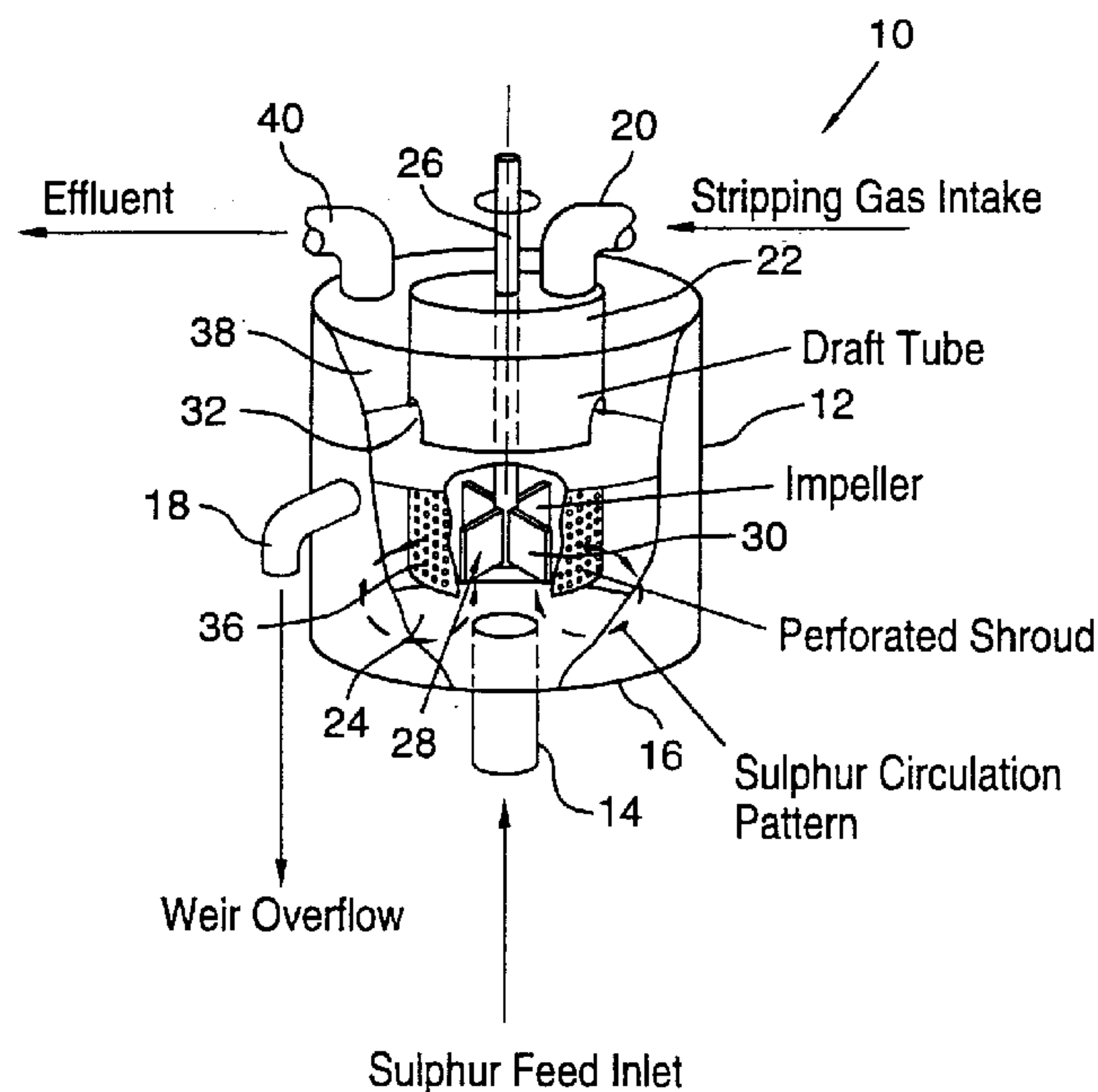




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(54) **DEGAZAGE DE SOUFRE LIQUIDE**
(54) **LIQUID SULFUR DEGASSING**



(57) Procédé de dégazage du soufre liquide en vue d'en extraire rapidement et efficacement l'hydrogène sulfuré et les polysulfures d'hydrogène au moyen d'un dispositif de roue à palettes et tambour qui diffuse dans le soufre liquide un gaz extracteur, qui peut être de l'air ou de la vapeur, sous forme de petites bulles, en présence d'un catalyseur fortement basique de Bronsted-Lowry, de telles conditions assurant un transfert de masse rapide et efficace. Ce dégazage peut être combiné à une épuration du gaz résiduaire par procédé Claus utilisant ledit gaz résiduaire comme gaz extracteur. De plus, l'épuration du gaz résiduaire par procédé Claus peut se faire dans le soufre liquide.

(57) Liquid sulfur is degassed to remove hydrogen sulfide and hydrogen polysulfides in rapid and efficient manner using a shroud and impeller combination which distributes a stripping gas, which may be steam or air, as small bubbles in the liquid sulfur in the presence of a strong Bronsted-Lowry base catalyst and provides parameters which provide rapid and efficient mass transfer. Such degassing may be combined with Claus plant tail gas clean up, by using the tail gas as the stripping gas. In addition, Claus plant tail gas clean up may be effected in liquid sulfur.



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(54) Title: LIQUID SULFUR DEGASSING			
(57) Abstract			
Liquid sulfur is degassed to remove hydrogen sulfide and hydrogen polysulfides in rapid and efficient manner using a shroud and impeller combination which distributes a stripping gas, which may be steam or air, as small bubbles in the liquid sulfur in the presence of a strong Bronsted-Lowry base catalyst and provides parameters which provide rapid and efficient mass transfer. Such degassing may be combined with Claus plant tail gas clean up, by using the tail gas as the stripping gas. In addition, Claus plant tail gas clean up may be effected in liquid sulfur.			

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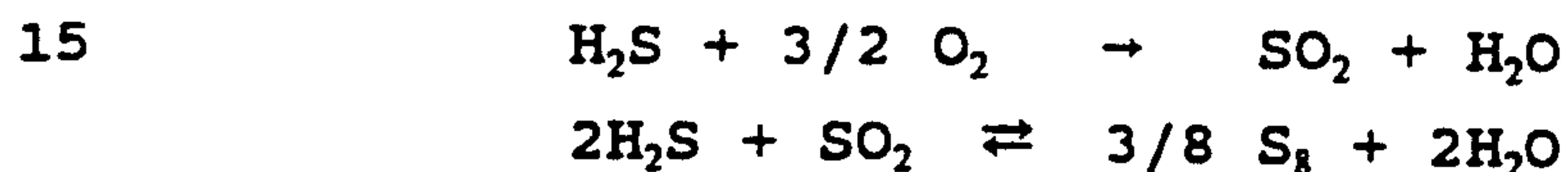
TITLE OF INVENTION
LIQUID SULFUR DEGASSING

FIELD OF INVENTION

5 The present invention is directed to the removal of hydrogen sulfide and hydrogen polysulfides from liquid sulfur in rapid and efficient manner.

BACKGROUND OF THE INVENTION

10 Hydrogen sulfide is a product which may be recovered during the production of natural gas, refining operations, or as a byproduct of a number of industrial operations. In many cases the hydrogen sulfide is converted to elemental sulfur by the Claus process. The basic chemistry of the Claus process is as follows:



20 Hydrogen sulfide is, to a limited degree, soluble in liquid sulfur. This dissolved hydrogen sulfide may react with the sulfur diradical chain species to form hydrogen polysulfides, as follows:



25 Liquid sulfur produced by the Claus process may contain from 50 to 550 ppmw of hydrogen sulfide species. The hydrogen sulfide and hydrogen polysulfides slowly reach an equilibrium in the liquid sulfur which is substantially influenced by temperature. Over time, the hydrogen sulfide diffuses out of the liquid sulfur, or in cases where the sulfur has been solidified, out of the solid product. Under ambient conditions, it can take 30 weeks for the hydrogen polysulfides to decompose to sulfur and hydrogen sulfide. The release of these hydrogen sulfide emissions may create a variety of nuisance, environmental and safety concerns, if not properly controlled. Examples of such concerns are:

- 35 - hydrogen sulfide concentrations can build up to lethal and explosive limits in the

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headspace when liquid or formed sulfur is stored or shipped within an enclosed space.

- low levels of hydrogen sulfide emissions coming off sulfur storage facilities can be environmentally objectionable due to the low odour threshold of only 0.003 to 0.02 ppmw (ref. 1).
- liquid sulfur is often processed into a solid formed product for transportation purposes, and very often hydrogen sulfide is released to the atmosphere during such forming, storage and transportation operations.

Over the years many systems have been designed to remove hydrogen sulfide and hydrogen polysulfides from liquid sulfur. A review of the prior art reveals that some systems favour the use of reagents or catalysts to effect the removal, while others use air or gas and agitation of the liquid sulfur to promote the release of hydrogen sulfide. It has been found that the use of some catalysts or reagents, such as urea, may assist in the removal of hydrogen sulfide, but it is believed that the friability of formed sulfur product may be impacted negatively by these compounds. Many systems use large vessels to hold the liquid sulfur and sparge air or an inert gas through the liquid sulfur to provide circulation and a carrier gas to remove the hydrogen sulfide from the liquid sulfur. The residence time for such processes generally ranges from several hours to, in some cases, several days. The inert gas may consist of steam or nitrogen, and is generally used to reduce the risk of fire or explosion. In addition, such prior art systems often result in residual hydrogen sulfide and hydrogen polysulfide levels of at least about 50 ppmw.

The prior art also comprises mechanical systems with air or gas sparging which decrease the residence time of the liquid sulfur by increasing the degree of agitation.

U.S. Patent No. 4,612,020 describes a system where liquid sulfur is sprayed into two or more chambers, which are joined in series, and the liberated hydrogen sulfide is scavenged from the head space by an inert gas, such as nitrogen. The residence time for the liquid sulfur as noted in this patent varies from 12 to 32 hours. Such residence times require extremely large degassing vessels to be employed. This residence time can be decreased to about 7 hours by using hetrocyclic amine catalysts. The present invention employs a new approach to the degassing of liquid sulfur, which in turn facilitates dramatically reduced residence times. While reference was made earlier to liquid sulfur produced by a Claus plant, the present invention may be applied to liquid sulfur produced by other processes as well.

Searches of the prior art have revealed several patents relating to the removal of hydrogen sulfide from liquid sulfur and the use of various catalyst materials in such procedures, as described above. As a result of such searches, the applicants are aware of the following references:

USP 3,364,655 USP 3,447,903 USP 3,807,141
USP 4,131,439 USP 4,612,020 USP 4,755,372
USP 4,844,720 USP 4,849,204 USP 5,030,438
USP 5,080,695 GB 1,067,815 CA 964,040
FR-A-2185587 (=GB-A-1,433,822) and DD-A-292635
describe processes for the removal of hydrogen sulfide from liquid sulfur by the introduction of a stripping gas beneath the surface of the liquid in the presence of an agitating means. While both documents additionally describe a cylindrical liquid-flow regulating part positioned around the agitator, which may have openings in the lowest part, as described in DD-A-292635, there is no flow of a liquid-gas mixture from the interior to the exterior of a cylindrical shroud through openings, as required herein.

In addition, the reaction of hydrogen sulfide and sulfur dioxide in liquid sulfur in columns containing static catalyst beds is known from EP-A-0030447.

SUMMARY OF INVENTION

5 The present invention provides an improved procedure for the removal of hydrogen sulfide and hydrogen polysulfides from liquid sulfur which enables the normal degassing time to be reduced from hours or days to minutes while achieving residual H_2S and H_2S_x levels below
10 5 ppmw. The procedure of the invention may employ the gas-liquid contactor generally described in USP 5,174,973 for the removal of a gaseous component from a gas stream in an aqueous system using parameters developed for

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aqueous systems, but modified for the removal of hydrogen sulfide and hydrogen polysulfides from liquid sulfur.

In one aspect of the present invention, there is provided a method of removing hydrogen sulfide and hydrogen polysulfides from liquid sulfur, which comprises providing a rotary impeller comprising a plurality of blades at a submerged location in the liquid sulfur surrounded by a shroud through which are formed a plurality of openings, feeding a stripping gas for hydrogen sulfide to the submerged location while providing a Bronsted-Lowry base catalyst for the conversion of hydrogen polysulfides to hydrogen sulfide thereat, rotating the impeller about a substantially vertical axis at a speed sufficient to draw liquid sulfur into the interior of the shroud and to distribute the stripping gas as bubbles in the liquid sulfur to the interior of the shroud and to form a gas-liquid mixture of bubbles of the gas in the liquid sulfur contained within the shroud, flowing the gas-liquid mixture from within the interior of shroud through and in contact with the openings to external of the shroud, and removing the stripping gas from the liquid sulfur.

