

# **Fundamentals of Sulfur Recovery**

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## **HAZARDS OF MOLTEN SULFUR STORAGE & HANDLING**

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## INTRODUCTION

In recent years there has been an increased interest to reduce vagrant emissions from sulfur recovery facilities, an effort to further improve safety during handling of the molten sulfur product, and a move to improve the final formed solid product quality by degassing the molten sulfur product. Such activities have required close attention to previously known characteristics of molten sulfur produced from Claus Sulfur Recovery Units (SRU) and the soluble H<sub>2</sub>S contained in the raw product. In this same time period, improvements or extensions to the Claus SRU technology, such as oxygen enrichment, and an increased need for molten sulfur degassing have in many cases changed the operating conditions and operating procedures for molten sulfur collection, storage and handling facilities.

This paper addresses the important parameters for the safe handling of molten sulfur produced in an SRU. A process flow diagram for a typical sulfur handling system is presented to review the major unit operations. Material balances have been developed for cases both with and without sulfur degassing to quantitatively follow the molten sulfur H<sub>2</sub>S content as the sulfur is collected, stored, degassed, pumped and transferred to the final user. Particular emphasis is given to the design features and startup and shutdown considerations that are necessary to avoid explosive mixtures and fire conditions in associated equipment, and to avoid toxic gas release to the atmosphere in operating areas. The discussion reflects the results of recent HAZOP Reviews and startup and operating experience gained from several types of SRU and Degassing facilities.

## SULFUR HANDLING SYSTEM PROCESS FLOW DESCRIPTION

Figure 1 represents a process flow diagram for a typical molten sulfur storage and handling system based upon a composite industry-wide perspective. Unit operations that are not always encountered have been represented with dashed lines. Material balances both with and without degassing have been included for the represented system, with emphasis on H<sub>2</sub>S content as the sulfur is transferred to the final user. The approach in developing the material balance has been to generally follow the H<sub>2</sub>S evolution at key handling junction points based on the calculation method outlined in Reference 1. A nominal 100 long tons per day production of sulfur has been assumed.

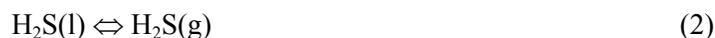
Sulfur produced in the Claus process (streams 1-3) contains soluble hydrogen sulfide (H<sub>2</sub>S) and hydrogen polysulfides (H<sub>2</sub>S<sub>X</sub>). More H<sub>2</sub>S is present at higher temperatures (i.e., sulfur from the #1 thermal-stage condenser) than at lower temperature (such as in subsequent sulfur condensers). This anomaly of physical adsorption is attributed to the formation of hydrogen polysulfides (H<sub>2</sub>S<sub>X</sub>) under the Claus reaction conditions. H<sub>2</sub>S<sub>X</sub> is a weakly-bound polymeric sulfur compound formed by the equilibrium reaction between sulfur and H<sub>2</sub>S:



This reaction proceeds to the right under increasing temperature and explains why the total H<sub>2</sub>S content is higher in molten sulfur from the waste heat boiler than in the subsequent condensers.

Claus sulfur flows by gravity into the sulfur rundown pit (stream 4), which is typically sized to contain at least one to two days sulfur production. The combined sulfur product from all SRU condensers has been considered to typically contain about 300 parts per million by weight (ppmw) of H<sub>2</sub>S and H<sub>2</sub>S<sub>X</sub>. For purposes of developing the material balances, the H<sub>2</sub>S and H<sub>2</sub>S<sub>X</sub> content of rundown sulfur from each condenser was estimated from equilibrium data.

During storage of the sulfur in the pit, the H<sub>2</sub>S<sub>X</sub> compounds will decompose (reverse of reaction 1) as the sulfur cools and is agitated. This results in the formation of dissolved H<sub>2</sub>S in the liquid sulfur which will pass to the gaseous phase by physical desorption:



Ambient air (stream 6) is drawn into and through the sulfur pit vapor space by a steam eductor (or blower) to sweep the pit vapor space of liberated  $\text{H}_2\text{S}$ . The contaminated vent air (stream 9) is discharged to the Thermal Oxidizer; however some designs allow for recycle of the vent to the SRU Reaction Furnace for improved recovery. For purposes of the material balance, it was assumed that about  $\frac{1}{2}$  of the total  $\text{H}_2\text{S}$  ( $\text{H}_2\text{S}_x + \text{H}_2\text{S}$ ) in the Claus rundown sulfur evolves in the sulfur pit or collection vessel into the vapor space (stream 7). This evolution is strongly dependent upon the storage temperature, residence time, degree of agitation, sweep gas source and degree of contact between the molten sulfur and sweep air<sup>2</sup>. Typically about 150 ppmw total  $\text{H}_2\text{S}$  and  $\text{H}_2\text{S}_x$  may remain in the sulfur being discharged from the pit (stream 5), although some plants with good sulfur pit residence time have reported total  $\text{H}_2\text{S}$  and  $\text{H}_2\text{S}_x$  concentration less than 100 ppmw.

#### Case 1: No Degassing

Sulfur from the rundown pit may be pumped directly to loading or may be directed to an above-grade storage tank (stream 5). The above-grade storage tank is typically designed to store at least the sulfur pit volume but more commonly up to 5-10 days storage, depending upon truck or ship availability. We have assumed that an additional 50 ppmw  $\text{H}_2\text{S}$  evolves in the storage tank as the sulfur cools and is possibly agitated while in residence in the tank. The storage tank is typically equipped with multiple intake vents around the roof line and a center vent pipe at the apex of the cone roof to allow a natural convection air sweep of the tank vapor space to the atmosphere. A recirculating blower system might be used to improve vapor traffic in the large cone roof volume. Alternatively, an eductor or blower can also be used to draw the vent stream through a Caustic Deodorizer before entering the atmosphere, or may direct the tank vent to the Thermal Oxidizer or to the SRU inlet.

Finally, molten sulfur is transferred intermittently (stream 13) to a tank truck (shown), ship, or a sulfur forming unit as product.  $\text{H}_2\text{S}$  will be released during this transfer and evolve into the truck vapor space. Most often, the vapor mixture ( $\text{H}_2\text{S}$  and air) in the vapor space is displaced to the atmosphere through the truck's loading and vent nozzles. Loading arms are now available with a method of sealing the loading nozzle and collecting the displaced vapor for improved vent dispersion, oxidation, treatment, or recycle. A caustic deodorizer has been shown as an option to control the odor in the tank or loading area. Ventilation fans may be used in the loading area to dilute and disperse vagrant emissions from loading operations.

#### Case 2: With Degassing

Our approach has been to show a degassing system that is external to the sulfur pit to allow discussion of this second case within the framework of a single process flow diagram. The partially degassed sulfur from the pit is pumped through the degassing system (stream 5) where it is intimately contacted with air. Depending upon the process, a chemical catalyst may or may not be used to accelerate the decomposition of hydrogen polysulfides ( $\text{H}_2\text{S}_x$ ) to dissolved  $\text{H}_2\text{S}$ . The degassing air rate shown (stream 10) is typical for many air-sparged degassing systems. The contaminated degassing off-gas is vented to the Thermal Oxidizer (stream 11) but can also be directed to the front-end of the SRU to reduce total sulfur emissions.

Note this calculation assumes no conversion of  $\text{H}_2\text{S}$  and  $\text{H}_2\text{S}_x$  to sulfur in the degassing step. Several of the process licensors report high levels of conversion so this assumption may be conservative (high) regarding actual measured emissions.

