Molten Sulfur Storage and Handling Instrumentation

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Abstract

Molten sulfur produced in Claus sulfur recovery units (SRUs) is often stored in pits, sub-surface collection vessels, collection headers, and above-ground molten sulfur storage tanks. From these vessels, the molten sulfur is loaded onto barges, trucks, or rail cars for transport. Molten sulfur storage tanks may also be common in other parts of the sulfur value chain (e.g., transloading facilities, sulfur forming facilities, sulfuric acid plants). While storage tanks and loading systems are not unique to molten sulfur, the properties and hazards associated with molten sulfur make the design of these systems unique from other applications in many regards. This paper will focus on the unique aspects of measurement and instrumentation in molten sulfur storage and handling systems; specifically, the paper will review the application and requirements of common instrumentation used in molten sulfur applications. For example, the several level measurement options in use on molten sulfur are generally different, or differently applied, than for storage of other types of refinery products. Temperature measurement may use the same technology, but the location for measuring temperatures may be different due to the reasons for measuring temperature (e.g., sometimes temperature may be used to detect a fire, dictating a different location than normal). H_2S may also be measured in the headspace of sulfur storage equipment, which comes with special considerations given the temperature and presence of sulfur vapor that can condense and foul instrumentation. Vapor flow may also need to be measured on air intakes and/or vent out flows, and molten sulfur flow may be measured at different points in the process. The choice of the instruments and their services can have a strong effect on operability and control, as well as safety. This paper will seek to explain best practices for sulfur tank instrumentation based on Trimeric's collective knowledge from consulting and engineering work with molten sulfur clients in oil refineries, sulfur merchant facilities (e.g., transloading), sulfur end users and others. Many of the measurement requirements for molten sulfur tanks would also apply to sulfur pits.

1.0 Overview of Molten Sulfur Handling

Sulfur Recovery Units (SRU) are used in refineries and gas processing plants to convert hydrogen sulfide (H_2S) into elemental sulfur. The most common process is the modified Claus process, which uses a thermal reactor followed by catalytic stages to recover sulfur from acid gas. After each catalytic stage, the sulfur vapor is condensed and removed as molten sulfur. The molten sulfur produced in a Claus SRU is stored and handled in several steps, as illustrated in the example in Figure 1. Key operating requirements and safety considerations for handling and storing molten sulfur are outlined below.

The molten sulfur from the condensers flows through rundown lines to storage in a concrete sulfur pit or steel collection vessel. All flows combined, the molten sulfur flowing into the pit may contain on the order of 300 ppmw H_2S and H_2S_x ([1], [2], [3]) although oxygen enrichment and sub-dewpoint operation can produce higher levels, e.g., 450 ppmw [4]. The sulfur may be degassed in the pit or in a separate degassing unit to remove H_2S down to perhaps 10 ppmw or lower. Even if there is no degassing equipment, some H_2S will evolve in the pit; however, the amount of H_2S evolution depends on several factors including the pit temperature, residence time, degree of agitation, and whether sweep gas is used [5]. When degassing systems are present or air sweep is used in the sulfur pit, an H_2S-laden vapor stream will be produced that must be managed safely. The H_2S poses a flammability / explosion hazard, as well as a health risk for operators (OSHA PEL of 10 ppmv, 8-hr TWA for construction and maritime industries; and 20 ppmv ceiling limit for general industry [6]).

Typically, sulfur pumps transfer molten sulfur from the pit to a tank, where it can be stored long-term prior to loading into railcars, trucks, barges, or, occasionally, pipelines for transportation to customers. The storage tank provides a buffer capacity for sulfur storage, allowing for intermittent tank filling or off-loading of sulfur without disrupting sulfur plant and refinery or gas plant operations. Due to the higher H_2S content in undegassed molten sulfur, the H_2S concentrations in the headspace of a tank storing undegassed sulfur can reach dangerous levels – on the order of tens of volume percent levels [2]. However, even with degassed sulfur (e.g., H_2S =10 ppmw), the equilibrium concentration of H_2S in the tank vapor space can be hundreds of ppmv to low volume percent levels and still extremely hazardous [2].

Sweep air may be used in the tank and pit to keep the vapor space H_2S concentration below 25% of the lower explosive limit (LEL is ~3.0 vol% at 330°F [7][1]). Alternative methods can also be implemented to prevent explosive gas mixtures from forming in the tank headspace [5]; for example, blanketing tanks with an inert gas such as nitrogen (special considerations are required for this, which are discussed later) or using alternate sweep gas media (e.g., steam) are known to be used in industry.

[1] Literature data indicates the LEL of H_2S is approximately 3.9 vol% at 68°F; when the LEL is adjusted for elevated temperature (i.e., 330°F), the LEL of H_2S is approximately 3.0 vol%.

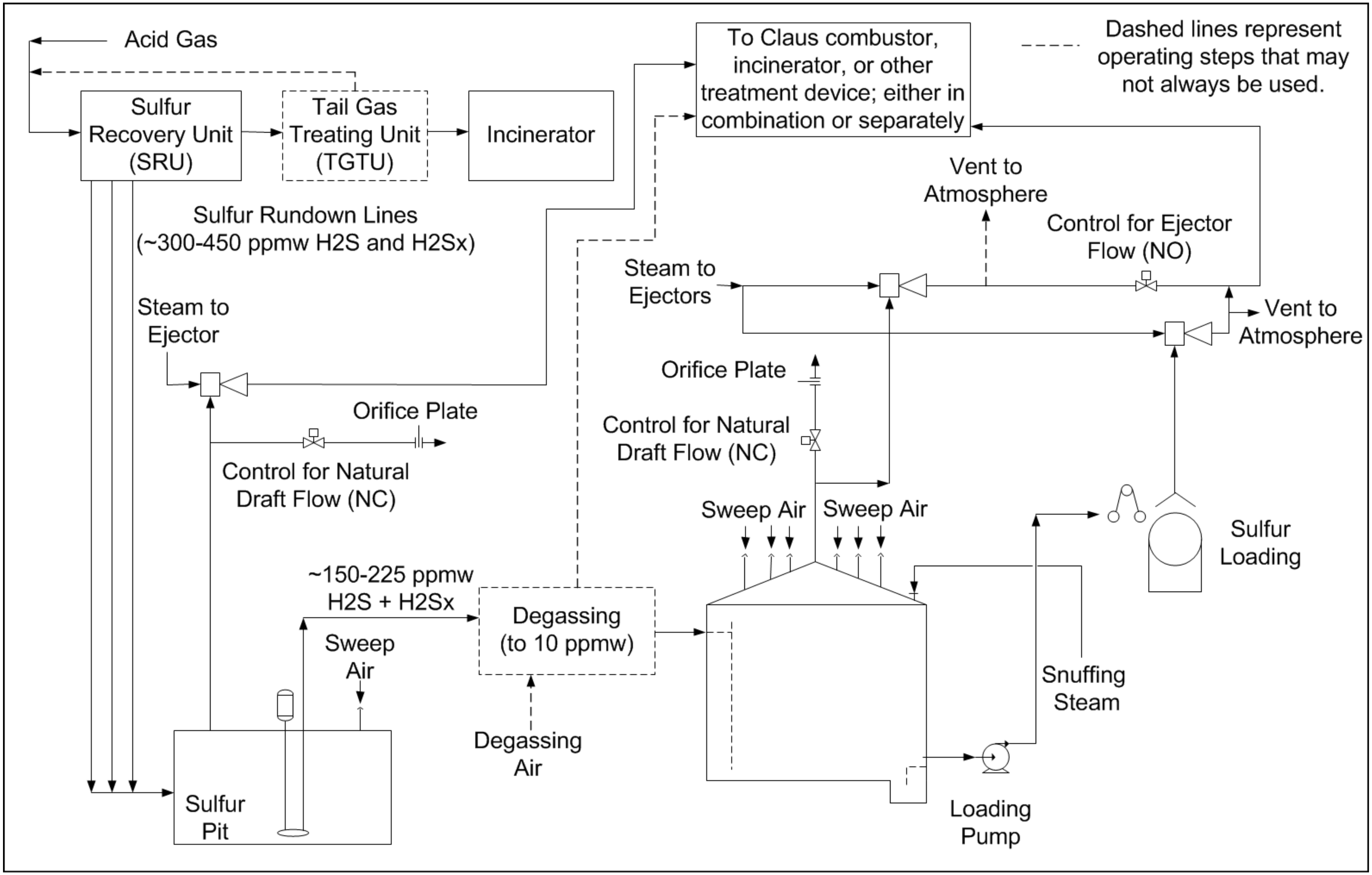

Figure 1: Molten Sulfur Storage and Handling System.