In one preferred aspect, the present invention provides a method of removing hydrogen sulfide and hydrogen polysulfides from liquid sulfur, which comprises a plurality of steps. A rotary impeller comprising a plurality of blades is provided at a submerged location in the liquid sulfur surrounded by a shroud through which are formed a plurality of openings. A stripping gas for stripping hydrogen sulfide from the liquid sulfur is fed to the submerged location. The impeller is rotated about a substantially vertical axis at a speed corresponding to a blade tip velocity (v_t) of at least about 150 in/sec (at least about 4 m/s), preferably at least about 350 in/sec (at least about 9 m/s), so as to draw liquid sulfur into the interior of the shroud and to generate sufficient

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shear forces between the impeller and the plurality of openings in the shroud to distribute the stripping gas as bubbles in the liquid sulfur to the interior of the shroud and to effect intimate contact of the stripping gas and the liquid sulfur at the submerged location so as to form a gas-liquid mixture of bubbles of the stripping gas in the liquid sulfur contained within the shroud while effecting shearing of the gas-liquid mixture within the shroud. The gas-liquid mixture is flowed from within the interior of the shroud through and in contact with the openings to external of the shroud at a gas velocity index (GVI) of at least about 4 per second per opening in the shroud, preferably at least about 18 per second per opening, so as to effect further shearing of the gas-liquid mixture and further intimate contact of the stripping gas and the liquid sulfur.

The gas velocity index (GVI) is determined by the expression:

$$GVI = \frac{QP}{4nA^2}$$

where Q is the volumetric flow rate of gas into the impeller (m³/s), n is the number of openings in the shroud, A is the area of the opening (m²) and P is the length of the perimeter of the opening (m).

The method is carried out:

(a) at an Effective Shear Index (ESI) value of about 1 to about 6000 as determined by the relationship:

$$ESI = \frac{GVI}{v_i} \times \frac{(D_s - D_i)}{2} \times 100$$

where D_s is inside diameter (m) of the shroud and D_i is the outside diameter (m) of the impeller,

(b) at a Shear Effectiveness Index (SEI) value of about 1 to about 25 as determined by the relationship:

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$$SEI = \frac{Q}{\pi h D_i V_i} \times 100$$

where h is the height of the impeller (m), and

(c) at a Density Index (DI) value of about 1 to about 8 as determined by the relationship:

$$DI = \frac{Q}{Q_i} \left[\frac{\rho_L - \rho_g}{\rho_{H_2O} - \rho_{air}} \right]$$

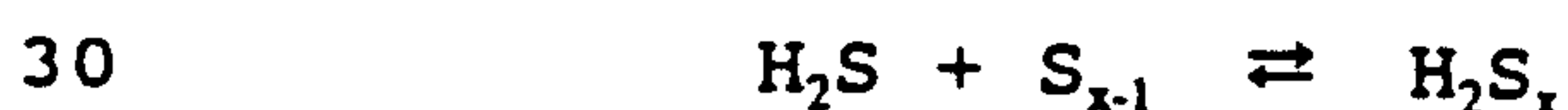
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where Q_i is the self-induction flow rate of air into water (m^3/s), ρ_L is the density of the liquid sulfur (kg/m^3), ρ_g is the density of the gas (kg/m^3), ρ_{H_2O} is the density of water (kg/m^3) and ρ_{air} is the density of air (kg/m^3).

10 The accelerated and efficient degassing process provided herein enables equipment size to be significantly decreased for a given mass throughput and avoids the necessity and expense of constructing large holding tanks or storage pits to accommodate the
15 traditional longer degassing residence times. Since small vessels suffice to give the needed residence time, the equipment necessary for effecting the present invention may be very compact in relation to its production rate, and requires a small capital investment
20 and possesses low operating costs.

The process of the invention may be used to effect hydrogen sulfide and hydrogen polysulfide degassing of individual batches of liquid sulfur in a treatment vessel, or, alternatively, to effect degassing of a
25 continuous feed of liquid sulfur to a treatment vessel or a series of treatment vessels.

In the process of the invention, the removal of hydrogen sulfide from the liquid sulfur produced by the stripping gas shifts the equilibrium of the equation:



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to the left, causing decomposition of hydrogen polysulfides. This reaction is catalyzed, as outlined below, by the presence of a Bronsted-Lowry base, with stronger Bronsted-Lowry bases causing a greater catalytic effect. Such Bronsted-Lowry base may be provided by utilizing steam as the stripping gas.

In general, the hydrogen sulfide and hydrogen polysulfide stripping operation is effected in the presence of a strong Bronsted-Lowry base catalyst soluble in the liquid sulfur. Such catalyst may be provided to the interior of the shroud and impeller combination in any convenient manner. For example, the catalyst may be introduced into the liquid sulfur flowing into a treatment vessel and/or may be introduced into the vessel, so as to be transported to the submerged location by the general circulation of liquid sulfur. Alternatively, the catalyst may be transported by any other convenient manner to the liquid sulfur at the submerged location. For example, the catalyst may be injected into the stripping gas or may be injected directly into the vortex of liquid sulfur formed inside the shroud. In this latter procedure, the difference in temperature generally existing between the stripping gas and the liquid sulfur may promote stripping of hydrogen sulfide in the presence of the catalyst. When the stripping operation is carried out in this way, a temperature differential between the cooler stripping gas and liquid sulfur of at least about 20°C is maintained. In general, the stripping gas may have a temperature of at least about 20°C and preferably less than about 100°C.

During the course of a series of experiments in two different sulfur processing plants to determine the efficacy of an impeller and shroud combination for the agitation of liquid sulfur and the stripping of hydrogen sulfide, it was observed that the addition of steam in one of the plants to the stripping gas stream which was

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intimately and vigorously contacted with the sulfur facilitated rapid decreases in the concentration of hydrogen polysulfides. Two different reaction vessels were used in the tests in each of the two sulfur plants, but each reaction vessel was designed in the manner illustrated in Figure 1 and described below. In both series of tests conducted at sulfur plants A and B, when air alone was passed through the contacting system, the free hydrogen sulfide was stripped from the liquid sulfur with a half-life of approximately one minute or less, and the rate of reaction for the removal of the hydrogen polysulfides ranged from a half life of 40 to 60 minutes. However, when steam from plant A was contacted with the liquid sulfur, the rate of reaction for the removal of the hydrogen polysulfides increased to a half-life of less than 6 minutes. Subsequent experiments using different proportions of steam to air resulted in a half-life of about 9 to 15 minutes. In all cases, where steam was employed, the hydrogen sulfide was rapidly stripped from the liquid sulfur, with an estimated half-life of 60 seconds or less.

When this series of experiments was repeated under more readily controlled conditions at sulfur plant B, using a smaller reactor system, it was discovered that the steam did not have the same effect as that used in sulfur plant A. In fact, the effect of steam appeared to be relatively minor, and once again, the half-lives for the decomposition of hydrogen polysulfides were in the order of 30 to 50 minutes, depending on experimental conditions. However, the period represents a considerable improvement over the 3 to 6 hours observed using an inert gas for the stripping.

A careful investigation lead to the finding that certain organic amines were used in the treatment of boiler water at gas plant A, but were not used at gas plant B, leading to the surprising and unexpected

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discovery that these additives, although present in only trace amounts in the steam, increased the reaction rate to half lives of 1 minute or less, in very great contrast to prior art, and even to our earlier results.

5 Almost any amine, including ammonia, promotes the rapid conversion of hydrogen polysulfides to hydrogen sulfide, which then are readily and efficaciously removed by the contactor system provided herein. It is observed that such amine materials have been used and are recorded
10 in prior art, but the half lives reported for the degassing process have been significantly longer than those noted herein. This surprising and unexpected discovery that a combination of vigorous agitation and stripping, such as with a contactor operating under the
15 conditions described herein, with the very high mass transfer rates, together with the addition of trace amounts of Bronsted-Lowry bases enables a process to be designed which will degas liquid sulfur much more quickly and with reduced equipment size compared to that of
20 competing systems, because the residence time for the sulfur can be significantly shortened.

It was observed during the series of experiments at sulfur plant B that steam, in effect, played a relatively minor role in the change in the removal of the hydrogen
25 polysulfides over that of air. The addition of a mixture of morpholine and cyclohexylamine resulted in half lives for the decomposition of hydrogen polysulfide in the order of 25 seconds or less. An analysis by FTIR showed the catalyst, water and volatile impurities also are
30 rapidly and nearly completely removed, resulting in a very pure sulfur product.

BRIEF DESCRIPTION OF DRAWINGS

Figure 1 is a schematic perspective view, with parts broken away, of a liquid sulfur degassing device,
35 provided in accordance with one embodiment of the invention;

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Figure 2 is a FTIR spectrum of liquid sulfur showing residual concentrations of H_2S and H_2S_x prior to treatment;

Figure 3 shows in graphical manner the decrease in concentration of H_2S and H_2S_x in a pilot plant contactor operating in accordance with one embodiment of the present invention using an air stripping gas in the absence of catalyst;

Figure 4 shows the concentration of hydrogen sulfide with time for the effluent from a pilot scale contactor operating in accordance with one embodiment of the present invention using an air stripping gas in the absence of a catalyst;

Figure 5 illustrates the effect of catalyst on the rate of decrease in concentration of H_2S_x in a bench-scale contactor operating in accordance with one embodiment of the invention using an air stripping gas;

Figure 6 shows FTIR spectrum of liquid sulfur for H_2S and H_2S_x with time post-catalyst injection in the results shown in Figure 5;

Figure 7 contains a comparison of FTIR spectra before and after catalyst addition;

Figure 8 contains a comparison of FTIR spectra for feed and product samples for a steady state flow-through test;

Figure 9 is a graphic representation of the H_2S_x half-life vs catalyst concentration in a pilot-scale contactor with continuous flow-through tests;

Figure 10 contains a comparison of FTIR spectra for an initial feed sample and final degassed sample for a liquid sulfur degassed according to the present invention and having residual H_2S and H_2S_x concentrations below 2 ppmw, and

Figure 11 is a graphical representation of the effect of catalyst concentration on the quantity of SO_2

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in the off-gas from a pilot plant contactor using an air stripping gas.

GENERAL DESCRIPTION OF INVENTION

The present invention is concerned with a procedure
5 for removing hydrogen sulfide and hydrogen polysulfides
from liquid sulfur in a rapid and efficient manner to
result in low residual hydrogen sulfide and hydrogen
polysulfide levels. The liquid sulfur processed herein
may be that produced by a Claus plant converting hydrogen
10 sulfide recovered from oil refining or sour gas to
elemental sulfur. However, liquid sulfur containing
dissolved hydrogen sulfide and hydrogen polysulfides from
any other source may be treated using the procedure of
the present invention.

15 Accordingly, the present invention provides a method
of removing hydrogen sulfide and hydrogen polysulfides
from liquid sulfur, comprising a plurality of steps. The
method of the invention is carried out at a temperature
at which sulfur is in a molten state. At temperatures
20 above about 160°C, liquid sulfur becomes more viscous.
The preferred temperature range for a system as described
herein is about 120°C to about 160°C, more specifically,
about 125°C to 155°C. The proportion of hydrogen
polysulfides to hydrogen sulfide also increases with
25 increases in temperature, and, therefore, it is
preferable to degas liquid sulfur within the temperature
ranges noted above.

A rotary impeller comprising a plurality of blades
is provided at a submerged location in the liquid sulfur
30 surrounded by a shroud through which are formed a
plurality of openings, within a range of impeller to
shroud diameter ratios as discussed below.

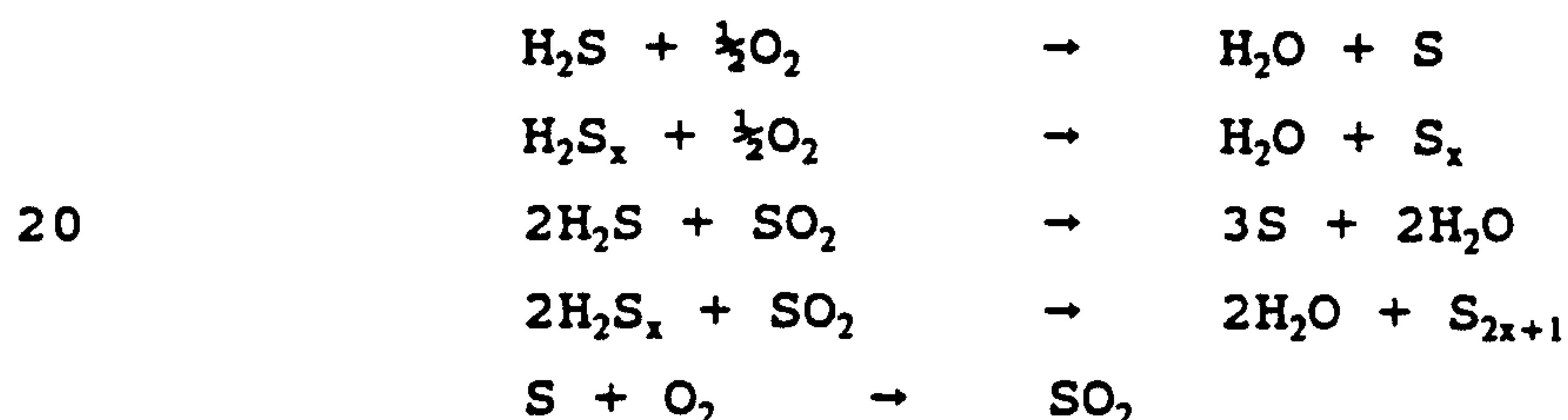
A stripping gas for hydrogen sulfide is fed to a
submerged location in the liquid sulfur within the
35 shroud. The stripping gas may comprise steam and removes
from the reaction vessel hydrogen sulfide released from

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the liquid sulfur in the stripping operation. An amine catalyst preferably is utilized in conjunction with the steam, provided in any convenient manner in the liquid sulfur within the shroud. An oxidizing gas also may be employed in the stripping gas. When such oxidizing gas is used, the oxidizing gas may be oxygen or sulfur dioxide, generally transported in an inert carrier gas, such as carbon dioxide or nitrogen or in steam. An amine catalyst preferably is used in conjunction with the oxidizing gas.