The degassed sulfur, containing 10 ppmw total  $\text{H}_2\text{S} + \text{H}_2\text{S}_x$ , is directed to an above-grade storage tank for long-term storage, but may also be sent directly to loading.

Even with sulfur degassed down to 10 ppmw total  $\text{H}_2\text{S}$ , some additional  $\text{H}_2\text{S}$  will evolve under the residence time in the tank. For purposes of developing the material balance for this case, we have assumed a 50% approach to

equilibrium to calculate the H<sub>2</sub>S evolution. On this basis, about 6 ppmw H<sub>2</sub>S remains in the sulfur and the vent contains up to 160 ppmv H<sub>2</sub>S. Sulfur is pumped as required to a sulfur truck, ship or forming unit as product from the molten sulfur handling system (stream 13).

### **RELEVANT PHYSICAL PROPERTIES OF SULFUR AND H<sub>2</sub>S**

The polymeric sulfur chain chemistry accounts for some of molten sulfur's unusual physical properties. Properties that are relevant to the safety discussion in this paper are described in Table 1.

Table 1 – Important Safety Related Characteristics of Sulfur Handling System Compounds<sup>3,4</sup>

<b>Molten Sulfur</b>		
<b>Physical Property or Characteristic</b>	<b>Hazards</b>	
Melts @ 240°F	Hot enough at process conditions to cause severe burns	
Boils @ 851°F	Exhibits measurable vapor pressure at molten sulfur handling process conditions	
Flash Point 334-369°F	Not much higher than normal storage temperatures	
Autoignites @ 478-511°F	Sulfur fires can easily occur in the presence of oxygen	
Viscosity Transition @ 318°F	<ul style="list-style-type: none"> <li>Increasingly difficult to pump above this temperature</li> <li>Magnitude is dependent upon H<sub>2</sub>S content of the molten sulfur</li> </ul>	
Highly flammable in air	Sulfur fires	
Solid dust cloud autoignites at 375°F	<ul style="list-style-type: none"> <li>Important variable in solid forming operations</li> <li>Deflagration possible</li> </ul>	
Excellent Insulator	Static discharge @ 11% or higher oxygen atmosphere	
<b>Hydrogen Sulfide (H<sub>2</sub>S)</b>		
Highly flammable in air between approximately 3.4% and 45% at process conditions	Deflagration possible	
Exposed carbon steel forms iron sulfide corrosion product under wet, reducing conditions	<ul style="list-style-type: none"> <li>Excessive corrosion in carbon steel piping in some cases</li> <li>Iron Sulfides are highly pyrophoric</li> </ul>	
Hydrogen sulfide is extremely toxic with rapid effects!		
<b>Approximate Concentration, ppmv</b>	<b>Duration</b>	<b>Effects</b>
0.01 - 0.15		Odor Threshold
Above 5		Eye irritation
10		Threshold limit value
50 – 500		Respiratory irritation
100 – 150	3 - 15 minutes	Paralysis of sense of smell (can be permanent)
	1 hour or more	May cause dizziness (falls), headache and drowsiness
250 – 600		May cause lung complications
Above 200	Many hours	Danger to life due to lung irritation
700 - 1,000	Immediate	Loss of consciousness, respiratory paralysis, rapidly fatal
Above 1,000	Immediate	Fatal

Sulfur Dioxide (SO <sub>2</sub> )		
Physical Property or Characteristic	Hazards	
Corrosive when wet	Forms sulfurous acid in presence of water at low temperatures	
<b>Sulfur Dioxide is even more toxic than H<sub>2</sub>S !</b>		
Approximate Concentration, ppmv	Duration	Effects
2	8 hours	Threshold limit value Eye, nose, throat irritation
3-5		Choking odor
5	10 minutes	OSHA Permissible Exposure Limit
400-500		Eye burns, pulmonary edema
1000	10 minutes	Lethal

## KNOWN HAZARDS OF MOLTEN SULFUR STORAGE AND HANDLING

### Sulfur Fires

Sulfur fires are not a common occurrence in plant operation. However, they do occur occasionally, principally during startup, shutdown or during maintenance periods<sup>5</sup>. An important item that is often overlooked is that sulfur fires can generate large amounts of SO<sub>2</sub> gas. SO<sub>2</sub> is a highly toxic respiratory irritant and can be lethal in sufficient concentrations. Sulfur fires burn very hot and can readily damage the mechanical integrity of plant equipment.

### H<sub>2</sub>S Concerns

H<sub>2</sub>S is highly toxic and even low concentrations can be fatal as indicated in Table 1. As a minimum, H<sub>2</sub>S leakage can create an odor nuisance. Therefore, release should be avoided.

H<sub>2</sub>S is also highly flammable and can form explosive mixtures in air as shown by the flammability triangle in Figure 2. As temperature increases, so will the flammability range by the following relationships<sup>6</sup>:

$$\text{LFL (T)} = \text{LFL (25}^\circ\text{C)} \times \{1 - [0.75 \cdot (T - 25)] / \Delta H_c\} \quad (3)$$

$$\text{UFL (T)} = \text{UFL (25}^\circ\text{C)} \times \{1 + [0.75 \cdot (T - 25)] / \Delta H_c\} \quad (4)$$

The temperature dependence of the lower flammability limit for H<sub>2</sub>S in air is represented as the top curve in Figure 3. It is important to consider the actual operating conditions of the process when evaluating the hazards of the system.

Despite the fact that both H<sub>2</sub>S and sulfur are flammable in air, the current safe and proven industry practice is to use an air sweep of the sulfur pit (or rundown vessel) vapor space to maintain the H<sub>2</sub>S level to well below the LEL, which is 3.4% volume at a storage temperature of 330°F<sup>1</sup>. **Why has the industry adopted an air purge when both H<sub>2</sub>S and sulfur are flammable?**

- First, air is generally less expensive and more readily available than inert gases such as CO<sub>2</sub> and nitrogen.
- More importantly, with the configuration shown, a slight vacuum is pulled on the sulfur pit, which ensures that toxic H<sub>2</sub>S will not leak out of the pit in the event of a leaky roof.

- In addition, air allows for better degassing in the sulfur pit, which means less H<sub>2</sub>S will evolve during downstream storage, handling, forming and loading operations.
- Finally, the presence of oxygen maintains an oxidizing atmosphere on the sulfur pit, which prevents the formation of highly pyrophoric iron sulfides on any carbon steel surfaces that may ignite under ambient conditions<sup>5,7</sup>.
- There are many plants that either have no gas purge or air sweep of the vapor space (therefore the vapor space fills with evolved H<sub>2</sub>S), or have employed fuel gas, nitrogen, or other inert gas purge of the sulfur storage vapor space. Thus, the system normally operates under a reducing environment. Such systems can easily experience deflagrations when air either leaks into the system or enters during inspection or maintenance activity.

### SO<sub>2</sub> Concerns

Although not flammable, SO<sub>2</sub> is even more toxic than H<sub>2</sub>S and will also be encountered in the headspace of sulfur storage vessels. It also forms highly corrosive sulfurous acid in the presence of water. Sulfurous acid will condense at an elevated dewpoint over that of pure water. Recent data indicates that SO<sub>2</sub> is actually formed by the reaction of oxygen with molten sulfur in the liquid phase of sulfur that has been deeply degassed of H<sub>2</sub>S (<10 ppmw H<sub>2</sub>S)<sup>18</sup>. Quantification of these emissions is discussed later in this paper.

### Corrosion

A reducing, wet sulfidic atmosphere can lead to severe corrosion of carbon steel and even stainless steel components typically found in sulfur handling systems<sup>8</sup>. This can lead to loss of containment and the risk of personnel exposure. Aluminum materials do not experience corrosion and may be used wherever possible to prevent such problems.

