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Molten sulphur storage design practices to manage and mitigate environmental, health, and safety risks of sulphur storage and system vapours

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This paper discusses best practices around molten sulphur storage and handling. The article covers scrubbers (e.g., caustic scrubbers) for controlling H_2S and particulate in vent streams from molten sulphur tanks and other operations. Practices for maintaining the head space of molten sulphur tanks in a safe zone relative to explosion limits are also discussed. Instrumentation for detecting conditions in molten sulphur tank head spaces and other molten-sulphur-handling equipment is reviewed. Practices for extinguishing molten sulphur fires in tanks will be touched upon. The paper will also discuss the effect of degassing molten sulphur on each of these best practices. The paper will also review some recent fires and/or explosions in molten-sulphur-handling equipment for which data is publicly available.

1. INTRODUCTION

Hydrogen sulphide (H_2S) associated with molten sulphur from a modified Claus sulphur recovery unit (SRU) unit may present environmental, health, and safety risks. The molten sulphur from the SRU may contain between 300 ppmw [1], [2], [3] to 450 ppmw of H_2S and H_2S_x [4], depending on the specific design and operation of the SRU (e.g., oxygen enrichment). As this molten sulphur leaves the SRU and enters the storage, handling, and transport areas of the facility, the composition of the molten sulphur is not constant or fixed at the composition leaving the SRU. Instead, the conditions of these downstream processes, including the temperature, extent of agitation and mixing, exposure to air, and residence time (among other conditions) impact the composition of the molten sulphur at each step of the process and over time. Figure 1 presents a simplified representation of the chemistry, vapour-liquid equilibrium, and mass transfer processes occurring with the molten sulphur in this downstream equipment.

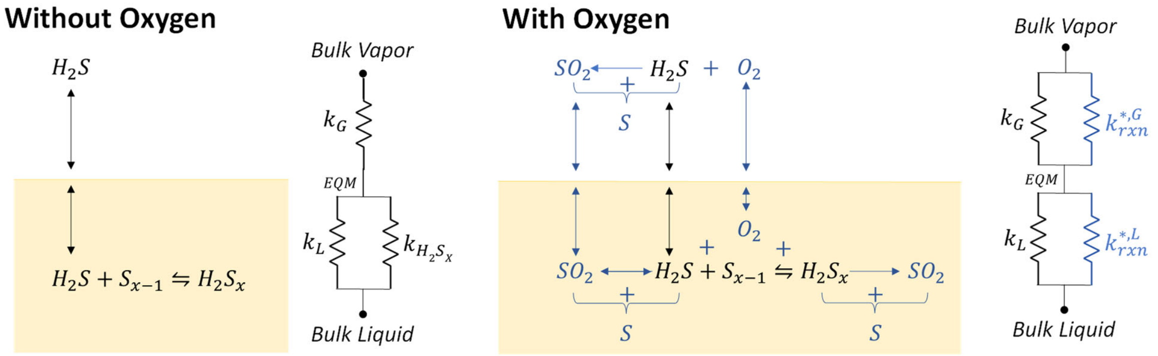

Fig. 1: **H_2S and SO_2 transport in sulphur handling systems with and without oxygen. Originally published in [5].**

As the figure indicates, the H_2S itself can take more than one form in the sulphur as free H_2S or hydrogen polysulfide (H_2S_x), with the speciation depending on temperature and the solubility of free H_2S in sulphur. Free H_2S is able to move between the molten sulphur and vapour phase based on equilibrium conditions, with the rate dictated by the transport conditions between the sulphur and vapour (e.g., agitation of sulphur, area of the vapour-liquid interface). When oxygen is present in the system (e.g., when the sulphur is exposed to air), SO_2 can form in multiple ways and can move between the sulphur and vapour phases as well. The chemistry and transport process are more complex than depicted in Figure 1; for example, molten sulphur itself can exist as a vapour and sulphur vapour will be present in the vapour space of equipment – this creates additional challenges such as freezing, plugging, and corrosion of equipment, which can create or exacerbate hazards of the molten sulphur handling. Regardless of the complexity in the real system, the implication for sulphur handling is the same – the presence of H_2S and SO_2 and the ability for these species to enter the vapour space, and often surrounding environment, represent a key source of hazards for the storage, handling, and transport systems. The design and operating conditions in the sulphur handling equipment have a strong impact on the behaviour and fate of H_2S and SO_2 and will be a core focus of this paper.

The specific approach to handling, storage, and transport of the sulphur leaving the SRU will vary based on the specific site design and requirements. Figure 2 presents a generic, but common, sulphur handling sequence of operations downstream of the SRU.

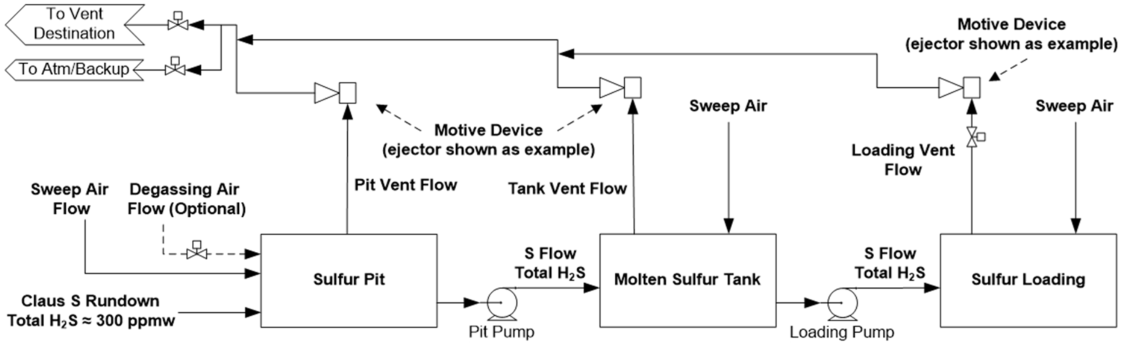

Fig. 2: **Example Molten Sulphur Handling System. Numbered points denote potential H_2S measurement points in molten sulphur or associated vapour. Originally published in [6].**

The molten sulphur leaving the SRU enters a short-term storage pit or collection header/vessel (the term sulphur pit will be used generally in this work), often located below grade to allow gravity draining of sulphur from the SRU. The sulphur temperature may be maintained in the pit (nominally around 280 - 300°F) with steam coils and the vapour space of the pit may be swept with air to mitigate accumulation of H_2S. The sulphur pit represents the first place in the handling system where molten sulphur will have extended residence time with an enclosed vapour space, will potentially interact with air, and represents an emissions point to the atmosphere. H_2S and SO_2 will evolve in the sulphur pit.

Some facilities will have a molten sulphur degassing unit integrated within or downstream of the sulphur pit; there are a wide range of commercial molten sulphur degassing technologies with different approaches to degassing the sulphur (i.e., actively removing H_2S from the sulphur). These technologies will be discussed briefly in this paper with a focus on the impact of sulphur degassing on the overall hazards and design requirements for the sulphur handling system.

The molten sulphur (degassed or not) will then often be transferred to an above-ground storage tank to provide longer-term interim storage of sulphur; the considerations and operation of the sulphur tank will be similar to the sulphur pit. The sulphur temperature (nominally around 260 - 300°F) will be maintained (either by internal steam coils or external engineered heat tracing), the vapour space will be managed for the accumulation of H_2S (via sweep air or an inert blanket gas), and the tank will represent a potential location for the accumulation or emission of H_2S and/or SO_2.