A plurality of simultaneous reactions take place in the liquid sulfur resulting in decomposition of hydrogen polysulfides to hydrogen sulfide and stripping of hydrogen sulfide or conversion of hydrogen sulfide to sulfur. Some of the reactions which may take place, depending on the nature of the stripping gas employed may be designated by the following equations:



The stripping gas which is passed through the liquid sulfur should comprise sufficient volume so as to remove the hydrogen sulfide released from the liquid sulfur. It is possible to use Claus process tail gas as the stripping gas since this gas contains nitrogen, water, hydrogen sulfide, sulfur dioxide, carbon dioxide and other constituents. As noted below, the use of a Claus process tail gas stream may result in simultaneous sulfur degassing and Claus plant tail gas clean up.

The catalyst employed herein may be any of the components known to assist the decomposition of hydrogen polysulfides to hydrogen sulfide and sulfur. In general, the catalyst is a Bronsted-Lowry base having a pK_b value of less than about 10, preferably less than about 6.

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Added catalyst materials in general are Bronsted-Lowry bases soluble in liquid sulfur, so as to promote the decomposition of the hydrogen polysulfides. Such materials preferably are sufficiently volatile to permit
5 their stripping from the degassed liquid sulfur. Specific examples of such added catalyst materials include volatile aliphatic, cyclic and heterocyclic amines sparingly soluble in the liquid sulfur, as well as ammonia. Such catalysts are preferably organic amines,
10 such as cyclohexylamine and morpholine. Only small quantities of added catalyst are needed, generally in the low parts per million range, generally about 10 ppbw to about 10 ppmw relative to the weight of sulfur, preferably about 1 to about 5 ppmw.

15 The amine catalysts used herein also catalyze conversion of hydrogen sulfide to sulfur in the presence of sulfur dioxide, so that, when a Claus plant effluent or tail gas stream is utilized as the stripping gas for removal of hydrogen sulfide from the liquid sulfur, the
20 presence of the amine enables Claus plant tail gas clean up and liquid sulfur degassing to be effected in a single step with a single catalyst species.

Such degassing operation in the presence of the amine catalyst and sulfur dioxide in the Claus plant tail
25 gas stream may result in almost complete elimination of hydrogen sulfide from the Claus plant tail gas and from liquid sulfur degassing, leaving a gaseous effluent stream free from hydrogen sulfide emissions. In addition to its presence in a Claus plant tail gas stream, the
30 sulfur dioxide employed in this procedure may be provided by burning elemental sulfur or as a slip stream from the Claus plant H₂S burner. It may be preferred to provide a stoichiometric excess of sulfur dioxide to ensure that all hydrogen sulfide, whether in the Claus plant tail gas
35 stream or in the liquid sulfur, is converted to sulfur, to provide a effluent stream which may contain residual

sulfur dioxide. Some oxygen may be added to the tail gas stream to effect sulfur dioxide formation in situ in the sulfur and reaction with hydrogen sulfide and/or hydrogen polysulfides to sulfur and water.

5 The ability to effect complete conversion of hydrogen sulfide to sulfur in the presence of liquid sulfur enables there to be provided, in accordance with another aspect of the invention, a Claus plant tail gas clean up procedure in which the hydrogen sulfide content
10 thereof is converted to sulfur by reaction with sulfur dioxide in liquid sulfur, irrespective of the degassing of the liquid sulfur to remove hydrogen sulfide and hydrogen polysulfides therefrom, using the shroud and impeller combination described herein under the process
15 conditions described.

Accordingly, in another aspect of the present invention, there is provided a method for the processing of a hydrogen sulfide- and sulfur dioxide-containing gas stream to remove at least one of the components
20 therefrom, which comprises providing a rotary impeller comprising a plurality of blades at a submerged location in the liquid sulfur surrounded by a shroud through which are formed a plurality of openings, feeding the gas stream to the submerged location, rotating the impeller
25 about a substantially vertical axis at a speed sufficient to draw liquid sulfur into the interior of the shroud and to distribute the gas stream as bubbles in the liquid sulfur to the interior of the shroud and to form a gas-liquid mixture of bubbles of the gas stream in the liquid
30 sulfur contained within the shroud, and flowing the gas-liquid mixture from within the interior of the shroud through and in contact with the openings to external of the shroud, thereby to effect reaction between hydrogen sulfide and sulfur dioxide to sulfur. The hydrogen
35 sulfide- and sulfur dioxide-containing gas stream preferably is a tail gas stream from a Claus plant.

In general, Claus plants are operated to provide a small stoichiometric excess of hydrogen sulfide over sulfur dioxide, which normally would result in residual hydrogen sulfide after reaction of the sulfur dioxide and hydrogen sulfide. The composition of the gas stream may be adjusted, however, as described herein, to ensure essentially complete reaction of both hydrogen sulfide and sulfur dioxide to sulfur in the liquid sulfur. For example, it may be desirable to add oxygen to the tail gas stream to provide for conversion to hydrogen sulfide and hydrogen polysulfide in the liquid sulfur to sulfur. The Claus plant tail gas clean up procedure generally is carried out in the presence of a catalyst for the reaction of hydrogen sulfide with sulfur dioxide. Any of the Bronsted-Lowry bases mentioned herein may be employed. It may be desirable to provide a non-volatile catalyst in the sulfur.

The impeller generally is rotated about a substantially vertical axis at the submerged location within the liquid sulfur at a blade tip velocity (v_i) of at least about 150 in/sec (at least about 4 m/s), preferably at least about 350 in/sec (at least about 9 m/s), more preferably at least about 500 in/sec (preferably at least about 12.5 m/s), for example, up to and greater than about 700 in/sec (up to and greater than about 18 m/s), and draws liquid sulfur into the interior of the shroud effecting vigorous circulation of liquid sulfur through the impeller and shroud combination.

The shear forces between the impeller blades and the plurality of openings in the shroud distribute the stripping gas in the liquid sulfur as bubbles to the interior of the shroud and forms a gas-liquid mixture of bubbles of the stripping gas in liquid sulfur contained within the shroud and effects intimate contact of the stripping gas and liquid sulfur at the submerged location while effecting shearing of the gas-liquid mixture within

the shroud and initiating and sustaining rapid mass transfer.

The gas-liquid mixture flows from within the shroud through and in contact with the openings therein to
5 external of the shroud at a gas velocity index (GVI) generally of at least about 4 per second per opening, preferably at least about 18 per second per opening, more preferably at least about 24 per second per opening, which causes further shearing of the gas liquid mixture
10 and further intimate contact of gaseous phase and liquid sulfur. The gas velocity index (GVI) more preferably is at least about 30 per second per opening, and may range to very high values, such as up to about 500 per second per opening, and often is in excess of about 100 per
15 second per opening.

The gas velocity index (GVI) is determined by the expression:

$$GVI = \frac{QP}{4nA^2}$$

where Q is the volumetric flow rate of gas into the
20 impeller (m^3/S), n is the number of openings in the shroud, A is the area of the opening (m^2) and P is the length of the perimeter of the opening (m).

An important operating parameter of the process is the relationship of the shroud diameter relative to the
25 impeller diameter for a given GVI and impeller tip speed and this parameter may be termed the Effective Shear Index (ESI). The ESI is determined by the expression:

$$ESI = \frac{GVI}{v_i} \times \frac{(D_s - D_i)}{2} \times 100$$

wherein GVI is the gas velocity index (/s), v_i is the
30 impeller blade tip velocity (m/s), and D_s and D_i are inside diameter of the shroud and outside diameter of the

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impeller respectively (m). The method of the invention generally is carried out at an ESI value from about 1 to about 6000, preferably from about 10 to about 250, optimally about 50.

5 Another important operating parameter of the process of the invention is the relationship of the impeller diameter and height for a given volumetric gas flow rate and impeller tip speed and this parameter may be termed the Shear Effectiveness Index (SEI). The SEI is
10 determined by the relationship:

$$SEI = \frac{Q}{\pi h D_i v_i} \times 100$$

wherein Q is the volumetric gas flow rate into the impeller (m³/s), h is the height of the impeller blades (m), D_i is the outside diameter of the impeller (m) and
15 v_i is the impeller blade tip velocity (m/s). The present invention generally employs an SEI value in the range of about 1 to about 25 and preferably about 2 to about 5. These parameters apply to self-induced systems without internal baffles as described in U.S. Patent No.
20 3,993,563 (Degner) and/or to those with externally-sparged gas. The ESI and SEI values employed herein preferably are determined for impeller tip speed velocities of at least about 350 in/sec (at least about 9 m/s).

25 A further important operating parameter of the process is the interrelationship of the density of the liquid phase, namely the liquid sulfur, processed by the present invention and the volumetric flow rate of gas into the reactor and this parameter may be termed the
30 Density Index (DI). The DI is determined by the expression:

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$$DI = \frac{Q}{Q_i} \left[\frac{\rho_L - \rho_g}{\rho_{H_2O} - \rho_{air}} \right]$$

where Q is the volumetric flow rate of the stripping gas to the impeller (m^3/s), Q_i is the self-induction flow rate of air into water (m^3/s), ρ_L is the density of the liquid sulfur (kg/m^3), ρ_g is the density of the gas (kg/m^3), ρ_{H_2O} is the density of water (kg/m^3) and ρ_{air} is the density of air (kg/m^3). The method of the invention generally is carried out at a DI value from about 1 to about 8, preferably about 3.

For efficient operation, the procedure is preferably effected at a Gas Displacement Index (GDI) value of about -0.50 to about 0.95 as determined by the relationship:

$$GDI = \frac{2V_i \cdot \omega - Q}{2V_i \cdot \omega}$$

where V_i is the volume swept by the impeller (m^3), ω is the rate of rotation of the impeller (rad/s) and Q is the volumetric flow rate of the gas to the impeller (m^3/s), and a Mixing Index (MI) value of at least about -5 as determined by the relationship:

$$MI = \frac{2V_i \cdot \omega - Q}{V_r}$$

where V_i is the volume swept by the impeller (m^3), ω is the rate of rotation of the impeller (rad/s), Q is the volumetric flow rate of gas to the impeller (m^3/s) and V_r is the volume of the reactor (m^3).

DESCRIPTION OF PREFERRED EMBODIMENT

Referring to the drawings, Figure 1 shows a novel liquid sulfur degassing device 10 operated in accordance with one preferred embodiment of the invention. The device 10 includes an enclosed housing 12 illustrated to

be of cylindrical shape but any convenient geometric shape may be adopted. It is preferred that the geometric shape of the housing may be such as to avoid dead zones in the liquid phase contained within the housing.

5 A liquid sulfur feed inlet pipe 14 communicates with the interior of the housing 12 through the lower wall 16 and an overflow outlet 18 for liquid sulfur allows liquid sulfur to overflow from the interior of the housing 18 following processing therein.

10 A gas inlet 20 communicates with a standpipe 22 or draft tube extending downwardly inside the housing 12 to below the level of the body of liquid sulfur 24 contained within the vessel to enable a stripping gas for hydrogen sulfide. The stripping gas generally is air but a sulfur
15 dioxide-containing gas stream may be employed. Steam may be employed in place of or in addition to air. The tail gas stream from a Claus plant also may be employed as the gas feed, thereby also effecting Claus plant tail gas clean up. As mentioned earlier, Claus plant tail gas
20 clean up also may be effected in liquid sulfur from which the stripping of hydrogen sulfide and/or polysulfide is not required, such as liquid sulfur which has been degassed according to the invention.