### Static Discharge

Due to the excellent insulating properties of molten sulfur, static discharge can build up where free fall of sulfur is allowed. Several incidents have been reported where static buildup was believed to have initiated a sulfur fire or explosion<sup>5</sup>.

### Sulfur Temperature

#### *Low:*

Sulfur must not be allowed to fall below the solidification temperature. Either online maintenance or a costly shutdown may be required to rod out lines, equipment and instruments; or a lengthy remelt effort may be required.

#### *High:*

Sulfur temperature should be maintained below about 315°F to avoid high viscosity and handling problems. Many sulfur plants today are utilizing oxygen enrichment. Oxygen enrichment produces more sulfur at higher temperatures which can create a viscous sulfur mixture in the pit. At temperatures above the sulfur flash point of 334°F, sulfur fires also become an increasing concern.

## **SULFUR DEGASSING AS A MEANS OF REDUCING STORAGE & HANDLING HAZARDS**

Because of the safety and environmental concerns associated with H<sub>2</sub>S release, or to improve formed sulfur product quality, many companies have opted to employ sulfur degassing. Various technologies are available, but

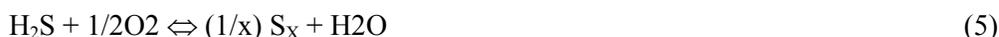
a complete comparison is beyond the scope of this paper. However, many of the elements between the technologies are common, and they have similar safety aspects that will be expanded upon later in this paper. Table 2 summarizes the important differentiators between the commercially available degassing technologies.

Table 2 – Commercially Available Sulfur Degassing Technologies<sup>9,16</sup>

Technology	In-Pit	External	Catalyst Used?	Air Sparging?	Mechanical Agitation?
Aquisulf	X		Chemical addition	Air sweep of vapor space only	Spray nozzles and sulfur recirculation
Exxon	X		Chemical addition optional	X	Induced recirculation through proprietary air sparger
Shell	X		Chemical addition optional	X	No moving parts and proprietary fabricated contacting device
Amoco	X	X	Fixed bed Claus Catalyst	X	No moving parts and proprietary fixed bed contactor
Hy-Spec		X	Chemical addition	Air sweep of vapor space only	Mixing impellor in multiple vessels
D'GAASS		X	None	X	No moving parts and proprietary contacting device

The principle of any sulfur degasification process is to accelerate the decomposition of hydrogen polysulfides to H<sub>2</sub>S and to release the dissolved H<sub>2</sub>S gas in a controlled manner. Sulfur temperature, residence time in the pit or degassing vessel, and the degree of agitation influence degassing. Chemical catalysts that accelerate the rate of H<sub>2</sub>S<sub>x</sub> decomposition have also been employed.

It is important to note that all of these processes generally employ air as the degassing medium. Research done by Alberta Sulphur Research Ltd. has demonstrated that air is a superior degassing agent compared to nitrogen, steam or other inert gases due to the presence of oxygen<sup>10</sup>. The oxygen present in air also promotes some level of direct oxidation of H<sub>2</sub>S to elemental sulfur as described below:



A portion of the H<sub>2</sub>S may also be over-oxidized as evidenced by small amounts of SO<sub>2</sub> measured in the vent gas:



Finally, the familiar Claus Reaction may take place to some extent:



All of these mechanisms reduce the H<sub>2</sub>S partial pressure in the degassing system vent stream, which improves the driving force for degassing by Le Chatlier's principle. Total H<sub>2</sub>S and H<sub>2</sub>S<sub>x</sub> conversion to sulfur is reported high in the Amoco (BP), Shell and D'GAASS processes.

Temperature is also an important variable to achieve optimal degassing and improve handling characteristics. The optimal degassing temperature is approximately 280°F. Without taking credit for the oxidation reactions 5-8, degassing to below a 10 ppmw total H<sub>2</sub>S (H<sub>2</sub>S + H<sub>2</sub>S<sub>x</sub>) specification cannot be thermodynamically achieved unless temperatures are below approximately 300-310°F.

Many global sulfur producers have adopted a 10 ppmw H<sub>2</sub>S specification for molten sulfur. However, even at this low H<sub>2</sub>S concentration there are still concerns for safety. Assume sulfur with a given H<sub>2</sub>S concentration is loaded into a storage vessel and closed off with only a very small vapor space. The vapor space volume is small enough so that any H<sub>2</sub>S that evolves from the molten sulfur does not appreciably change the amount of H<sub>2</sub>S remaining in the molten sulfur. The sulfur and air are then allowed to equilibrate at a given temperature assuming atmospheric pressure is present in the vapor space. Results are presented in Table 3.

For example, molten sulfur containing 300 ppmw total H<sub>2</sub>S is loaded into a tank truck. After a long haul, the sulfur has been agitated and cools to 280F allowing the contents to approach equilibrium as in Table 3. Assuming a 30 ton sulfur truck with 5% vapor volume is used, the H<sub>2</sub>S content in the tank truck vapor space at the end of the haul is 40.5% volume, which is within the flammable range. The H<sub>2</sub>S content in the sulfur only drops by 20 ppmw to 280 ppmw after the haul, so the assumption of constant molten sulfur H<sub>2</sub>S content is reasonable.

Table 3 – Equilibrium Vapor space H<sub>2</sub>S Concentrations above Sulfur with no Air Purge

ppmw total H <sub>2</sub> S in loaded sulfur	Equilibration temperature, °F	vol% H <sub>2</sub> S in vapor space
300	300	29.7
300	280	40.5
150	300	14.0
150	280	20.4
50	300	4.3
50	280	6.9
10	300	0.7
10	280	1.4

From Table 3, only sulfur degassed to something less than 50 ppmw H<sub>2</sub>S results in a nonflammable vapor space mixture. In reality, these equilibrium conditions might parallel a long haul in a sulfur truck or rail car. Another real life parallel could be loss of purge on a sulfur storage pit or vessel. Pressure can build up over time and create a dangerous release when the truck is opened for unloading. Finally, please note that even though the vapor space generated during transport of sulfur degassed to 10 ppmw H<sub>2</sub>S is not flammable, it will still be highly toxic.

## **SO<sub>2</sub> EMISSIONS FROM SULFUR HANDLING EQUIPMENT**

Jacobs Comprimo Netherland, ASRL and Shell Global Solutions recently collaborated to collect and interpret field measurements and laboratory studies to better understand formation and handling of H<sub>2</sub>S and SO<sub>2</sub> emissions from liquid sulfur during storage and transportation. The following is a brief summary of their findings<sup>18</sup>.

### SO<sub>2</sub> in the Vapor Space of Storage Tanks and the Stored Sulfu

The data collected from air-blanketed storage tanks show SO<sub>2</sub>, COS, and CS<sub>2</sub> are present in the produced sulfur from an SRU, due principally to solubility at the operating conditions. However, the SO<sub>2</sub> in particular has been shown to concentrate at certain locations in the plant. These compounds accumulate above the liquid surface in

rundown lines, storage tanks and in product transport vessels as a result of diffusion of these species from the liquid. The highest concentrations have been noted immediately above the liquid surface in the particular vessel or tank. Since SO<sub>2</sub> is highly toxic and the predominant component of the three, it poses some risks to operating personnel if not recognized.

The primary mechanism for SO<sub>2</sub> formation is through reaction with the air in the storage tank and shipping vessels. Both vapor phase and liquid phase reaction likely occur, but the liquid reaction is the dominant process. Therefore it is important to minimize the turbulence in storage and shipping vessels during either filling or removal of sulfur from the system. SO<sub>2</sub> formation may be limited in a tank where there is no turbulence, hence mixing of air with the sulfur. In the absence of turbulence, most of the SO<sub>2</sub> will be concentrated in only the surface liquid layer.

