Beyond Triazines: H_2S Control via Downhole Injection of a New, Non-scaling H_2S Scavenger Chemistry

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ABSTRACT

The corrosive effects of hydrogen sulfide result in significant additional operating costs for production, including higher pipeline failure rates as well as souring of the produced oil and gas. In order to mitigate the effects of hydrogen sulfide and produce in spec oil and gas, chemical scavengers are often applied, typically topside and post-separation. However, application of the appropriate chemistry requires knowledge of the impact of system parameters, such as temperature and pH, on the performance of the scavenging chemical. In this regard, a novel product has been developed that exhibits good hydrogen sulfide scavenging performance in aqueous conditions over a wide temperature and pH range. The kinetics and mechanism of H_2S scavenging are discussed and compared with current technologies employed for hydrogen sulfide scavenging, such as triazines. In addition, we report case histories of this new, non-scaling chemical scavenger that exhibits excellent hydrogen sulfide scavenging performance for downhole applications. Further, we show how detailed knowledge of the system parameters and the kinetics and mechanism of H_2S scavenging provide a means for predicative performance and optimization within individual systems.

Key words: hydrogen sulfide, chemical scavenger, downhole injection, kinetics, non-triazine

INTRODUCTION

Hydrogen sulfide (H_2S) is a highly toxic, highly corrosive gas found in many oilfield production system, that can originate either from geologic sources or bacterial sources. The existence of H_2S within a production field has a significant economic impact not only upon the cost of production, but also on the ability to sell the oil and gas. Further, H_2S exhibits a considerable negative impact on the lifetime of installation assets due to corrosion failure, significantly compromising personnel safety, and having negative impacts on the environment. [1,2,3]

Minimizing the impact of hydrogen sulfide in production, transportation, and transmission of oil and gas, often requires the implementation of mechanical and chemical methods to remove H_2S in the system.

Chemical methods reduce the negative effects of H_2S by converting it to less corrosive, more innocuous products. The most common types of chemicals used to scavenge H_2S includes alkyl, aminoalkyl, and hydroxyalkyltriazines, or scavengers such as aldehydes and di-aldehydes.

The proper choice of chemical scavenger requires a detailed understanding of the production system and conditions as well as an understanding of the detailed kinetics of scavenging for the chemical option. Such understanding allows both the service provider and operator to design and implement a treatment program that not only achieves the reduction of H_2S in a cost effective manner, but also avoids additional operational challenges imposed by selecting the incorrect chemistry or applying a chemical at an improper point in the system.

An example of a concern of particular relevance exists for the commonly used triazines. Since triazines are cyclic amine compounds and alkylamines are released in either the scavenging reaction or the hydrolysis of triazines, an abrupt jump in localized pH can occur at the point of injection or if there is a local concentration build-up within a system. This has the potential to create scale deposition problems and impact a variety of applications ranging from water flooding, hydrotesting, production, and potentially even refining operations.[4] Although the use of triazines in combination with scale inhibitors has shown to be effective in some operations, the selection of the scale inhibitor and dose is dictated by the water parameters and does not take into account the risk of local pH effects from a high pH chemistry being applied into the system. This is important since a primary risk of scale is the formation of nucleation sites that destabilize the system with respect to saturation, leading to increased deposition.

In this regard, we report on the development of a novel H_2S scavenger formulation which shows pH-independent scavenging rates and linear temperature dependence under pseudo-1st order conditions. In addition, we report the successful use of this new scavenger chemistry in the field for scavenging H_2S via downhole injection. This methodology was utilized in part due to the non-scaling properties of the scavenger chemistry. Further, proper application was conducted via consideration of H_2S partitioning, well depth, production levels, mass transfer rates, and scavenging kinetics. An alogrithm was developed that provided the ability to model the system and apply an effective scavenger dose to reduce the H_2S to target levels, achieving sales gas specifications (< 4 ppm) in some fields in topside operations just after the wellhead.

EXPERIMENTAL PROCEDURE

Hydrogen Sulfide Scavenging Experiments

H_2S scavenging experiments were performed via addition of a known quantity of NaSH standard solution to a deoxygenated water solution. For instance, 1.000 mL of a 20,000 ppm as H_2S (33,000 ppm as NaSH) to water with a final total volume of 400 mL provided a 50 ppm solution as H_2S. Solutions containing 200 ppm alkalinity as $CaCO_3$ saturated with CO_2 prior to addition of NaSH (targeting ~45 – 65 ppm as H_2S) provided a solution with pH = 5, therefore, primarily existing as H_2S (99%; pKa_1 ~ 7), whereas no CO_2 saturation provided solutions of pH = 9.5 (therefore, 99.7% as HS^-). Solution pH was further adjusted with NaOH solution after sparging in order to obtain the target pH, if needed. For comparative purposes, reaction was also run in phosphate buffered solution (0.05 M KH_2PO_4 / 5.6 mM NaOH, pH = 6)[5] to determine if CO_2 or salt conditions exhibited an impact on the reaction. In addition, water samples were deoxygenated with a stoichiometric amount of ammonium bisulfite. A minimum was used in order to prevent reaction with H_2S in solution.

Sulfide scavenging products were qualitatively analyzed via GC of dichloromethane extracted material from aqueous test solutions. Reaction rates were evaluated under pseudo-1st order conditions such that the total product:H_2S mole ratio was approximately $\geq$ 10:1. Correspondingly, the residual H_2S was measured at specified time intervals using a HACH®[6] Hydrogen Sulfide Test Kit[7] via dilution of a 0.100 mL sample aliquot in 10.0 mL DI water in order to bring the sulfide concentration within the range of the test protocol. The reaction was monitored over the course of ~ the first 2 – 3 half-lives if the reaction went to completion.

Field Test Conditions

Baseline H_2S was measured in each of the test fields using gas analysis tube at the nearest accessible point to the wellhead. Production levels of gas, oil, and produced water were obtained, as well as water chemistry when available. The data were fit to an overall kinetic algorithm according to a nonlinear least-squares regression analysis using the Microsoft® Excel Solver[8]. Performance in the field was evaluated via measurement of both the dose rate of the scavenger chemical (in gallons per day, gpd and liters per day, lpd) and residual H_2S.