Finally, the molten sulphur will be pumped from the sulphur tank to a transport vessel (e.g., truck, rail, barge) for sulphur offtake from the site. This final sulphur loading operation has unique challenges and considerations associated with the transfer of the sulphur from a process vessel to a transport vessel (rapid transfer of sulphur, non-uniform transport vessels/loading equipment, direct personnel interaction with operations). As with other areas in the system, H_2S and SO_2 will evolve, can accumulate in vapour spaces, and may be emitted to the atmosphere with associated environmental and personnel risks.

Throughout the operations described above, the molten sulphur conditions must be managed to i) mitigate and limit emissions, ii) minimize operator exposure to toxic H_2S and SO_2, iii) and prevent fire / explosion hazards. This paper provides an overview of these hazards and outlines operating and design best practices and options for hazard prevention and mitigation.

2. HAZARDS OF MOLTEN SULPHUR HANDLING

2.1 Health and personnel exposure

As discussed in the preceding section, exposure to vapours containing H_2S and/or SO_2 are possible throughout the molten sulphur handling process; these vapours may be emitted from equipment as a normal part of operations (i.e., equipment designed to vent to atmosphere). Therefore, it is important to understand the specific risks to personnel around these operations. Table 1 summarizes exposure limits/hazards for H_2S and SO_2. The table illustrates the risks associated with H_2S and SO_2, and the exposure limit levels are well-within ranges that can occur in the sulphur handling system and may exist within the equipment or near the equipment during normal operations. Therefore, it is essential to consider personnel exposure risks in the design and operation of the molten sulphur handling facility.

General prevention and mitigation strategies may include the use of personal H_2S detectors for any personnel in the area, area H_2S monitors mounted at various strategic locations throughout the sulphur handling facility (e.g., located at grade/personnel access levels near expected emissions points, placed based on dispersion modelling), and dispersion studies to aid in the design of emissions points (e.g. vent stacks) to limit the risk of personnel exposure. Ideally, these general strategies to protect personnel should mirror the same approaches used in the SRU upstream where personnel exposure is also a known risk. More specific prevention and mitigation strategies, such as vapour recovery from emissions sources, are discussed later in this paper.

Table 1
Exposure limits of H_2S and SO_2

Parameter	H_2S, ppmv [7]	SO_2, ppmv [8]
Threshold Limit Value, Short-Term Exposure Limit (TLV-STEL) per ACGIH^Note 1	5	**0.25**
OSHA Permissible Exposure Limit (PEL), 8-hr Time Weighted Average (TWA)	10 for construction and maritime industries 20 ppm ceiling limit for general industry	**5**
Immediately Dangerous to Life and Health (IDLH)	100 (loss of smell > 100)	**100**
Other Relevant Exposure Levels	700 – 1000 = Rapid unconsciousness, “knockdown” >1000 = Nearly instant death	

1: ACGIH = American Conference of Governmental Industrial Hygienists

2.2 Fire and explosion hazards

H_2S can concentrate in the vapour space of storage and transport equipment to dangerous levels. H_2S is flammable and can present an explosion risk when present in concentrations within the explosive limits for H_2S with oxygen and an ignition source present. The following sections review the conditions that can lead to fires and explosions in molten sulphur handling systems. Mitigation and prevention associated with the design of the molten sulphur handling system will generally be discussed in a later section, but where general or straightforward mitigation approaches are known, they will be discussed with the hazards in this section.

2.2.1 H_2S flammability

In sulphur handling equipment, hydrogen sulphide (H_2S) will be present as a dissolved gas within the liquid sulphur phase. Over time, it will evolve to the vapour phase in significant quantities. H_2S is a flammable component with its flammability typically denoted by upper and lower explosive limits (UEL and LEL). However, only the LEL is of practical concern in sulphur handling equipment since the concentrations approaching the UEL are not expected based on equilibrium concentrations of H_2S in the vapour. Figure 3 depicts the LEL of H_2S as a function of temperature.

As shown in the figure, increasing temperature corresponds to a larger explosive limit window for H_2S, so temperature is a key parameter quantifying the risk of fires and explosions in molten sulphur systems. It is important to note that literature on this topic is limited, and recent data has updated the understanding of LEL limits of H_2S. For example, at 330°F, the newer literature data indicates the LEL of H_2S to be ~3 vol% H_2S [9]. It is a common industry practice to use a temperature (330°F) that is above the normal operating conditions of the equipment (e.g., sulphur storage at 280 - 300°F) when estimating the LEL of H_2S to incorporate a margin of safety directly into the estimate [2]. Therefore, operation should not risk being too close to the LEL in Figure 3. Methods for maintaining a safe margin from the LEL are discussed later in this paper.

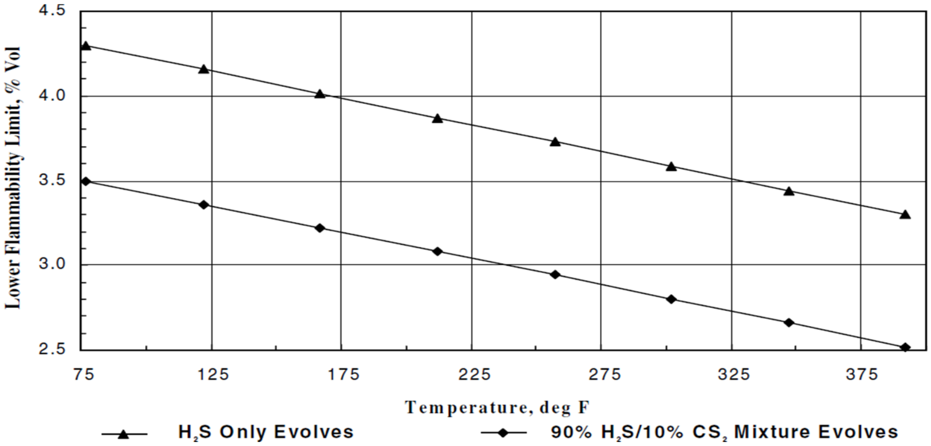

Fig. 3: Lower Flammability Limit of H_2S [2]

2.2.2 Ignition sources

For molten sulphur below its auto-ignition temperature (~450°F) but within its flammability window, fires require an ignition source. One known source is from static discharge accumulated by free-falling sulphur, which can occur because molten sulphur is an electrical insulator and can accumulate static charge when falling through air [2, 10]. Incidents have been reported where static buildup was identified as the source of a sulphur fire or explosion [11]. This is a particular concern in molten sulphur loading operations where sulphur is entering an empty vessel and the loading equipment configuration may make it difficult to feed sulphur consistently below or near the liquid interface level. Minimizing the distance for free-falling sulphur (or ideally eliminating by feeding the sulphur below the liquid level e.g., with a dip pipe in molten sulphur tanks or an extended drop pipe in loading operations) and proper grounding of all equipment [12] associated with the sulphur transfer operations are important, proven mitigation measures.

Excessively high temperatures due to localized hot surfaces in process equipment (e.g., failing pump bearings) or due to improper design or operating temperatures (e.g., using saturated steam above the flash point of sulphur) can also provide the energy required for ignition. In many incidents, the exact ignition source was not determined. For safety, it should be assumed that an ignition source could always be present.