Other sulfur species, such as SO_2, elemental sulfur vapor, and COS and CS_2, can be present around molten sulfur operations as well. SO_2, while not flammable, is toxic, and elemental sulfur vapor can cause plugging and corrosion issues (with liquid water) if condensed. Although CS_2 is not normally present in significant quantities, molten sulfur storage systems can potentially operate at temperatures above the auto-ignition temperature of CS_2. The auto-ignition temperature of CS_2 depends on the concentration of CS_2 present and the overall composition of the gas (e.g., the presence of H_2S suppresses autoignition of CS_2/raises the auto-ignition temperature); an auto-ignition event also requires the presence of air [8].

In addition, molten sulfur, with its unique properties, presents other specific challenges that must be carefully considered for safe handling and management. As shown in Figure 2, the properties of pure sulfur create a narrow operating window (260-300°F) due to freezing, flash point, and autoignition concerns. Field experience in the literature [9] also reports that temperatures above 309°F are more prone to sulfur fires, even though this temperature is well below the flash point. The viscosity of pure sulfur also increases above a certain temperature (318°F), at which point there could be difficulty in pumping/handling the molten sulfur. Lastly, the total H_2S solubility also increases with temperature, which in turn increases environmental, health, and safety (EHS) risks, the potential for explosions, and impacts on other physical properties.

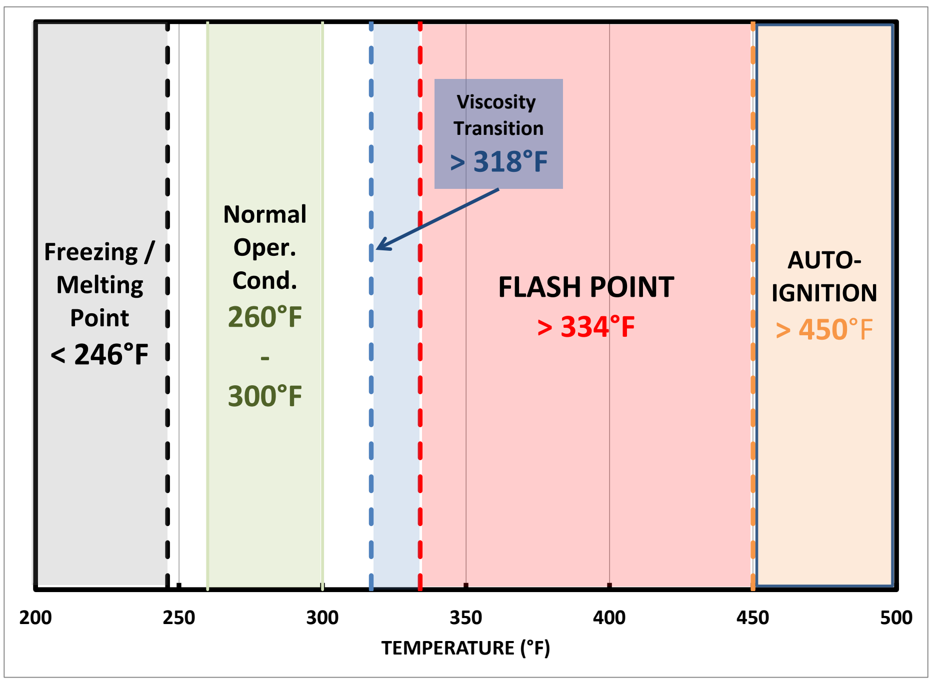

Figure 2: Pure Sulfur Properties.

As such, the properties and hazards associated with molten sulfur make the design of the storage and handling systems unique, requiring careful monitoring and control of operating conditions to ensure optimal performance and safety. This paper provides an overview of the important process measurements in different areas of the molten sulfur handling equipment to illustrate the key types of measurements that are common across sulfur handling processes.

Then, best/common practices for molten sulfur instrumentation are reviewed, with a focus/emphasis on molten sulfur tanks.

2.0 Overview Important Process Parameters and Measurements

This section discusses the important process parameters that are measured in different areas of the molten sulfur storage and handling system. A summary table of the process parameters/measurements is provided at the end of this section for reference. Then, subsequent portions of the paper discuss the instrumentation used to measure those parameters.

2.1 Molten Sulfur Rundown from SRU

The goal for sulfur transfer from the SRU to the pit/storage vessel is to provide a reliable, continuous outlet for sulfur with minimal impact on the reliability or operability of the SRU. To this end, while individual SRU designs may vary, molten sulfur typically flows by gravity via sloped lines from the condensers in the Claus sulfur recovery unit to a molten sulfur pit or similar storage vessel. Equipment (e.g., pumps) and instrumentation are minimized or avoided altogether in these lines to reduce the risk of failures/reliability issues. Where process data is needed from the rundown system, proxy data from other parts of the process are often used in lieu of direct measurements in the rundown lines. For example, the molten sulfur production rate might be calculated indirectly from the gas-side SRU system sulfur balance and validated with molten sulfur level measurements in downstream storage equipment rather than attempting to measure the flow in the rundown lines directly.

Two types of equipment/instrumentation are common in sulfur rundown lines: sulfur seals of some sort and sulfur look boxes and/or sight glasses. These items are covered briefly here for completeness but are not the focus of this paper and are unique to the rundown system.

- Sulfur seals are used to create a liquid sulfur seal between the main SRU process equipment and the downstream storage equipment for molten sulfur. This seal prevents gas blow-by from the SRU to downstream sulfur collection equipment, which could pose serious hazards to personnel and the surrounding environment and facility. There are a variety of sulfur seal designs, generally grouped into in-ground/underground seal legs and above ground mechanical seals.
    - Underground seal legs have been more common historically and rely on a vertical leg of liquid sulfur to provide sufficient liquid head to prevent gas blow-by. The seals effectively serve as a relief path when the seal is blown, although this carries the risks associated with gas blow-by that the seal was intended to prevent.
    - Above-ground seals typically use an internal float and/or orifice and other proprietary internals that seal when pressure increases during a potential gas blow-by event. As long as the seal device performs as designed, there is no path for gas blow-by; therefore, the seal cannot be relied upon for gas relief unless the seal design includes a specific supplementary relief feature as part of the design. Above-ground seals provide easier access for maintenance and cleaning.
- Sulfur look boxes/sight glass are used to allow visual verification of flowing sulfur from the SRU through the rundown lines. Traditional look boxes were often hatches that would open directly to the process fluid; this represented both a significant convenience and a

potential hazard for personnel from the high temperature molten sulfur itself and the associated hazardous dissolved gases (H_2S and SO_2). Attempts to enclose the viewing port utilized sight glass devices – these devices can suffer from fogging and issues that reduce or eliminate visibility over time. The sight glass devices have been modified over time with proprietary upgrades and reliability features (e.g., heating) to address concerns. Sight glasses are often integrated into above ground seals.