 The gas stream generally is of a volume to permit
25 purging of hydrogen sulfide released by the liquid sulfur following decomposition of hydrogen polysulfide from the vessel 10. Suitable Bronsted-Lowry catalysts included those having pK_b values of less than about 10 and preferably less than about 6, and may comprise amines
30 sparingly soluble in the liquid sulfur. The catalyst may be introduced to the interior of the shroud by any convenient means, such as transportation in the stripping gas stream or by direct introduction to the vortex in the shroud. If desired, a catalyst which is soluble in the
35 liquid sulfur may be contained therein.

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Generally, the flow rate of the stripping gas stream may range upwardly from a minimum of about 50 cu.ft/min. (about 25 dm³/s), for example, in excess of about 3000 cu.ft/min. (about 1400 dm³/s), although much higher or lower flow rates may be employed. The pressure drop across the unit may be quite low and may vary from about -3 to about +40 in. H₂O (from about -75 to about +1000 mm H₂O), preferably from about 0 to less than about 10 in. H₂O (250 mm H₂O). For larger units, employing a fan or a blower to assist the gas flow rate to the impeller, the pressure drop may be greater. The flow rate is further defined by the DI values discussed above.

A drive shaft 26 extends into the vessel 10 and has an impeller 28 mounted at its lower end just below the lower extremity of the standpipe 22. A drive motor (not shown) is operatively connected to the drive shaft 26 to effect rotation of the shaft 26 and hence the impeller 28.

The impeller 28 comprises a plurality of radially-extending blades 30. The number of such blades may vary and generally at least four blades 30 are employed, as illustrated, with the blades being equi-angularly spaced apart. The impeller 28 is illustrated with the blades 30 extending vertically. However, other orientations of the blades 30 are possible.

Generally, the standpipe 22 has a diameter dimension related to that of the impeller 28 and the ratio of the diameter of the standpipe 22 to that of the impeller 28 generally may vary from about 1:1 to about 2:1. However, the ratio may be lower, if the impeller is mounted below the standpipe. The impeller 28 generally has a height which corresponds to an approximately 1:1 ratio with its diameter, but the ratio generally may vary from about 0.3:1 to about 3:1. As the stripping gas is drawn down through the standpipe 22 by the action of the rotating impeller 28 and the liquid sulfur is drawn into the

impeller 28 thereby, the action of stripping gas and liquid sulfur flow and rotary motion produce a vortex of liquid sulfur in the standpipe and the region of the impeller 28. Alternatively, the stripping gas may be introduced below the impeller 28 and drawn into the interior of the shroud 32 by the action of the impeller 28.

The ratio of the cross-sectional area of the shrouded impeller 28 to the cross-sectional area of the apparatus 10 may vary widely.

Another function of the impeller 28 is to distribute the introduced stripping gas bubbles within the liquid sulfur in the interior of the shroud 34. This result is achieved by rotation of the impeller 28, resulting in shear of liquid sulfur and stripping gas to form bubbles of relatively wide size distribution dimensioned so that the largest are no more than about $\frac{3}{4}$ inch (20 mm) in diameter.

A critical parameter in determining an adequate shearing to form the gas bubbles is the velocity of the outer tip of the blades 32. A blade tip velocity (v_t) of at least about 150 in/sec (at least about 4 m/s) is required to achieve efficient (i.e., 99%+) and rapid (of the order of minutes residence time) removal of hydrogen sulfide from liquid sulfur and conversion of hydrogen polysulfide, preferably at least about 350 in/sec (at least about 9 m/s), more preferably at least about 500 (at least 12.5), and up to and greater than 700 in/sec (about 18 m/s).

The impeller 28 is surrounded by a cylindrical stationary shroud 34 having a uniform array of circular openings 36 through the wall thereof. The shroud 34 generally has a diameter slightly greater than the standpipe 22. Although, in the illustrated embodiment, the shroud 34 is right cylindrical and stationary, it is possible for the shroud 34 to possess other shapes. For

example, the shroud 34 may be tapered, with the impeller 28 optionally also being tapered. In addition, the shroud 34 may be rotated, if desired, usually in the opposite direction to the impeller 28. Further, the shroud 34 is shown as a separate element from the standpipe 22. However, the shroud 34 may be provided as an extension of the standpipe 22, if desired.

Further, the openings 36 in the shroud are illustrated as being circular, since this structure is convenient. However, it is possible for the openings to have different geometrical shapes, such as square, rectangular or hexagonal. Further, all the openings 36 need not be of the same shape or size.

The shroud 34 serves a multiple function in the device. Thus, the shroud 34 prevents gases from bypassing the impeller 28, assists in the formation of the vortex in the liquid sulfur necessary for gas induction, assists in achieving shearing as well as providing additional shearing and confines the gas-liquid mixture and hence maintains the turbulence and agitation produced by the impeller 28. The effect of the impeller-shroud combination may be enhanced by the employment of a series of elongate baffles, provided on the internal wall of the shroud 34, preferably vertically extending from the lower end to the upper end of the openings in the shroud. The gas-liquid mixture flows through and in contact with the openings 36 in the shroud which results in further shearing of the gas bubbles and further intimate contact of the gaseous and liquid phases.

The combined action of the impeller and shroud results in a rapid mass transfer of hydrogen sulfide into the stripping gas and catalyst into the sulfur, resulting in very rapid removal of dissolved hydrogen sulfide and decomposition of hydrogen polysulfides to sulfur and hydrogen sulfide. The reactions are very rapid, in contrast to the prior art liquid sulfur degassing

operations. The combined action of the impeller and shroud effects circulation of liquid sulfur within the housing 12 and adequate mixing of the liquid sulfur in the body of liquid sulfur external to the shroud 32 to ensure the release of the stripping gas.

The openings 36 are dimensioned to permit a gas flow rate therethrough corresponding to a gas velocity index of at least about 4 per second per opening in the shroud, preferably at least about 18 per second per opening and more preferably at least about 30 per second per opening.

The shroud 34 is spaced only a short distance from the extremity of the impeller blades 30, in order to provide and promote the above-noted functions. Generally, the ratio of the diameter of the shroud 34 to that of the impeller 28 generally is about 3:1 to about 1.1:1, preferably approximately 1.5:1. The relationship of the shroud and impeller diameters may be further particularized by the ESI and SEI indices discussed above.

The openings 36 in the shroud 34 generally are circular, although an equivalent effect can be achieved using openings of large aspect ratio, such as slits. When such circular openings are employed, the openings 36 generally are uniformly distributed over the wall of the shroud 34 and usually are of equal size. The equivalent diameter of the openings 36 often is less than about one inch (25 mm) and generally should be as small as possible without plugging, preferably about $\frac{1}{4}$ to about $\frac{1}{2}$ inch (about 10 to about 15 mm) in diameter, in order to provide for the required gas flow therethrough. When the openings 36 are of non-circular geometrical shape and of aspect ratio which is approximately unity, then the area of each such opening 36 generally is less than the area of a circular opening having an equivalent diameter of about one inch (25 mm), preferably about $\frac{1}{4}$ to about $\frac{1}{2}$ inch (about 10 to about 15 mm). The openings have sharp

corners to promote shearing of the gas bubbles passing through the openings and contacting the edges.

The shroud 34 is illustrated as extending downwardly for the height of the impeller 28. It is possible for
5 the shroud 34 to extend above the height of the impeller 28 or for less than its full height, if desired.

In addition, in the illustrated embodiment, the impeller 28 is located a distance corresponding to approximately half the diameter of the impeller 28 from
10 the bottom wall of the reactor 10. It is possible for this dimension to vary from less than about 0.25:1 to about 1:1 or greater of the proportion of the diameter dimension of the impeller. This spacing of the impeller 28 from the lower wall allows liquid sulfur to be drawn
15 into the area between the impeller 28 and the shroud 34 from the body of liquid sulfur 24 in the reactor 10. If desired, a draft tube may be provided extending into the body of the liquid sulfur from the lower end of the impeller 28, to guide liquid into the region of the
20 impeller.

Following passage of the gas-liquid mixture through the openings in the shroud a relatively quiescent zone is provided in the body of liquid sulfur 24 where gas bubbles disengage from the sulfur and provide a gaseous
25 phase in the head space above the liquid level of molten sulfur in the vessel 10. A further shroud may be provided surrounding the shroud 34 to promote additional mixing.

The liquid sulfur degassing apparatus 10 provides a
30 very compact unit which rapidly and efficiently removes hydrogen sulfide and hydrogen polysulfides from liquid sulfur, in contrast to the large size holding tanks commonly employed in the art.

The novel sulfur degassing procedure employed herein
35 may be effected as a single operation, or more preferably, a plurality of individual contactors

connected in series, with each contactor having a hold-up volume commensurate with the production rate of liquid sulfur, thereby providing continuous processing of the liquid sulfur. The individual contactors may vary in
5 hold-up capacity and residence time, depending on the degassing requirements of the sulfur producing plant. A typical operation effected in accordance with the invention produces a high quality product with a total hydrogen sulfide content of a few ppmw or less, or such
10 higher value as can be accepted. A polishing contactor, designed to remove catalyst from the liquid sulfur can further contribute to improve product quality. The gas phases may be circulated countercurrently through the contactors.

15 Any sulfur dioxide which may be formed as a result of oxidation with air in later stages of processing with low residual hydrogen sulfide levels may be reacted with hydrogen sulfide in earlier stages, converting some of the hydrogen sulfide present to elemental sulfur.

20 The processing of the liquid sulfur in the apparatus 10 produces a low volume effluent gas which is relatively high in hydrogen sulfide content, along with some volatile impurities and traces of sulfur vapor, which is vented from the head space 38 above the body of liquid
25 sulfur 24 in the vessel 10 through a gaseous outlet 40. This effluent stream may be disposed of by incineration, or may be used as feed stream to a Claus plant for the oxidation of hydrogen sulfide, or to another hydrogen sulfide control process, such as by utilizing the
30 procedure of USP 5,174,973, referred to previously.

EXAMPLES

Example 1:

An experimental apparatus was set up having the structure illustrated in Figure 1. Two contactors
35 differing in size were tested. A bench scale unit having a volume of 9 litres (16 kg) was used primarily to

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measure the removal rates of H_2S and H_2S_x by measuring concentrations over time. A 970 litre pilot scale unit (1.75 tonnes) was used primarily to measure reaction rates using a steady-state flow of liquid sulfur. Sulfur produced by the Claus process from two different gas plants was used as feedstock for the two units.

The operating parameters for the apparatus employed in generating the data shown in Figure 3 to 8 and 10 are set forth in the following Table:

10	Figures	3, 4	5, 6	7	8, 10
	v_i (in/sec)	640	356	640	640
	GVI (/s)	28.9	9.7	53.1	43.4
	ESI	23.7	3.1	43.6	35.6
	SEI	1.6	0.9	2.9	2.3
15	DI	1.9	1.0	3.5	2.9

The apparatus used to generate the data in Figure 5, 6 had an impeller 2 in. in diameter and 2 in. in height and a shroud having an inside diameter of 4.25 in., with 100 x $\frac{1}{8}$ in. diameter openings therethrough. The apparatus used to generate the data in the other Figures had an impeller 12.75 in. in diameter and 13 in. in height and a shroud having an inside diameter of 23.25 in., with 1824 x $\frac{1}{8}$ in. diameter openings therethrough.

Samples of liquid sulfur were withdrawn at various locations and times and the concentration of the H_2S and H_2S_x measured using a Fourier Transform Infra Red (FTIR) spectrophotometer within a few minutes after sampling. An accessory system developed by Alberta Sulphur Research had permitted non-intrusive determination of the total residual H_2S ($H_2S+H_2S_x$) concentrations in liquid sulfur down to 4 ppmw. The absorptions by H_2S at 2570 cm^{-1} and

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H_2S_x at 2497 cm^{-1} were calibrated and displayed in units of ppmw.

The contactors were operated using air as the stripping gas with and without added catalyst OPTI-MEEN 2200 (Betz Chemicals, Ltd, a proprietary aqueous solution of morpholine (10 to 30%) and cyclohexylamine (40 to 70%). A typical FTIR spectrum showing H_2S and H_2S_x in liquid sulfur feed is shown in Figure 2. The curves shown in Figure 3 present the H_2S and H_2S_x concentration plotted logarithmically against time for a typical experiment conducted with the pilot reactor using air as the stripping gas in the absence of catalyst. The steeply falling line at the left edge of the graph depicts the decrease in concentration of dissolved hydrogen sulfide, while the gradually sloped curve depicts the decrease in the concentration of polysulfides. Based on the slopes of these lines, the half-lives for H_2S and H_2S_x were calculated to be 0.75 and 48 minutes respectively.

Figure 4 contains a semi-logarithmic plot of the gaseous hydrogen sulfide concentration in the effluent gas against time. The very fast initial decrease in H_2S concentration can be attributed to the rapid degassing of hydrogen sulfide while the slow decrease in H_2S concentration corresponds to decomposition of H_2S_x .