2.2.3 Pyrophoric iron sulphide formation

When solid sulphur solidifies or condenses, it can allow moisture (from steam leaks or other sources) to condense and accumulate between the sulphur and carbon steel surface of molten sulphur handling equipment. The condensed water then causes corrosion and promotes the formation of pyrophoric iron sulphide in anaerobic / oxygen-deprived conditions. This is a particular safety risk in tanks that are purged or blanketed with inert gas (e.g., nitrogen, discussed later) when the pyrophoric iron sulphide is subsequently exposed to air (oxygen) during maintenance or cleaning. In molten sulphur tanks that use sweep air, the iron sulphide tends to oxidize quickly and in a controlled manner so that it cannot build up to the levels where pyrophoric reactions can take place [13]. However, even in sweep air systems, if enough solid sulphur deposits, pyrophoric iron sulphide can form beneath the solid sulphur and later ignite if fragments break loose and become exposed to oxygen in the sweep air [14], [15]. Thus, it is important to i) maintain the tank vapour space at the appropriate temperature to prevent the freezing of sulphur and ii) eliminate sources of water ingress such as by using external steam heat tracing instead of internal tank steam coils.

2.3 Emissions considerations

Beyond personnel exposure risks, many sites may face limitations on environmental emissions of H_2S and SO_2; the quantitative limits will be specific to each facility. In general, however, molten sulphur pits, tanks, and loading operations will all contribute to air emissions of H_2S and SO_2; even vapour recovery (discussed later in this paper) may result in elevated emissions if the vapour from the molten sulphur handling equipment is ultimately recycled to SRU and some part of the pollutants are emitted from the back-end of the SRU (e.g., via incinerator). Emissions mitigation approaches are discussed further in the vapour disposition section of this paper.

2.4 Role of temperature

The introductory discussion in this paper alluded to the importance of temperature on the safe and reliable handling of molten sulphur. The temperature of the sulphur directly impacts how much H_2S and SO_2 can be present at different steps in the molten sulphur handling system, how much will accumulate in specific places in the system, and how much enters the atmosphere as emissions. For example, lower temperatures favour the equilibrium conversion of H_2S_x to H_2S; the free H_2S can move from the molten sulphur phase to the vapour. This can both represent a hazard (e.g., higher emissions/accumulation of H_2S) and a design opportunity (degassing of H_2S as discussed later).

However, temperature also impacts other properties of molten sulphur which in turn may create or worsen hazards in the system. Figure 4 highlights several of these key temperature transition points for properties of pure sulphur. An operating window (260-300°F), as depicted in the figure, can be defined based on the unique properties of pure sulphur (freezing, viscous transition, flash point, and autoignition concerns) and leaving adequate margins between the operating window and temperature limits. Some of the hazards of the property transitions are clear (e.g., flash point and fires); others, like freezing of sulphur create multiple hazards indirectly – for example, sulphur expands upon heating and may rupture piping when re-melted.. Solid sulphur may also prevent air/vapour flow as designed leading to dangerous accumulations of H_2S in equipment, or initiate the formation of pyrophoric iron sulphide. Similarly, the viscous transition of sulphur (which occurs over a very narrow temperature range and increases the viscosity of sulphur by several orders of magnitude) creates operational challenges (i.e., difficulty to pump/transfer sulphur, which in turn may create other hazards (e.g., viscous heating).

Further, NFPA-655 cites 309°F as a transition temperature for the design of molten sulphur storage equipment. Field experience reported in the literature [10] and Trimeric's knowledge of operator experience supports the implication that temperatures above 309°F, which is below the sulphur vapour flash point, are nonetheless associated with increased frequency of sulphur fires [6].

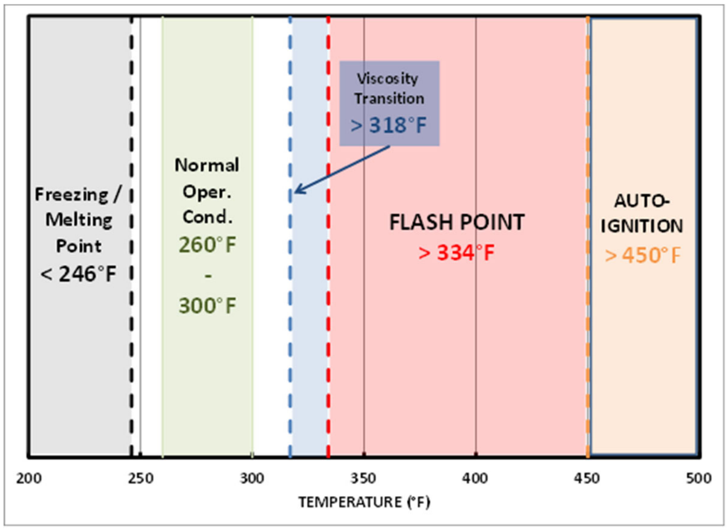

Fig. 4: Pure Sulphur Properties [12]

Maintaining the temperature of sulphur will not be discussed in detail in subsequent sections of this paper but it represents a core operating and design principle to minimize risks. A few select best practices and operational considerations for temperature maintenance are outlined below:

- Heating with jacketed pipe or piping with engineered bolt-on heat tracing and insulation is best practice for safe and proper operation of a molten sulphur handling system. For critical process lines or equipment (e.g., vent line from tank or pit), jacketing is recommended. It is also important to heat trace and insulate relief devices to avoid rendering these devices inoperable.

- The tank must also be properly heat traced and insulated to avoid the formation of solid sulphur on the tank inner walls and roof, which could lead to fires in the tank due to pyrophoric iron sulphide formation.
- Equipment, instrumentation, and steam traps serving the heating systems in a molten sulphur handling system should be periodically inspected for proper heat tracing and insulation or function.
- To prevent sulphur solidification, a minimum steam pressure for steam jacketing and tracing should be 35-40 psig (50 psig steam is common in SRUs and is a good source for heating requirements). However, the steam should not be higher than 80 psig since this temperature is approximately the flash point for sulphur (334°F). Several incidents have been reported where use of high-pressure steam was believed to have started a sulphur fire [12].
- Where steam coils are used, the sulphur level in pits and tanks should be maintained above the coils in these vessels. When heating coils are exposed to air, ignition can occur from the pyrophoric iron sulphide that can form in this area.

2.4 Industry experience with molten sulphur hazards

Different hazards exist when handling molten sulphur. Fires and/or explosions can occur due to the unique characteristics of molten sulfur and the evolution of H_2S into the vapour space of pits, tanks, and loading vessels. In some cases, the sweep air rate may be insufficient to maintain the H_2S below the LEL, and an ignition source subsequently results in a fire or explosion. Plant personnel can also be exposed to hazardous concentrations of H_2S or SO_2, and the high temperature of the molten sulphur can burn operators and damage equipment. Several examples of hazardous situations in the industry are presented in the table below. These examples highlight the importance of regularly monitoring the molten sulphur handling equipment to ensure safe operating conditions and to prevent operator exposure to toxic gas.