RESULTS

Hydrogen Sulfide Scavenging of the Non-Triazine Product

In the development of the product, it was of interest to investigate the mechanism and rate of reaction of the new scavenger product with H_2S in detail. Sulfide scavenging was followed by measuring the residual H_2S concentration at specific time intervals over a wide concentration range (2,500 – 20,000 ppm as product), over a large pH range applicable to oilfield applications (5 – 9.5 ~ 5 – 10), and over a wide temperature range (7 – 50 ^{o}C). In each case, the initial H_2S concentration was ~ 50 ppm. Aqueous solutions were buffered with either sodium bicarbonate and slightly purged with either N_2 or CO_2 in order to achieve a specific pH and to remove oxygen (< 0.1 ppm measured). In contrast to some previously reported patent and literature data[9,10,11,12], experiments were conducted under pseudo-1st order conditions in total product (i.e., [Product] > 10[H_2S]), which provides the pseudo-1st order rate constants at different concentrations, corresponding to an observed rate of H_2S scavenging according to:

$$[Scavenger\ Product]_{total} + H_2S \overset{k_5}{\rightarrow} Sulfur\ containing\ products$$

where,

$$\frac{-d[H_2S]}{dt} = k_5[H_2S]_{total}[Scavenger\ Product]_{total}$$

$$= k_{scavenging}[H_2S]_{total} \quad (1)$$

and,

$$k_{scavenging} = k_5[Scavenger\ Product]_{total} \quad (2)$$

$$[H_2S]_{total} = [H_2S] + [HS^-] + [S^{2-}] \quad (3)$$

Linear least-squares regression analysis over ~ 3 half-lives at pH 5 shows that the reaction of H_2S with the new product is 1st order with respect to H_2S concentration (since $[H_2S]_{total} \approx [H_2S]$ at pH =5; pKa_{1H2S} = 6.9)) and the rate increases with increasing product concentration (Figure 1).

Figure 1– Psuedo-1st Order Reaction with H_2S as a function of the New Scavenger Product Concentration at pH = 5

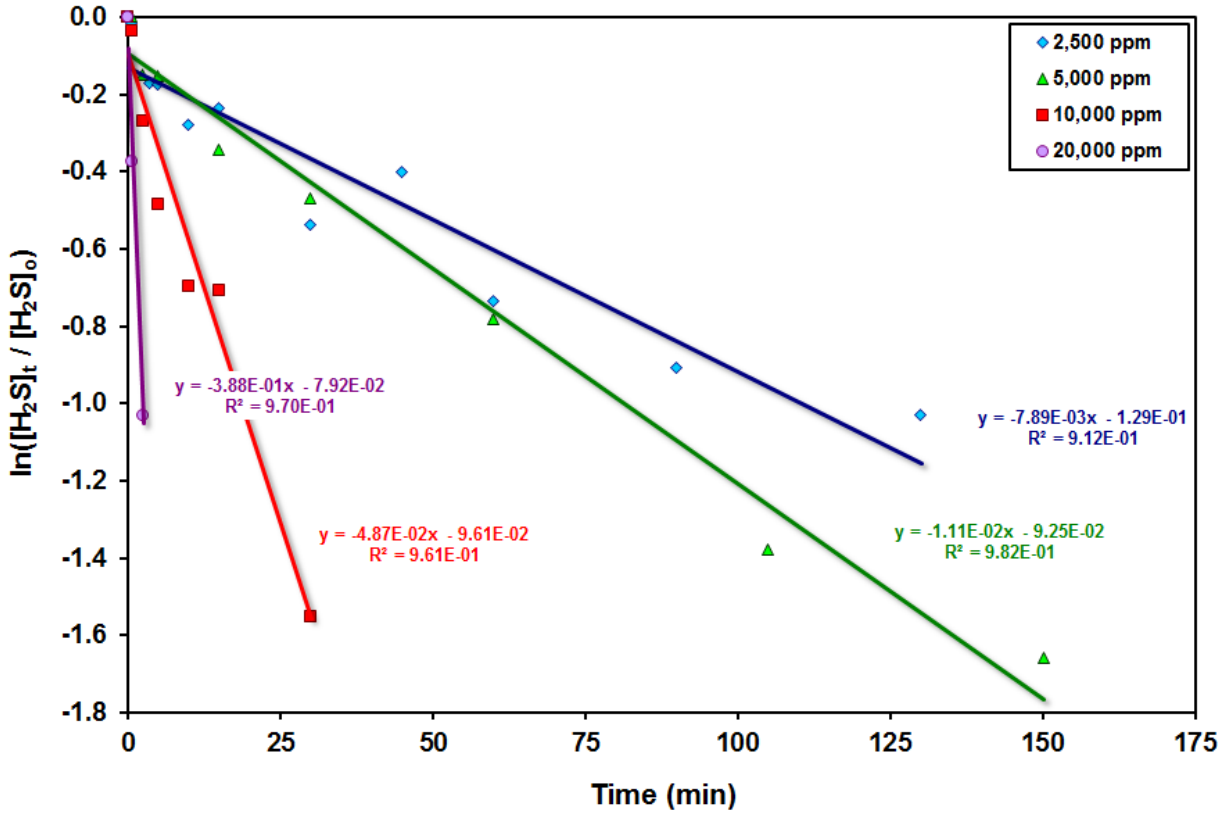


Table 1 – Pseudo-1st order Rate Constants for H_2S scavenging by the New Scavenger Product (pH 5, T = 22 °C)

Product Dose (ppm)	k_{obsd} (min^{-1})	$t_{1/2}$ (min)
2,500	7.89×10^{-3}	88
5,000	1.11×10^{-2}	62
10,000	4.87×10^{-2}	14
20,000	3.88×10^{-1}	2