#### *Degassing Does Not Solve the Problem*

Degasification of liquid sulfur prevents explosions and has contributed significantly to reducing malodorous emissions, especially around sulfur rundown pits. It has been proven that handling of degassed sulfur is much safer. However, it has been mistakenly assumed that all associated environmental problems were also solved. However, it is clear from the field and laboratory data collected for this study that SO<sub>2</sub> emissions from tanked, highly H<sub>2</sub>S-degassed sulfur can be considerable. Several facilities operators in the industry, with both degassed and un-degassed sulfur, have complained about SO<sub>2</sub> emissions and visible plumes about storage and loading equipment. Measurements at one facility indicated that the maximum allowable concentration for SO<sub>2</sub> of 5 ppmv was exceeded considerably.

#### *SO<sub>2</sub> Solubility in Molten Sulfur*

The solubility of SO<sub>2</sub> in liquid sulfur collected by Touro and Wiewiorowski showed that the solubility of SO<sub>2</sub> in sulfur decreases with increasing temperature. This data showed that SO<sub>2</sub> solubility in sulfur ranges from about 500 ppmw to 400 ppmw over a range of temperature from 255°F to 270°F in an SO<sub>2</sub> atmosphere of 1 bar absolute.

To corroborate this data, several measurements were made to determine the actual amount of SO<sub>2</sub> dissolved in liquid sulfur at various locations throughout a sulfur plant. The SO<sub>2</sub> concentrations, measured at various times, in the three run down pits varied from 13 - 18 ppmw. The SO<sub>2</sub> levels are similar (9 - 14 ppmw) as the liquid sulfur enters the air degassing unit, but are reduced considerably (3 - 7 ppmw) as the sulfur leaves the degassing unit. These results imply that SO<sub>2</sub> is actually removed by the sparging action of the air degasser. The liquid SO<sub>2</sub> levels increase, however, in the product shipping vessel (14 - 18 ppmw sulfur). This increase is likely due to formation of SO<sub>2</sub> from reaction of sulfur with air in the sulfur storage tank before it is loaded. Thus, again, it seems clear that degassed liquid sulfur reacts readily with O<sub>2</sub> in air-blanketed tanks.

#### *SO<sub>2</sub> in the Headspace of Tank Cars Transporting Molten Sulfur*

Data was also obtained for the SO<sub>2</sub> quantities found in tank cars transporting liquid sulfur. Two of the tankcars had considerable quantities of SO<sub>2</sub> before loading! Residual liquid sulfur (from a previous trip) likely reacted with air (O<sub>2</sub>) on the return trip, forming SO<sub>2</sub>. But according to the data, the amount of SO<sub>2</sub> in the headspace actually drops during loading of fresh sulfur. This observation can be explained by SO<sub>2</sub> re-dissolving in the liquid during the loading process. For this to occur, the liquid sulfur in the storage tank must be under-saturated with SO<sub>2</sub>. This is likely true since the loading pumps take suction from the bottom of the storage tank. The sulfur below the air-sulfur interface has limited opportunity to see and react with air unless the tank is circulated.

## OTHER SULFUR PLANT DESIGN CONSIDERATIONS

### Avoiding Static Discharge

Free fall of sulfur entering storage vessels should be avoided<sup>5</sup>. Dip tubes should be provided to allow sulfur to enter below the lowest anticipated liquid level in the storage vessel or pit. Spray nozzles, sulfur loading arms, etc. should all be grounded. All instrumentation should be designed with explosion proof housings in accordance with the area electrical classification.

### Sweep Air System Design Recommendations

Industry practice for design of sweep air streams 6 and 14 is to introduce enough air to dilute any H<sub>2</sub>S evolved to below ¼ of the lower explosive limit in air, which is 3.4% volume at 330°F<sup>1</sup>. This corresponds to 0.85% volume H<sub>2</sub>S. Commonly accepted design practice is to generally assume *all* H<sub>2</sub>S present in the initial rundown sulfur evolves at each point in the process. This may seem like double jeopardy, but there is no guarantee how well the sweep air is distributed throughout the large vapor spaces of storage pits and tanks. This volume of sweep air is also considered to provide a reasonable velocity to avoid dead space and pockets of high H<sub>2</sub>S in the system.

H<sub>2</sub>S will continue to evolve even if the sulfur is degassed down to 10 ppmw total H<sub>2</sub>S. **Accumulation of H<sub>2</sub>S to toxic levels should be taken into consideration even if levels are well below the flammability limits.**

Design and operating conditions of enhanced sulfur recovery processes should also be accounted for. For example, subdewpoint processes produce sulfur in a cyclic manner. At periods during the cycle such as regeneration, more sulfur may be produced and with a higher H<sub>2</sub>S/H<sub>2</sub>S<sub>x</sub> content than the cycle average.

Oxygen-enriched operation also introduces unusual conditions. During oxygen use, more sulfur will be produced. In addition, a larger percentage of sulfur will be produced near the front of the plant which operates at higher temperature and with a higher H<sub>2</sub>S partial pressure in the vapor. Both of these variables favor higher equilibrium dissolved H<sub>2</sub>S/H<sub>2</sub>S<sub>x</sub>.

Recent data collected by Alberta Sulphur Research Ltd. from actual operating plants indicates that “actual” concentration of H<sub>2</sub>S may be up to 100-200 ppmv higher than values calculated by the theoretical basis represented in the material balance<sup>11</sup>. Design of the sulfur handling system and sweep air requirements of the sulfur pit or storage vessel should be conservative to allow for this actual plant experience.

If the sulfur handling design is a revamp for an existing system, actual measurements should be taken for the H<sub>2</sub>S<sub>x</sub>/H<sub>2</sub>S content of the rundown sulfur. Total H<sub>2</sub>S in the rundown sulfur can be estimated with a Tutweiler laboratory analysis. Alberta Sulphur Research has developed an analyzer that provides more accurate, online H<sub>2</sub>S/ H<sub>2</sub>S<sub>x</sub> measurement. Brimstone Engineering and Enersul both offer commercialized versions of the analyzer.

The sulfur handling sweep air design needs to accommodate Claus system upsets. Poor operation of the Claus Reaction Furnace can result in the following hazards to the sulfur handling system.

- Higher H<sub>2</sub>S:SO<sub>2</sub> ratio operation favors higher equilibrium solubility of H<sub>2</sub>S in molten sulfur, which increases sweep air requirements.

- Unburned heavy hydrocarbons, tars, soot, and CS<sub>2</sub> formation exiting the Claus Reaction Furnace can allow a mechanism for CS<sub>2</sub> to evolve from the rundown sulfur. CS<sub>2</sub> is highly combustible<sup>12</sup> and can increase the mixture flammability range with H<sub>2</sub>S as shown in the bottom curve of Figure 2. This curve was estimated with the following relationship<sup>6</sup>:

$$LFL_{mix} = \frac{1}{\sum_{i=1}^n \frac{y_i}{LFL_i}} \quad (9)$$

A refinery sulfur plant experienced an incident in a recent in-pit degassing system startup that was believed to be initiated by such a Claus system upset<sup>13, 14</sup>. A fire and subsequent pit deflagration caused damage to the sulfur pit and degassing system. Capability of the degassing system to be bypassed in the event of a Claus system upset is one of the major advantages offered by the external degassing systems. In addition, pit or tank vapor space H<sub>2</sub>S can be analyzed to confirm that adequate sweep air is being introduced online and provide indication of such upsets.