Table 2
Industry examples of molten sulphur hazards

Event	Description
Fires / Explosions	
1	Low air flow to the sulphur tank (indicated by alarm) caused the H_2S vapour concentration to exceed the LEL. Pyrophoric iron sulphide may have ignited a fire, resulting in an explosion that ruptured the roof joint and caused flames to protrude from the tank [16].
2	Sulphur buildup plugged the tank vacuum breakers, preventing proper sweep gas flow which caused H_2S concentrations to rise above the LEL. The grounding system for truck loading malfunctioned, and a spark ignited a flashback through the vent line to the tank [16].
3	H_2S vapour accumulated in the calibration chamber of a level instrument. When an operator opened the inspection hatch, pyrophoric iron sulphide was exposed to air and ignited, triggering a fire [16].
4	A sulphur tank was ventilated with sweep air from a sulphur pit, but air flow was insufficient to keep the tank vapour H_2S below the LEL. The ignition source was likely pyrophoric iron sulphide or inadequate grounding (truck loading) [16].
5	A sulphur tank with internal heating coils was filled with nozzles that did not extend into the tanks. H_2S was released from the molten sulphur during agitation upon filling, resulting in an explosive mixture with air in a confined space. The source of ignition was thought to be from i) an electric spark from the static electricity built up by the falling stream of sulphur, ii) the use of superheated steam in the heating coil above the flash point, or iii) pyrophoric iron sulphide [17].
6	A refinery experienced fires in their sulphur pit when routinely operating at bulk temperatures of 315-320F. To mitigate this risk, the facility implemented cooling measures to maintain the pit at 300F where the frequency of fires is reduced [10].
Personnel Exposure	
7	An employee was taking a sample from a molten sulphur tank truck and reached into the tank to collect a sample without wearing a respirator. The employee fell off a platform and was found unconscious. The H_2S levels were estimated after the incident to be on the order of 800 ppm [18].
8	During transfer of sulphur into a tank trailer, a minor flash occurred, and sulphur was ejected from the open dome of the trailer tank. Some of the sulphur hit operators that were 10 to 12 feet from the dome, resulting in burns that required medical attention [17].

3. INCORPORATING PREVENTION AND MITIGATION APPROACHES INTO MOLTEN SULPHUR HANDLING DESIGN AND OPERATIONS

The preceding section highlighted key hazards associated with molten sulphur handling. Select prevention or mitigation approaches were discussed alongside the hazards in some cases (e.g., mitigating static discharge risks). However, there are several design and/or operating approaches that can be used to mitigate the risks with molten sulphur handling that merit more detailed review and discussion. These approaches will be the focus of this section.

3.1 Molten sulphur degassing

Molten sulphur degassing is an industrially established practice with multiple commercially available technologies The principal behind sulphur degassing is to use an engineered system to remove H_2S in a controlled manner to yield a molten sulphur product for downstream handling and transport that has much lower levels of H_2S (typically < 10 ppmw $H_2S + H_2Sx$). Reducing the H_2S in molten sulphur reduces risks and hazards associated with handling molten sulphur in the downstream equipment and throughout the sulphur value chain. Degassed sulphur will have less H_2S present to evolve in downstream equipment and create associated hazards (Exposure, explosion risks) and the lower concentrations of H_2S in the molten sulphur will also generally reduce the rate at which H_2S evolves from the molten sulphur (i.e., less driving force for H_2S to evolve from molten sulphur into a vapour space).

The general process of degassing involves several distinct steps [19]:

- H_2S present in the molten sulphur as polysulfides (H_2S_x) must be converted to free H_2S by shifting the chemical equilibrium between the two forms of H_2S. The equilibrium can be shifted, or the rate of conversion can be enhanced in several ways:
  - Cooling the sulphur: Lower temperature favours dissociation of H_2Sx to H_2S
  - Introduction of oxygen into the molten sulphur: Oxygen enhances the rate of H_2Sx decomposition; facilitating oxygen transfer to the molten sulphur (e.g., via operating at elevated pressure) will in turn enable H_2Sx decomposition.
  - Use of chemical catalysts in molten sulphur: Catalysts enhance the rate of H_2Sx decomposition
- Free H_2S must be stripped from the molten sulphur into the gas phase, and the gas phase must be continuously swept or removed to maintain low concentrations of H_2S in the vapour (i.e., maintain driving force for H_2S transfer).
- The molten sulphur must experience good surface-to-bulk mixing to ensure that free H_2S continuously reaches the molten sulphur-vapour phase interface to be effectively stripped from the molten sulphur.

Commercial degassing technologies will include design features (e.g., sparging of air, elevated pressure operation, mixing enhancement, catalysts, gas-liquid contacting equipment) to incorporate some combination of the above mechanisms for degassing [20]. Degassing technologies may be incorporated in the molten sulphur pit itself or may be a standalone technology outside of storage equipment.

Table 3 presents vapour-liquid equilibrium of H_2S between molten sulphur and a stagnant/quiescent vapour space (i.e., H_2S accumulates in the vapour until equilibrium is reached).

Table 3:
Vapour-liquid equilibrium of H_2S in molten sulphur [20]

Total H_2S in Sulfur	Temperature	H_2S in Vapour Space	Notes
ppmw H_2S +H_2S_x	F	vol%	
300	300	29.7	> LEL for H_2S
300	280	40.5	> LEL for H_2S
150	300	14	> LEL for H_2S
150	280	20.4	> LEL for H_2S
50	300	4.3	> LEL for H_2S
50	280	6.9	> LEL for H_2S
10 (Degassed)	300	0.7	Degassed Sulfur. Lethal H_2S levels.
10 (Degassed)	280	1.4	Degassed Sulfur, > 25% of LEL of H_2S

Degassing molten sulphur moves the H_2S concentration in the molten sulphur outside of the range where H_2S can accumulate to concentrations above the LEL of H_2S; however, H_2S is still present at lethal levels and may be above designated safe margins below the LEL concertation (e.g., above 25% of LEL of H_2S). Therefore, while degassing has notably reduced risks associated with H_2S in molten sulphur, it has not eliminated hazards. Furthermore, degassing technologies (particularly ones introducing oxygen/air) may generate additional $SO_2$with associated hazards and potential emissions. Finally, the degassing system will generate an offgas stream with potentially significant amounts of H_2S and SO_2that must be managed to mitigate the hazards of these streams.

3.2 Prevention and suppression of fires and explosions

3.2.1 Industry standards

NFPA-655 [21] provides industry standards and guidance for the prevention of sulphur fires and explosions in a “normal” handling temperature range (246°F-309°F) and at temperatures above 309°F where the frequency of fires tends to increase. NFPA 68 is relevant for deflagration venting to be considered at high temperature operation (>309°F), and NFPA 69 contains information on preventing and managing explosions/deflagrations. Details of these standards are provided in the source documents and summarized in the literature [12]. Other standards and industry documentation may also be relevant (e.g., API standards for tank design) and should be considered on a case-by-case basis.

3.2.2 Managing the vapour space of molten sulphur storage equipment

Two distinct approaches have been used in industry to attempt to prevent the formation of an explosive atmosphere in molten sulphur storage equipment (i.e., pits, tanks, vessels):

1. Sweep gas to limit H_2S concentration: A sweep gas (often air) is purposely introduced to the headspace of storage equipment to ensure that the H_2S concentration of the vapour space does not exceed the LEL of H_2S (and typically stays below a safe margin of LEL, e.g., 25% of LEL, some use 15-35% [22], [21]). Different methods for introducing sweep gas exist, including pressurized gas, sweep air flow induced by motive device (e.g., ejector, blower), and sweep air induced by natural draft [23]. See additional sources for more details of sweep gas tank design [24, 6, 23].
2. Purge or blanket gas to exclude air/limit oxygen concentration: The vapour space of storage equipment is maintained as an inert atmosphere (often nitrogen) at positive pressure to prevent air ingress and ensure that oxygen concentrations stay below thresholds [25] to form an explosive mixture with H_2S. H_2S in the vapour will generally be present at or near equilibrium concentrations with the molten sulphur, which would far exceed LEL concentrations for undegassed sulphur (see Table 3). The positive pressure is maintained in the tank by feeding inert gas (e.g., when drawing down the tank level) or venting (e.g., when filling the tank) as required by tank operation. There is no continuous sweeping or venting of the tank when a blanket or purge is used.