2.2 Molten Sulfur Short-Term Storage – Sulfur Pit, Collection Header, Vessel

Molten sulfur from the SRU rundown lines typically flows to a sulfur pit for short-term storage and sometimes in-pit degassing. A sulfur pit is a large, underground structure designed to temporarily store sulfur in a liquid state using steam coils or other methods. It is constructed using corrosion-resistant materials, such as reinforced concrete or protective liners, to withstand the harsh conditions created by molten sulfur, impurities, and potential water intrusion from leaks. Sulfur pits also typically operate at a slight vacuum.

Other types of short-term containment devices can be used such as a collection header or vessel. A collection header is a larger diameter section of pipe that gathers molten sulfur from the SRU. A collection vessel is usually a steel vessel in a concrete sump. It can be a pressure vessel to withstand deflagrations, and a sweep air system is typically not used. The challenge with collection vessels or headers is that they typically have less storage capacity than sulfur pits. Similar instruments and measurements would be used as appropriate for these types of containment devices.

2.2.1 Key Process Measurements for Sulfur Pits

Sulfur pits present several operational and safety hazards such as high temperature, toxic gas release, fire danger, and corrosion. Specific instrumentation and safety systems to monitor, control, and mitigate risk that may be present follow.

- Molten Sulfur Level Measurement: Molten sulfur levels are monitored to ensure the pit does not overfill or drain to low levels that could cause the transfer pump to cavitate or expose steam coils.
- Molten Sulfur Temperature Measurement: The temperature of the molten sulfur is measured to maintain the sulfur in the liquid form at the appropriate temperature range (typically 260-300°F) to prevent solidification or overheating that can increase the risk of fire.
- Vapor Space Temperature Measurement: The vapor space temperature is monitored to detect a sulfur fire.
- Vapor Space Pressure Measurement: The vapor space pressure is monitored to detect for overpressure or poor ventilation.
- Vapor Space H_2S Measurement (uncommon): The H_2S content in the vapor space may be measured to provide indication of potentially dangerous/hazardous H_2S concentration accumulation in the vapor space.
- Verification of Sweep Air Flow: There are multiple potential approaches to verifying sweep air flow in sulfur pits; these approaches may include direct measurement of

sweep air flow or indirect approaches such as pressure measurements in the pit or at the motive device for the sweep air.

2.3 Molten Sulfur Degassing

As shown in Figure 1, some SRUs will include a sulfur degassing technology to proactively remove H_2S from the molten sulfur before it reaches storage and transport areas. While sulfur degassing does not eliminate all hazards associated with molten sulfur handling (H_2S can still reach potentially lethal levels in downstream equipment), it does reduce hazards associated with H_2S and reduces the amount of H_2S that can evolve in the downstream process, and potentially reduces the rate at which H_2S evolves.

This paper is not focused on a review of molten sulfur degassing technologies; there are many technologies that have been developed, offered commercially, and applied in industry. Rather, this paper identifies key process measurements that may be required for sulfur degassing technologies.

2.3.1 Key Process Measurements for Sulfur Degassing

Degassing technologies can be implemented in the sulfur pit or collection vessel itself or applied externally as a standalone unit operation. Depending on the specific implementation and technology, the instrumentation requirements can vary widely. The focus of this paper is to highlight common key instrumentation that is present in the molten sulfur portion of the process or in the gas containing stripped H_2S and SO_2 of many sulfur degassing systems:

- Molten Sulfur Temperature Measurement: Reliable molten sulfur temperature measurement is critical for many degassing technologies that control and monitor the sulfur feed temperature to the degassing system to optimize performance.
- Vapor-Phase Pressure Measurement: Some sulfur degassing technologies may operate at elevated pressure – reliable measurement and control of the gas-side pressure is important in these systems. For example, the gas leaving the degassing system may be particularly susceptible to corrosion (presence of SO_2, elevated pressure) and plugging (entrainment of molten sulfur, presence of sulfur mist).
- Molten Sulfur Level Measurement: Some degassing technologies may maintain a molten sulfur liquid level in the degassing contactor or vessel; selecting appropriate molten sulfur level measurement and a level control valve are important for these degassing technologies.
- Molten Sulfur Flow Measurement: Some degassing technologies may operate with flow control to feed the degassing unit; selection of appropriate molten sulfur flow measurement and flow control valve are required for these systems.

While degassing technologies are targeting a specific residual H_2S content in molten sulfur (typically < 10 ppmw total $H_2S + H_2S_x$), continuous composition measurements are not common on the gas or liquid side of the degassing system. An initial performance test followed by periodic sampling can be used to verify composition and a sulfur material balance data around the broader system may be used to assess degasser performance.

2.4 Molten Sulfur Long-term Storage – Sulfur Tank

The molten sulfur from the pit or degassing unit is often stored in an above-ground molten sulfur tank before further processing or transportation. The required storage capacity should account for the worst-case shipping scenario. A sulfur production capacity of 1 to 5 days is typical with 5 to 10 days being preferred. The capacity can be split between the sulfur pit and tank. The molten sulfur tank is typically significantly larger than the sulfur pit and is usually an above-ground, steam-heated, carbon steel tank. The tanks are often designed to follow API-650 specifications. The tank has several key design elements as shown in Figure 3 and summarized below:

- Heating System: To keep the sulfur molten, tanks are usually equipped with either i) multiple, parallel, internal steam coils (common) or ii) external heating with steam-trace panels (newer, increasingly common) to avoid the risk of internal steam leaks and corrosion.
- Insulation: The tank and all attachments to it are insulated to reduce heat loss and prevent the occurrence of frozen solid sulfur.
- Headspace Sweep System or Blanketing System: The headspace of a molten sulfur tank may be managed to prevent explosions either by using a sweep gas to dilute headspace vapors to maintain the H_2S concentration to less than 25% of the LEL or by using an inert blanket gas to prevent oxygen ingress and avoid explosive headspace conditions.
- Fire Mitigation: Fires may be suppressed by introducing sealing steam, or possibly water sprays in some situations [10], to inert the vapor space.
- Other Safety Features: Frangible roofs and deflagration vents (above 309°F per NFPA 655 [11]) are used to prevent catastrophic tank failure and protect plant personnel from explosion risk.