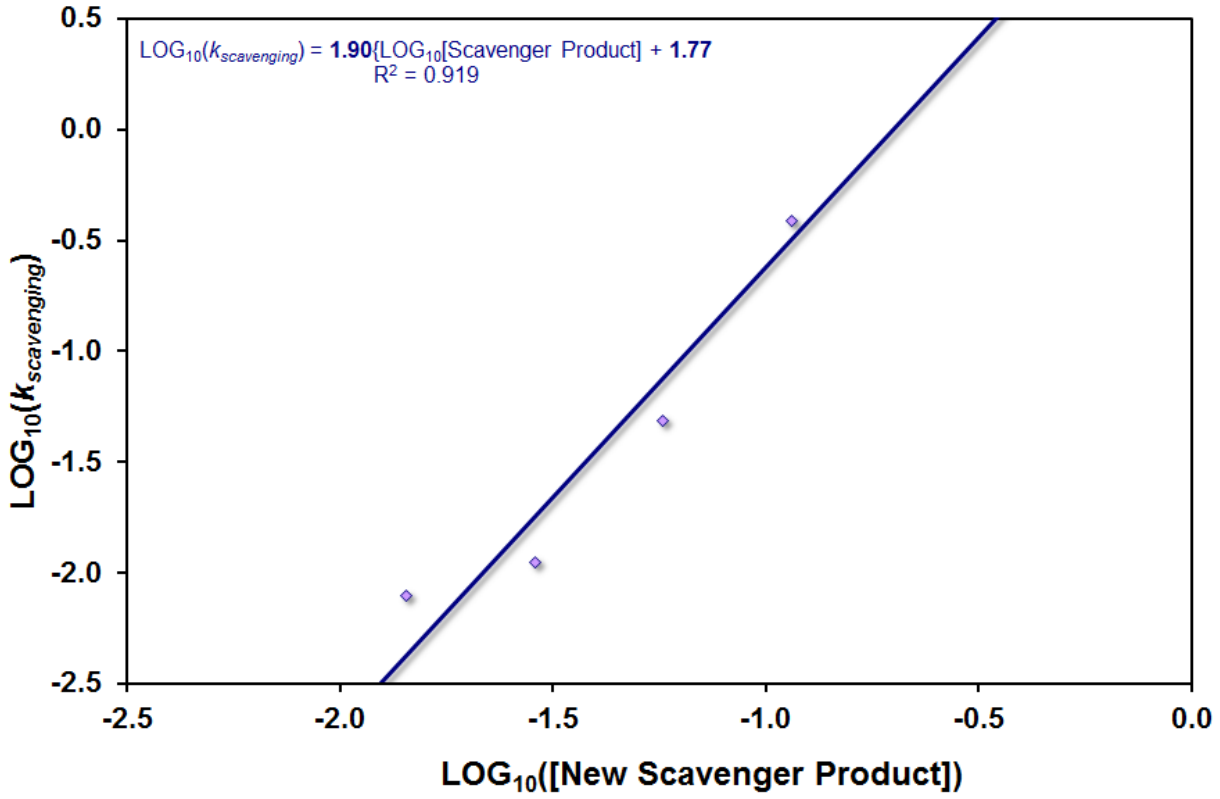
Interestingly, the observed increase in rate as a function of increasing product concentration is non-linear, suggesting the order of reaction with respect to the scavenger product concentration is greater than 1. The order of reaction is readily determined by plotting $LOG_{10}(k_{scavenging})$ vs. LOG_{10}([Scavenger Product]), giving a slope equal to the order of reaction with respect to product concentration and an intercept equal to the concentration independent rate constant, according to:

$$LOG_{10}(k_{scavenging}) = LOG_{10}(k_5[Scavenger\ Product]^n)$$

$$= nLOG_{10}([Scavenger\ Product]) + LOG_{10}(k_5) \qquad (4)$$

As shown in Figure 2, the slope is ~ 2, indicating the reaction is 2nd order with respect to total product concentration and gives a k_5 = 15.8 M^{-1} s^{-1}. This is in contrast with the reaction of H_2S with triazines, which has been reported to be 1st order with respect to triazine concentration.[11]

Figure 2 – Plot of $k_{scavenging}$ vs. New Scavenger Product Concentration



In addition to monitoring the reaction as a function of product concentration, the reaction was monitored as a function of pH at 5,000 ppm over a pH range of 5 – 9.5 at 22 °C. A plot of the logarithm of the observed rate constant, $k_{scavenging}$, vs. pH provides the pH-dependence of the reaction with a slope >0 if there is a dependence.

However, as shown in Table 2, the near zero slope indicates that *the reaction of H_2S with the new scavenger product is not pH dependent, in contrast with the reaction of H_2S with triazines*.

