

Combustibles Measurement in Sulfur Recovery Unit Acid Gas with a Combined NDUV / NDIR Analyzer

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COMBUSTIBLES MEASUREMENT IN SULFUR RECOVERY UNIT ACID GAS WITH A COMBINED NDUV/NDIR ANALYZER

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KEYWORDS

NDUV, NDIR, H₂S Measurement, Feed Forward, Hydrocarbon Measurement.

ABSTRACT

The paper describes the development of a gas analyzer that uses both ultraviolet and infrared spectroscopy for the measurement of hydrogen sulfide (H₂S) and Total Hydrocarbon Combustibles (THC) in the acid gas feed to the reaction furnace of a Claus Sulfur Recovery Unit (SRU). The analyzer combines a Non-Dispersive Ultraviolet (NDUV) optical bench with a Non-Dispersive Infrared (NDIR) sensor for the simultaneous measurement of H₂S and Hydrocarbons (HCs) from a single sample point. This measurement requires a very fast response time for effective feed-forward control. A total hydrocarbon measurement is done rather than attempting speciation of the hydrocarbon mixture. The prototype analyzer will be field tested in several different SRU acid gas streams.

INTRODUCTION

Amine Gas Treating, or sweetening, is a process using amine solutions to remove acid gases (H₂S and carbon dioxide (CO₂)) from hydrocarbon gas in refineries and gas plants. Once the acid gases have been removed, they are typically further treated in a Claus SRU. A perfect version of this process would completely separate acid gases from the HCs, so that the treated gas would contain no acid gases and the gas sent to the SRU would have no HCs. HCs do end up in the acid gas, creating SRU operating issues including unstable off-ratio operation and reduction in catalyst activity. The unstable operation can reduce efficiency and also potentially damage downstream equipment such as tail gas clean up units.

DESCRIPTION OF A TYPICAL AMINE TREATING UNIT

Different versions of this process are used, depending on the quantity and type of gas to be processed. Figure 1 shows a simplified process flow for natural gas sweetening^{1,2}. An inlet filter-separator is installed at the front of the amine treating unit to minimize the amount of liquids and particulate in the gas. Gas from the inlet separator flows into the bottom of an absorber tower and travels upward, countercurrent to a stream of lean amine solution. The amine solution flows down the tower and absorbs H₂S and CO₂ from the gas, with sweetened natural gas exiting the top of the tower, and rich amine solution flowing out the bottom.

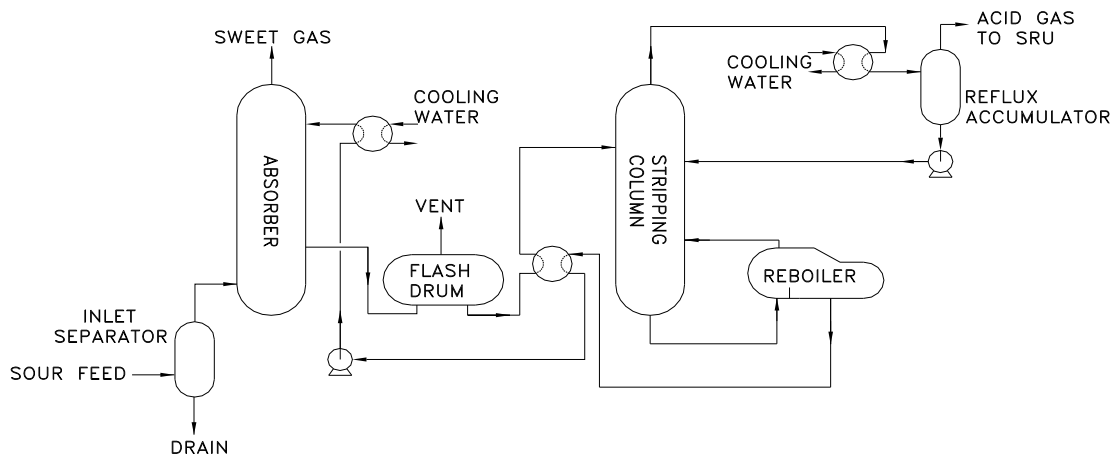


FIGURE 1 – SCHEMATIC OF A TYPICAL AMINE TREATING PROCESS

A flash drum after the absorber removes HCs from the rich amine and prevents them from carrying over to the downstream SRU. The flash drum is a horizontal vessel where the pressure is reduced to allow light HCs to flash out of solution and heavy HCs to be skimmed from the top. The HC removal efficiency increases with flash drum size, with residence times of at least 20 to 30 minutes being recommended. Lower flash drum pressure results in maximum HC removal from the amine, but the minimum pressure is sometimes limited by the treatment system for the flash drum vent stream. The rich amine then passes through a lean/rich amine heat exchanger and then flows into the top of the stripping/regenerating column, where low pressure and high temperature conditions reverse the reaction between the acid gases and the amine. Acid gases are stripped from the amine solution as it flows down the column and is sparged by steam from the reboiler. Lean amine from the bottom of the vessel is heated in the reboiler and then is cooled by the lean/rich exchanger and a lean solution cooler. The lean amine is then circulated back to the top of the absorber tower. Acid gases that have been stripped from the rich amine flow out of the top of the stripping column and into the reflux accumulator, after being cooled in the reflux condenser. The reflux accumulator minimizes the amount of water that is carried over to the SRU with the acid gases.