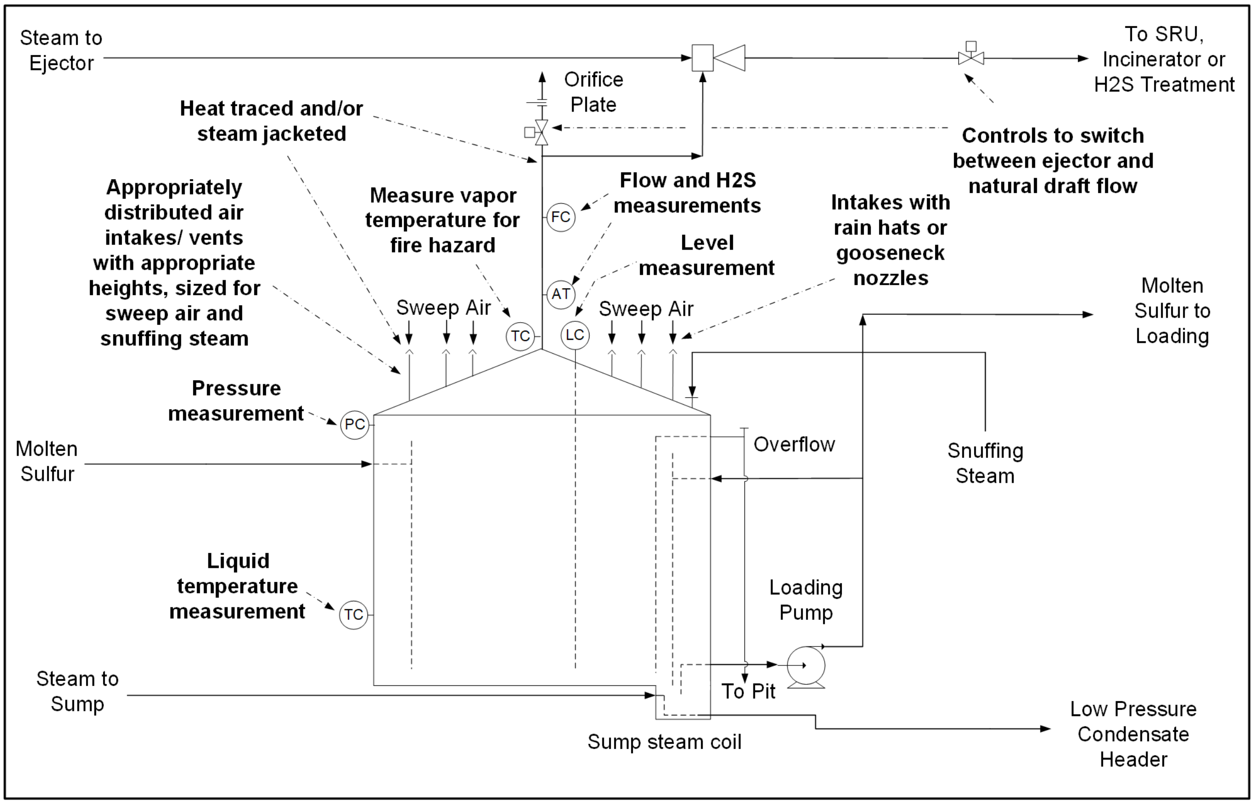

Figure 3: Schematic of Molten Sulfur Tank.

2.4.1 Key Process Measurements for Molten Sulfur Tanks

Similar hazards exist in molten sulfur tanks as in sulfur pits, and similar process measurements and instrumentation would be used as well. A unique challenge for molten sulfur tanks is that due to its large size and different designs, multiple measurement points may be needed to assess the operating conditions in the tank.

- Molten Sulfur Temperature Measurements: Sulfur should ideally be stored in an "optimal" temperature range as depicted in Figure 2; in other words, the temperature should not be too high or too low. On the low end, sufficient margin should be maintained above the freezing temperature of sulfur. On the high end of the temperature range, excessive heating can lead to fire/explosion risks and/or potentially sharp transitions in the physical properties of sulfur (e.g., viscosity).
- Vapor Space Temperature Measurements: The roof and vapor space portion of the tank should be adequately heated and the temperature of the tank vapor space should be measured to determine if there is potential for sulfur condensation and freezing on the roof or tank walls above the liquid level. When sulfur solidifies or condenses, it can trap moisture between the sulfur and metal surface that causes corrosion on the metal surface and promotes the formation of pyrophoric iron sulfide in anaerobic environments. This becomes a safety concern in tanks blanketed with inert gas (e.g., nitrogen) when the pyrophoric iron sulfide is exposed to air during maintenance. A similar hazard exists in sweep air systems, where pyrophoric iron sulfide can form beneath solid sulfur and later

ignite if fragments break loose and come into contact with oxygen in the sweep air [12]. Monitoring the tank vapor headspace or outlet vent gas temperature for high temperature may also provide indication of fire.

- Vapor Space Pressure Measurement: The vapor space pressure should be monitored to ensure the tank is operating at the intended operating pressure and to detect/avoid overpressure/excessive conditions.
    - In the case of tanks that are swept with air (or other media), the headspace will normally operate at a slight vacuum to maintain flow through the headspace of the tank. Monitoring the pressure may provide indication of loss of sweep gas, plugging, or other problematic conditions associated with the sweep gas/ventilation system.
    - In the case of blanketed tanks, the vapor space pressure measurement may be used directly/integrated with the controls that supply and/or vent the pad/blanket gas from the tank during operations to maintain a constant, slightly positive, tank pressure and prevent oxygen ingress
    - Common potential overpressure scenarios in a tank include pressure-build during tank filling and pressure-build during application of snuffing/sealing steam. Vacuum conditions may occur during tank unloading.
    - When using a motive device (e.g., ejectors) on a tank, it is important to monitor the headspace pressure and suction pressure of the motive device to ensure the tank is not drawn below its allowable vacuum limit. A significant drop in pressure at the tank and/or suction of the motive device may also be indicative of plugging/blockage in the system, which may correspond to loss of sweep air flow.
    - Finally, during a fire scenario, the tank pressure could be monitored for a positive pressure signifying the tank is sealed adequately with steam to prevent air ingress so the fire can be extinguished.
- Vapor Space H_2S Measurement: The H_2S content in the vapor space is sometimes measured to provide indication of potentially dangerous/hazardous H_2S concentration accumulation in the vapor space.
- Verification of Sweep Air Flow: There are multiple potential approaches to verifying sweep air flow in sulfur tanks; these approaches may include direct measurement of sweep air flow or indirect approaches such as pressure measurements in the tank or at the motive device for the sweep air.
- Molten Sulfur Level Measurement: The sulfur level is measured to help prevent the tank from overfilling or running dry.

2.5 Molten Sulfur Loading

Transfer and loading of molten sulfur for transport is often the final step in the molten sulfur handling system, e.g., in a refinery. The transport mode may vary (truck, rail, barge), but the loading operations will be similar at a high-level. In general, sulfur is pumped from the molten sulfur tank (or other sulfur storage equipment) to loading racks/stations, which include loading arms to supply the molten sulfur to the transport vehicle and sometimes include a weigh scale (e.g., truck loading) to verify the transfer of sulfur. In some cases, there are environmental or personnel safety considerations associated with the vapors evolved from the sulfur as it is loaded; field data corroborates that H_2S will evolve from the molten sulfur during loading due to agitations, splashing, etc. Therefore, some loading racks will include a vapor recovery system. These vapor recovery systems often consist of a specially designed "cover" or vapor plate assembly for the loading hatch to recover vapors but allow for air to be pulled in to dilute the vapors, a steam-jacketed vapor recovery line (ideally rigid line to ensure consistent flow/prevention of flow restrictions), and a motive device (e.g., steam-driven eductor) to recover vapors and send them to ultimate disposition [13], [14].

2.5.1 Key Process Measurements for Molten Sulfur Loading Operations

Sulfur loading system controls generally focus on ensuring that appropriate sulfur transfer has occurred from the storage system to the transport vehicle. Vapor recovery systems may require additional process measurements and controls (e.g., monitoring pressure/temperature/flow in the vapor recovery lines), but these are similar instruments required in other vapor systems associated with molten sulfur operations and are not considered explicitly for molten sulfur loading operations.

- Molten Sulfur Flow Measurement: Some sulfur loading operations will include measurements of sulfur flow to verify transfer rates and total volume to the transport system; this data may be useful for custody-transfer verification and also serves to identify issues with the loading system operations (e.g., pump issues, plugging, etc.).
- Molten Sulfur Level Indication: While some loading operations involve visual verification by the transport operator (e.g., truck driver) to ensure the transport vehicle is not over-filled, this approach presents a risk to the operator and is not a reliable method for monitoring level. Best practice would include level instrumentation associated with the loading arm assembly.

2.6 Summary of Key Measurements by Area

Table 1 shows a high-level summary of the types of measurements that may be taken at different areas of the molten sulfur handling and storage system. The following sections will review some of the key common instrumentation identified across all areas and some guidance regarding selection/application of instrumentation in molten sulfur service (with a specific focus on sulfur tanks/storage).

Table 1: Key Measurements by Area.