Finally, consideration should be given to maintaining sweep air flow at all times, even during periods when no molten sulfur production or transfer occurs. Eductor internals will wear over time. Several plants employ blowers to pull sweep air across the sulfur pit. Some have plugged or experienced mechanical failure over time due to accumulation of solid sulfur. Where feasible, redundancy should be provided in these services. Nearly all recent designs employ some sort of emergency natural draft vent to allow air purge on loss of the eductor(s) or blower(s).

### Sulfur Fires

The design of the system should accommodate means to detect and deal with sulfur fires<sup>5</sup>. The temperature of the storage pit or vessel vapor space can be a key indicator of a sulfur fire. Thermocouples immersed below the sulfur liquid level may not detect a fire burning at the surface. An SO<sub>2</sub> analyzer provided in the pit vapor space can also provide indication of a sulfur fire, although this is a more expensive option. In the event of a fire, the design should allow for injection of adequate snuffing steam or inert gas to cool the fire and to prevent air leakage to the storage pit or tank<sup>5</sup>. Pressurized sources of air should also be isolated or diverted from the storage pit, tank or degassing system in the event of a sulfur fire.

### Temperature Monitoring and Control

Degassed sulfur will become highly viscous and difficult to pump above 315-320°F. As the H<sub>2</sub>S is removed by degassing, viscosity will approach the viscosity of pure sulfur. Molten sulfur temperature should be monitored at key points in the process and maintained below the viscous transition and at the same time safely above the solidification temperature.

### Proper Steam Jacketing Pressure Level on Site-Specific Designs

Proper attention should be given to the site-specific design of steam jacketing and tracing. A minimum 35-40 psig steam pressure is recommended to prevent sulfur solidification. On the other hand, utilizing steam which is too hot can lead to safety related concerns. Several incidents have been reported where high pressure steam was believed to have started a sulfur fire<sup>5</sup>. In addition, the sulfur may be heated to a viscous range with high steam jacketing pressure, which can lead to difficulty in flowing and pumping and result in poor degassing. No higher than 75 psig nominal saturated steam should be used for jacketing and tracing.

Design consideration should also be given to the type of sulfur storage vessel and pumping configuration. A sulfur pit will lose much more heat than an insulated vessel. In addition, sulfur pumping energy efficiency should be accounted for to avoid high sulfur temperature. On one occasion following an initial SRU startup, sulfur coolers had to be installed to a sulfur storage system because of the following site-specific concerns:

- A large recycle sulfur flow was present from the degassing system.
- The climate was extremely hot.
- Heat loss was low from the insulated storage vessel used instead of a sulfur pit.
- The lowest pressure steam available in the plant was 75 psig.

On an oxygen-enriched SRU revamp design, boiler feed water jacketing was used on the sulfur rundown dip-legs to allow the sulfur to be cooled enough to pump.

### Corrosion Mitigation

Generally carbon steel metallurgy with a 1/8” corrosion allowance provides acceptable service in sulfur handling equipment. However, it is important to maintain the vapor space hot and dry and in an oxidizing atmosphere to prevent excessive corrosion and iron sulfide formation<sup>8</sup>. Adequate steam jacketing or tracing should be provided to avoid a wet environment. Where high velocities or potential of water vapor to condense exists, stainless steel, cladding or aluminizing have been used to provide extended life. A good alternative is to fabricate all possible splash zone and sulfur storage components of aluminum.

### Sulfur Handling Piping and Instrumentation

Table 4 provides a summary of typical recommendations for sulfur handling system piping and instrumentation. Designing for molten sulfur and sulfur vapor services requires special considerations.

Table 4 – Piping and Instrumentation Recommendations for Sulfur Liquid and Vapor Service

<b>Instrumentation</b>	<b>Do's</b>	<b>Don'ts</b>
Flow	Liquid – Wedgemeters, eccentric orifices, coriolis Vapor – Low ΔP vortex shedding, venturis or thermal massmeters	Moving parts such as turbine meters
Level	Bubblers and Radar	Displacer type
Pressure	Diaphragm	
All	Adequate tracing and or purging	
<b>Piping</b>	<b>Do's</b>	<b>Don'ts</b>
Valves	Jacketed full port plug and ball to allow rodout	Reduced port, gate or globe valves
General	<ul style="list-style-type: none"> <li>• Jacketed or traced</li> <li>• Crosses to allow rodout in both directions</li> <li>• Free draining</li> <li>• Avoid check valves and use seal loops where possible</li> </ul>	<ul style="list-style-type: none"> <li>• Pocketed, poorly traced or jacketed lines</li> </ul>

## DEGASSING SYSTEM STARTUP AND SHUTDOWN CONSIDERATIONS

### *Degassing Introduces Added Complexity and Its Own Safety Considerations.*

It was stated earlier that the principle of any air-sparged sulfur degasification process is to release the dissolved H<sub>2</sub>S in a “controlled” manner. One important consideration that is often overlooked is how the design will perform during startup and shutdown. As an example, degassing was being installed into a large, existing above-grade sulfur storage tank with 10 days production capacity. The rate of degassing upon initial air sparger startup is much higher because of the high concentration of total H<sub>2</sub>S in the mass of undegassed sulfur which can allow H<sub>2</sub>S levels to exceed the lower explosive limits. Operations personnel have observed Thermal Oxidizer stack SO<sub>2</sub> spikes from numerous degassing systems on startup, even for systems with much less residence time. This hazard exists in any air-sparged degassing process with a large mass of undegassed sulfur that is present on startup.

Data published by Alberta Sulphur Research Ltd. Has been used to develop a kinetic unsteady-state CSTR degassing model to better quantify this hazard<sup>10, 15</sup>. The assumptions in the model are that perfect mixing takes place, and that the rate-limiting step is H<sub>2</sub>S<sub>x</sub> decomposition. H<sub>2</sub>S evolved into the gas phase is assumed to be at equilibrium based on Henry’s law. While perfect mixing is never achieved and for most cases significant degassing will occur prior to entering the degassing system, the results of the model provided us with conservative results to allow safeguards to be evaluated.

Figure 4 contains the results of the model, which predict that a hazard exists when the air is turned on. The initial vapor space concentration in the tank is well above the lower explosive limit of 3.4% volume for a significant amount of time. The model predicts that the time spent in the flammable range is directly proportional to the amount of undegassed sulfur initially present. In reality, degassing prior to air sparger startup will improve as the sulfur spends more residence time in the tank. Based on these results, design improvements and operating procedures in Table 5 were developed to safeguard the system:

Table 5 – Design and Operating Procedure Safeguards for Degassing System Startup

<b>Option</b>	<b>Advantages</b>	<b>Disadvantages</b>
Have air flowing through the sparger when undegassed sulfur first enters the tank.	Simple and no additional cost	Never assured that this will be possible 100% of the time
Pump out tank sulfur inventory prior to initial air sparger startup or following extended shutdowns when undegassed sulfur accumulates.	Simple and no additional cost	<ul style="list-style-type: none"> <li>• An operating procedure is never fail-safe</li> <li>• Additional sulfur rail cars must be available during these periods</li> </ul>
<ul style="list-style-type: none"> <li>• Turn on sparging air slowly initially</li> <li>• H<sub>2</sub>S release is controlled by the amount that can be held at equilibrium by the sparging air</li> <li>• External sweep air dilutes the vapor space below the LEL</li> </ul>	Simple and no additional cost	<ul style="list-style-type: none"> <li>• Still possible that operating above the LEL below the sulfur liquid level</li> <li>• Cannot guarantee that the sparging air can or will be started up slowly enough.</li> </ul>
Redesign the tank for more sweep air on startup (e.g. operate an installed spare blower or eductor)	More failsafe than a procedure	<ul style="list-style-type: none"> <li>• Eductors had already been purchased and construction was underway</li> <li>• Highest cost and time would be lost in a redesign effort</li> </ul>