Figure 5 shows the effect of the addition of a small amount of catalyst in a batch test, namely 4.8 ppmw of OPTI-MEEN 2200. As can be seen, the addition of the catalyst had a remarkable effect on the rate at which hydrogen polysulfides were converted to H_2S . Sulfur containing high initial concentrations of H_2S and H_2S_x was degassed using air as the stripping gas in the bench scale apparatus. Measurement of the hydrogen polysulfide concentration commenced about 30 minutes into the run, once the concentration fell within the calibrated range (<195 ppmw) and at various times over the next 25 minutes

to establish a reference half-life using air as the stripping gas. Fifty-eight minutes into the run, 0.08 mL of catalyst, corresponding to a nominal concentration of 4.8 ppmw, was injected into the stripping gas intake line using a syringe. As can be seen from Figure 5, the half-life for the decrease in H_2S_x concentration with catalyst present was reduced to less than one minute from about 45 ± 15 minutes generally observed with air alone, corresponding to about a 40-fold increase in decomposition rate.

Figure 6 contains FTIR spectra showing the history of H_2S and H_2S_x peaks at around the time of catalyst addition in Figure 5. A small H_2S peak, absent before the introduction of catalyst, appeared simultaneously with the onset of the rapid depletion of H_2S_x . Concomitant with the progressive decrease in the H_2S_x peak, the H_2S rose to a maximum a short time later and then declined in parallel with the H_2S_x peak.

Batch tests using catalyst in the larger pilot-scale contactor yielded results comparable with those obtained with the bench-scale apparatus. Figure 7 shows the spectra of sulfur samples taken from the contactor prior to and 70 seconds after injecting an amount of catalyst, nominally equivalent to 1.2 ppmw, into the air intake of the large contactor. In the test of Figure 7, polysulfides concentration decreased with a half-life of around 0.5 minutes, again indicative of the powerful influence a very small amount of catalyst has on the polysulfide decomposition rate in the process of the invention. As with the bench-scale unit, H_2S remained observable while H_2S_x was decomposing at a rapid rate. Moreover, the agreement between declining concentration rates observed with the two contactors widely different in size confirmed the validity of the scale-up factors used in the design of the larger unit.

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A visual comparison of the two spectra in Figure 7 reveals that many of the peaks, like the H_2S peak, rapidly decrease in height over a short time. The majority of the peaks declined exponentially with half-lives between 0.3 and 0.8 minutes, quite within the range seen for H_2S . This is the case for water, which absorbs in the region over 4000 cm^{-1} to 3400 cm^{-1} and at 1591 cm^{-1} . It may be reasonably generalized that any volatile substance, such as water CO_2 , SO_2 , COS , CS_2 and hydrocarbons also may be effectively purged, along with the catalyst itself.

Example 2:

Although batch studies of the time dependency of the H_2S and H_2S_x concentrations provided a clear picture of the capabilities of the process as outlined in Example 1, the performance of the pilot-scale contactor using a steady-state flow of feed sulfur gave a much better idea of the process in its practical application. Sulfur flowrates were varied between 34 and 53 tonnes/h. Catalyst was introduced directly into the liquid sulfur in quantities ranging from 4.8 to 10.8 ppmw. No significant effect on the rates of decrease in concentration of H_2S and H_2S_x was observed on varying the sulfur temperature from 140° to 152°C .

The spectrum of contactor feed and contents for a typical flowthrough test are presenting in Figure 8. The marked decrease in the absorption by H_2S , H_2S_x and other volatiles qualitatively suggests that efficient degassing of the various substances is taking place.

Measurement of the respective concentrations indicate that 61% of the H_2S and 79% of the H_2S_x was removed during the hold-up time of 2.5 minutes, corresponding to half-lives of 0.76 and 0.46 minutes, respectively. It is noted in Figure 8 that other volatile substances in the feed are as readily degassed

in flowthrough tests as in batch tests (see Figure 7), with rates comparable to the degassing of H_2S .

Figure 9 summarizes the results of the continuous flow experiments, showing the half-life of the H_2S_x decomposition reaction as a function of catalyst concentration, expressed in ppmw with respect to the sulfur. The vertical lines represent the range of values for several sets of measurements taken at a particular catalyst concentration. In many instances where the catalyst concentration was above 8 ppmw, the H_2S_x concentrations in the contactor were reduced to levels below the measurement sensitivity (< 2 ppmw) of the FTIR system. Since the final concentration was not known in these cases, quantitative rate data were not acquired, although it is noted that the half-life was below 0.5 minutes. This Figure suggests that the half life of polysulfide concentration decrease varies approximately linearly with catalyst concentration in the region between 4 and 8 ppmw. The curve, however, rises very steeply as the catalyst concentration approaches zero, since the half-life measured in batch tests with no catalyst present was approximately 45 ± 15 minutes.

Degassification of sulfur down to very low levels of H_2S and H_2S_x was routinely achieved with the pilot scale contactor. Figure 10 compares the spectrum of a sulfur sample taken at the start of one of the tests to one taken after approximately three passes through the pilot-scale contactor. Very low residual H_2S and H_2S_x concentrations were achieved, (since the H_2S peak is hardly detectable, and the H_2S_x concentration is well below the measurement accuracy) using a flowthrough contactor of a size that would comfortably handle the production of many existing sulfur plants.

Example 3:

During the experiments reported in Examples 1 and 2, it became apparent that the strength properties of

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(solidified) sulfur samples taken from the contactor varied significantly. Those samples that contained a modest amount of residual catalyst were brittle and crumbly whereas those samples that contained less of the catalyst were very hard to break.

This effect was substantiated in an experiment where the H_2S and H_2S_x concentrations in 75 tonnes of sulfur were reduced below 2 ppmw by degassing using a catalyst concentration of approximately 10 ppmw. After the H_2S and H_2S_x were essentially eliminated, half of the liquid sulfur was solidified using a granulation process, and samples of the product set aside for friability testing according to SUDIC procedures (ref. 2). With liquid sulfur feed containing residual amounts of catalyst but very little H_2S and H_2S_x , the friability averaged 3.1%. The remaining sulfur was then circulated once more through the contactor to degas as much of the volatile catalyst as possible by contacting the sulfur with air only.

After this simple treatment, the sulfur was solidified as before. The friability of the granulated product, in this case produced from liquid sulfur with a reduced amount of catalyst, averaged 0.7%.

Based on these comparative results, it appears that the presence of the catalyst and possibly other impurities, even in concentrations of a few ppmw, can have a significant effect on product quality. However, the degassing process provided herein has the capability to overcome any detrimental effects linked to catalyst contamination by dedicating a polishing contactor to the task of degassing the residual catalyst from the sulfur.

Example 4:

During the course of sulfur degassification tests with the pilot-scale unit described in Example 1 using air as the stripping gas and continuous sulfur flows, a determination was made of SO_2 content of the off-gas

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stream as a function of catalyst concentration. During these experiments, the following operating parameters were employed:

$V_i = 350$ in/s, $GVI = 34.8/s$, $ESI = 11.2$, $SEI = 3.3$
5 and $DI = 5$. The data obtained was plotted graphically and appears as Figure 11.

As is seen therein, in the absence of catalyst (OPTI-MEEN 2200) 25 to 42 ppmw or approximately 0.01 mol/min of sulfur dioxide is produced by the reaction:



This production rate decreased to 0 mol/min when the catalyst concentration was in the range of 4.4 to 5.0 ppmw. A likely explanation for this observation is an increased rate of a catalysed Claus reaction in the
15 liquid sulfur:



In another series of experiments, on this occasion using the bench-scale unit described in Example 1, with nitrogen containing a small amount of sulfur dioxide as
20 the stripping gas and a batch sulfur with no catalyst, the effluent concentrations of sulfur dioxide and hydrogen sulfide were determined. The parameters of operation are those for the bench-scale unit enumerated in Example 1 for Figures 5 and 6. The effluent
25 concentrations were as set forth in the following Table:

Time (min)	[SO ₂] ppmv	[H ₂ S] ppmv
0	120	0
4	>120	0
13	1000	0
26	200	0

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The data presented in this Table provides additional evidence of the existence of a Claus reaction in the liquid sulfur.

SUMMARY OF DISCLOSURE

5 In summary of this disclosure, the present invention provides a novel method of degassing liquid sulfur to remove hydrogen sulfide and hydrogen polysulfides therefrom by a stripping gas in the presence of a catalyst. The hydrogen sulfide is rapidly and
10 efficiently stripped and the hydrogen polysulfides are decomposed to sulfur and hydrogen sulfide, using an impeller-shroud combination, generally in conjunction with specific operating parameters. The present invention further provides a novel method of effecting
15 the Claus reaction, particularly for Claus plant tail gas clean up, by employing the impeller-shroud combination and liquid sulfur as the medium in which sulfur dioxide and hydrogen sulfide react to form sulfur. Claus plant tail gas also may be used as the stripping gas for liquid
20 sulfur containing hydrogen sulfide and hydrogen polysulfides. Modifications are possible within the scope of this invention.

REFERENCES

1. NIOSH, "Criteria for a Recommended Standard, Occupational Exposure to Hydrogen Sulfide", National Institute for Occupational Safety and Health, Cincinnati OH, 1977.
2. Sampling and Testing of Sulphur Forms, Sulphur Development Institute of Canada, 1978.

CLAIMS

What we claim is:

1. A method of removing hydrogen sulfide and hydrogen polysulfides from liquid sulfur, which comprises:

providing a rotary impeller comprising a plurality of blades at a submerged location in said liquid sulfur surrounded by a shroud through which are formed a plurality of openings,

feeding a stripping gas for hydrogen sulfide to said submerged location while providing a Bronsted-Lowry base catalyst for the conversion of hydrogen polysulfides to hydrogen sulfide thereat,

rotating said impeller about a substantially vertical axis at a speed sufficient to draw liquid sulfur into the interior of the shroud and to distribute said stripping gas as bubbles in said liquid sulfur to the interior of said shroud and to form a gas-liquid mixture of bubbles of said gas in said liquid sulfur contained within said shroud,

flowing said gas-liquid mixture from within the interior of shroud through and in contact with said openings to external of said shroud, and

removing said stripping gas from the liquid sulfur.

2. The method of claim 1 wherein said stripping gas is steam and said steam also provides said Bronsted-Lowry base catalyst.

3. The method of claim 1 wherein said stripping gas is an oxidizing gas.

4. The method of claim 3 wherein said oxidizing gas is air.

5. The method of claim 1 wherein said stripping gas is a tail gas stream from a Claus plant.

6. The method of claim 5 wherein said Claus plant tail gas stream contains a stoichiometric excess of sulfur dioxide with respect to hydrogen sulfide to effect

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substantially complete conversion of hydrogen sulfide to sulfur in said liquid sulfur.

7. The method of claim 5 wherein said Claus plant tail gas stream contains sufficient oxygen to provide a stoichiometric excess of sulfur dioxide with respect to hydrogen sulfide in said liquid sulfur.

8. The method of claim 5 wherein a catalyst for conversion of hydrogen sulfide to sulfur by reaction with sulfur dioxide.

9. The method of claim 3 wherein said oxidizing gas is sulfur dioxide in sufficient quantity to convert substantially all hydrogen sulfide present in the liquid sulfur and produced by decomposition of hydrogen polysulfides to sulfur.

10. The method of claim 1 wherein said Bronsted-Lowry base catalyst has a pK_b value of less than about 10.

11. The method of claim 10 wherein said pK_b value is less than about 6.

12. The method of claim 11 wherein said Bronsted-Lowry base catalyst comprises ammonia or an amine.

13. The method of claim 12 wherein said amine is cyclohexylamine or morpholine.

14. The method of claim 12 wherein said catalyst is used in an amount of about 10 ppbw to about 10 ppmw relative to the weight of sulfur.

15. The method of claim 12 wherein said catalyst is employed in an amount from about 1 to about 5 ppmw relative to the weight of sulfur.

16. The method of claim 12 wherein said Bronsted-Lowry base catalyst is provided at said submerged location by transportation thereto.

17. The method of claim 16 wherein said transportation is effected by introducing said catalyst into the liquid sulfur flowing into a vessel containing the same and/or by introducing the catalyst into the vessel, so as to be

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transported to said submerged location by circulation of liquid sulfur in the vessel.

18. The method of claim 16 wherein said transportation is effected by said stripping gas.

19. The method of claim 16 wherein said transportation is effected by introducing said catalyst directly into a vortex formed by said rotation of said impeller within the shroud.

20. The method of claim 19 wherein said stripping gas has a temperature at least about 20°C cooler than said liquid sulfur.

21. The method of claim 20 wherein said stripping gas has a temperature of less than about 100°C.

22. The method of claim 12 wherein said Bronsted-Lowry base catalyst is a volatile amine soluble in said liquid sulfur to facilitate removal from said liquid sulfur.