Location	Temperature	Pressure	Level	Sulfur Flow	Vapor Space Analysis
Rundown Line	Minimal instrumentation				
Sulfur Pit	X	X	X		X
Degassing	X	X	X	X	
Storage Tank	X	X	X		X
Loading	X		X	X	
Transfer Pumps	X	X		X	

3.0 Molten Sulfur Level Measurement

Measuring the molten sulfur level in the sulfur pit and storage tank is important to prevent overfilling, which can result in spills, safety risks, and damage to equipment since molten sulfur is hot (260-300°F). Monitoring level can also prevent operating at low-level conditions, which could expose heating coils (if used), result in lower temperatures in tanks, and cause transfer pumps to cavitate. Level measurements also help ensure a steady flow of molten sulfur to downstream units.

Level instruments used in molten sulfur service include bubble, radar (guided wave and non-contact radar), capacitance, and diaphragm-type level measurements. The experience with each of these instruments varies in the industry and is impacted, in part, by the design and implementation of the instrument. These devices need to be able to withstand the high temperature and corrosive environment as well as be resistant to sulfur plugging.

A bubbler-type device uses air or inert gas pressure to estimate the liquid level in the tank or pit (see Figure 4). A constant flow of air or inert gas flows through a dip tube inserted to near the bottom of the container. At higher liquid levels, the pressure required to push bubbles out of the dip tube increases. A pressure transmitter measures the backpressure that is proportional to the liquid height above the tube opening. Typical bubbler tubes are often sized at 1-inch diameter (schedule 40). However, for viscous products such as molten sulfur, a 2-inch tube (stainless steel) may be used. The larger diameter makes the tube easier to clean and less prone to clogging, resulting in a longer operational life and more reliable performance. The bubbler design may also allow for blow down with high pressure gas (e.g., air, nitrogen) to clear plugs, and a v-notch or tapered outlet may be included at the bottom of the bubbler tube to promote a constant/consistent stream of bubbles. Some designs favor steam jacketing the bubbler tube down to near where the bubbles exit the bottom of the tube. Finaly, a rod-out feature to clean the bubbler tube should be considered.

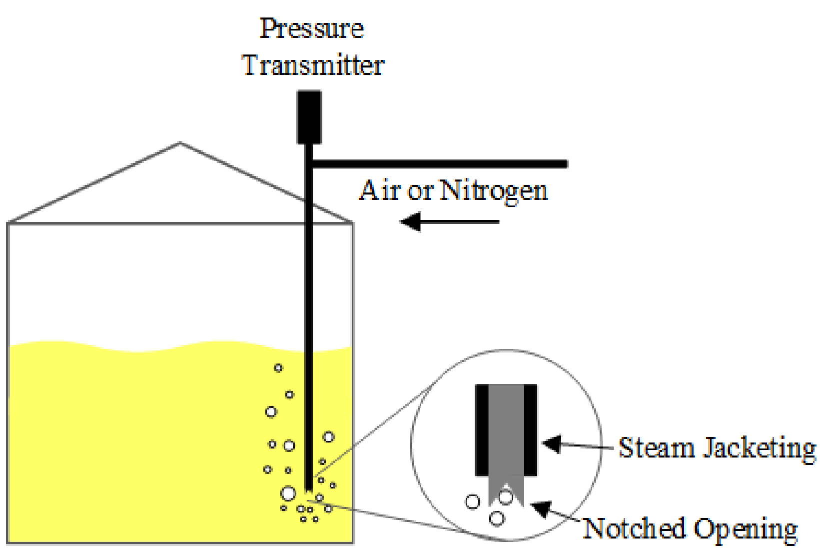

Figure 4: **Simplified Schematic of Bubbler-Type Level Device (excluding steam condensate drain, rod-out connection, and other details).**

Radar level instruments (guided wave or non-contact) operate on similar principles (see Figure 5). The instruments rely on Time Domain Reflectometry (TDR) which measures the time between emission and detection of the pulse generated by the instrument to calculate the distance to the liquid surface being measured. Guided wave radar (GWR) uses a probe inserted into the tank or pit; the radar signal travels down the probe and reflects off the liquid surface. By guiding the pulse to the liquid surface, GWR probes are suited to perform well when there is a risk of scattering or absorption of the signal at the liquid surface (e.g., agitation at the surface or low dielectric fluids, such as molten sulfur). Molten sulfur can build up and coat the probe, potentially impacting the level measurements. However, the authors know of one refinery that has had good operating experience with GWR in their existing molten sulfur tank and GWR is used in other fouling services. Non-contact radar sends radar pulses from a sensor mounted at the top of the tank; the time required for the pulse to reflect off the liquid is used to estimate the distance and liquid level. Non-contact radar uses special antennas to direct and receive the signals, and the device is mounted on the roof of the tank. Some designs do not extend into the vessel, while others do. Accumulation of sulfur deposits on the radar instrument is a potential problem with these units, and occasional cleaning may be necessary. If the unit does not extend into the tank vapor space, any build-up on the roof itself or nozzle may interfere with the measurement. The nozzle used for installation of the device should be well insulated and heat traced to prevent sulfur condensation. Some instruments feature full steam tracing of the radar antenna itself [15].

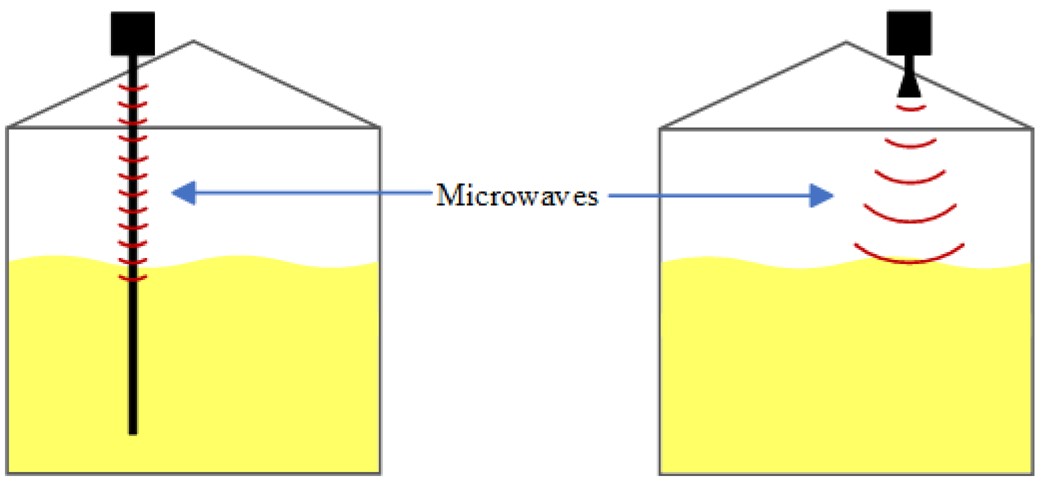

Figure 5: **Simplified Schematic of Radar Level Devices.**

Diaphragm-type level instruments need special consideration for molten sulfur service. When sweep air is used, the tank or pit operates at a slight vacuum to ensure air flow into the headspace. A differential pressure transmitter with remote diaphragm seals and capillary tubing can be used for level measurement in molten sulfur tanks. The diaphragm seal isolates the detecting components from contact with the molten sulfur, protecting it from corrosion and high temperatures. The high-pressure side of the transmitter is connected to the diaphragm seal at the bottom of the tank that measures the total pressure from the molten sulfur column and vapor space. The low pressure-side is connected to the diaphragm seal mounted in the vapor space. Both seals are connected to the differential pressure transmitter by sealed capillaries filled with a fluid that can withstand elevated temperatures. The seal faces should be heated and insulated to keep the diaphragm hot to prevent sulfur from solidifying. The transmitter calculates the differential pressure from the high and low-pressure sides, which represents the hydrostatic head which is converted to sulfur level. Other diaphragm-type methods may also be used.

Additionally, depending on the process control philosophy for the site, redundant level measurement may be employed. The high-level measurements could be used to trigger additional actions such as upstream shutdown, or diversion of the molten sulfur, among others. This may also create an opportunity to use two different level measurement instruments, if desired, to avoid common failure modes.