Table 5 – Design and Operating Procedure Safeguards for Degassing System Startup

Option	Advantages	Disadvantages
		<ul style="list-style-type: none"> <li>• 10 times the design sweep air would have been required for a full tank</li> </ul>
Initiate pre-startup N <sub>2</sub> sparging in place of air sparge	<ul style="list-style-type: none"> <li>• Oxygen content can be brought down to below the MOC for H<sub>2</sub>S combustion</li> <li>• Most of the H<sub>2</sub>S can be removed in a more controlled manner as N<sub>2</sub> is a less efficient degassing medium</li> </ul>	Additional operating cost of nitrogen usage and capital for nitrogen line

The pre-startup nitrogen sparge option was the best fit for the example system. A pre-startup nitrogen sparge is initiated prior to admitting air through the sparger on initial startup and following extended shutdowns. The nitrogen sparge duration was derived with the referenced model as a function of the level of undegassed sulfur that had accumulated.

### Planned Shutdown

Most of the recommendations provided below are not new to the industry. However, the consequences of poor procedures are high enough that they are worth mentioning.

Following any planned shutdown, steam should be maintained to all jacketing & tracing to prevent sulfur solidification and water condensation, which can lead to excess corrosion. A routine inspection of steam traps should be continued in these critical services. If vessel entry or inspection is necessary, special precautions should be taken. Following drainage, system piping and equipment should be purged with hot inert gas to remove as much residual sulfur as possible. Air should not be readmitted until the plant has had time to cool down and should then be introduced slowly in case iron sulfides have had time to form<sup>7</sup>. Oxygen levels and H<sub>2</sub>S levels should be verified as safe. Upon entry, respirators should still be used whenever full ventilation is not provided because isolated pockets of toxic gas may still be present.

On planned shutdowns, it is advisable to physically monitor the corrosion in the plant. Areas where internal steam lines can leak should be especially watched as the presence of liquid water can increase corrosion by orders of magnitude. The importance of maintaining instruments cannot be overstressed as the safety of the plant may be put into jeopardy. All steam traps, jacketing and tracing should be monitored regularly for proper function, particularly during shutdown periods.

### Emergency Shutdown

The primary emergency shutdown concerns for a sulfur handling system are loss of containment, sulfur fires, explosions and toxic gas release. The following emergency shutdown table is based on results of recent HAZOPS, safety reviews and site visits. It is important to note that many of the items are somewhat client and site-specific so this list should not be taken as all encompassing nor as directly applicable to all situations.

Table 6 – Common Sulfur Handling Emergency Shutdowns

<b>Initiator</b>	<b>Possible Hazard</b>	<b>Responses</b>	<b>Additional Operator Action Required</b>
Low-low sweep air flow	<ul style="list-style-type: none"> <li>• Backflow through sweep air inlet</li> <li>• Flammable H<sub>2</sub>S mixture</li> </ul>	<ul style="list-style-type: none"> <li>• Start spare eductor or blower</li> <li>• Isolate eductor from Thermal Oxidizer</li> <li>• Open emergency draft stack</li> <li>• Isolate degassing air if in-pit degassing is present</li> </ul>	<ul style="list-style-type: none"> <li>• Verify eductor/blower mechanical integrity</li> <li>• Check for pit steam coil rupture</li> </ul>
Low-low sulfur level	Damage to sulfur pumps	Stop sulfur transfer or degassing pumps	Check level control valve if applicable
Low sulfur temperature	Sulfur solidification	Alarm only	Check steam tracing & jackets
High-High Sulfur temperature	Sulfur fire	<ul style="list-style-type: none"> <li>• Stop degassing feed pumps</li> <li>• Isolate sweep air eductor or blower</li> <li>• Isolate degassing air</li> </ul>	Open snuffing steam or inert gas to air free and cool the system.
High-High vapor space temperature	Sulfur fire	<ul style="list-style-type: none"> <li>• Stop degassing feed pumps</li> <li>• Isolate sweep air eductor or blower</li> <li>• Isolate degassing air</li> </ul>	Open snuffing steam or inert gas to air free and cool the system
Thermal Oxidizer S/D	<ul style="list-style-type: none"> <li>• H<sub>2</sub>S out stack</li> <li>• Operator exposure on restartup</li> </ul>	<ul style="list-style-type: none"> <li>• Stop degassing feed pumps</li> <li>• Isolate sweep air eductor or blower</li> <li>• Open sweep air emergency vent</li> <li>• Isolate degassing air</li> </ul>	Restart Thermal Oxidizer before attempting to restart sweep air eductor/blower or degassing
High-high sulfur flow to external degassing	<ul style="list-style-type: none"> <li>• Poor degassing</li> <li>• Flammable H<sub>2</sub>S mixture</li> </ul>	<ul style="list-style-type: none"> <li>• Stop degassing feed pumps</li> <li>• Isolate degassing air</li> <li>• Initiate N<sub>2</sub> purge to degassing</li> </ul>	Check flow control valve, meter, etc. for proper operation
High-High level in Sulfur truck or above-grade storage	<ul style="list-style-type: none"> <li>• Loss of containment</li> <li>• Personnel exposure</li> </ul>	<ul style="list-style-type: none"> <li>• Isolate sulfur source</li> <li>• Open to alternative storage or emergency blocking (if available)</li> </ul>	<ul style="list-style-type: none"> <li>• Check high level trip for proper operation</li> <li>• If no other route for sulfur is possible, shutdown the SRU until loadout becomes possible</li> </ul>

## CONCLUSIONS

The hazards associated with molten sulfur storage and handling systems were reviewed. Results of recent HAZOPS and operating experiences have been shared to emphasize that new hazards will continue to arise as technologies advance. In addition, changes to existing designs, facilities and operating conditions and methods require careful analyses.

It is important to determine accurate H<sub>2</sub>S/H<sub>2</sub>S<sub>x</sub> content of the rundown sulfur and make conservative assumptions for sweep air design to stay out of flammability range and prevent toxic gas exposure. The sweep air venting system needs to accommodate all anticipated operating scenarios including startup and shutdown and upstream Claus unit upsets.

Many recent innovations or extensions to the basic Claus system technology such as oxygen enrichment, sulfur degassing, and tail gas cleanup have changed the operating conditions and procedures for molten sulfur collection, storage and handling characteristics. We have presented this paper with hope that the industry as a whole will continue to give the safety of molten sulfur storage and handling systems a thorough review.