Figure 5 provides an example depiction of the two approaches to managing the atmosphere of a molten sulphur tank.

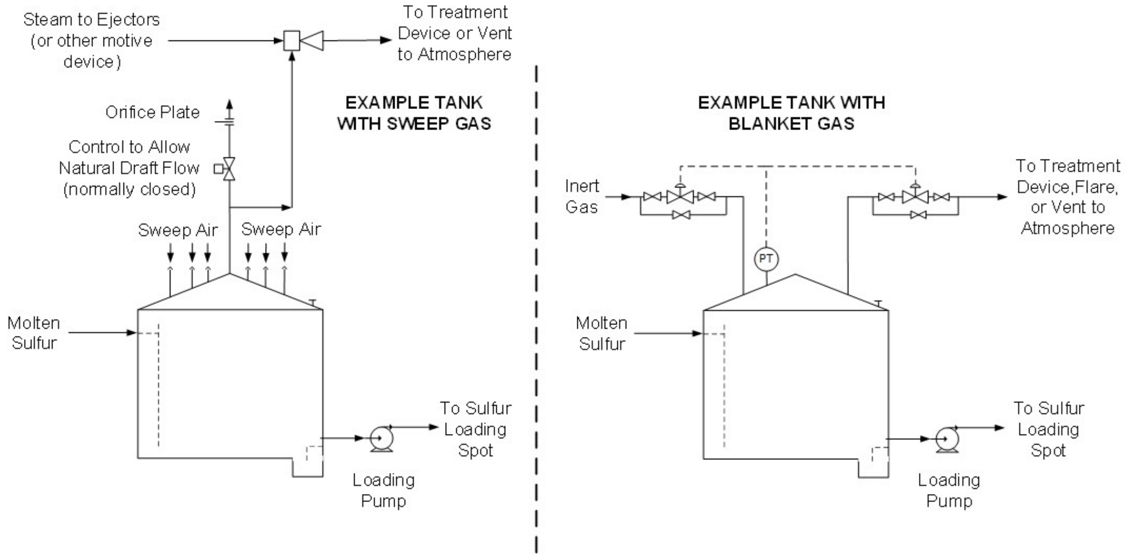

Fig. 5: Molten sulphur tank configurations with sweep air and with inert gas blanket [12]

The use of sweep gas or blanket gas has specific trade-offs that must be considered – Table 4 provides a summary of some of the key considerations. Both approaches are used in industry and can be designed to operate safely, but sweep air appears to currently be viewed as best practice in some industries (e.g., petroleum refining).

Table 4
Comparing sweep air vs. inert gas blanket

	Air (note 1)	**Inert gas (e.g., nitrogen)**
Tank Use	Sweep	Blanket
Flammability	Introduces oxygen, creates risk for ignition.	No air present during normal operation.
Explosion Risk	Maintaining adequate sweep air (H_2S < 25% of LEL) mitigates risk. Loss of sweep air (e.g., via plugging of intakes with sulphur) results in explosive environment.	Pyrophoric iron sulfide formation in reducing environment - increases risk if air enters vessel (Note 2). Unexpected air ingress could result in explosive atmosphere.
Tank Pressure	Operates at slight vacuum.	Operates under slight positive pressure.
Monitoring	Measure H_2S (directly measure hazard), monitor sweep air flow, monitor tank pressure. See [15] for additional details.	Monitoring tank positive pressure is a reliable indicator of functionality. O2 concentration could be monitored, but not commonly done.
Vent Gas	Continuous vent stream is produced that must be safely managed. May include elevated SO_2levels due to air sweep.	Intermittent venting only, limits emissions and amount of gas that must be handled/managed, gas may have high concentration of H_2S.
Cost	Readily available. Some cost may be required for heating.	Tie into existing system or add new source of inert gas. Usually higher cost option.

Notes:

1. *Alternative sweep gases such as N_2, fuel gas, or steam are not as common due to their expense, risk of pyrophoric iron sulfide formation, or introduction of combustibles to the tank.*
2. *An alternative blanketing technique using gas with oxygen below the limiting oxygen concentration (LOC) is also presented in the literature (15; 14; 18; 19; 20). This approach can help mitigate the pyrophoric iron sulfide formation risk.*

Note that there are known examples in industry of facilities that have used an approach that is effectively a hybrid of the air sweep vs. inter gas blanketing in the preceding table; these facilities use a low-oxygen vapour stream (e.g., 2 vol% O2) for the tank sweep or blanket media. This approach ensures that the oxygen content stays below the minimum oxygen concentration to form an explosive mixture with H_2S but still provides sufficient oxygen to readily oxidize and iron sulphide that may form. This approach is contingent on have the appropriate supply of gas that meets the oxygen content requirements.

3.2.3 Fire suppression and explosion protection

Fire suppression and explosion protection measures are used to limit the consequences when a hazardous condition is not prevented by the means described in the preceding discussion in molten sulphur handling equipment. The topic is covered in relevant industry standards (e.g., NFPA-655,-68, -69) and in industry literature [12], [26]. This paper will briefly describe a few key aspects of fire suppression and explosion protection, but the reader is encouraged to review standards and industry best practices in detail to determine appropriate implementation for specific scenarios.

3.2.3.1 Snuffing and sealing steam

NFPA-655 makes a distinction between snuffing and sealing steam. Snuffing steam is used to directly extinguish a fire by displacing air at the fuel-fire interface with steam, removing the oxygen needed for combustion. Sealing steam is used to effectively “seal” the tank/pit by continuously introducing steam to the headspace and generating a positive pressure in the vessel. This prevents additional air ingress, extinguishing the fire after any oxygen in the headspace is consumed.

The addition of “sealing” (as opposed to “snuffing”) steam in NFPA-655 was based on the overpressure risk for typical air-swept tank and pit designs subject to the snuffing steam requirement [26]. Analyses indicated that the snuffing steam requirement of 2.5 lb/min/100 ft3 of tank volume was often impractical to vent from air swept tanks and pits when balancing overpressure risk from the steam (large air inlets required to vent steam) against normal air intake (smaller inlets to prevent reverse flow) [26]. A lower sealing steam rate (1 lb/min/100 ft3) was proposed based on industry feedback and steam flow evaluation via CFD. In Trimeric’s experience, some sites do not have enough steam to supply the 1lb/min/100 ft3 sealing step as recommended in NFPA-655 but are nonetheless able to successfully extinguish fires with lower steam rates.

Sealing and snuffing steam systems have several considerations outside of the fire suppression function/flow requirements. Key design considerations include:

- Location of the Steam Activation Valve: Industry practice is that the valve should be at least 50 ft from the tank and located in a place where the operator has a clear line of sight from the valve to the tank [26, 1].
- Verification of Dry Steam: The design should include provisions for blowdown of steam prior to activation to ensure only dry steam is present. Wet steam can create a tank rupture risk. The steam system should also include a drip leg and steam trap upstream of the valve to ensure condensate does not accumulate in the line and the line stays warm [26].
- Minimize Risk of Plugging: To prevent sulphur plugging of the steam line, the line may have rupture disks at the tank. Alternatively, the line can use a small purge gas flow to prevent back flow of sulphur vapour and/or be thoroughly steam jacketed or traced to prevent plugging. The sealing steam line should be verified periodically to ensure plugging has not occurred.