For loading operations, bubbler-type level devices could be used; additionally, RF capacitance or transmittance type sensors may be appropriate if supported by field experience in molten sulfur service.

For all level instrumentation, and for any instrumentation in molten sulfur in general, the key is careful design and attention to the details of installation to maintain temperature and robustness.

4.0 Vapor-Space H_2S Measurement and Indirect Measurement of Explosive Atmosphere Risk

Molten sulfur storage equipment and handling equipment is susceptible to the accumulation of H_2S to dangerous levels [5], including concentrations that may be at or above the low explosive limit (LEL) of H_2S (~3.0 vol% at 330°F [7]). Even H_2S evolved from degassed sulfur can reach levels above 1.0 vol% H_2S which are lethal and may exceed allowable operating limits for approach to LEL (e.g., above 25% of LEL).

While there are several design approaches to mitigating risks associated with H_2S in the headspace of sulfur storage and/or transport equipment (e.g., [16], [17], [10]), including sweeping the headspace of equipment to maintain low H_2S concentrations, blanketing of equipment to limit oxidant concentrations, limiting ignition sources, providing response measures to fires/explosive environments (e.g., sealing/snuffing steam), etc., operators may want verification that these systems are operating properly and may want early detection of accumulation of H_2S in equipment to minimize risks of incidents.

The most direct approach to detecting risks associated with H_2S accumulation in the headspace is to measure the H_2S concentration itself. There are a variety of instruments offered to measure H_2S concentration in the gas phase, but also many considerations for the practical implementation of this sort of measurement. Depending on the specific equipment and mitigation measure(s) used to prevent dangerous levels of H_2S accumulation, there may be secondary process measurements that can provide an indirect measurement of H_2S/LEL risks. The following sections cover the direct measurement of H_2S followed by examples of indirect measurement for specific types of sulfur storage systems.

4.1.1 *Direct Measurement of H_2S – H_2S Analyzers*

H_2S can be measured in the gas-phase directly by a variety of commercial analyzers. H_2S analyzers are widely used in industry, including measurement of H_2S in natural gas, treated gas streams in a range of applications (e.g., refinery fuel gas systems), and within the SRU itself in applications such as the feed gas or tail gas (i.e., tail gas air demand analyzer). However, the use of H_2S analyzers specifically in sulfur storage and handling is less common; there are known applications of sulfur pit H_2S measurements and other similar measurement applications, but in Trimeric's experience, this is not a universally accepted practice. Some of the challenges with H_2S measurement in sulfur handling and storage are outlined in this section and may, in part, explain the less extensive use. In addition, as noted previously, although the sulfur handling part of the SRU is critical for the safe and reliable operation of the SRU, it does not represent the core operating area with the SRU; therefore, additional instrumentation and controls in the molten sulfur areas may be more difficult to justify.

Prior to considering the analyzers themselves, there are common challenges associated with the implementation of analyzers in practice:

- <u>Suitability for the Service/Conditions:</u> As with all instrumentation in sulfur service, the ability of the instrument to withstand the environment and perform as designed is a critical consideration.
    - H_2S analyzers utilized to sample the vapor space of a molten sulfur tank will need to be robust to withstand a potentially corrosive environment (particularly if any cold surfaces are exposed to the vapor space, leading to condensation of water and acid gas species). At minimum, this involves the appropriate selection of materials of construction and proper heating of all components exposed to the vapor space.
    - The analyzers are directly sampling gas from the vapor space and will be directly exposed to elemental sulfur (as vapor or mist) – therefore, the instrument must have means to manage elemental sulfur to prevent plugging/solids instrumentation in the equipment.
        - Some analyzers may transport collected gas over some distance from the probe to the analyzer – this requires careful design to prevent condensation of sulfur where possible and consider how to prevent accumulation of any liquid sulfur (e.g., mist) that is present from collecting in the sample line. Trimeric is aware of industry reports of plugging of sample lines for some operators. The operators reported this a critical concern regarding installation of H_2S analyzers in molten sulfur service.
        - Some instruments may include mechanisms or sample conditioning systems to remove elemental sulfur prior to reaching the sensitive measurement components – however, any attempt to separate elemental sulfur (condensation, filtering, etc.) introduces a potential failure point and may introduce other unintended consequences (e.g., corrosion risks) if not implemented properly.
    - The measurement point will be exposed to the high temperatures of the molten sulfur vapor (in excess of 200°F, much higher near heated surfaces of the system).

Many commercial H_2S analyzers used in other applications (e.g., sampling natural gas) are not designed to operate with gas samples at these conditions. Conditioning the gas to support the use of these analyzers will require managing all the aforementioned risks with low temperatures associated with the vapor samples in sulfur service (temperatures will be below sulfur condensation temperature and may also be below acid and water dew points in the system).
    - Because air swept tanks operate under a slight vacuum, a sample pump may be used to draw a small, controlled flow of vapor to the analyzer for certain devices. The sample extraction equipment is a high-risk area for plugging, condensation of sulfur, etc., which makes extractive sampling particularly challenging in sulfur applications and requires a fit-for-purpose design proven in the field.
    - In Trimeric's experience, the variability and severity of the conditions in molten sulfur storage vapor spaces makes it a highly challenging application for direct continuous gas composition measurement. The design of an analyzer may appear to address major reliability concerns; however, actual field operating experience in the same service (molten sulfur storage vapor space) is essential to confirm that an analyzer will actually perform as designed and does not create undue maintenance burdens or costs (e.g., high frequency replacement of components).
    - In contrast to continuous measurement, periodic vapor space sampling is a common practice in many molten sulfur applications and many operators consider this sufficient for their operational needs; for a continuous H_2S analyzer to justify the relatively high upfront cost, it must operate with high reliability and accuracy and low recurring costs over the design lifetime of the instrument.
- <u>Location of H_2S Sample Probe:</u> Sulfur storage equipment can include a large vapor headspace (e.g., molten sulfur pit or tank). The concentration of H_2S can vary in the headspace of these systems as a function of the geometry and the systems may be prone to "dead" or stagnant zones where H_2S may accumulate. Therefore, identifying a representative point measurement for H_2S in the headspace may be difficult. If the headspace is vented or vapor is recovered from the storage system, the vent/vapor recovery line may be a good location to get a representative H_2S concentration, particularly if the vapor space is expected to be relatively well-mixed. However, this approach may miss the highest risk or early detection areas for H_2S accumulation in the system.
    - Computational Fluid Dynamics (CFD) can be (and has been) used to identify potential areas for elevated H_2S concentrations in storage systems such as tanks and pits. However, CFD relies on the quality of input data from the system (e.g., information on H_2S evolution rates) and is typically more robust for an existing system where real field data is available to support/validate the model vs. a new system design where only expected performance information is available.
    - Multiple sample points may be possible with certain analyzers; however, each measurement point will add cost (potentially significant costs) and complexity, and given the size of the vapor space, it is likely impractical to install a sufficient number of sample points to ensure representative sampling of the full vapor space.