DESCRIPTION OF A TYPICAL CLAUS SRU

A flow diagram of a Modified Claus SRU is shown in Figure 2. The acid gas flows through a knock out drum to remove any entrained liquids and catch any liquid slugs. The acid gas is mixed with air in the reaction furnace, and partially oxidized to create SO₂, and a large portion of the H₂S is converted directly to sulfur and is removed from the reaction furnace effluent by the waste heat exchanger. Additional sulfur is condensed from the gas in the first condenser. The gas is reheated before flowing into the catalytic converter, which reacts the H₂S and SO₂ as per Equation 1³. Sulfur generated in the first converter is removed from the gas in the next condenser. The reheat, converter, condenser sequence is repeated once or twice more and then the gas from the final condenser is sent to a tail gas clean up unit or directly to an incinerator stack. The second and subsequent converter beds are run at temperatures just above the outlet sulfur dew point temperature to maximize the conversion to sulfur rather than optimizing COS and CS₂ hydrolysis (Equations 2 and 3), which was addressed in the first converter.

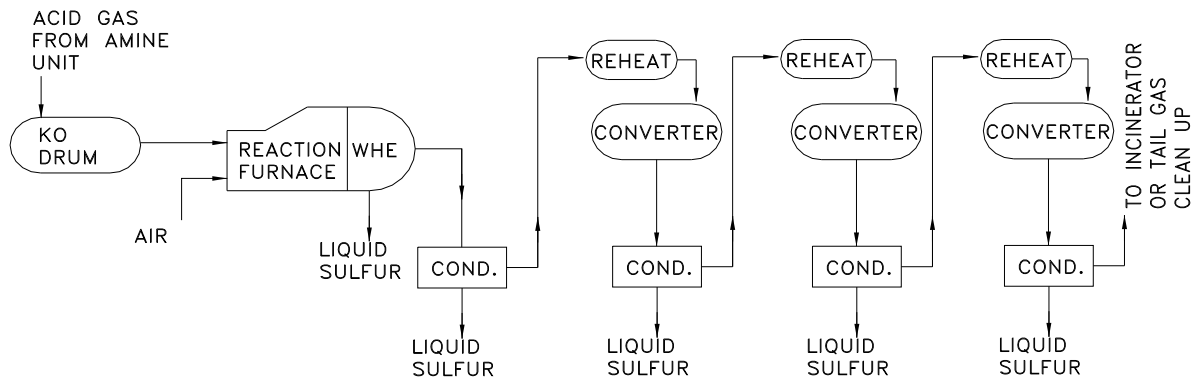


FIGURE 2 – SIMPLIFIED FLOW DIAGRAM OF MODIFIED CLAUS SRU

CONTROL OF SRU REACTION FURNACE AIR

Sulfur recovery is maximized at a 2:1 H₂S to SO₂ ratio as per the reaction shown in Equation 1. Reaction of the H₂S in the reaction furnace is sub-stoichiometric, meaning that there is only enough air introduced to “burn” 1/3 of the H₂S. Control of the air flow to the reaction furnace is done with a combination of feed forward and feedback control. The main air flow valve is controlled by feed forward control using the acid gas flow rate and the H₂S concentration, if that measurement is available. Approximately 10% of the air is supplied with a trim air valve, controlled via feedback from an air demand analyzer located after the final condenser. The feedback control using tail gas air demand

provides the most precise control of the reaction furnace air. However it is limited by the ~30 second process lag time if the flow rate or composition of the acid gas changes rapidly. A good feed forward control system can adjust for changes in acid gas composition or flow rate and prevent upsets or damage in the SRU Claus section or tail gas clean up unit^{4,5,6,7}.

The analysis time for the flow rate and H₂S concentration measurements of the acid gas must be much faster than the process lag time for the tail gas air demand measurement to make the feed forward control of the main air valve effective^{3,4,6}. Total process residence time for the acid gas flow between the sample point at the outlet of the knock out drum and when it enters the waste heat exchanger is typically less than 3 seconds. A fast responding feed forward system that controls using the acid gas flow and H₂S concentration is better than feedback control only, however it is still limited because the hydrocarbon content of the acid gas is ignored unless a hydrocarbon analyzer is installed.