### Nomenclature

- LFL – Lower flammability limit, %volume  
UFL – Upper flammability limit, %volume  
T – Temperature in °C  
 $\Delta H_c$  – Heat of Combustion, kcal/gmol  
 $y_i$  – mole fraction of component i on a combustible basis  
n – number of combustible species

### Subscripts

mix - mixture

### Acknowledgements

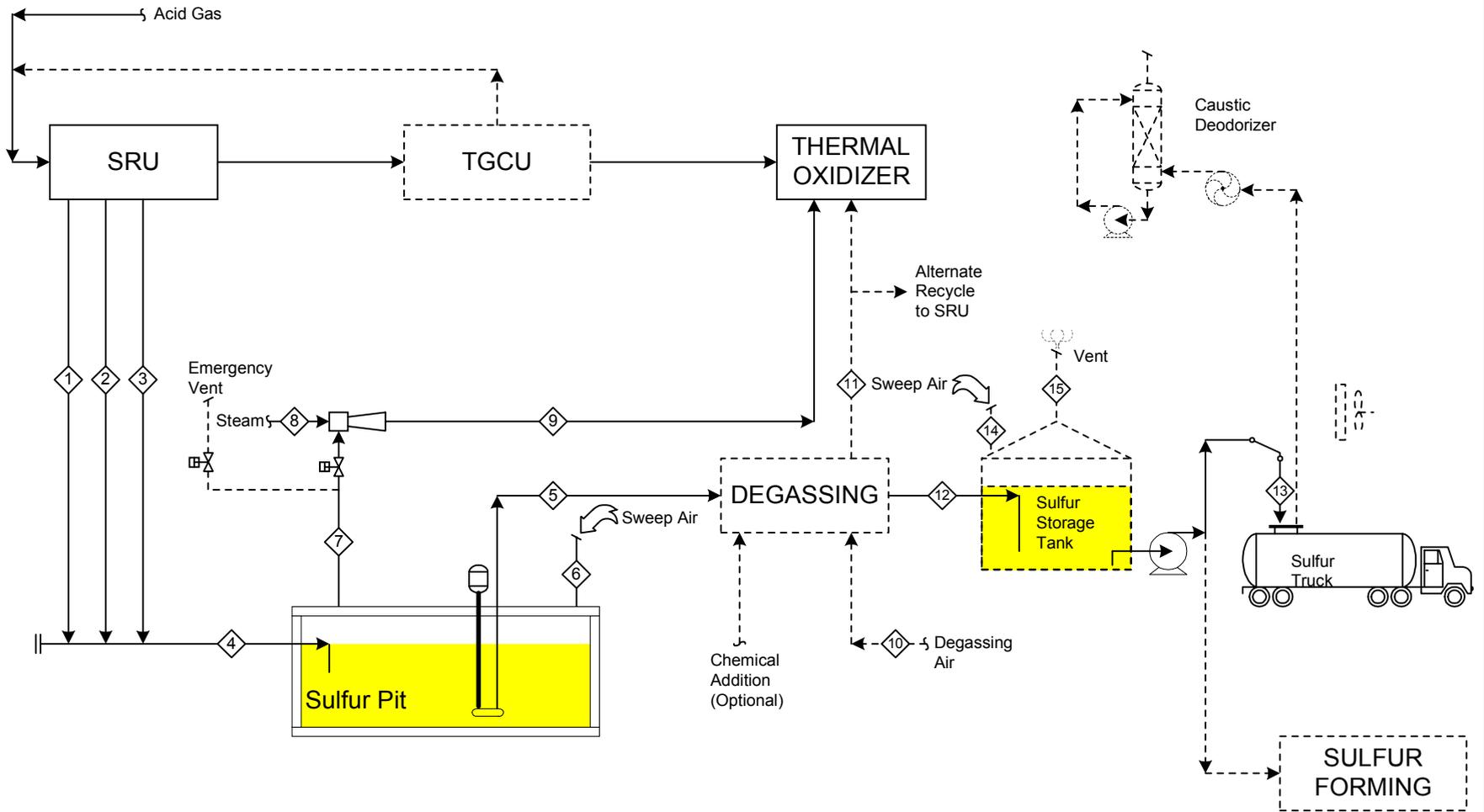
The authors would like to thank Alberta Sulphur Research Ltd. for all of the valuable input they have provided. We would also greatly appreciate hearing the experiences of others to improve the safety of our industry as a whole.

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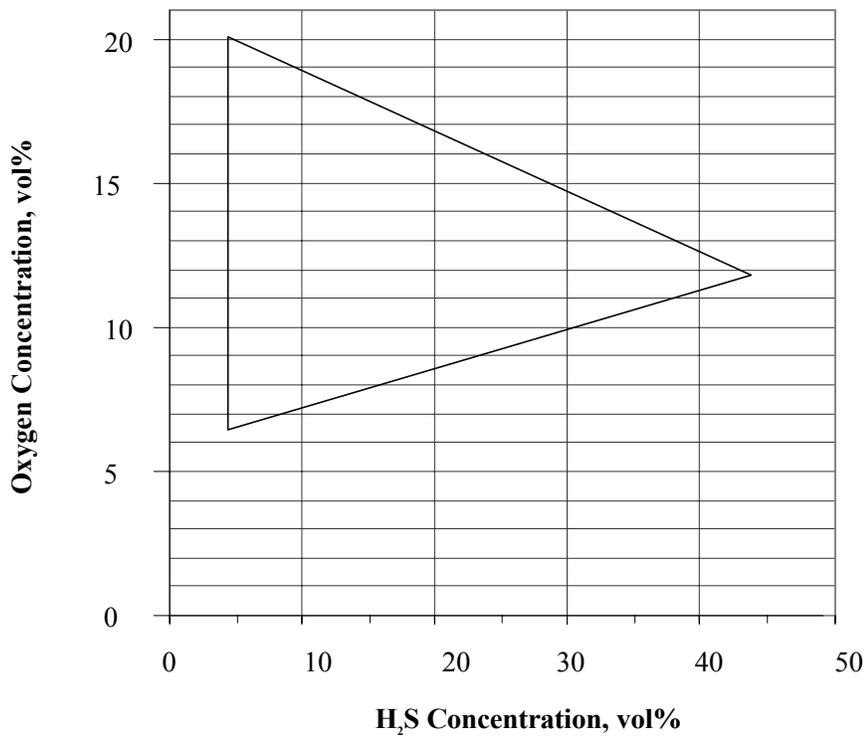
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**FIGURE 1 - MOLTEN SULFUR STORAGE & HANDLING SYSTEM PFD**