23. A method of removing hydrogen sulfide and hydrogen polysulfides from liquid sulfur, which comprises:

providing a rotary impeller comprising a plurality of blades at a submerged location in said liquid sulfur surrounded by a shroud through which are formed a plurality of openings,

feeding a stripping gas for hydrogen sulfide to said submerged location,

rotating said impeller about a substantially vertical axis at a speed corresponding to a blade tip velocity (v_i) of at least about 150 in/sec (at least about 4 m/s) so as to draw liquid sulfur into the interior of the shroud and to generate sufficient shear forces between said impeller and said plurality of openings in said shroud to distribute said stripping gas as bubbles in said liquid sulfur to the interior of said shroud and to effect intimate contact of said gas and said liquid sulfur at said submerged location so as to form a gas-liquid mixture of bubbles of said gas in said liquid sulfur contained within said shroud while

effecting shearing of said gas-liquid mixture within said shroud, and

flowing said gas-liquid mixture from within the interior of shroud through and in contact with said openings to external of said shroud at a gas velocity index (GVI) of at least about 4 per second per opening in said shroud so as to effect further shearing of the gas-liquid mixture and further intimate contact of said gas and said liquid sulfur,

said gas velocity index (GVI) being determined by the expression:

$$GVI = \frac{QP}{4nA^2}$$

where Q is the volumetric flow rate of gas into the impeller (m³/s), n is the number of openings in the shroud, A is the area of the opening (m²) and P is the length of the perimeter of the opening (m),

said method being carried out:

(a) at an Effective Shear Index (ESI) value of about 1 to about 6000 as determined by the relationship:

$$ESI = \frac{GVI}{v_i} \times \frac{(D_s - D_i)}{2} \times 100$$

where D_s is inside diameter (m) of the shroud and D_i is the outside diameter (m) of the impeller,

(b) at a Shear Effective Index (SEI) value of about 1 to about 25 as determined by the relationship:

$$SEI = \frac{Q}{\pi h D_i v_i} \times 100$$

where h is the height of the impeller (m), and

(c) at a Density Index (DI) value of about 1 to about 8 as determined by the relationship:

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$$DI = \frac{Q}{Q_1} \left[\frac{\rho_L - \rho_g}{\rho_{H_2O} - \rho_{air}} \right]$$

where Q_1 is the self-induction flow rate of air into water (m^3/s), ρ_L is the density of the liquid sulfur (kg/m^3), ρ_g is the density of the gas (kg/m^3), ρ_{H_2O} is the density of water (kg/m^3) and ρ_{air} is the density of air (kg/m^3).

24. The method of claim 23 wherein said blade tip velocity is at least about 350 in/sec (at least about 12.5 m/s).

25. The method of claim 23 wherein said blade tip velocity is at least about 500 in/sec. (at least about 12.5 m/s).

26. The method of claim 23 wherein said gas velocity index is at least about 18 per second per opening.

27. The method of claim 23 wherein said gas velocity index is at least about 24 per second per opening.

28. The method of claim 23 wherein said gas velocity index is from about 30 to about 500 per second per opening.

29. The method of claim 23 wherein said blade tip velocity is at least about 500 in/sec. (about 12.5 m/s) and said gas velocity index is at least about 30 per second per opening.

30. The method of claim 23 wherein said SEI value is determined at a v_i value of at least about 350 in/sec. (at least about 9 m/s).

31. The method of claim 23 wherein said SEI value is determined at v_i value of at least about 350 in/sec. (at least 9 m/s).

32. The method of claim 23 wherein said SEI value is about 10 to about 250 and is carried out at a Shear Effectiveness Index (SEI) value of about 4 to about 5.

33. The method of claim 32 wherein said SEI value is about 3.

34. The method of claim 23 which is carried out in the presence of a Bronsted-Lowry base catalyst for the conversion of hydrogen polysulfide to hydrogen sulfide.
35. The method of claim 23 wherein said stripping gas is steam, an oxidizing gas, or a tail gas stream from a Claus plant.
36. The method of claim 34 wherein said catalyst is ammonia or an amine.
37. The method of claim 36 wherein said amine is cyclohexylamine, morpholine or a mixture thereof.
38. The method of claim 36 wherein said catalyst is used in an amount of about 10 ppbw to about 10 ppmw based on the weight of sulfur.
39. The method of claim 36 wherein said catalyst is employed in an amount from about 1 to about 5 ppmw based on the weight of sulfur.
40. The method of claim 34 wherein said Bronsted-Lowry base catalyst is provided at said submerged location by transportation thereto.
41. The method of claim 34 wherein said transportation is effected by introducing said catalyst into the liquid sulfur flowing into a vessel containing the same and/or by introducing the catalyst into the vessel, so as to be transported to said submerged location by circulation of liquid sulfur in the vessel.
42. The method of claim 40 wherein said transportation is effected by said stripping gas.
43. The method of claim 40 wherein said transportation is effected by introducing said catalyst directly into a vortex formed by said rotation of said impeller within the shroud.
44. The method of claim 43 wherein said stripping gas has a temperature at least about 20°C cooler than said liquid sulfur.
45. The method of claim 44 wherein said stripping gas has a temperature of less than about 100°C.

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46. The method of claim 34 wherein said Bronsted-Lowry base catalyst is a volatile amine soluble in said liquid sulfur to facilitate removal from said liquid sulfur.

47. The method of claim 1 or 23 which is effected at a sulfur temperature of about 120° to about 160°C.

48. The method of claim 47 wherein said sulfur temperature is about 125° to about 155°C.

49. The method of claim 1 or 23 wherein said liquid sulfur is treated in a series of procedures as defined in claim 1 effected in series-connected vessels.

50. The method of claim 49 wherein any sulfur dioxide formed in a later one of said series-connected vessels is recycled to an earlier one of said series-connected vessels for conversion to sulfur by reaction with hydrogen sulfide therein.

51. The method of claim 49 wherein residual soluble catalyst is stripped from the liquid sulfur following said series of procedures.

52. The method of claim 23 which is carried out at a Gas Displacement Index (GDI) value of about -0.5 to about 0.95 as determined by the relationship:

$$GDI = \frac{2V_i \cdot \omega - Q}{2V_i \cdot \omega}$$

where V_i is the volume swept by the impeller (m^3), ω is the ratio of rotation of the impeller (rad/s) and Q is the volumetric flow rate of gas in the impeller (m^3/s).

53. The method of claim 23 or 52 which is carried out at a Mixing Index (MI) value of at least about -5 as determined by the relationship:

$$MI = \frac{2V_i \cdot \omega - Q}{V_r}$$

where V_i is the volume swept by the impeller (m^3), ω is the rate of rotation of the impeller (rad/s), Q is the

volumetric flow rate of gas to the impeller (m^3/s) and V_r is the volume of the reactor (m^3).

54. A method for the processing of a hydrogen sulfide- and sulfur dioxide-containing gas stream to remove at least one of the components therefrom, which comprises:

providing a rotary impeller comprising a plurality of blades at a submerged location in liquid sulfur surrounded by a shroud through which are formed a plurality of openings,

feeding said gas stream to said submerged location, rotating said impeller about a substantially vertical axis at a speed sufficient to draw liquid sulfur into the interior of the shroud and to distribute said gas stream as bubbles in said liquid sulfur to the interior of said shroud and to form a gas-liquid mixture of bubbles of said gas stream in said liquid sulfur contained, within said shroud, and

flowing said gas-liquid mixture from within the interior of shroud through and in contact with said openings to external of said shroud, thereby to effect reaction between hydrogen sulfide and sulfur dioxide to sulfur.

55. The method of claim 54 wherein said hydrogen sulfide and sulfur dioxide-containing gas stream is a tail gas stream from a Claus plant.

56. The method of claim 55 wherein said Claus plant tail gas stream contains a stoichiometric excess of sulfur dioxide with respect to hydrogen sulfide to effect substantially complete conversion of hydrogen sulfide to sulfur in said liquid sulfur.

57. The method of claim 55 wherein said Claus plant tail gas stream contains sufficient oxygen to provide a stoichiometric quantity or excess of sulfur dioxide with respect to hydrogen sulfide in said liquid sulfur, whereby substantially all of the hydrogen sulfide and

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sulfur dioxide in said tail gas stream is removed in the liquid sulfur.

58. The method of claim 55 wherein a catalyst for conversion of hydrogen sulfide to sulfur by reaction with sulfur dioxide is provided in said liquid sulfur at said submerged location.

59. The method of claim 58 wherein said catalyst is a Bronsted-Lowry base catalyst having a pK_b of less than about 10.

60. The method of claim 59 wherein said pK_b value is less than about 6.

61. The method of claim 60 wherein said Bronsted-Lowry base catalyst is provided at said submerged location by transportation thereto.

62. The method of claim 60 wherein said transportation is effected by introducing said catalyst into the liquid sulfur flowing into a vessel containing the same and/or by introducing the catalyst into the vessel, so as to be transported to said submerged location by circulation of liquid sulfur in the vessel.

63. The method of claim 61 wherein said transportation is effected by said tail gas stream.

64. The method of claim 61 wherein said Bronsted-Lowry base catalyst is a volatile amine soluble in said liquid sulfur to facilitate removal from said liquid sulfur.

65. The method of claim 58 wherein said catalyst is substantially non-volatile and is dissolved in said liquid sulfur.

66. A method for the processing of a hydrogen sulfide- and sulfur dioxide-containing gas stream, which comprises:

providing a rotary impeller comprising a plurality of blades at a submerged location in said liquid sulfur surrounded by a shroud through which are formed a plurality of openings,

feeding said gas stream to said submerged location,

rotating said impeller about a substantially vertical axis at a speed corresponding to a blade tip velocity (v_t) of at least about 150 in/sec (at least about 4 m/s) so as to draw liquid sulfur into the interior of the shroud and to generate sufficient shear forces between said impeller and said plurality of openings in said shroud to distribute said gas stream as bubbles in said liquid sulfur to the interior of said shroud and to effect intimate contact of said gas stream and said liquid sulfur at said submerged location so as to form a gas-liquid mixture of bubbles of said gas in said liquid sulfur contained within said shroud while effecting shearing of said gas-liquid mixture within said shroud, and

flowing said gas-liquid mixture from within the interior of shroud through and in contact with said openings to external of said shroud at a gas velocity index (GVI) of at least about 4 per second per opening in said shroud so as to effect further shearing of the gas-liquid mixture and further intimate contact of said gas stream and said liquid sulfur, whereby reaction is effected between hydrogen sulfide and sulfur dioxide contained in said gas stream in the presence of liquid sulfur to form sulfur,

said gas velocity index (GVI) being determined by the expression:

$$GVI = \frac{QP}{4nA^2}$$

where Q is the volumetric flow rate of gas into the impeller (m^3/s), n is the number of openings in the shroud, A is the area of the opening (m^2) and P is the length of the perimeter of the opening (m),

said method being carried out:

(a) at an Effective Shear Index (ESI) value of about 1 to about 6000 as determined by the relationship:

$$ESI = \frac{GVI}{V_i} \times \frac{(D_s - D_i)}{2} \times 100$$

where D_s is inside diameter (m) of the shroud and D_i is the outside diameter (m) of the impeller,

(b) at a Shear Effective Index (SEI) value of about 1 to about 25 as determined by the relationship:

$$SEI = \frac{Q}{\pi h D_i V_i} \times 100$$

where h is the height of the impeller (m), and

(c) at a Density Index (DI) value of about 1 to about 8 as determined by the relationship:

$$DI = \frac{Q}{Q_i} \left[\frac{\rho_L - \rho_g}{\rho_{H_2O} - \rho_{air}} \right]$$

where Q_i is the self-induction flow rate of air into water (m^3/s), ρ_L is the density of the liquid sulfur (kg/m^3), ρ_g is the density of the gas (kg/m^3), ρ_{H_2O} is the density of water (kg/m^3) and ρ_{air} is the density of air (kg/m^3).

67. The method of claim 66 wherein said blade tip velocity is at least about 350 in/sec (at least about 9 m/s).

68. The method of claim 66 wherein said gas velocity index is at least about 18 per second per opening.

69. The method of claim 66 wherein said hydrogen sulfide and sulfur dioxide-containing gas stream is a tail gas stream from a Claus plant.

70. The method of claim 69 wherein said Claus plant tail gas stream contains a stoichiometric excess of sulfur dioxide with respect to hydrogen sulfide to effect substantially complete conversion of hydrogen sulfide to sulfur in said liquid sulfur.

71. The method of claim 69 wherein said Claus plant tail gas stream contains sufficient oxygen to provide a stoichiometric quantity or excess of sulfur dioxide with respect to hydrogen sulfide in said liquid sulfur, whereby substantially all of the hydrogen sulfide and sulfur dioxide in said tail gas stream is removed in the liquid sulfur.

72. The method of claim 66 wherein a catalyst for conversion of hydrogen sulfide to sulfur by reaction with sulfur dioxide is provided in said liquid sulfur at said submerged location.