THE EFFECT OF HC UPSETS ON THE REACTION FURNACE

The acid gas feed to the SRU reaction furnace will contain small amounts of hydrocarbons in normal operation, which can increase to several percent during upset conditions in the amine unit. The hydrocarbon fraction is composed of C1 through C6 alkanes, plus aromatics (benzene, toluene, etc.). Other compounds such as methanol may be present. Actual concentrations of these HCs will depend on various factors including their concentration in the sour natural gas feed, type and strength of the amine, the circulation rate in the amine absorber, pressure and temperature in the absorber, flash tank operation conditions, and what type of upset, if any, is occurring^{1,2,8,9}.

The hydrocarbons in the amine have either been physically absorbed or have condensed in the solution. Higher molecular weight amines (diglycolamine (DGA), methyldiethanolamine (MDEA), diethanolamine (DEA), and diisopropylamine (DIPA)) absorb more HCs than monoethanolamine (MEA) (MW 61 g/mol). Upset conditions often result in the concentration of the heavier HCs in the rich amine increasing because they have higher solubility in the amine than methane and also are more likely to condense. Upsets caused by foaming in the amine absorber can increase lighter HCs in the acid gas, because foaming amine tends to hold more gas in solution. An increase in the heavy HC content of the gas feed will usually result in higher concentrations of the same HCs in the acid gas. A decrease in sour natural gas flow rate to the amine unit, without an adjustment in amine flow, will result in less acid gas flow to the reaction furnace, but usually with a higher HC concentration. Correct operation of the amine unit flash drum will minimize the HC content of the acid gas, but any lighter hydrocarbons that are not flashed off will end up in the SRU reaction furnace. Hydrocarbons can condense if the sour natural gas feed temperature is higher than the amine absorber temperature and these condensed hydrocarbons can end up in the acid gas if they are not skimmed off in the flash drum^{1,2,4,5,8,9}.

It is important that the HCs in the acid gas get destroyed in the reaction furnace because they can deactivate the catalyst if they reach the first converter. Studies suggest that at typical reaction furnace temperatures, HCs oxidize more slowly than H₂S, which consumes most of the oxygen in the burner flame. Although a small fraction of the HCs may react directly with oxygen to form carbon monoxide (CO) and hydrogen (H₂), a larger portion is consumed in other side reactions either in the main flame or in the combustion chamber downstream of the flame. It is possible that a fraction is oxidized by SO₂ or reacts with water to form CO, but it appears that the majority forms CS₂, probably by the reaction shown in Equation 4:



In reaction furnaces with a sufficiently high temperature, most of the CS₂ then goes on to react with other species such as SO₂, CO₂, water or H₂, and the largest portion of the carbon eventually ends up as CO₂, although some fraction forms CO because of the reducing conditions in the reaction furnace. Since the H₂ portion of the HC eventually ends up as water, the amount of O₂ it requires is the same as if it was burned directly with oxygen. The net result is that if the intermediate reactions are ignored, the amount of O₂ required by the HCs is basically the same as if they were undergoing direct combustion, although a small correction is required to account for the amount of CO that is formed. This CO correction will vary between plants, but it has a typical effect of reducing the air requirement for the HC by about 20%⁹. This CO correction doesn't matter in applications where the HCs are measured and used to estimate the change in the air requirement, since it can be accounted for when setting the gain to apply to the HC measurement in adjusting the main air control valve. It is important that the HC measurement be proportional to the air requirement for the mixture, because many HC species are present.

The effect of changing HC concentration on the air requirement to stay on a 2:1 H₂S to SO₂ ratio is much higher than for a change in H₂S concentration. Table I shows the moles of O₂ required by combustion of one mole of various HCs and compares it to the O₂ requirement for one mole of H₂S if the SRU is on ratio. Ethane requires 7 times more air than H₂S, which means that if the ethane went from 0% to 1% during an upset, the increase in air requirement is the same as if the H₂S concentration increased by 7%.

**TABLE I – OXYGEN REQUIRED TO BURN HYDROCARBONS
COMPARED TO THAT FOR AN EQUAL AMOUNT OF H₂S**

Compound	Moles of O ₂ per mole HC	Ratio of O ₂ needed per mole HC compared to mole of H ₂ S
Methane	2	4
Ethane	3.5	7
Propane	5	10
Butane	6.5	13
Pentane	8	16
Hexane	9.5	19

ANALYZER DESIGN

Based on experience of supplying analyzers for measurement of H₂S in acid gas and occasionally being asked for a separate analyzer to measure hydrocarbons, it seemed that there would be several advantages to a single analyzer that was capable of doing both measurements. The primary advantage of combining these measurements is a savings in both the cost of the analyzer and also installation costs. Manufacturing costs are lower with elimination of duplicate items like micro-controllers, sample system, enclosures, power supplies, etc. Further cost savings can be realized during the installation because of reduced number of sample lines, probes, electrical connections and also space savings. The NDUV photometric portion of the analyzer was designed around an optical bench which has been used many times for the measurement of H₂S in SRU acid gas. The optical bench has been designed to provide excellent baseline stability, linearity and sensitivity. The typical full-scale measuring range in SRU acid gas applications is 0-100% H₂S, so a sample cell length of only 1 mm is used. This very short path-length results in a very low volume, which helps to maintain a fast analyzer response time. A schematic for the optical bench, which has no moving parts, is shown in Figure 3. Further details on the design of this spectrometer are available in Reference 13.