- Installation of Analyzers: Analyzers can be costly, sensitive pieces of equipment that require protection from the environment, utilities to operate as designed, and other specific considerations for proper/reliable field installation.
    - Protective Structures: While most H_2S analyzers offered commercially do not require a conditioned space, most analyzers do require some level of protection from ambient conditions. This may include a structure e.g., shed or three-sided cover to protect the analyzer from direct sunlight and precipitation.
    - Utilities: Many H_2S analyzers will need power, steam, and/or instrument air. The analyzer will also need communications connections to the local DCS at the site.
    - Weight: The analyzer cabinet/enclosure can have substantial weight (e.g., several hundred pounds) before accounting for any of the protective structure. If the analyzer is installed at grade, this may not be an issue. However, many analyzers are limited in the distance they can be located from a sample point; this is due to considerations regarding sample reliability, quality, and accuracy, with the specific technical reasons for the limitations being specific to each analyzer/vendor. If the sample point is in a difficult to access location well above grade (e.g., sulfur tank vent), this may require considering the option to install the analyzer on the equipment structure itself (e.g., tank roof or supporting structure with the tank). This may add additional cost/complexity to the analyzer installation.
    - Access for Maintenance: Any analyzer used in molten sulfur service will require access to the sample probe and analyzer cabinet for routine maintenance activities (e.g., cleaning of components, replacement of consumable parts, calibration). While many state-of-the-art analyzers include capabilities for remote/on-line cleaning or calibration steps, the analyzer and sampling system still require access for direct maintenance, with a frequency that will almost certainly require accessing the probe while the tank or storage system is still operational/handling sulfur. Therefore, safe access for personnel to perform maintenance activities must be considered explicitly in the installation of the analyzer.
- Cost of Analyzers: Analyzer vendors should be consulted directly for the cost of a specific system in a specific application. However, analyzers can exceed $100,000 USD even prior to considering costs associated with any protective/support structures, utilities, and installation costs. Therefore, they can represent a significant expense.

There are a variety of H_2S analyzers available commercially; this paper is not focused on providing a detailed technical review of all analyzers available or the theory of operation; the vendors/manufacturers of the analyzers should be consulted directly for technical information on their analyzers as they will have subject matter expertise regarding the design and intended operating principles of the analyzer. A few general classes of H_2S analyzers are described below:

- UV-Vis (Ultraviolet-visible spectrophotometry): This type of analyzer is offered by multiple vendors and has been used on molten sulfur service. As indicated by the name, the method relies on analysis of light absorption (in the visible and ultraviolet range) of gas samples to assess H_2S content. These instruments will include light sources, optics, and a sample probe/collection system.

    - Many of the UV-Vis type analyzers have been used in gas streams in the Claus process (e.g., tail gas analyzers). While conditions are similar to a molten sulfur handling system, the conditions are not identical; therefore, the application in other areas of the Claus unit may not be a perfect proxy to establish reliability in sulfur storage applications.
- TDL (Tunable Diode Laser): These analyzers are offered for direct, in-situ measurement of H_2S in a range of applications, including harsh environments such as coke oven gas and catalytic reformer gas. However, Trimeric is not directly aware of this type of analyzer being used in molten sulfur service. Like UV-Vis, TDL relies on a spectroscopic measurement using a laser light source (producing infrared light) to measure gas species absorption.
    - TDL, in particular, may be susceptible to interference from moisture when measuring H_2S, which may be a key limitation for use in sulfur service.
- Electrochemical Sensors: These sensors are known to be used in wastewater, biogas, and oil and gas applications for measurement of H_2S. Electrochemical sensors rely on redox-type chemistry when H_2S reacts with the sensor substrate/electrode to produce electrical current which is measured and related to H_2S content. Trimeric is not aware of these types of sensors being used in molten sulfur service.
    - The sensors are typically limited to relatively low temperatures (e.g., <120°F), which would require sample conditioning/cooling to be used in molten sulfur service.

Other approaches (lead acetate tape, stain tubes, etc.) have been used for field measurement of H_2S in a variety of industrial applications, but are not generally suited for the continuous sampling provided by other H_2S analyzers discussed in this report.

In summary, it is possible to reliably measure H_2S in the headspace of a molten sulfur tank, but only if one uses a very high level of attention to detail in the design and implementation of the measurement systems. Sometimes, molten sulfur storage is not given that level of detail.

Aside: Note that for blanketed tanks, which are designed to prevent oxygen ingress, measuring oxygen concentration in the headspace could theoretically be done with analyzers in a manner similar to the H_2S detection described in this section. However, in blanketed tanks, maintaining and monitoring tank positive pressure is a robust, common, and reliable approach to detecting the risk of air ingress.

4.1.2 Indirect Measurement of LEL/Explosive Atmosphere Risk – Secondary Process Measurements

Sulfur storage systems may include specific mitigation measures to prevent an explosive headspace environment in sulfur pits, vessels, and tanks. These systems can include sweeping the headspace of the storage system with a diluent gas (air is most common, but steam and nitrogen have been used in industry) or can include blanketing the system with an inert gas (e.g., nitrogen and maintaining positive pressure to prevent air ingress and avoid sufficient oxidant concentration to yield an explosive headspace even in the presence of H_2S at levels at or above the LEL. As noted, the blanketed tank systems can use the tank headspace pressure as a reliable approach to detecting the risk of oxygen ingress; thus, this indirect measurement is a common approach to detect explosive atmosphere risk.

For air swept tanks, there are other options for potentially detecting process conditions that are correlated with likely elevated H_2S and/or explosive atmosphere risks. The following summarizes a few of these options, the possible types of instrumentation that could be used, challenges, and the known use in similar industrial applications:

- Sweep Air Flow Measurement (at air inlets): This approach monitors incoming sweep air flow at each air inlet and alarms/interlocks when air rate drops below level sufficient to maintain 35% of LEL of H_2S or below per NFPA-655 recommendations.
    - Detection of LEL Risk: Directly measures a single contributing process variable (sweep air), but not the risk (H_2S level) directly. Allows for total sweep air monitoring and air flow at each air intake to facilitate troubleshooting.
    - Challenges: Does not detect other changes that may initiate high headspace H_2S (e.g., higher than normal H_2S entering tank/evolving). In the event of reverse flow, meters are at risk of potentially accumulating sulfur vapor and may become unreliable. In addition, some types of flow meters may have issues with the small pressure difference available for measuring flow (see Table 2).
    - Instruments: See Table 2.
    - Used in Molten Sulfur Applications: Yes, known examples for pits and sulfur tanks. Not known how common.
- Sweep Air Flow Measurement (at outlet/vapor recovery equipment suction): This approach monitors incoming sweep air flow/vent vapor flow in the vent line from the tank (e.g., at a suction of a motive device for the sweep air such as an eductor, if present) and alarms/interlocks when air rate drops below level sufficient to maintain 35% of LEL of H_2S or below per NFPA-655 recommendations. Detection of LEL Risk: Directly measures a single contributing process variable (sweep air), but not the risk (H_2S level) directly.
    - Challenges: Does not detect other changes that may initiate high headspace H_2S (e.g., higher than normal H_2S entering tank/evolving). The meters are exposed to sulfur vapor and corrosive components continuously and are at risk of potentially accumulating sulfur vapor and may become unreliable.
    - Instruments: See Table 2.
    - Used in Molten Sulfur Applications: Yes, known examples for sulfur pits. Not known how common.
- Other Approaches:
    - The vapor space pressure of tanks and sulfur pits is often monitored. In air swept systems, the headspace pressure should be at a slight vacuum. Monitoring for positive pressure could indicate risk for reverse flow from tank and loss of air sweep.
        - This approach is an indirect measure of air flow - loss of suction pressure/positive pressure indicates a risk of dangerous headspace conditions, but does not quantify the risk.
        - Pressure measurement at a single location may not be indicative of a conditions everywhere in the vent system. A pressure measurement at the

outlet of the system may indicate negative pressure, but at the air inlets, positive pressure may be possible. Even for pressure measurements on the tank, there are risks of misleading measurements. For example, a crack or open gauge hatch could introduce air that "short-circuits" the flow from inlets. The tank pressure measurement may still indicate negative pressure/vacuum, but at specific air intakes, little or no air may be entering.

    - The normal operating vacuum condition of the tank/pit is typically very small, so detection of real changes vs. false alarms/measurement variability may present a challenge.
    - Used in Molten Sulfur Applications: Yes, known examples for sulfur pits..