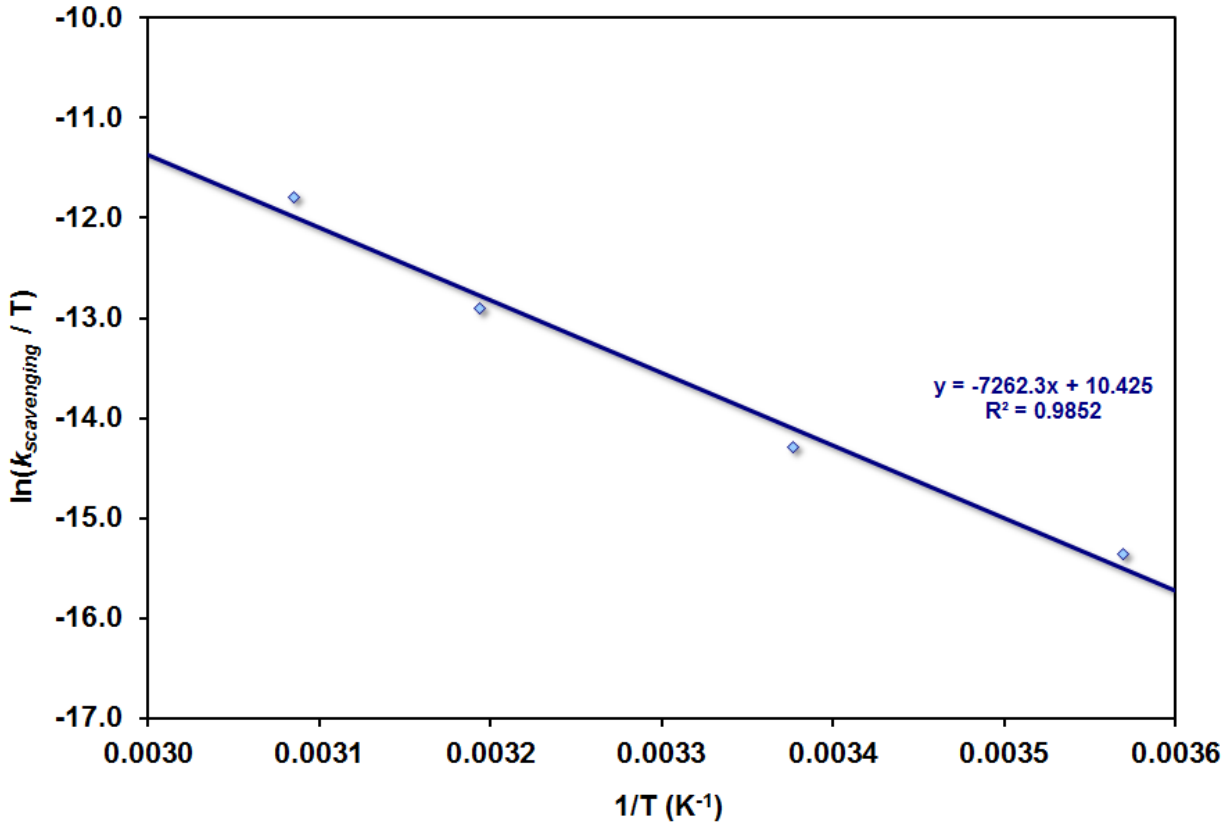
Table 2–pH-Independent Rate Constants for the Reaction of H_2S with New Scavenger Product

pH	Temp (°C)	k_{obsd} (min^{-1})	$t_{1/2}$ (min)
5.0	22	1.11 x 10^{-2}	62
7.2	22	1.30 x 10^{-2}	53
8.4	22	1.56 x 10^{-2}	44
9.4	22	1.24 x 10^{-2}	56
5.0	40	4.68 x 10^{-2}	15
6.1	40	4.77 x 10^{-2}	15

The lack of dependence on pH suggests that H_2S scavenging the new scavenger product could be more versatile for a variety of applications. Further, *the rate of reaction with H_2S is faster than hydrolysis at low pH (~100 times faster at pH = 5), another notable difference with respect to triazines*.

In addition to studying the rate as a function of pH, the H_2S scavenging reaction was monitored as a function of temperature at 5,000 ppm at pH 5. An Eyring plot ($\ln(k_{scavenging} / T)$ vs. 1/T) provides both the activation enthalpy ($\Delta H^{\ddagger}$) and entropy ($\Delta S^{\ddagger}$), from the slope and intercept, respectively. The plot of the data provided activation parameters such that $\Delta H^{\ddagger}$= 60.2 ± 5.4 kJ mol-1 and $\Delta S^{\ddagger}$ = -111 ± 17 J mol^{-1} K^{-1} (Figure 3).

Figure 3 – Eyring Plot for the Reaction of H_2S with the New Scavenger Product



The large, negative entropy is consistent with the overall observed 3rd order reaction (2nd order in scavenger product, 1st order in H_2S), and indicates that the rate is very temperature dependent. This information allows for additional prediction of H_2S scavenging rates over a broader range of temperatures. Hence, for comparison, the calculated scavenging rates ($k_{scavenging}$)and half-lives ($t_{1/2}$) as a function of temperature are listed in Table 6, showing that the H_2S scavenging rate increases dramatically with increasing temperature. Importantly, the scavenging rate is still rapid even at lower temperatures.

Table 3 – Calculated $t_{1/2}$ from Experimental Activation Parameters for H_2S Scavenging by the New Scavenger Product as a Function of Temperature

Temperature (°C)	Calculated k_{obsd} (min^{-1})	Calculated $t_{1/2}$ (min)
-5	1.00×10^{-3}	691
10	4.42×10^{-3}	157
25	1.69×10^{-2}	41
40	5.67×10^{-2}	12
55	1.71×10^{-1}	4.1
70	4.69×10^{-1}	1.5
85	1.18	0.6

Hydrogen Sulfide Phase Partitioning

Recently, Burger et al reported on the development of a "flash partitioning" model that employed Henry's Law approach for the aqueous solubility of hydrogen sulfide and the Peng-Robinson equation of state for the non-aqueous phases to calculate the mole fraction of hydrogen sulfide in a gas, oil, and water phase.[13,14,15,16] Importantly, the model incorporated the effect of ionic strength and pH on the partitioning of H_2S.

In multi-phase systems, the H_2S concentration between the phases is effectively described by partition coefficients. The overall partitioning is a function of the partial pressure of H_2S, as traditionally described by Henry's Law[17,18,19], water phase ionic strength, and water phase pH. An elegant description of the relationship of each of these factors has been recently reported.[13] Utilizing these equations allows for the calculation of the amount of molecular H_2S in each phase if you know the H_2S concentration in one phase, the masses and molecular weights of the phases, and the partition coefficients. In general, this is expressed according to:

$$K_{ow} = \chi_{H2Soil} / \chi_{H2Saq} \quad (6)$$

$$K_{gw} = \chi_{H2Sg} / \chi_{H2Saq} \quad (7)$$

$$K_{go} = \chi_{H2Sg} / \chi_{H2Soil} \quad (8)$$

Where χ_{H2Si} = mole fraction of H_2S in phase *i*.