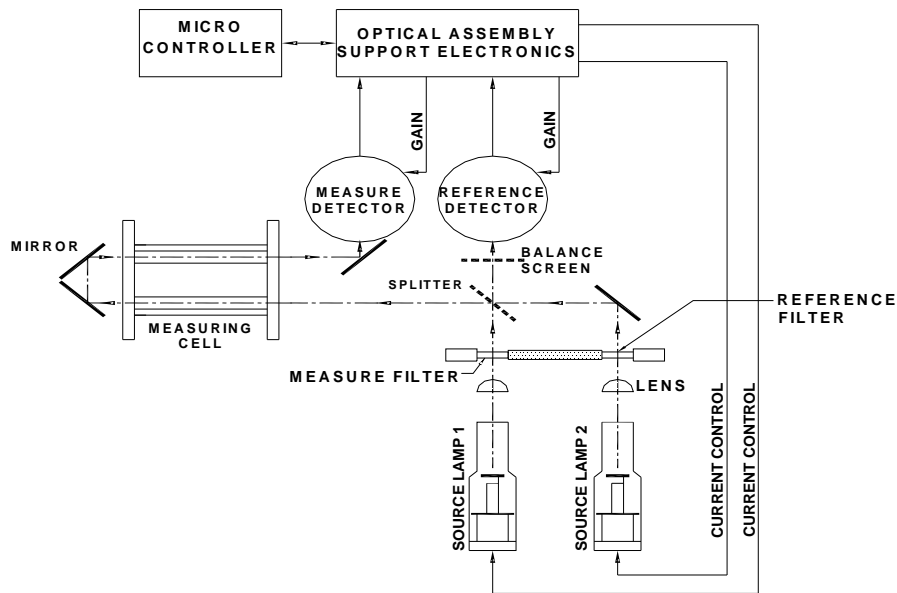


FIGURE 3 – DUAL BEAM DUAL-WAVELENGTH SPECTROMETER

NDIR SENSOR FOR HYDROCARBON MEASUREMENT

Saturated HCs do not absorb at UV wavelengths where the NDUV optical bench measures H₂S, so a different detection method must be used. There are a number of HC measurement techniques, and there have been a few attempts over the years to use some of them in this application. These methods have included Gas Chromatography (GC), Fourier Transform Infrared spectroscopy (FTIR), Mass Spectrometry (MS), and Non Dispersive Infrared (NDIR)

All of these technologies except for NDIR, are capable of individual speciation, which may be desirable for amine unit diagnostics, but is not absolutely necessary for the feed forward control. Individual speciation typically requires more time to carry out and the accuracy of the total hydrocarbon air demand requirement can be limited because the measurement errors for each individual component are accumulated in the final result. Speciation of the hydrocarbons in a complex mixture isn't necessary as long as the THC measurement is proportional to the combustion air requirement for the mixture. A survey on the type and number of HC analyzers installed in SRU applications doesn't seem to be available. Based on experience in supplying NDUV H₂S analyzers for feed forward applications and also visits to SRU installations, it appears that NDIR and GC are the most widely applied technologies for the HC measurement (with at least 10 installations between the two methods), but there has been at least one MS installation and three others that tried FTIR. The GC and FTIR do not have fast enough response times to meet feed forward control requirements and are also expensive (approximately double the cost of an NDUV analyzer measuring H₂S only). Mass spectrometers have very fast response times, but are expensive (approximately three times the cost of an NDUV analyzer measuring H₂S only), the data analysis usually requires special software, and high skill is needed for setup, calibration and maintenance¹⁴. One attempt was made to determine an empirical value for feed forward air requirements using stoichiometric combustion of the acid gas, but ran into problems with metering complexity and the formation of sulfur trioxide in the combustor¹⁵.

The most economic technology for HC measurement is NDIR (with a cost approximately equal to a NDUV analyzer measuring H₂S only). NDIR uses fixed bandpass optical filters to isolate regions of the infrared output from an IR light source. The radiation absorbance in these narrow wavelength regions is measured and used to calculate the gas concentration. Hydrocarbons absorb light in the IR region between 3 and 3.5 microns mainly through stretching modes of the C-H bond. Generally, the more C-H bonds that are present, the stronger the absorption lines or bands^{16,17}. An NDIR analyzer and sample system, packaged for hazardous areas, is still relatively expensive, so it is more common to see SRU plants with NDUV H₂S measurement only for feed forward control. More plants might integrate this into their feed forward control if an inexpensive NDIR based HC measurement could be added to the NDUV H₂S analyzer.