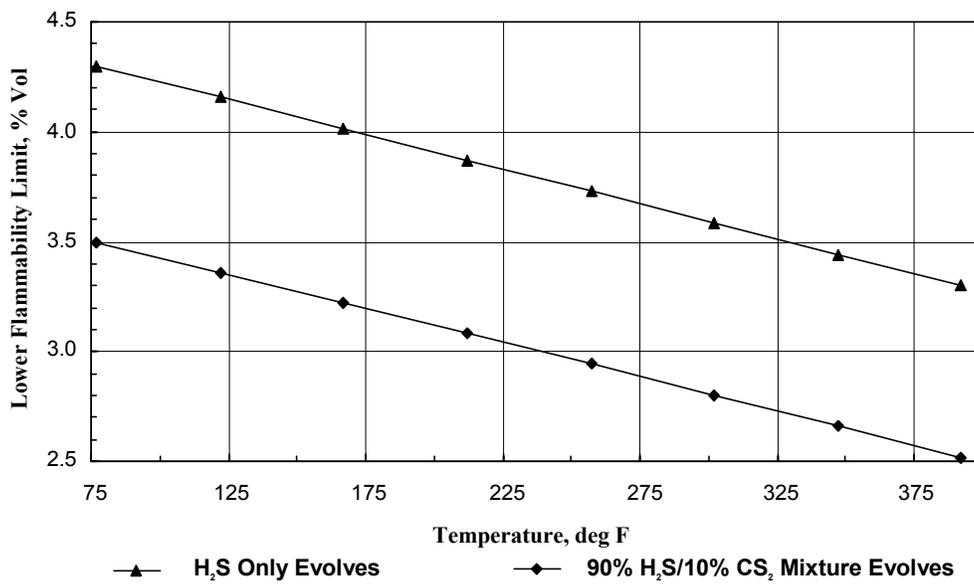


**Figure 2: Flammability Triangle for H<sub>2</sub>S**

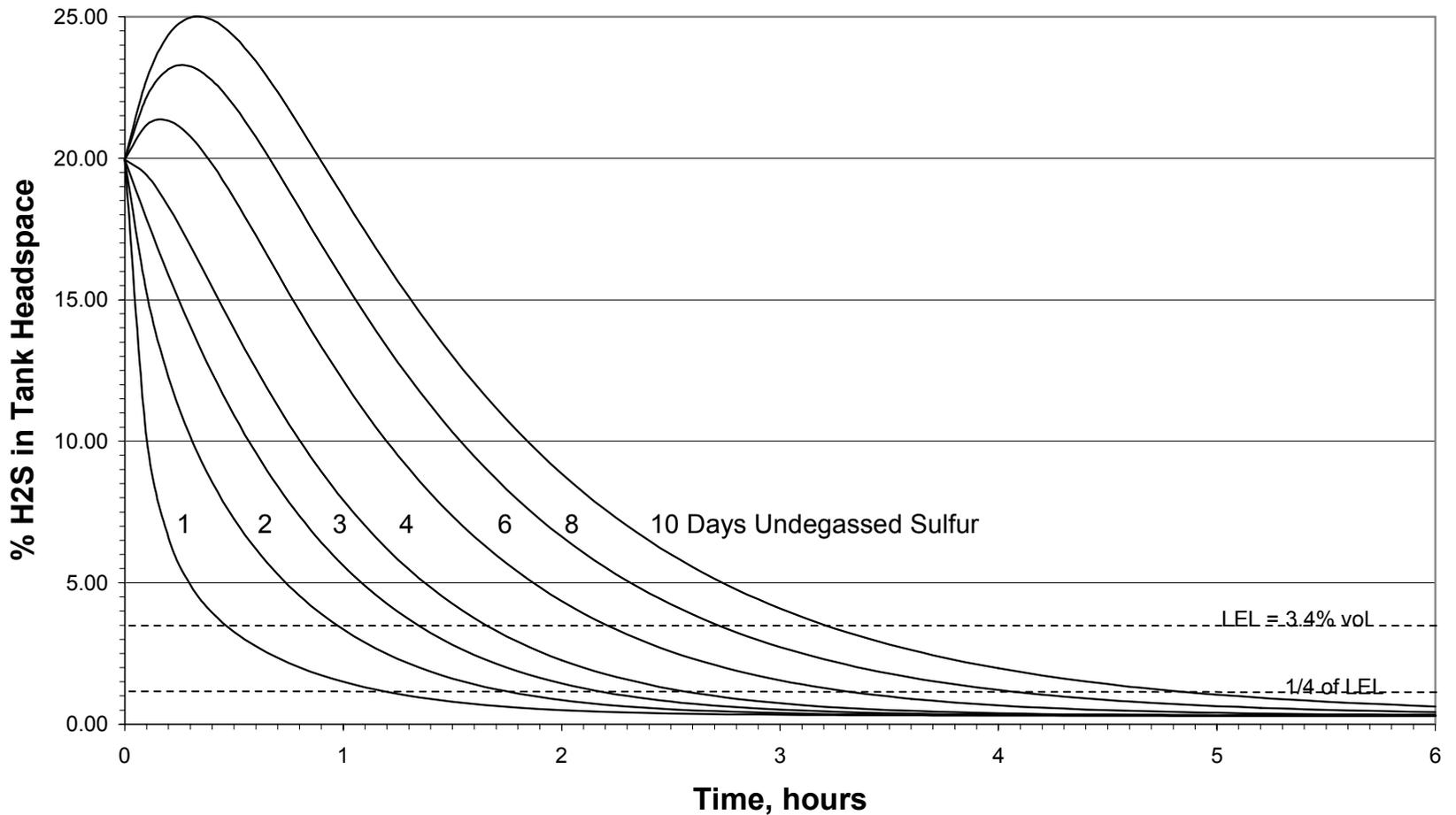
@ 77 deg F



**Figure 3: Flammability Limits as a Function of Temperature**



**FIGURE 4: Degassing Headspace H<sub>2</sub>S Concentration After Initial Sparger Startup**



**Assumptions:**

1. 300 ppmw total H<sub>2</sub>S present in sulfur prior to startup (180 ppmw H<sub>2</sub>Sx & 120 ppmw H<sub>2</sub>S).
2. Perfect mixing is achieved.
3. Air is initiated suddenly at design rate.

SULFUR HANDLING MASS BALANCE - Case 1: No Degassing  
 FLOWS IN LBS/HR

	STREAM NUMBER	1	2	3	4	5	6	7	8
VAPOR	WATER						7.2	7.2	117.4
	OXYGEN						52.7	52.7	0.0
	NITROGEN						173.5	173.5	0.0
	H2S (NOTE 1)						0	4291	0
	SULFUR VAPOR,S1						0.0	0.3	0.0
	TOTAL						233.4	234.7	117.4
LIQUID	SULFUR,S1 (NOTE 2)	5732.6	2693.5	904.0	9330.6	9330.3			
	H2S (NOTES 1,3)	422	171	6	276	150			
	TOTAL	5735.0	2694.0	904.0	9333.0	9331.7			

	STREAM NUMBER	9	10	11	12	13	14	15
VAPOR	WATER	124.5	0.0	0.0			7.2	7.2
	OXYGEN	52.7	0.0	0.0			52.7	52.7
	NITROGEN	173.5	0.0	0.0			173.5	173.5
	H2S (NOTE 1)	2860	0	0			0	1996
	SULFUR VAPOR,S1	0.3	0.0	0.0			0.0	0.1
	TOTAL	352.1	0.0	0.0			233.4	234.0
LIQUID	SULFUR,S1 (NOTE 2)				9330.3	9329.8		
	H2S (NOTES 1,3)				150	100		
	TOTAL				9331.7	9330.8		

Notes

1. H2S in PPMW
2. Sulfur liquid exists as S6 and S8 but is shown as equivalent S1 for simplicity.
3. H2S flow represents total dissolved H2S + H2Sx.

SULFUR HANDLING MASS BALANCE - Case 2: With Degassing  
 FLOWS IN LBS/HR

	STREAM NUMBER	1	2	3	4	5	6	7	8
VAPOR	WATER						7.2	7.2	117.4
	OXYGEN						52.7	52.7	0.0
	NITROGEN						173.5	173.5	0.0
	H2S (NOTE 1)						0	4291	0
	SULFUR VAPOR,S1						0.0	0.3	0.0
	TOTAL						233.4	234.7	117.4
LIQUID	SULFUR,S1 (NOTE 2)	5732.6	2693.5	904.0	9330.6	9330.3			
	H2S (NOTES 1,3)	422	171	6	276	150			
	TOTAL	5735.0	2694.0	904.0	9333.0	9331.7			

	STREAM NUMBER	9	10	11	12	13	14	15
VAPOR	WATER	124.5	23.3	23.3			7.2	7.2
	OXYGEN	52.7	98.6	98.6			52.7	52.7
	NITROGEN	173.5	370.9	370.9			173.5	173.5
	H2S (NOTE 1)	2860	0	2642			0	161
	SULFUR VAPOR,S1	0.3	0.0	0.5			0.0	0.1
	TOTAL	352.1	492.8	494.6			233.4	233.5
LIQUID	SULFUR,S1 (NOTE 2)				9329.8	9329.8		
	H2S (NOTES 1,3)				10	6		
	TOTAL				9329.9	9329.8		

Notes

1. H2S in PPMW
2. Sulfur liquid exists as S6 and S8 but is shown as equivalent S1 for simplicity.
3. H2S flow represents total dissolved H2S + H2Sx.