Once the distribution of H_2S is determined from the model, the influence of ionic strength and pH can be incorporated to determine the relative levels. An additional level of simplification can be utilized if the calculations are performed assuming infinite dilution, since in this case the activity coefficients are equal to unity and, correspondingly, the pH of the water determines the ionic species present. This provides a means to evaluate the equilibrium ionization of H_2S according to:

$$H_2S_{(aq)} \leftrightarrow HS^-_{(aq)} + H^+_{(aq)} \quad (9)$$

$$HS^-_{(aq)} \leftrightarrow S^{2-}_{(aq)} + H^+_{(aq)} \quad (10)$$

Pressure and temperature effects need to be taken into account when determining the partitioning. Nontheless, as a simplified example at STP, an oilfield (Oilfield A) with a production quantity of 250 MSCF (7,100 m^3) gas, 415 barrels of oil produced per day (BOPD; 66 m^3 per day), and 40 barrels of water produced per day (BWPD; 6.4 m^3 per day) at pH 6.5, with an H_2S_g concentration of 150 ppm_v, results in approximate partitioning values as shown in Table 4. The calculations indicate a total of 1.9 kg of H_2S produced per day.

Table 4 : H_2S partitioning between gas, oil, and water phases according to a model production scenario.

Phase	Total H_2S concentration
Gas	150 ppm_v
Oil	7 ppm_w
Water	3 ppm_w (including ionization)

Modeling and Simulation of the New Hydrogen Sulfide Scavenger for Oilfield Systems

The calculations described above allow for the estimation of the amount of scavenger one would require per day to remove H_2S. However, simply dosing the calculated amount into the system does not necessarily indicate that it will be sufficiently remediated. As described above, it is equally important is an understanding of the mechanism and rates of H_2S scavenging. The kinetic equations allow for the determination of the scavenging rate within an aqueous phase. However, if H_2S is partitioning among 3 phases, it is important to consider the following rates for an overall assessment of the scavenging rate within a system:

- Scavenging rate in the water phase (aqueous scavenging rate; if applicable)
- Scavenging rate in the oil phase (if applicable)
- Scavenging rate in the gas phase (if applicable)
- H_2S mass transfer rate between phases
- Scavenger mass transfer rate between phases (if applicable)

Complex kinetic profiles will exist if the scavenger used is able to react with H_2S in both the oil and water phases and is soluble in both phases. These can be fit according to a series of differential equations via nonlinear least-squares regression algorithms. Gas phase reactivity is primarily a consideration if the scavenger has a particularly low vapor pressure (atypical) or if it is injected as droplets into the gas stream.[20]

In the present case, the new scavenger is insoluble in the oil phase and its reaction with H_2S in the gas phase is sufficiently slow so that it can be effectively ignored. Therefore, the two primary considerations are the aqueous scavenging rate and H_2S mass transfer rate between phases. As depicted in Figure 4, mass transfer between the gas and aqueous phases can be simulated by a penetration model via flux across the gas-liquid film boundaries and the diffusion rates of H_2S within the corresponding medium.

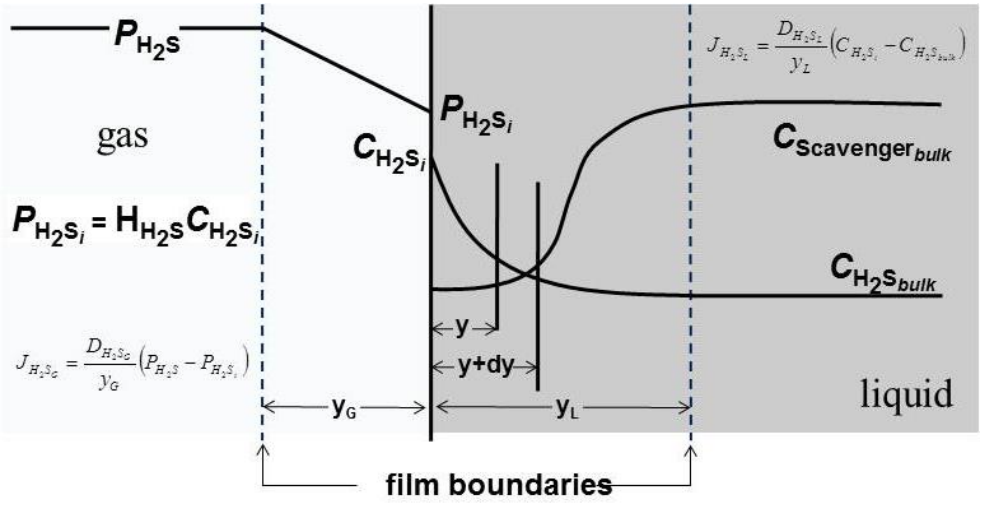


Figure 4 : Depiction of Mass Transfer of H2S Between a Gas an Liquid Phase

D_{H2Si} is the diffusion rate of H_2S in the corresponding medium, *i* (1.60 x 10^{-5} cm^2/sec in water).[5] In effect, the rate of removal of H_2S is estimated using a model that combines calculation of mass transfer resistance of H_2S through the phases and the rate of reaction in the aqueous phase. This can be estimated if it is assumed that H2S undergoes an absorption process from the gas or oil phase into the aqueous phase, followed by an irreversible scavenging reaction according to equations 11 – 13:

$$H_2S_{(g)} \leftrightarrow H_2S_{(aq)} \quad (11)$$

$$H_2S_{(g)} \leftrightarrow H_2S_{(oil)} \quad (12)$$

$$H_2S_{(aq)} + [Scavenger]_{(aq)} \rightarrow Products_{(aq)} \quad (13)$$

While the overall rates are affected by diffusion and interfacial films, calculation of the equilibrium values provides boundaries for the reversible diffusion across each layer and the scavenging rates provide a means to calculate the overall flux.

An important parameter that characterizes how the mass transfer is affected by the chemical reaction is the reaction-diffusion modulus, or Hatta number:[21]

$$H_a = \frac{\sqrt{k_{scav} D_{H2S}}}{k_l} \quad (14)$$

Using the production values for Oilfield A listed above, and the rate of scavenging reaction for the new scavenger chemistry, one can calculate the overall concentration in each phase as a function of distance from injection / chemical contact and chemical dose rate, as shown in Figure 5.