Since only a total hydrocarbon measurement is required, the NDIR measurement only needs two optical filters. The measurement has been developed around a very simple, low cost optical bench which can be considered as an NDIR sensor. The basic design of the NDIR sensor is shown in Figure 4. The light source is an incandescent lamp with a glass envelope, located within the measuring cell. The light from the lamp travels through the gas, reflects off an IR mirror and onto two pyroelectric detectors. These detectors are sealed and have built-in optical filters, one for a reference and the other for a measure wavelength. Wavelengths for the optical filters on the detectors were selected in the region between 3 and 4 microns where water and CO₂ do not absorb. The reference wavelength is at 4 microns where there are no strong gas absorption lines. The measure wavelength is between 3.2 and 3.5 microns where there are strong absorption

lines from various HCs. These strong absorption lines allow a sample cell length of only 5 cm, which keeps the volume to a minimum. A sintered flame arrestor is used to obtain a flameproof hazardous area certification on the sensor while allowing the gas to diffuse into the cell. The low sample cell volume, along with the large area of the sintered flame arrestor, maintains a fast response time.

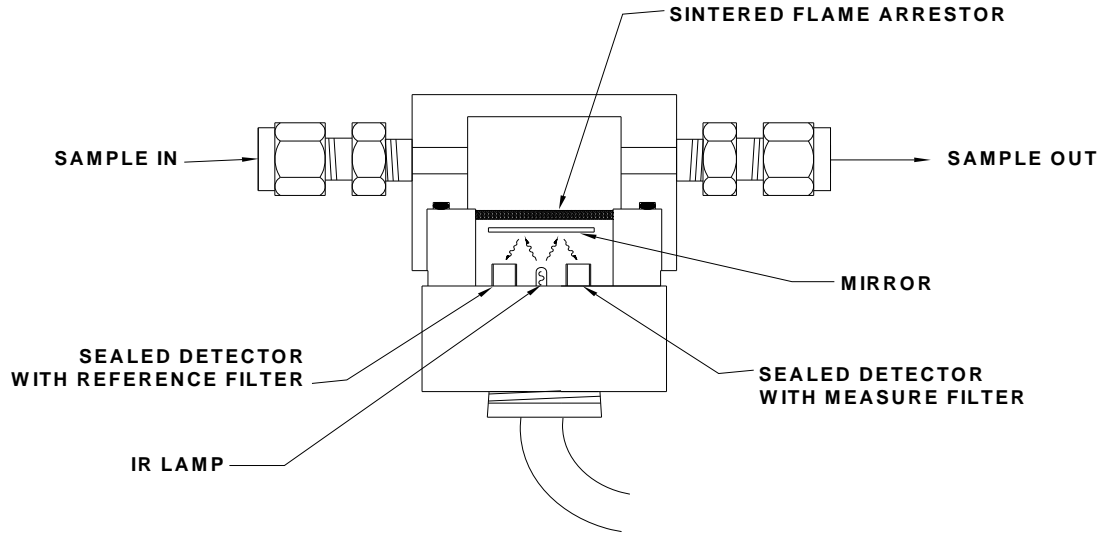


FIGURE 4 – LAYOUT OF INFRARED SENSOR

The NDIR sensor is connected to the NDUV optical bench micro-interface board, which provides power and signal processing. Pyroelectric detectors have high detection sensitivity, which is basically constant over the wavelength region of interest. The IR lamp voltage supply is a 4 Hz square wave pulsed between 0 and 5 VDC at a 50% duty cycle. Modulated lamp operation is required because the pyroelectric detectors detect changes in incident radiation. Heating and cooling time constants for the lamp filament result in a 4 Hz sine wave output from the detectors, with the peak-to-peak signal being proportional to the incident light intensity. The NDUV analyzer signal processing converts the detector signals to DC voltages which are read by the micro-controller.

Beer's law is used to calculate the concentration from the detector signals as shown in Equation 5 and 6 below. This method of calculating concentration from the NDIR Sensor signals is very similar to the principle used in the NDUV concentration calculation, but includes a second order linearization since the response of the NDIR measure detector is non-linear. This response is non-linear because there are wavelengths within the measure optical filter bandpass which have no HC absorption lines, so some light always reaches the detector, even at very high HC concentrations.

$$\text{HC Concentration} = N \cdot T (S \cdot \text{ABS}_{\text{diff}} + Q \cdot \text{ABS}_{\text{diff}}^2) / P \quad (5)$$

Where N is a constant which includes the cell length and molar absorbtivity, T is the sensor temperature (K), S is a linear gain term, Q is a quadratic linearization term,

P is the cell pressure (mm Hg), and ABS_{diff} is the differential absorbance calculated from the measure and reference wave length absorbance using Equation 4.

$$ABS_{diff} = \log(M_{zero}/M) - \log (R_{zero}/R) \quad (6)$$

Where M and R are the measure and reference detector signals on sample gas, and M_{zero} and R_{zero} are the measure and reference detector signals with zero gas. An adaptive filter is used on the absorbance for both the NDIR sensor and the NDUV optical bench. This method provides a high level of filtering when the signals stay within a tight band, and no filtering if the signals start to change quickly, resulting in a fast response time.

