) over TCP/IP Ethernet. This point raises an anomaly that is perhaps unique to sulfur recovery but in any case is worth revisiting on every new project. Remote digital communication has been available on 2nd generation tail gas analyzers (two models, ~1,800 units) for the past 17 years. Of that total fewer than 10% are tied into remote digital communications. At least half have been new SRUs, grass roots project. The question to ask of EPCs (and end users) why they deem it acceptable to go on to the sulfur unit when it could all be done from the safety of the analyzer shop using a remote digital connection. This has hazardous operation implications and should be on top of the list for any analyzer engineer on a project to say “and why not?”

UTILITIES

There was opportunity to reduce the utilities. The Division 2/Zone 2 analyzer does not require an EExP purge, this reduces the instrument air volume and eliminates damage from wet instrument air. Steam as a utility is reduced to a single pressure. The demister pads held firm in place on a shaft can now withstand medium pressure (MP) steam and can utilize the same MP steam required for the process connection valve to maintain 300° F (150° C).

SAFETY

Hazardous Operation (“HazOp”) review is becoming more prevalent in the world of process analyzers. It has been said that sulfur recovery is not as well understood because it is a chemical plant in a refinery world and that is largely true. Sulfur recovery while not any more inherently

dangerous than other refinery unit operations has the stigma of H₂S. With that in mind safety features were added for with future requirements in mind.

Three major refining companies in the USA often require double block isolation at their sites. This was not entirely possible in the 2nd generation analyzer but has been implemented in the 3rd generation analyzer with two isolation valves at the process access valve on both the sample and the vent. The sample conditioning components are external to the process and it is possible to remove the probe from the process under live conditions with zero egress through the Conax™ fitting. The sample handling components are a safe distance from the process (500 mm/18", which is considered the boundary for "safe distance")

To close this subject it is worthwhile to look at the safety implications of having to work with a compromised sample point. A modern SRU and tail gas treating unit when built as a single project is typically conjoined with a short vertical section of pipe. When analyzer professionals get zero input into at the piping design stage they are tied into a situation that can compromise both function and safety. For SRU-TGTUs the problems are confined quarters (egress), access, and process heat amongst others⁽⁹⁾. It is incumbent on our profession to interject ourselves at an early stage of a project to take a leading role in sample point selection and to take advantage of the available knowledge base

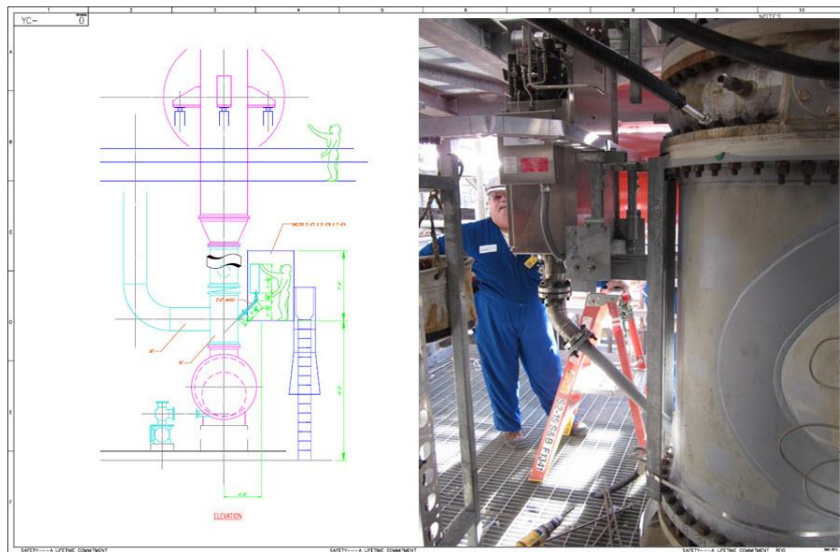


FIGURE 7. COMPROMISED SAMPLE POINT LOCATION

FIELD TRIAL OF 3rd GENERATION ANALYZER

A search for a suitable site to conduct field trials commenced in late 2013; the prime requirement being close proximity to the factory located in Newark DE. There are a number of refineries in the area but SRUs are normally only in turn around every 4 years and a pool of suitable sites is therefore quite limited. The PBF Paulsboro NJ refinery had an available sample point location

and consented to installing the new analyzer by hot-tapping a process connection valve. Like the 2nd generation analyzer the 3rd generation analyzer can be installed while the process is hot (i.e. ingress and egress of the probe via a Conax TM gland) as long as a one inch full port ball valve is in place as the process connection.