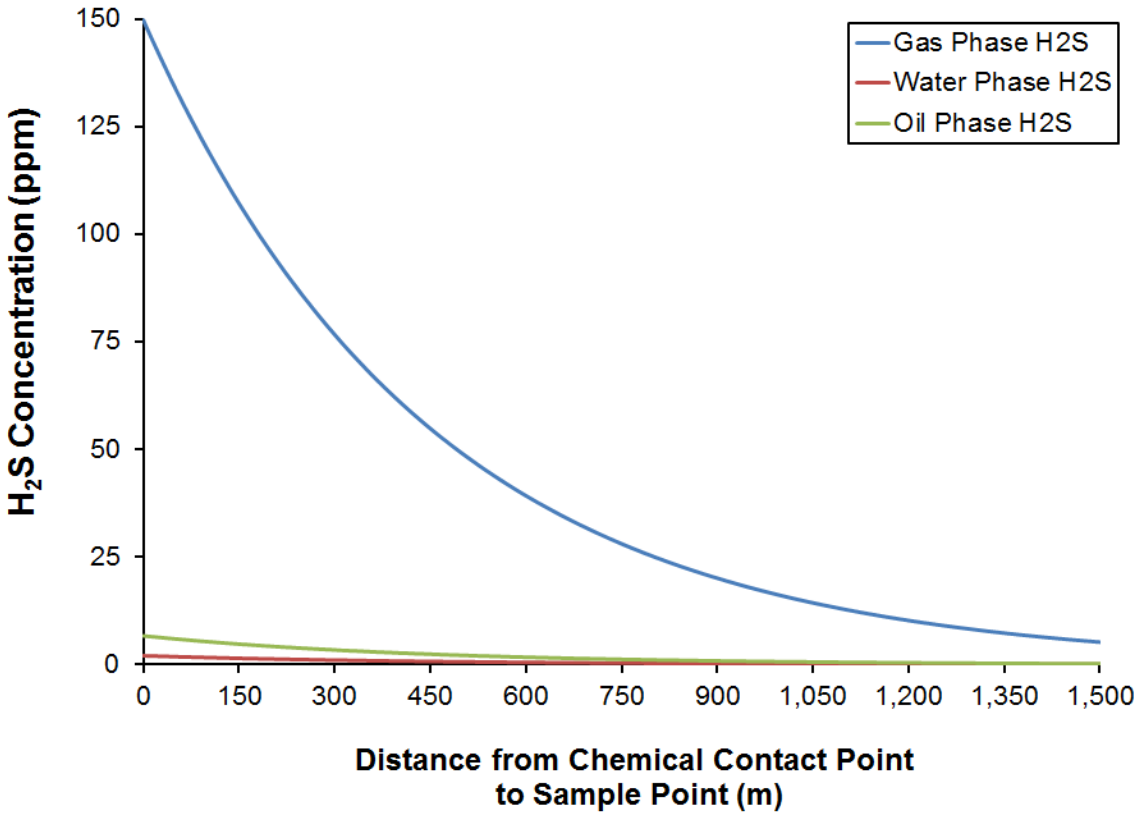


Figure 5 : Calculated H_2S Concentrations in Each Phase as a Function of Distance from Chemical Contact Point

Application of the Scavenging Model for the New Hydrogen Sulfide Scavenger for Oilfield Systems

The model above was then utilized to target an initial dose rate for removal of H_2S in the Oilfield A production system. Only the gas phase H_2S levels were able to be measured during the trial. However, since the model accounts for H_2S mass transfer between each phase and the scavenging reaction occurs only in the aqueous phase, any reduction of H_2S in the gas phase is a function of these two parameters.

As shown in Figure 6, the measured H_2S levels at each location fit well with that calculated based upon the chemical dose rate, scavenging rate, and estimate of mass transfer rates.