RESULTS

Two different sensors have been tested. Sensor A uses a measure center bandpass wavelength of 3.4 microns, while Sensor B uses 3.3 microns. These sensors were tested with different HCs to determine which one provides an output concentration proportional to the combustion air requirement for the HC mixtures in the SRU acid gas of different plants. Table II shows the ideal response to various HCs for methane and ethane sensor calibrations as a ratio of sensor response to HC concentration. These values were derived by taking the ratios of the entries in the second column of Table I (e.g. ethane response of a sensor with methane calibration should be $3.5/2 = 1.75$). Approximate values can also be determined from the ratio of gross heating values of the HC test gas to the HC that the sensor was calibrated for, which are available in Reference 18. Figure 5 shows the response of Sensor A to HCs with an ethane calibration. The lines in the graph indicate the ideal response given in Table II and the data points are the results from gas runs. The sensor response to ethane and heavier HCs is reasonable, but it under predicts for methane. This behavior suggests that the ethane concentration from this sensor would be proportional to the combustion air requirement in applications where most of the contribution to this requirement comes from ethane and heavier HCs. The air requirements would be over predicted by a large amount if the sensor was calibrated for methane and substantial quantities of other HCs were present.

TABLE II – IDEAL RESPONSE OF SENSOR CALIBRATED FOR METHANE AND ETHANE FOR A CHANGE IN CONCENTRATION OF VARIOUS HC

Hydrocarbon Being Varied	Sensor Response for Methane Cal	Sensor Response for Ethane Cal
Methane (CH ₄)	1	0.57
Ethane (C ₂ H ₆)	1.75	1
Propane (C ₃ H ₈)	2.5	1.43
n-Butane (C ₄ H ₁₀)	3.25	1.85
Pentane (C ₅ H ₁₂)	4	2.29

Figure 6 shows the response of Sensor B to different HCs when the sensor has been calibrated for methane. It is not suitable for use in applications where there are substantial quantities of various HCs present, because the output is not representative of the combustion air requirement for these mixtures. The methane reading from this sensor in streams that contain mostly methane and only trace quantities of other HCs would be

very close to proportional to the required combustion air, and much better than Sensor A for this type of application.