73. The method of claim 72 wherein said catalyst is a Bronsted-Lowry base catalyst having a pK_b of less than about 10.

74. The method of claim 73 wherein said pK_b value is less than about 6.

75. The method of claim 74 wherein said Bronsted-Lowry base catalyst is provided at said submerged location by transportation thereto.

76. The method claim 72 wherein said catalyst is provided in said liquid sulfur at said submerged location by introducing the catalyst directly into a vortex formed by rotation of the impeller within the shroud.

77. The method of claim 66 wherein said catalyst is substantially non-volatile and is dissipated in said liquid sulfur.

78. The method of claim 66 which is carried out at a Gas Displacement Index (GDI) value of about -0.5 to about 0.95 as determined by the relationship:

$$GDI = \frac{2V_i \cdot \omega - Q}{2V_i \cdot \omega}$$

where V_i is the volume swept by the impeller (m^3), ω is the ratio of rotation of the impeller (rad/s) and Q is the volumetric flow rate of gas in the impeller (m^3/s).

79. The method of claim 66 or 78 which is carried out at a Mixing Index (MI) value of at least about 0.1 as determined by the relationship:

$$MI = \frac{2V_i \cdot \omega - Q}{V_r}$$

where V_i is the volume swept by the impeller (m^3), ω is the rate of rotation of the impeller (rad/s), Q is the volumetric flow rate of gas to the impeller (m^3/s) and V_r is the volume of the reactor (m^3).

80. The method of claim 66 which is effected at sulfur temperature of about 120° to about 160°C.

81. A method of removal of hydrogen sulfide from a gas stream, which comprises:

providing a rotary impeller comprising a plurality of blades at a submerged location in liquid sulfur surrounded by a shroud through which are formed a plurality of openings,

feeding said gas stream to said submerged location,
feeding an oxidizing gas for said hydrogen sulfide to said submerged location,

rotating said impeller about a substantially vertical axis at a speed sufficient to draw liquid sulfur into the interior of the shroud and to distribute said gas stream and said oxidizing gas as bubbles in said liquid sulfur to the interior of said shroud and to form a gas-liquid mixture of the bubbles of the gas stream and oxidizing gas in the liquid sulfur contained within the shroud, and

flowing said gas liquid mixture from within the interior of the shroud through and in contact with the openings to external of the shroud, thereby to effect reaction between hydrogen sulfide and oxidizing gas to sulfur.

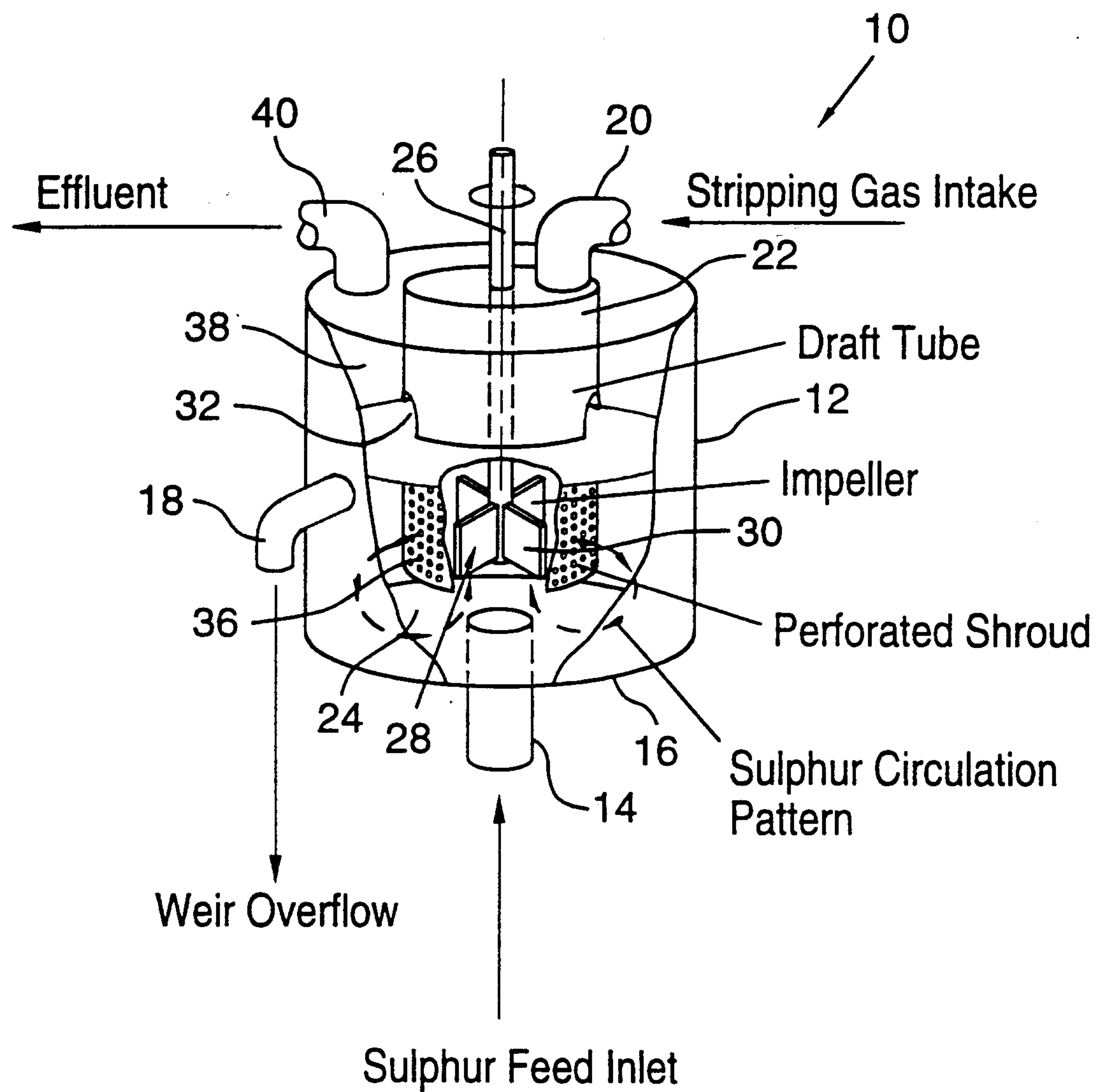
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FIG. 1

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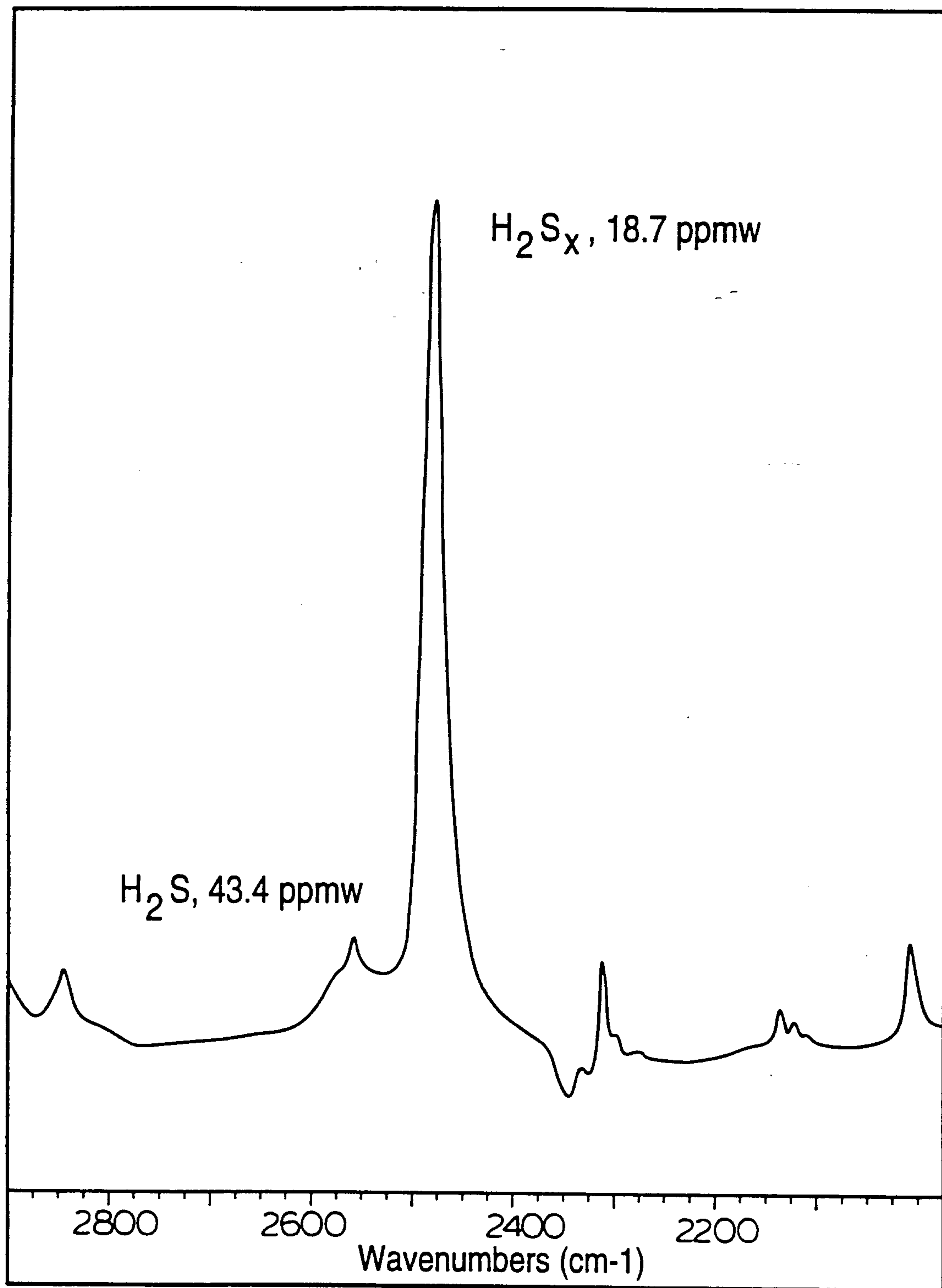
FTIR Spectrum of Liquid Sulphur With Residual H_2S and H_2S_x 

FIG.2.

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Concentration Decrease of H_2S and H_2S_x
Pilot-scale Contactor Using Air Sweep

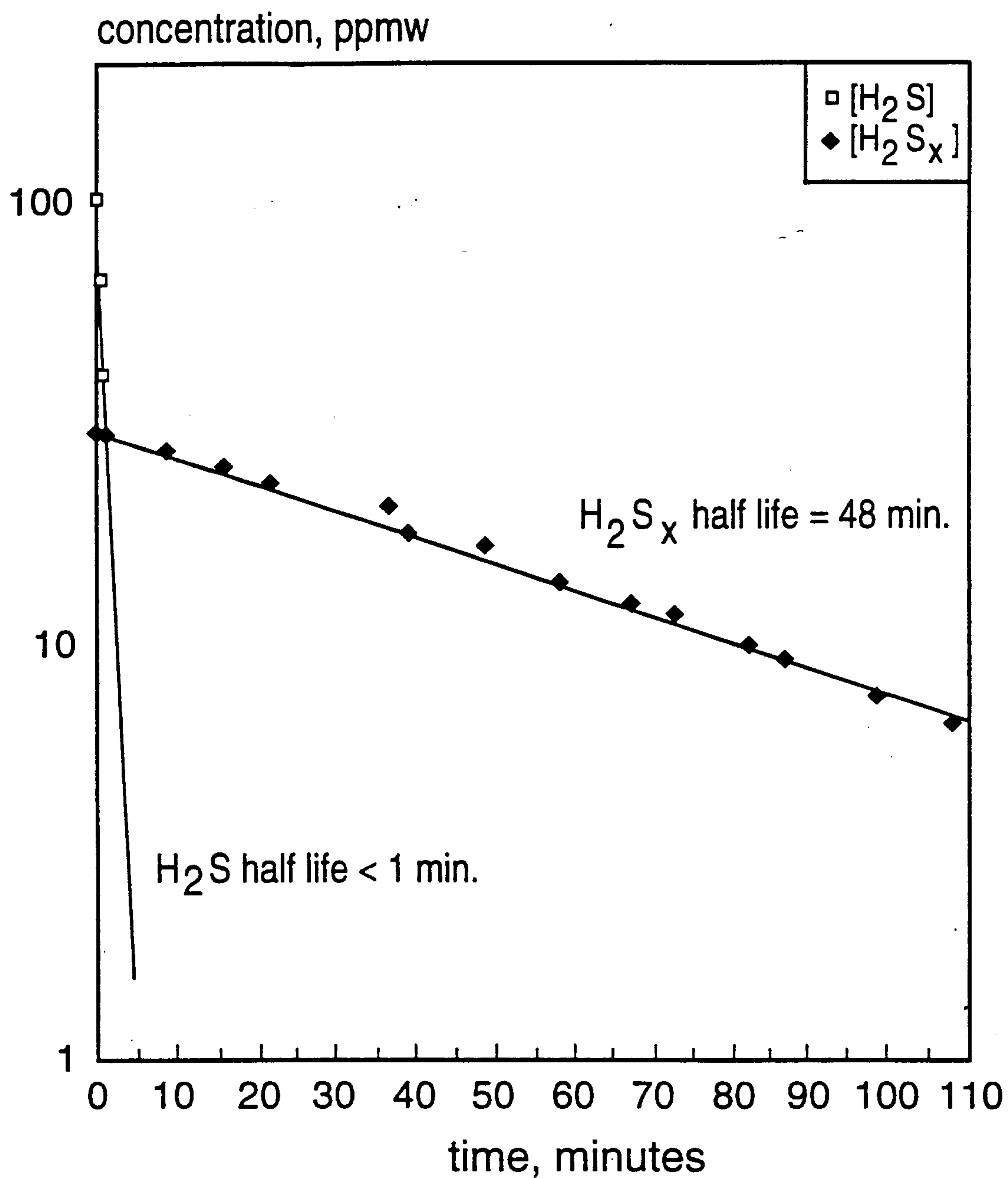


FIG.3.