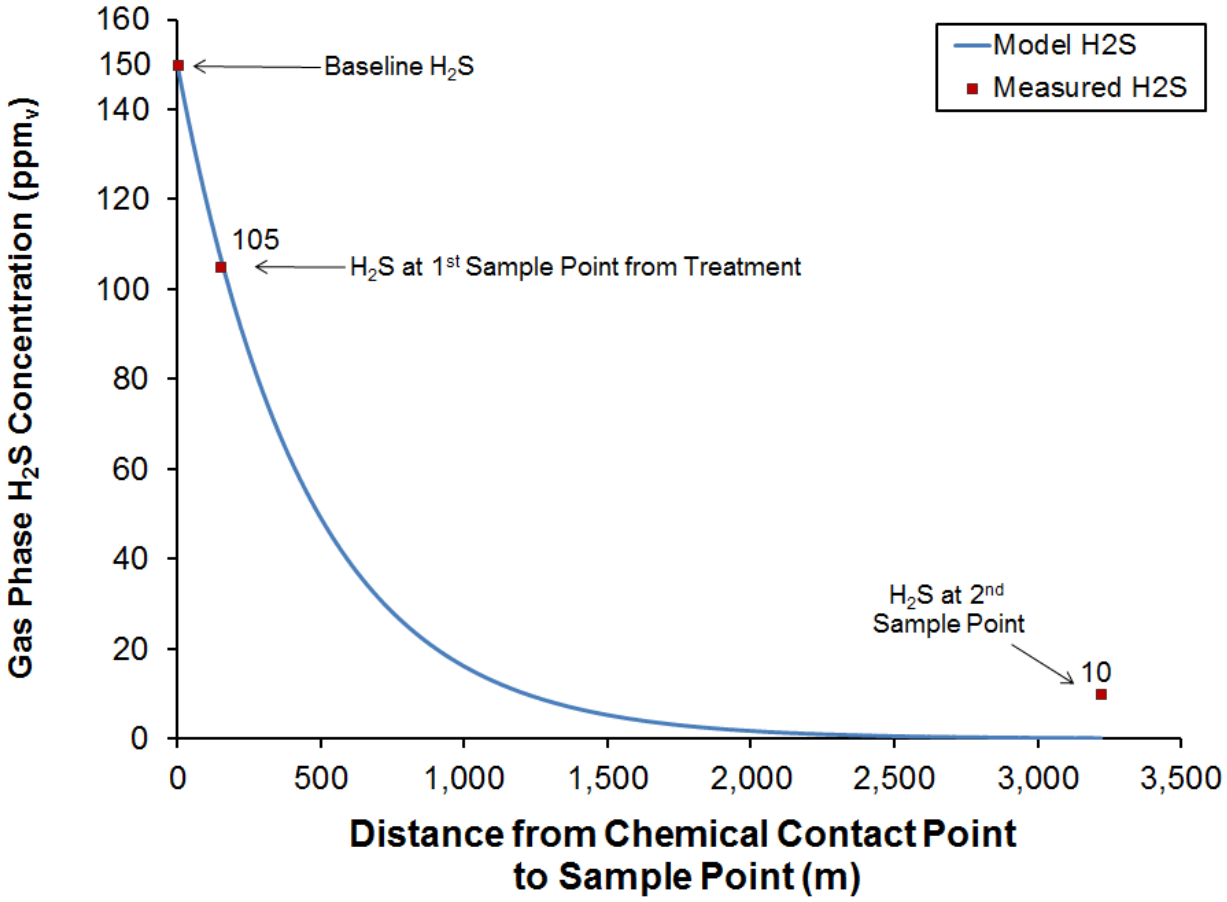


Figure 6 : Measured vs Estimated H_2S Levels in the Gas Phase via Application of the New Scavenger Chemistry in Oilfield A

Downhole injection of chemical H_2S scavengers is sometimes necessary in order to minimize the potential for increased corrosion of tubing, production facilities, iron sulfide deposition, and injection or wellbore plugging. Although triazines are a commonly utilized chemistry, operators can experience problems due to the formation of insoluble materials and the potential for scale deposits.[4]

Oilfield B exhibited high levels of H_2S but did not have topside facilities that could effectively handle the H_2S levels in the gas. Production levels were 240 MSCF (6,800 m^3), 240 BOPD (38 m^3), and 90 BWPD (14 m^3), with 2,200 ppm_v H_2S. Due to its non-scaling nature, the new scavenger chemistry was applied downhole and the H2S levels measured topside. The total distance from downhole chemical contact to

the point of measurement was ~8,100 m. As shown in Figure 7, the scavenging model provided a recommended dose rate to reduce the H_2S to 10 ppm_v.

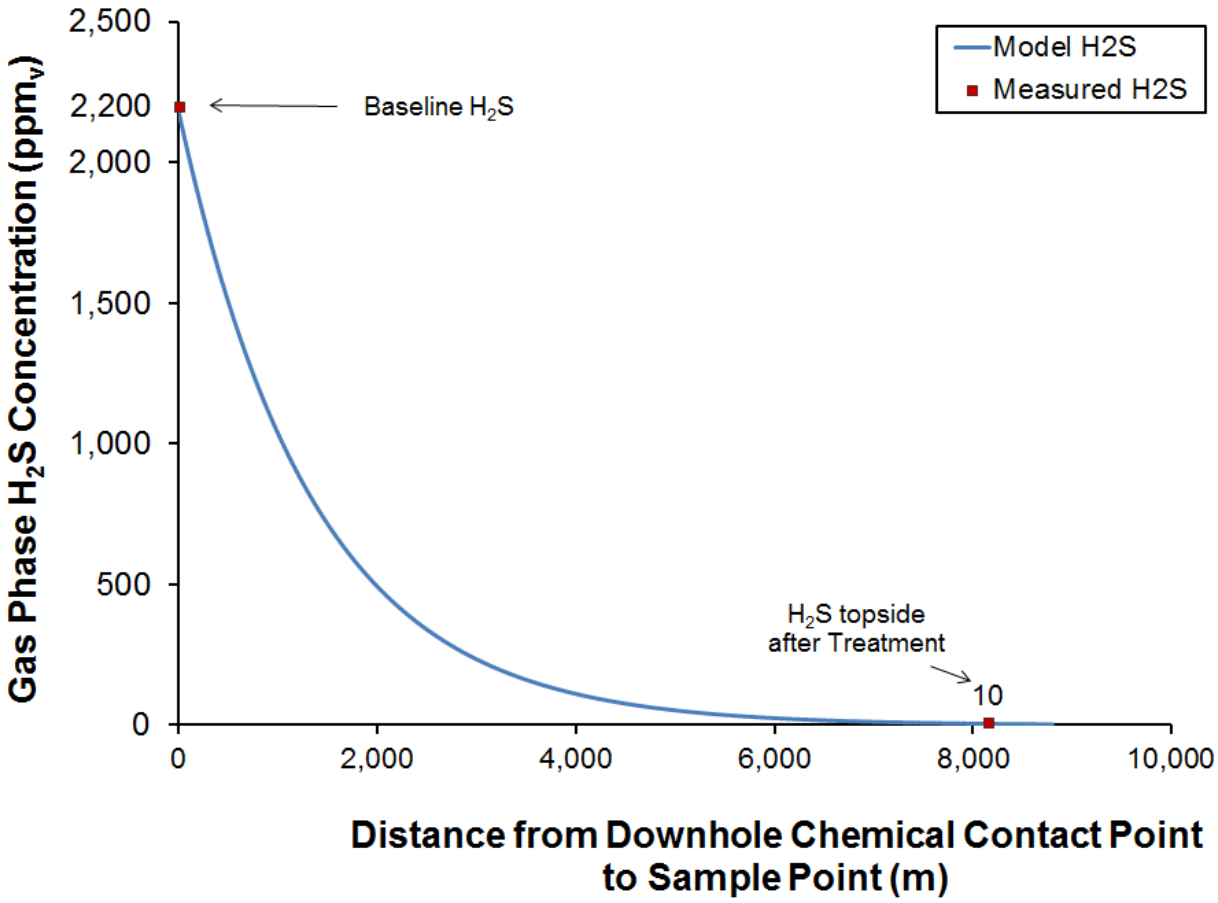


Figure 7 : Measured vs Estimated H_2S Levels in the Gas Phase via Downhole Application of the New Scavenger Chemistry in Oilfield B

Similarly, Oilfield C exhibited levels of H_2S of 9,500 ppm_v in the gas phase. Production levels were 134 MSCF, 270 BOPD, and 210 BWPD. Downhole injection of the new scavenger chemistry was applied the H_2S levels measured topside. The total distance from downhole chemical contact to the point of measurement was ~5,100 m. As shown in Figure 8, the scavenging model provided a recommended dose rate to reduce the H_2S to 800 ppm_v.