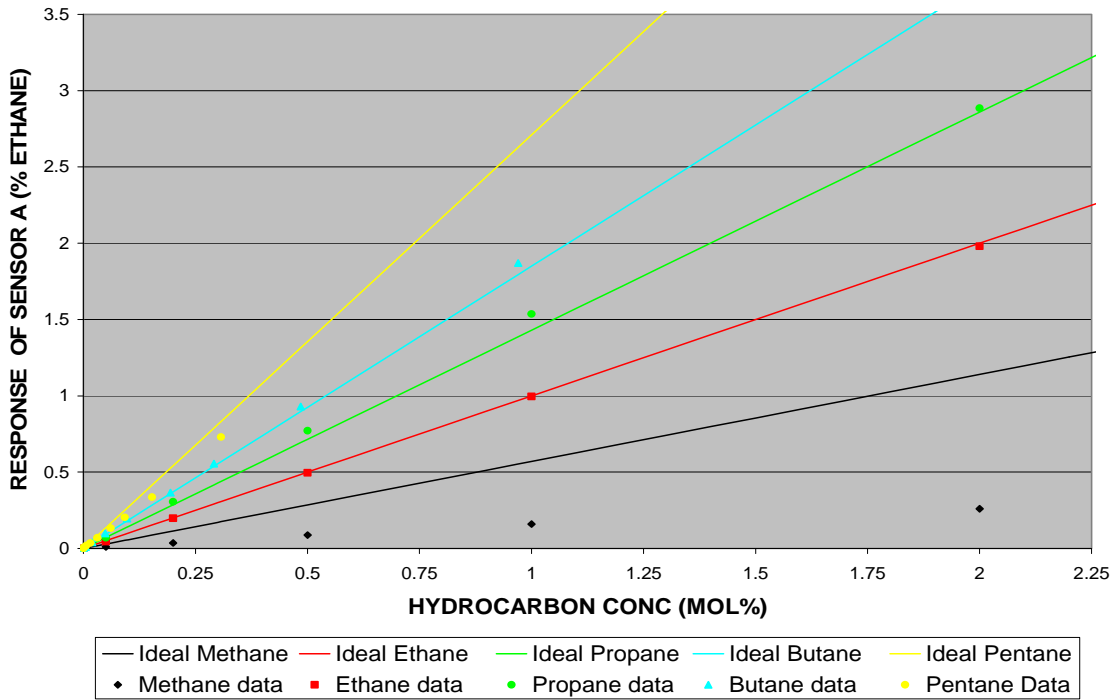


FIGURE 5 – RESPONSE OF SENSOR A WITH ETHANE CALIBRATION

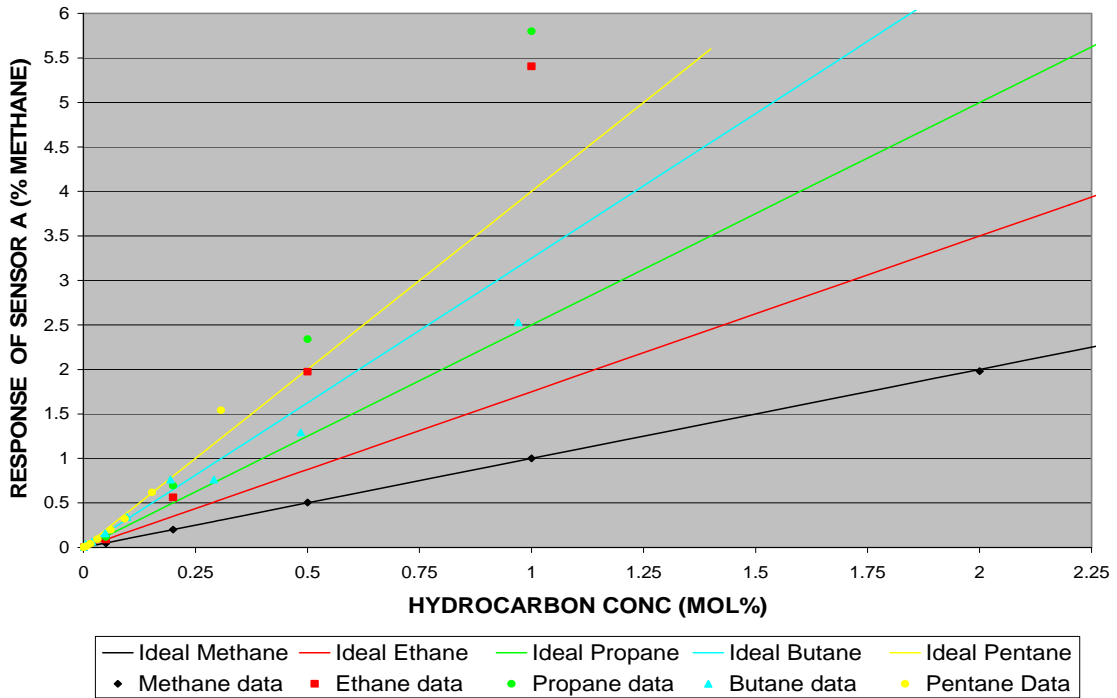


FIGURE 6 – RESPONSE OF SENSOR B WITH METHANE CALIBRATION

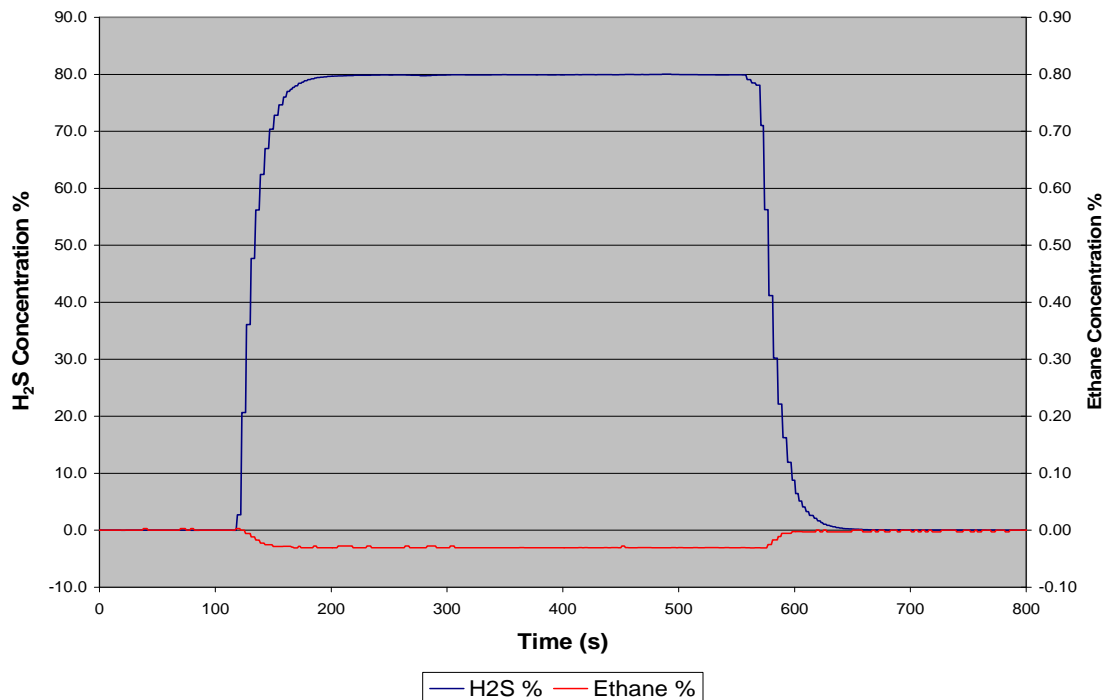


FIGURE 7 – ANALYZER H₂S AND HC OUTPUTS AT 80% H₂S

Carbon dioxide and water vapor have no effect on the readings of either sensor type, if the temperature of the sensor is kept above the water dew point. Hydrogen sulfide has only very weak absorption between 3 and 4 microns and testing has confirmed that there is only a very small effect of high H₂S concentrations on the HC concentration reading from either sensor. Figure 7 shows the HC concentration has a small negative reading when in the presence of 80% H₂S, for the prototype analyzer equipped with Sensor A calibrated for ethane.

ANALYZER INTEGRATION AND SAMPLE SYSTEM DESIGN

A prototype was built with the NDUV optical bench and associated electronics located in two flameproof enclosures which allow the analyzer to be certified for use in Division 1 and Zone 1 hazardous areas without requiring any purge gas. In this application the sample system was given special attention. The toxicity of high concentration H₂S is well known and so the sample system design is simple and compact to minimize the possibility of leakage. A sample probe with built-in particulate and membrane filters is used to extract the sample from the gas line, while removing any liquids or particulates. The probe can be isolated from the process and back flushed (along with the analyzer) so safety integrity is maintained during service intervention. Acid gas is saturated with water at a temperature between 30°C and 60°C, requiring heating of the probe and other sample handling components to avoid condensation. A heater mounted on the probe keeps all of the components, including the sample line connection fitting, at a temperature higher than the water dew point. This probe may allow the sample point to

be moved upstream of the knock-out drum, closer to the amine unit outlet, providing faster response time and early warning of changes in acid gas composition. An electrically heat traced sample line is used to transport the sample to the analyzer oven which contains the NDUV sample cell, the NDIR sensor, and the rest of the sample system. Oven temperature is maintained at 60°C to prevent water condensation and to eliminate potential temperature drift caused by ambient temperature changes. The maximum operation temperature for the NDIR sensor is 65°C, which is high enough for measuring HCs in gas plant SRU acid gas, but makes it unsuitable for refinery SRU feed. A refinery SRU has multiple sources of acid gas including sour water stripper (SWS) gas, which usually contains high concentrations of ammonia. Measurement of hydrocarbons in SWS gas is not possible because ammonia salts tend to form at temperatures less than 80°C.