The hot tap was made and the model 888 analyzer installed at PBF Paulsboro in April 2014. Operations considered keeping the other analyzer in service in case the new analyzer required intervention. This would have added considerable cable and utility work. Given the additional cost, the confidence in the new analyzer was high and the opinion of analyzer professionals that “two watches” is never good the decision was made to go solo with the new analyzer. The cut over was made in one day and the new analyzer was placed in control by Operations same day.

During the first 4 months of the trial there were no major outages for the analyzer. There was concern that the process connection valve used for the hot-tap was not steam jacketed but as it was close coupled there were no immediate problems. Later in July during a cold rain there was intermittent plugging and this was remedied with an off the shelf piece of Contra-Trace TM from Controls Southeast Inc (CSI) to match this specific valve body. CSI specializes in sulfur recovery applications but the product is well applied to any analyzer heat tracing requirement and is much preferable to wrapping in SS tubing which in this service serves no useful purpose.

There was only minimum intervention in the first seven months, modifications were restricted to software changes, availability was >99%. Operating conditions went through the normal range of events and the sample handling system performed to expectations with zero complaints from operations. Given the anomalies and the degree of difficulty associated with this type of process analyzer application the testing program allowed for up to 12 months of evaluation to observe long term effects on the components.

In early November a heater fuse failed, it was deemed to be under rated and a larger fuse installed. In late November/early December condensed water was discovered in the sealed photometer box as well as on the detector board and the mystery as to how water was getting in. Wet instrument air was quickly eliminated. After some investigation a leaking check valve from a well known supplier was determined to be the root cause, it was intermittently failing and allowing small amounts of wet process gas to find a pathway into the photometer. The check valve was replaced with one from another supplier.

In late early January there was observation of something off-gassing on the optics causing a slow decay of the measured light (compensated for but eventually causing low light level alarms). The photometer mirrors were replaced so the material could be extracted and analyzed. The root cause was a small silicon based seal which was replaced with another material. At the time of writing there were no other problems. The evaluation will run until March at which time the analyzer design will be turned over to production and manufacturing will commence.



FIGURE 8. FIELD TRIAL PBF PAULSBORO REFINERY

DISCUSSION AND CONCLUSIONS

Process analytics is a young industry (approximately 50-60 years) with a product life cycle that can be relatively long. Most vendors think in terms of 15-20 year operation before considering replacement. Reviewing an application with a known degree of difficulty such as sulfur recovery is not complete without addressing the external failure modes that potentially occur within the process. As the analyzer results are the point of contact between the process and operations, these external failure modes are more often ascribed as “analyzer failures” and in all cases Operations will always have the last word; “we trust it/we don’t trust it”.

First and 2nd generation tail gas analyzers have successfully concluded the measurement technique and closed loop control respectively. Now approaching the third generation of tail gas analyzers affords the opportunity to move beyond the borders of the analyzer itself and step into the process world, evaluating the process and responding to external failure modes. It takes a step further by educating the operators on the analytical technique, the sample system as well as the anomalies of the process to arrive at a holistic concept of reliability.

Finally, it is imperative when approaching the process industry with a 3rd generation of tail gas analyzer that a long term field trial (minimum of 12 months) has been concluded. Even with many years of experience it takes time for hidden flaws to be revealed. Only after a successful field trial can an analyzer be introduced with confidence to an industry that expects a high level of reliability.

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