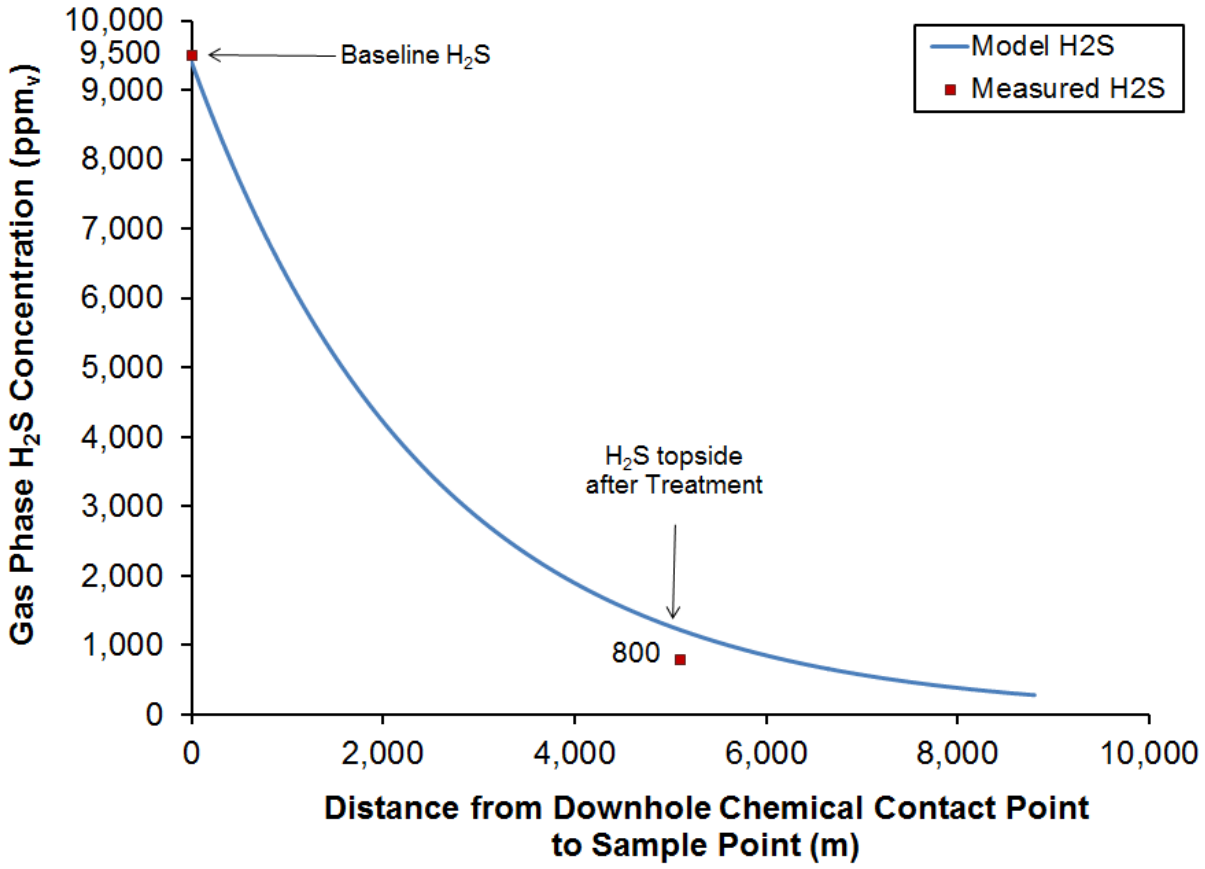


Figure 8 : Measured vs Estimated H_2S Levels in the Gas Phase via Downhole Application of the New Scavenger Chemistry in Oilfield B

Table 5 compares the chemical dose rates and calculated Hatta numbers for each production situation, indicating that the scavenging reaction for this chemistry is intermediate between proceeding predominantly near the gas-liquid interface (typically $H_a > 2$) and the bulk solution ($H_a < 0.2$). This shows that the chemical dose effectively creates a pseudo-first order reaction rate within the water phase that makes the scavenging reaction competitive with mass transfer of H_2S from the gas or oil phase to the water phase.

Table 5: Comparison of Field Performance and Hatta Numbers for the New Scavenger Chemistry

System	Initial H_2S_g (ppm_v)	Final H_2S_g (ppm_v)	Hatta number (Ha)	Scavenger Dose Rate (recommended)	Scavenger Dose Rate (actual)
Oilfield A	150	10	1.47	1.5 gpd (5.7 lpd)	2 gpd (7.6 lpd)
Oilfield B	2,200	10	1.20	19 gpd (72 lpd)	26 gpd (99 lpd)
Oilfield C	9,500	800	1.57	53 gpd (201 lpd)	59 gpd (224 lpd)

Conclusions

A new non-triazine H_2S scavenger has been developed and successfully applied to reduce H_2S levels in oilfield production systems, even via downhole injection. This was achieved by understanding the partition of H_2S into each phase, allowing the calculation of total H_2S produced within a system. Further, a simplified reaction model and algorithm has been developed to model and predict the scavenging efficiency and predictably optimize chemical dose rate.

In order to achieve this, an understanding the phenomenological kinetic equations for the scavenger of choice is required and, in particular, the rate in the phase or phase(s) it scavenges is important. This will dictate the complexity of the model used and the role mass transfer has on each of the potentially competing rates.

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