Some references also indicate that sealing steam should be introduced close to air inlet nozzles [1] so that the steam rapidly exits via the air inlets [26, 21]. In practice, if sufficient steam is introduced to generate positive pressure in the tank, the sealing effect of the steam should still be effective even if the vapour that initially leaves the inlets is headspace vapour as opposed to steam. However, limiting the rapid expulsion of the toxic headspace vapour to the atmosphere and quick visual verification that the steam has reached the tank are benefits of steam introduction near inlets.

Finally, the time for steam activation after fire detection is another important design consideration to prevent fire damage. The authors’ experience and some of the available literature [26, 27] suggest that the operators have activated steam within ~4 to ~10 minutes of detecting a fire. The data also suggest that the pits and tanks often suffered no known damage, sometimes despite multiple fires. However, the duration of steam applications to ensure complete extinguishing may vary greatly depending on many site-specific factors.

3.2.3.2 Other sealing methods

While NPA-655 has historically discussed sealing steam in the context of sealing methods, other methods discussed in the literature [12] include mechanical sealing and water mist spraying. However, both have their limitations and should be used with extreme caution. Rapidly sealing the tank by closing air intake and outlet vent control valves has been used but may result in high temperatures, overpressure, or vacuum conditions if the tank is not properly relieved. Explosion hatches may also be needed, and the molten sulphur should be allowed to cool below 309°F before reopening the vents. Direct contact with a solid stream of water is not recommended, but the use of a fine water mist spray has reportedly been used in the literature to control sulphur fires on merchant sulphur vessels [28] and in sulphur production and manufacturing industries [29]. NFPA-655 recognizes the use of a fine water spray to extinguish sulphur fires in an open container but not an enclosed tank. There are no known engineering standards for using this method to suppress molten sulfur fires.

3.2.3.2 Explosion protection

Explosion protection may take a variety of forms in practice; in general, the goal of explosion protection devices or design is to minimize damage to the structural integrity of the storage equipment in the event of an explosion, which in turn may minimize the secondary impacts to the facility and personnel in the event of an explosion (e.g., loss of containment, projectiles/impact from the failed equipment). However, there is no clear consensus in industry on the need for explosion protection for sulphur handling equipment or the specific design approach used for explosion protection and the cost and design of the vents can create impediments to implementation [30].

NFPA-68 specifically focuses on “deflagration venting” and is referenced by NFPA-655 in the context of molten sulphur storage systems. Deflagration venting devices are known to be used for sulphur pits and tanks, though they are not always present on sulphur storage systems; other approaches, such as frangible roofs in tanks or pressure vessels designed for pressure containment (referenced by NFPA-69) in lieu of concrete sulphur pits, may be used to mitigate consequences of an explosion in sulphur handling systems. Therefore, the use and design of deflagration venting is very specific to the application and storage system design.

When deflagration venting devices are used, they may vary in design, function, maintenance/reliability requirements, and industry terminology for the devices (e.g., explosion hatches, explosion doors, rupture disks). It is important to note that venting devices used for explosion protection/deflagration venting are not identical or interchangeable with other overpressure protection devices (e.g., relief valves). The purpose and design approaches are different as explosion protection devices must respond to the specific nature of deflagration events (e.g., the rate of pressure increase along with the extent of pressure increase). There are also specific considerations for the design and implementation of venting devices in sulphur service, including the fact that vents will release toxic, hazardous gas upon activation, vents may be prone to plugging/sticking due to molten sulphur accumulation (i.e., may require mitigation with heating, purge gas), and maintenance/inspection of vents may be challenging/hazardous. There are more detailed reviews of deflagration venting devices and design in molten sulphur service in literature e.g., [30].

3.3 Detection of hazardous conditions

The design of the molten sulphur pit or tank should include features to detect conditions that could lead to a fire or explosion or indicate that a fire may be occurring. Some of these same detection approaches may also serve as general hazard detection for exposure and environmental risks as well. Table 5 reviews select detection methods.

Table 5
Detection of hazardous condition in molten sulphur handling

Measurement	Hazard Detected	Instruments/Approaches	Other Considerations
H_2S or vapour concentration	Direct measurement of hazard (H_2S, SO_2) in vapour space of equipment. Rising/elevated SO_2 has been used as an indication of an active fire [26].	H_2S/SO_2 analysers commercially available (e.g., UV Vis Spectrophotometer). See [15] for additional instruments/details.	Cost, representative measurement in large vapour spaces, reliability in sulphur service (plugging, maintenance).
Field sampling of H_2S in molten sulphur	Indirect predictor of H_2S in vapour space, but critical for establishing basis for H_2S vapour space estimates.	Qualified/experienced sampling vendors required to perform sampling, provide analysers, interpret results.	Selecting appropriate measurement locations and ensuring facility operation is representative of design conditions (e.g., avoid transients/unusual off-design periods)
H_2S area monitors	Direct detection of dangerous H_2S levels in ambient space around equipment.	Instruments are widely used in industry. Located at grade for personnel exposure concerns, but can locate strategically (e.g., high risk areas from vents) for early detection of abnormal conditions.	
Temperature Indication	Indirect indication of fire (elevated temperatures).	Thermocouples or RTDs in storage equipment. Multiple strategic locations (e.g., in different parts of vapour space, vent lines) will provide more resolution for abnormal conditions. Rate of temperature rise can also be used - rates in the range of ~2 F/min to ~5 F/min have been experienced during a fire [24].	Molten sulphur temperature measurement is generally important to prevent abnormal operating conditions that create hazards.
Sweep air flow/vent flow	Indirect measure of elevated H_2S due to inadequate sweep air flow	Variety of instruments and approaches (various air flow meters, pressure as an indication of flow, etc.) Can also monitor motive device (e.g., steam flow to ejector) See [15] for more discussion.	Instrumentation requires special care for sulphur service (heating, prevent contact with sulphur vapours, moisture).
Visual Detection	Fire via plume/smoke	Observing a yellow plume emitting from the tank vent [24]	

Additional details on the types of instruments used and their benefits, challenges, and applicability to the refinery industry are discussed elsewhere in the literature [15].

4. DISPOSITION OF MOLTEN SULPHUR VENT STREAMS

There are multiple potential sources of vapour emissions from the molten sulphur handling system including the pit/collection header, degassing unit, storage tank, and/or loading. Depending on site-specific requirements and regulations, venting these vapours to atmosphere may be possible; however, personnel exposure and environmental risks require careful assessment of any emissions to atmosphere (e.g., dispersion studies). In many cases, an alternate disposition for the vapours may be preferred or desired. In addition to the known hazards associated with H_2S and SO_2, molten sulphur vent streams will contain sulphur vapour and water, creating challenges with plugging and corrosion that must be managed when transporting vapours. Detailed reviews of vapour disposition are available in the literature (e.g., [6], [31]). This paper provides a summary of vapour transport considerations and a high-level review of disposition options. Caustic scrubbers for vent vapour treating are reviewed in additional detail.

4.1 Transport of molten sulphur vent vapours

Any disposition option for molten sulphur vapours will require transporting the vapour from the emissions source (i.e., sulphur handling equipment) to the final disposition location via piping and will almost always require a motive device. Molten sulphur loading requires specialized components as part of the sulphur loading arm apparatus for vapour recovery (e.g., vapour recovery plate/hatch cover, jacketed vapour recovery piping [1]), but otherwise the vapour recovery process and principals are similar across sources of vapour.