Flow of nitrogen zero gas is controlled by a solenoid and is set up to auto-zero once a day. The sample gas flows through the NDUV sample cell and NDIR sensor in series, with a needle valve being used to control the sample flow. Gas pressure at the exit of the NDIR sensor will be just above the low pressure flare header to which it vents. A pressure transducer is connected at the exit of the NDIR sensor and is used for active pressure compensation of both concentration measurements. Operating most of the sample system at the low pressure gives a faster response time, reduces the risk of H₂S leaks, and minimizes sample flow requirements. Lower sample flow requirement and faster response time can be achieved by locating the analyzer closer to the sample point.

PLANS FOR FUTURE WORK

Now that a prototype analyzer has been constructed and lab tested, the second phase of the project will be field testing in several different SRU acid gas streams to determine if the analyzer provides an accurate combustibles measurement that is proportional to the air requirement of the reaction furnace.

CONCLUSION

A gas analyzer has been developed that combines a NDUV optical bench with a NDIR sensor for the simultaneous measurement of H₂S and HCs in SRU acid gas, from a single sample point. Both measurements are unaffected by common components in acid gas, including water and carbon dioxide. The HCs do not interfere with the NDUV H₂S measurement, and the interference of the H₂S on the HC measurement is almost zero.

Two different NDIR sensors have been evaluated to determine if they can be calibrated to provide a hydrocarbon concentration output which is proportional to the combustion air demand of the total hydrocarbon mixture that is present in the acid gas. Overlooking this aspect of the IR measurement may have prevented the successful use of this technology in some previous attempts at this application. The results of the in-house evaluation suggest that Sensor A can provide a suitable HC concentration measurement if large amounts of non-methane hydrocarbons are present in the gas mixture and the sensor is

calibrated for the predominant hydrocarbon. Sensor B may be suitable for applications where methane is the main hydrocarbon which changes concentration during upsets.

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