- If a motive device such as an eductor is present to provide the motive force for sweep air flow, there may be other indirect process measurements that can provide an indication of sweep air availability, particularly for cases of complete loss of sweep air (i.e., situations that require urgent response).
    - Loss of steam flow to an eductor would indicate loss of sweep air flow unless a backup motive force is available (e.g., natural draft).
    - Increases in the suction pressure at the motive device for the sweep air may indicate plugging or other similar issues that will ultimately lead to reduction or loss of sweep air flow. This approach may have many of the same limitations as monitoring the vapor space pressure (described immediately above).
    - Used in Molten Sulfur Applications: Yes, known examples for sulfur pits, often as a combination of approaches (e.g., steam flow and suction pressure for eductor). Appears to be common in sulfur pit applications.

These secondary measurement approaches may be useful for detecting major failure scenarios but are not likely to be reliable indicators of the sweep air flow rate itself and therefore are difficult to relate to LEL risk in the headspace in any meaningful way.

Table 2: Instrumentation for Monitoring Sweep Air Flow/Vent Flow.

Instrument	Notes
Thermal mass flowmeter (constant current/flux and constant temperature available)	Cannot be exposed to liquids and maintain reliable measurement. Accuracy for individual flow rates at each inlet may not be reliable enough for an alarm/interlock (can be used for monitoring), but aggregate flow across all meters may be acceptable for alarm/interlock.
Specialized differential pressure meter (e.g., venturi, V-Cone Flow meter)	Pressure difference is too small for orifice-type flow meter. Venturis and V-Cone have been mentioned for sulfur tank air flow measurement by operators, but not clear how common they are. Note that limited length of straight pipe for air intakes may impact feasibility of venturi flow meters. V-Cone documentation indicates ability to install in proximity to flow disturbances (e.g., entrance, gooseneck, etc.)
Ultrasonic flowmeter	Known to be used on air inlets for sulfur tank/pit application, not clear if they would be suitable or robust in the vent vapor measurement application. Accuracy may not always be reliable enough for alarm/interlock (can be used for monitoring). Possibility of checking flow regularly with hand-held anemometer at air inlets.
Venturi flowmeter (with close-coupled remote diaphragm pressure-sensing elements)	Known to be used in sweep air motive device suction lines. Meters should be steam jacketed. May require regular inspection and cleaning of sensing elements.

5.0 Temperature Measurement

Measuring the temperature of molten sulfur is important to ensure that the operating temperature is in the normal range as discussed previously. Sulfur pit and tank vapor space temperature measurements are also essential as an indication of the potential to freeze sulfur, or of sulfur fires.

Temperature devices used for molten sulfur service include thermocouples and resistance temperature detectors (RTD). For example, Pt100 has been used in this application. RTDs are more accurate and stable than thermocouples. They are often used where more precise control of sulfur temperature is needed. RTDs are typically more expensive than thermocouples. Thermocouples and RTDs are often installed inside thermowells made of corrosion resistant material to protect the sensors from direct contact with molten sulfur. Dial thermometers are sometimes used for local, visual backup to electric sensors.

Skin or surface-mounted sensors are sometimes also used on the tank shell. They provide an indirect measurement of the temperature of the tank contents. They may be used if direct insertion is difficult, or as a secondary temperature indication to other sensors.

Multiple temperature sensors are often used at different elevations in the liquid and roof locations in the vapor space of molten sulfur storage tanks to provide an indication of temperature stratification in large tanks.

5.1 Fire Detection

Temperature measurements also need to be located at multiple points in the vapor space to provide a greater chance of local fire detection. Literature showing vapor space temperature measurement suggests that temperature increase rates in the range of ~2°F/min to ~5°F/min have been experienced during a fire [18]. It has also been reported in the literature [18] that SO_2 measurements in an SRU thermal oxidizer stack have been used as an indication of sulfur pit fires, for units that route vent streams to the thermal oxidizer.

6.0 Pressure Measurement

Pressure measurement typically occurs in the sulfur pit and tank vapor space above the liquid level to detect abnormal conditions and control vapor handling systems. Pressure measurements may also be made in the molten sulfur downstream of transfer pumps. Pressure transmitters and dial gauges can be used with a remote seal to protect against molten sulfur, sulfur vapor condensing and plugging and corrosion.

7.0 Molten Sulfur Flow Measurement and Regulation/Control

Flow measurement of molten sulfur is not always utilized in SRUs and associated handling applications and therefore is not a focus of this paper. However, specific areas of the facility (degassing units, sulfur loading systems) may utilize flow measurement and control, so a brief overview of flow measurement and control instruments in molten sulfur is provided in this section.

7.1.1 Flow Measurement

The following provides examples of meters that have been reported to be used in molten sulfur service:

- Coriolis meters: These meters have been commonly used in molten sulfur service and can represent a reliable measurement approach, especially considering high temperatures and properties of molten sulfur. The meters will provide temperature and density alongside flow, which is particularly useful for accurate estimates of mass flow, for example.
    - Straight-tube Coriolis meter designs are preferred in molten sulfur applications to avoid forming pockets in the sulfur flow path; bent-tube design are susceptible to the introduction of a pocket in the flow path and may require special installation consideration to prevent this risk, and therefore, are not preferred in molten sulfur applications.
    - Availability and cost-effectiveness of Corolis meters in large diameter piping applications may limit their use in those specific cases.
- Wedge meters: Wedge meters are offered for molten sulfur service and have been used in similar challenging liquid service (e.g., sulfur slurry service in liquid redox plants). The meters can be used in high-temperature and fouling prone applications. The wedge design

also avoids the flow damming that can occur with orifice-type meters (see the following section for concerns with orifice plate design in sulfur service).

  - Designs for molten sulfur applications should include heating/steam-jacketing to prevent sulfur solidification and may include remote seal options to increase the robustness of the design for the molten sulfur service.

- Ultrasonic meters: Ultrasonic flowmeters have been reported to be used in molten sulfur flow metering applications (e.g., [19]). Ultrasonic meters can be non-intrusive and/or avoid contact with the process fluid altogether (i.e., clamp-on).
  - Specific designs for molten sulfur that have appropriate temperature tolerant components and jacketing/heating are available.

Other types of metering devices may have been used in molten sulfur service (e.g., orifice meters), but are not generally well-suited to accurate, robust measurement when considering the properties of sulfur and the best-known practices in molten sulfur system design.

7.1.2 Flow Regulation Control

Flow control devices are not the focus of this paper; however, many of same principles and concepts discussed throughout this paper apply when specifying flow control devices such as valves and orifice plates in molten sulfur service. Specifically, proper heating (e.g., jacketed valves) and utilizing designs that are robust to the risks of plugging and corrosion are critical. The flow control device itself may create an unintended consequence in the system design intent as well. For example, piping in molten sulfur service should generally be free-draining and pockets should be avoided to prevent the accumulation of sulfur in piping that may ultimately solidify and plug the piping. However, when an orifice plate is used in a molten sulfur piping system, the orifice plate may create a dam that allows sulfur to accumulate. Using a segmented or eccentric orifice with proper installation in the line can prevent this effect and allow sulfur to free drain.

8.0 Conclusions

Given the unique properties and hazards associated with molten sulfur, the storage and handling systems from Claus SRUs must be designed to monitor and control important operating parameters. This paper provides an overview of the important process measurements in different areas of the molten sulfur handling equipment. Specific types of measurement devices used in molten sulfur service are presented with guidance for maintenance and reliability given the corrosive service and potential for sulfur plugging. The molten sulfur tank is a central focus because of the potential to form an explosive atmosphere in the vapor space due to high H_2S concentration. Sweep air and inert gas blanketing are approaches used to mitigate this risk. Methods to directly measure the H_2S in the headspace are presented including the different types and suitability of analyzers for the service, location of the H_2S probe, and special installation requirements. Other indirect methods to measure the lower explosive limit in air swept tanks include air flow measurements and vapor space pressure monitoring. Many of the same types of measurements taken for molten sulfur tanks would apply to sulfur pits as well.

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