Key elements of vapour piping design for molten sulphur vapour transport include the following:

- The motive device should be located at the high point of the entire vapour recovery piping system, with free draining of piping back to the source or forward to the destination. This is essential for reliable operation of the motive device in this service as any accumulation of condensed sulphur at the motive device represents a high-risk failure mode for the system.
- The vapour piping should not contain pockets or intermediate low points and should be sloped for free draining to prevent accumulation of sulphur. This requires careful planning of the routing of vapour piping, especially when traversing long distances. Figure 6 presents a simplified example of vapour piping that meets the requirements of free-draining and high-point motive devices.
- All piping should be heat-traced (e.g., engineered steam-tracing) to maintain temperatures above the freezing point of sulphur but below problematic elevated temperatures (see temperature window in Figure 4). For vapour piping critical to the safe operation of the system, jacketed piping may be warranted (e.g., suction line to an ejector, where plugging is more likely than the discharge, which is mixed with steam).

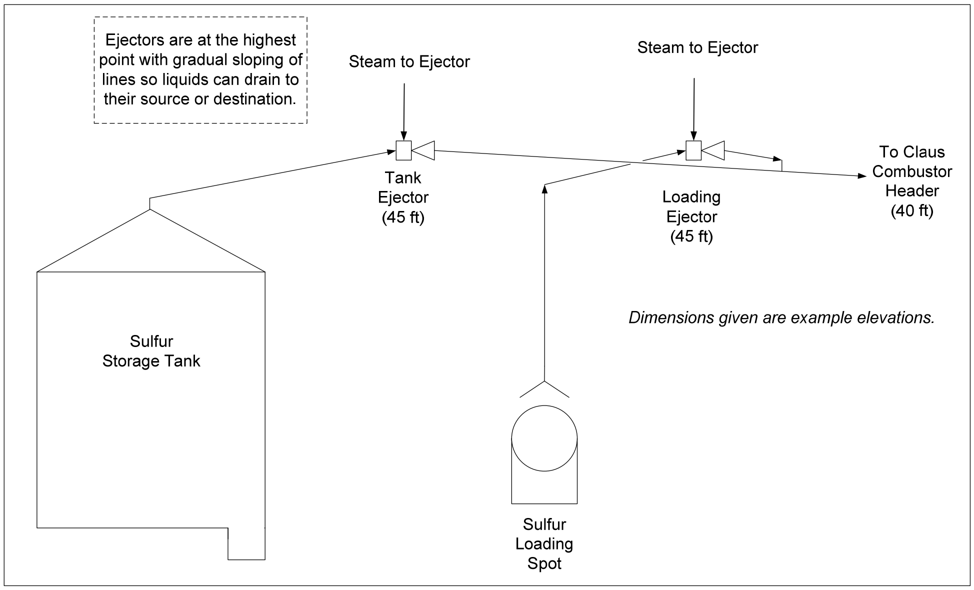

Fig. 6: Example free-draining configuration of vapour piping in molten sulphur vent application [1]

While there may be a variety of motive devices that can be used in molten sulphur vapour handling, the most common are blowers (i.e., rotating gas compression equipment) and ejectors (i.e., no moving parts, secondary motive fluid provides energy for transport). The following table presents a high-level comparison of these devices for molten sulphur service.

Table 6: Comparison of motive devices

Device	Mechanical Reliability	Operational Flow Flexibility	Cost / Utilities	Other Issues
Ejector	High (no moving parts): Erosion is biggest risk	None	Simple and less costly, but requires motive fluid (steam, air, N2)	If steam is motive, water is introduced to downstream processes
Blower	Lower: Corrosion, plugging, mechanical wear, etc.	Most Flexible (particularly with supporting controls such as VFD, valving, etc.)	Typically, more expensive and requires power to operate	No additional fluid is added that could impact downstream processes

The motive devices used in the vapour transport system must be reliable and robust in molten sulphur service – this typically requires properly designed heating (e.g., jacketing of ejector body, specialized heating design for blowers, including enclosures in cold climates), free-draining of any liquids accumulated at or near the equipment, and possible sparing of devices.

4.2 Vent vapour disposition options

Disposition options will be highly dependent on the specific SRU facility configuration, design, and equipment arrangement. Table 7 provides a general summary of possible disposition options; other literature provides more technical details on specific options in the SRU [6], [22], [31].

Table 7
Molten sulphur handling vent vapour disposition options

Option	Description	Advantages	Limitations
SRU Incinerator	Vent vapours are routed to incinerators at the back-end of the SRU that nominally manage tail gas from the SRU. Sulphur species are converted to SO_2.	Reduces personnel exposure risks by localizing emissions at the incinerator stack. Does not impact the upstream SRU operation and can operate when the SRU is down.	Does not reduce overall emissions (SO_2 still emitted). Sulphur handling equipment will be tied to a combustion source, so proper safety/isolation considerations required.
Recycle within SRU process (e.g., thermal reactor, reducing gas generator)	Vent vapours are routed to process combustion steps in SRU to be processed with other acid gas streams.	H_2S and SO_2 load is typically small compared to primary SRU acid gas content, limited impact on overall emissions.	Can impact the capacity and/or operation of SRU processes (e.g., steam from ejectors can impact chemical equilibrium/conversion, sweep air content can be significant relative to combustion air, O2 in sweep air can impact tail gas catalyst). Sulphur handling equipment will be tied to a combustion source, so proper safety/isolation considerations required.
Treating Vapours in Standalone Equipment (discussed further in following section)	Standalone treating equipment (solid/liquid scavengers, scrubbers, liquid redox, other similar technologies) receives vapours from sulphur handling equipment via motive device.	Flexibility to install near emissions points, removes H_2S and/or SO_2 to prevent emissions. Treated vapours can go to atmosphere. No impact on SRU.	Requires new equipment (CAPEX) and recurring treatment costs/chemicals (OPEX). New process step/equipment added complexity. Processes can be prone to reliability/operability issues if not designed properly for sulphur service.

4.2.1 Treating vapours - caustic scrubbing of molten sulphur vents

Treating molten sulphur vent gas to remove H_2S and/or SO_2 may be necessary or preferred when alternate vent gas disposition is not available, when other disposition options are not reliable or practical (e.g., too far from storage equipment), when impacts on other disposition options (e.g., SRU performance) are undesirable, or when the vent stream is small, intermittent, or relatively dilute (i.e., low acid gas content). Treating vapours may also serve as a backup to other disposition options.

Treating vapours from molten sulphur vents can be accomplished by various technologies (solid adsorbent media such as activated carbon or metal oxides, liquid scavengers such as triazines or similar media, liquid redox systems, etc.). Caustic scrubbing is a liquid scavenger treating option that may be well-suited to treating vapours from molten sulphur vents as it is effective at scrubbing H_2S and SO_2, is readily available and used in many industries such as petroleum refining, can be designed for robust operation in the service [32], [33]. A key characteristic of caustic for this application is its ability to react with and dissolve large amounts of elemental sulphur, opening up the possibility of designing a system that is relatively free of plugging.

Managing the sulphur vapour is of critical importance in the design of caustic scrubbers as caustic is an aqueous media which will operate well below the freezing temperature of elemental sulphur. Caustic scrubbers operate over a range of temperatures, but a nominal operating range of 120 - 140°F may be expected; lower temperatures require special care to avoid salt precipitation while higher temperatures require special attention for the ability to meet the treatment specification and for metallurgy selection to avoid corrosion in caustic service.

Because the elemental sulphur can exist in multiple forms within the vent gas and scrubber (vapour, aerosols/fogs, liquid sulphur, sulphur sols, and solid particulate), plugging may occur in the feed and exit gas piping as well as inside the scrubber itself. To address this, special design features can be incorporated to reduce sulphur buildup and manage the dissolution of elemental sulphur in the caustic solution as shown in Figure 7.

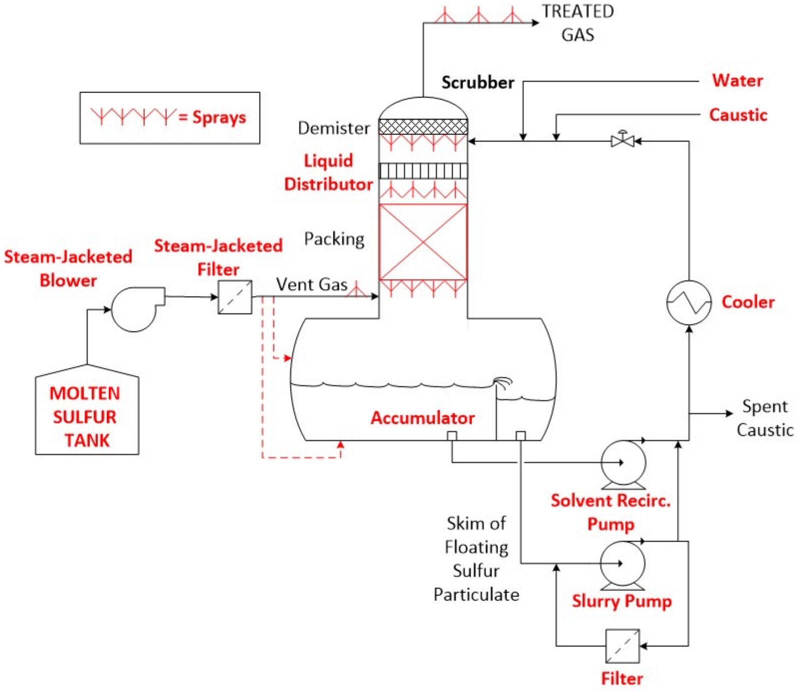

Fig. 7: Example Caustic Scrubber with Sulphur Deposition Management Strategies [32]

General design considerations [32] for handling elemental sulphur in a caustic scrubber include:

- Gas-Liquid Transfer: Design the system to transfer elemental sulphur from the gas into the liquid where it can be more easily managed.
- Suspension: Keep the sulphur particles suspended in solution or as a slurry until they dissolve or can be removed by allowing them to settle in a separator for removal or filtering the liquid.
- Circulation: Use high liquid circulation rates to remove sulphur from the gas, sweep solids off the packing, and keep solids from settling. It also eliminates dry surfaces in the packing and walls where sulphur may condense or solidify.
- Packing: A coarse spray distributor could be used to more forcefully distribute liquid over the packing to remove solids. Large, open packing should also be used to reduce the frequency of plugging.
- Sprays / Weirs: Incorporate spray designs at the scrubber inlet and outlet piping as well as around the packing and mist eliminator to loosen solids and prevent plugging. Submerging the gas entry in the sump can also aid in separating solids; a weir could also be installed in the sump for overflow of floating powder.
- Alternate Equipment Approaches:
  - A two-stage-scrubbing system with a first stage water scrubber (venturi or other design) or feed gas filter to remove particulate upstream of the main scrubber.
  - A venturi contactor (eductor) can be used with caustic as the motive fluid with a high circulation rate to ensure good contact between the gas and liquid phases.
  - Crossflow (horizontal) scrubbers may provide more stable operation and longer run times (particulate accumulation on internals reduced by gas-liquid contacting approach, easily integrated pre-wash section).
  - Installing redundant scrubbers allows one unit to stay online while the other is cleaned.

Other operational strategies can be made implemented to mitigate plugging as shown in Table 8. These design and operational modifications can improve performance, even in the most challenging applications.

Table 8
Operating strategies to mitigate sulphur plugging in caustic scrubber system [32]

Parameter	Description
Upstream sulphur vapour	Avoid unnecessarily high storage temperatures of sulphur in upstream equipment (increases sulphur vapor pressure, creates larger temperature change when vapours cool, favouring mist formation). Repair steam coil leaks to help decrease the amount of elemental sulphur in the headspace vapours.
Scrubber Additives	Surfactants increase the amount of sulphur suspended in solution. Oxidizing agents (e.g., bleach or hydrogen peroxide) change solids properties to enhance removal.
Scrubber Solution Temperature Control	Operating at higher solution temperature helps prevent sulphur vapour from condensing and improves the reaction rate of sulphur with caustic solution. Operating at higher solution temperature increases the vapour pressure of H_2S and SO_2 in the caustic solution making it more difficult to reach treatment specification. Depending on the operating temperature, high alloy material may be required (e.g., 300 series stainless or higher such as AL-6XN, or 20).
Caustic Strength	Higher operating caustic concentration enhances rates of elemental sulphur dissolution.
Exercising the Unit	Exercising the unit by changing flow rates, levels, and temperature as well as cycling control valves and motor speeds can remove accumulating solids.
Recovery and Cleaning	Warm caustic (e.g., 140°F) may enhance the rate of dissolution of sulphur and can be used for cleaning as an alternative to physically removing sulphur from the system. Can drain and clean with high strength caustic at elevated temperature or temporarily increase pH and temperature while operating to clean online. Material of construction needs to be considered for cleaning operations.

5. CONCLUSIONS

This paper reviewed hazards associated with molten sulphur handling equipment, including personnel exposure risks, environmental emissions, and fire/explosion risks due to the presence of H_2S and SO_2. The operational challenges of molten sulphur systems (operating temperature window, plugging/corrosion risks) were discussed, particularly in the context of creating or exacerbating the existing risks in the system. Direct industry examples of molten sulphur handling hazards and incidents were reviewed to contextualize the risks (i.e., risks turn to incidents in the real world if not adequately mitigated).

Approaches to manage and mitigate risks were reviewed, including identifying and maintaining appropriate operating temperatures, reducing risk of ignition sources (e.g., pyrophoric iron sulphide, free-falling sulphur), molten sulphur degassing to reduce H_2S content of sulphur in the storage and handling system, and the use of sweep air to dilute the H_2S concentration of the vent gas to a safe margin below the LEL (best practice) or blanket gas to exclude oxygen from vapour space of equipment to prevent fires and explosion.

In the event of a fire or explosion, fire suppression and explosion protection measures were reviewed, including the use of sealing steam (best practice) vs. other tank sealing methods to extinguish fires and the application of explosion protection devices (e.g., deflagration vents) in sulphur service. The paper also reviewed approaches to detect hazardous conditions (or the potential for hazardous conditions to form) in molten sulphur handling equipment, ranging from direct measurement of hazards (i.e., H_2S or SO_2 in vapour space) to indirect monitoring of process conditions (e.g., sweep air flow, temperature) to provide early indication of hazardous conditions.

Finally, the paper reviews disposition options to safely and effectively manage vapours/vent streams from sulphur handling equipment, particularly if the vapours cannot be emitted to atmosphere. Vent gas handling best practices and design considerations were reviewed for piping and motive devices to convey the vapours to their final destination. Disposition options that may be available within typical SRUs were compared (e.g., recycle to combustion devices, treatment of vapours to remove acid gases) followed by a more detailed review of the design and operation of caustic scrubbers to treat the vapours from molten sulphur handling system.

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