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Effluent $[H_2S]$ vs. Time
Pilot-scale Contactor Using Air Stripping Gas

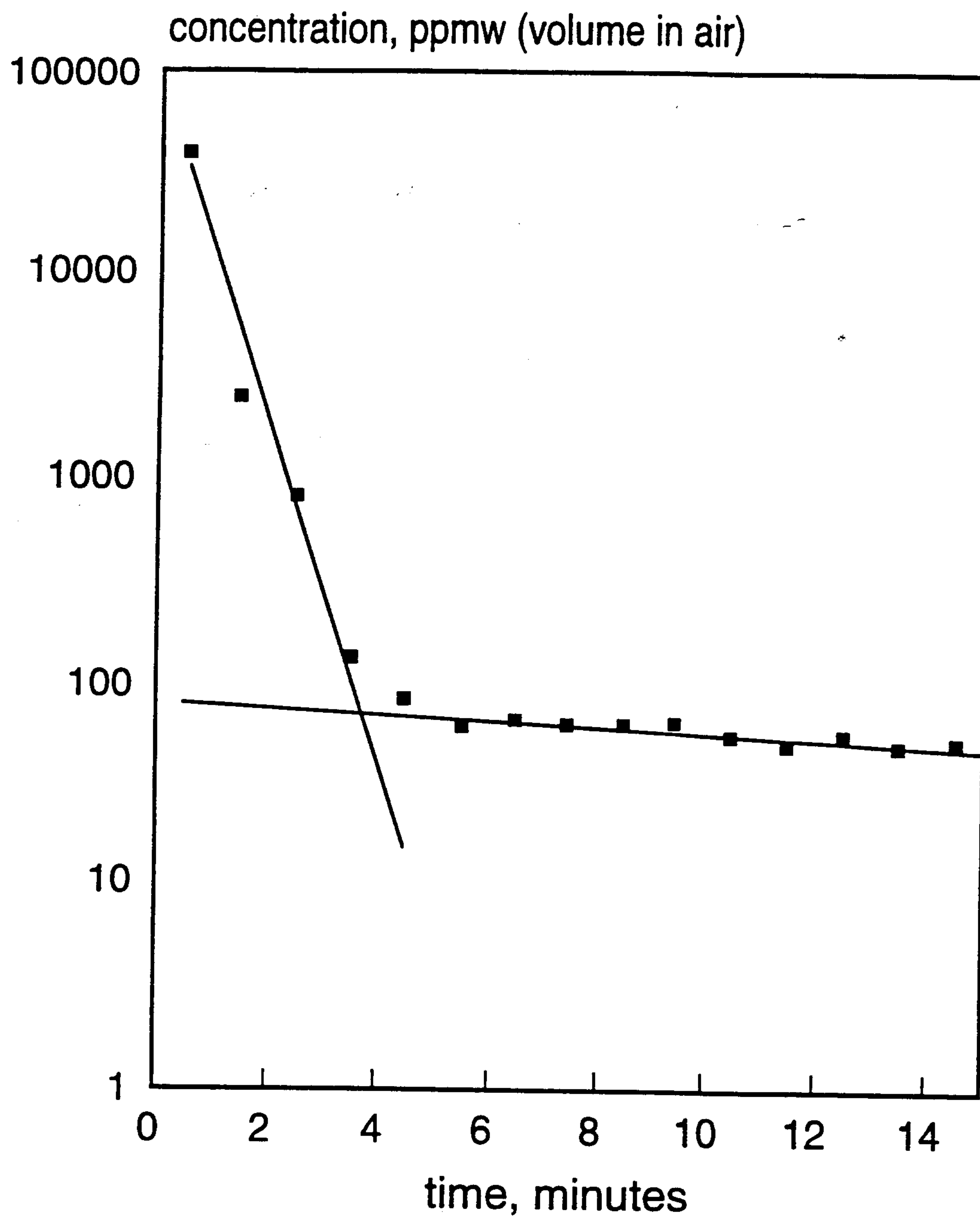


FIG.4.

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Effect of Catalyst on the H_2S_x Concentration Reduction Rate.
Bench-scale contactor using air stripping gas

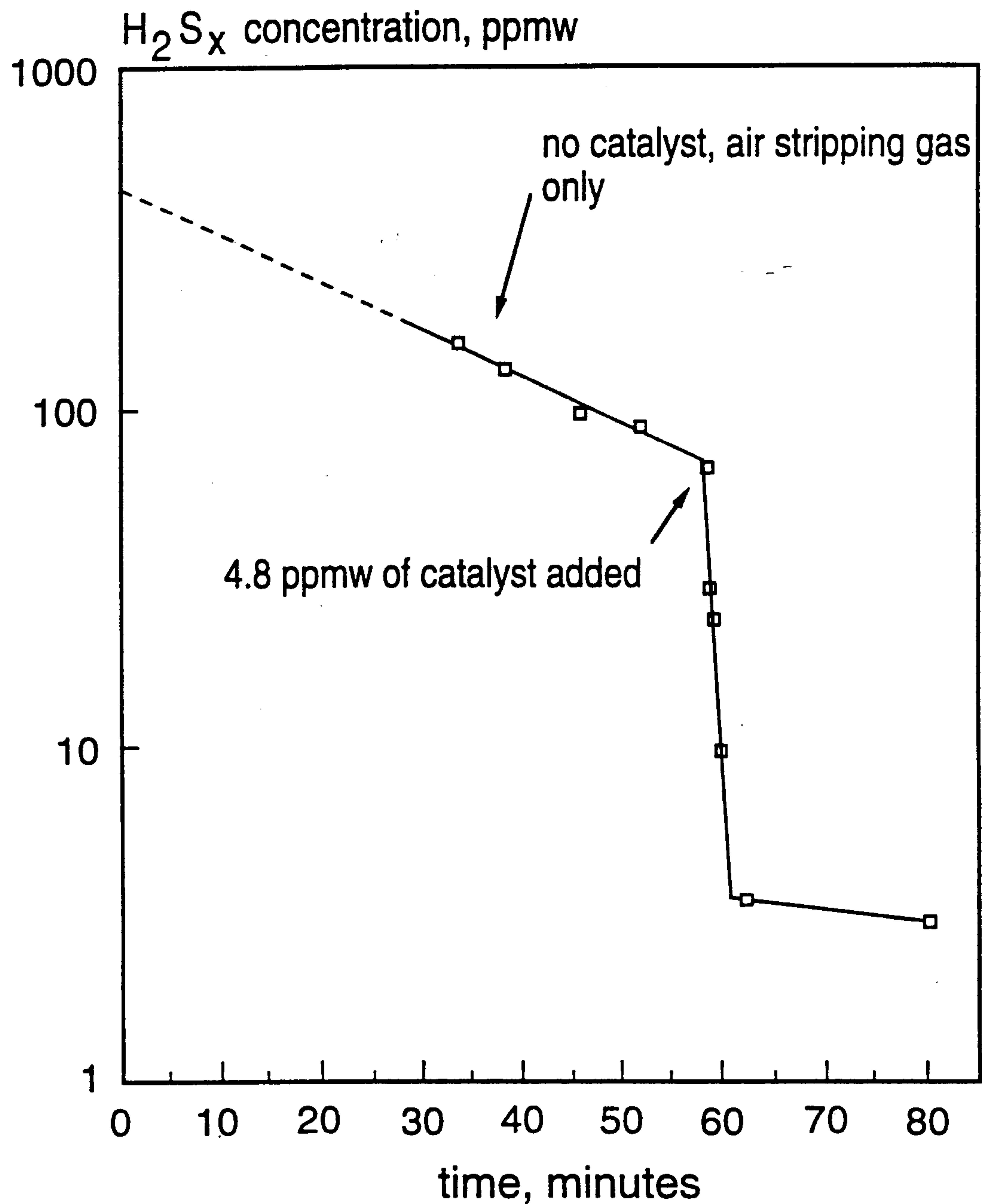


FIG. 5.

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History of H₂S and H₂S_x Peaks of FIG.5.

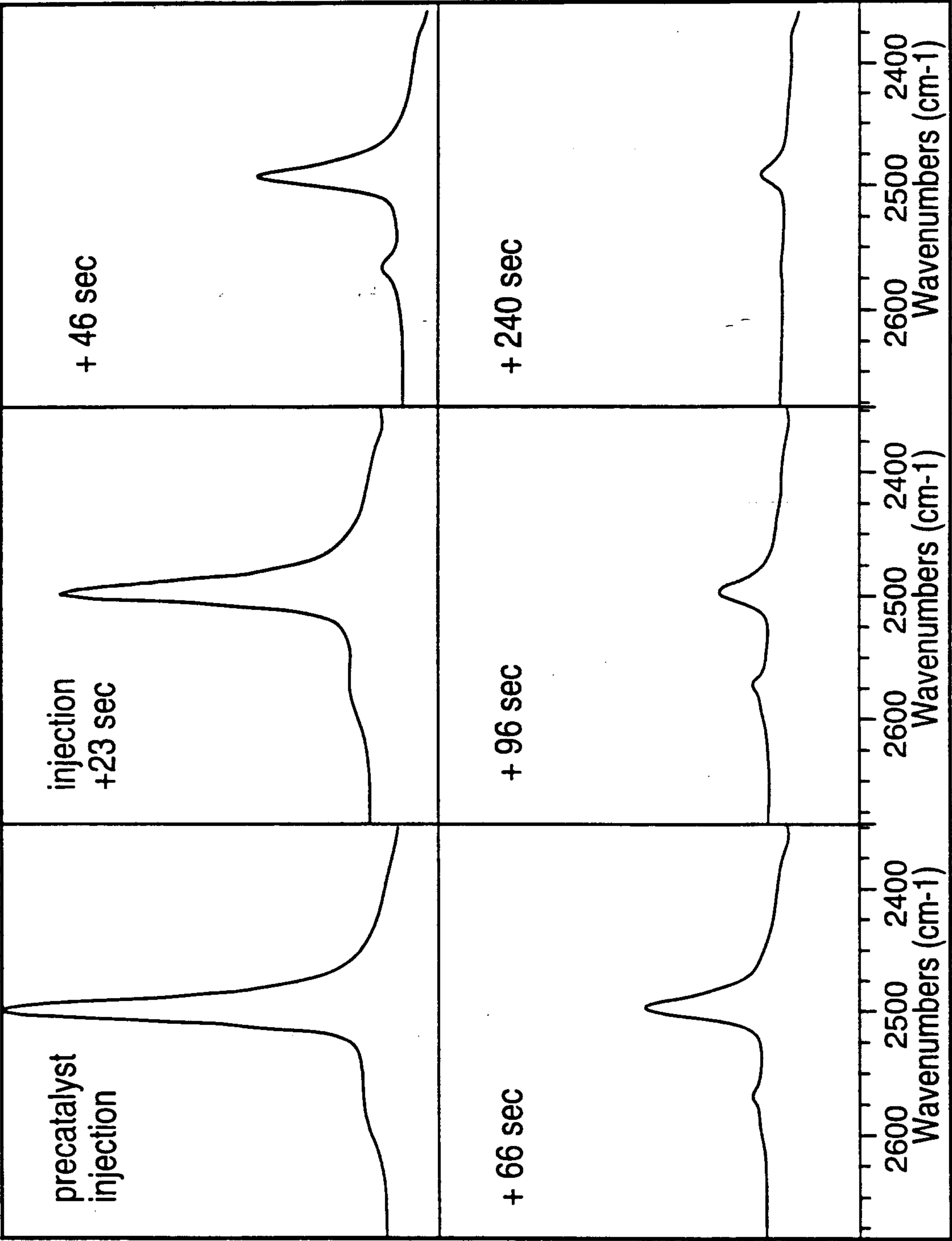


FIG.6.

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History of H₂S and H₂S_x Peaks Using the Pilot-scale Contactor

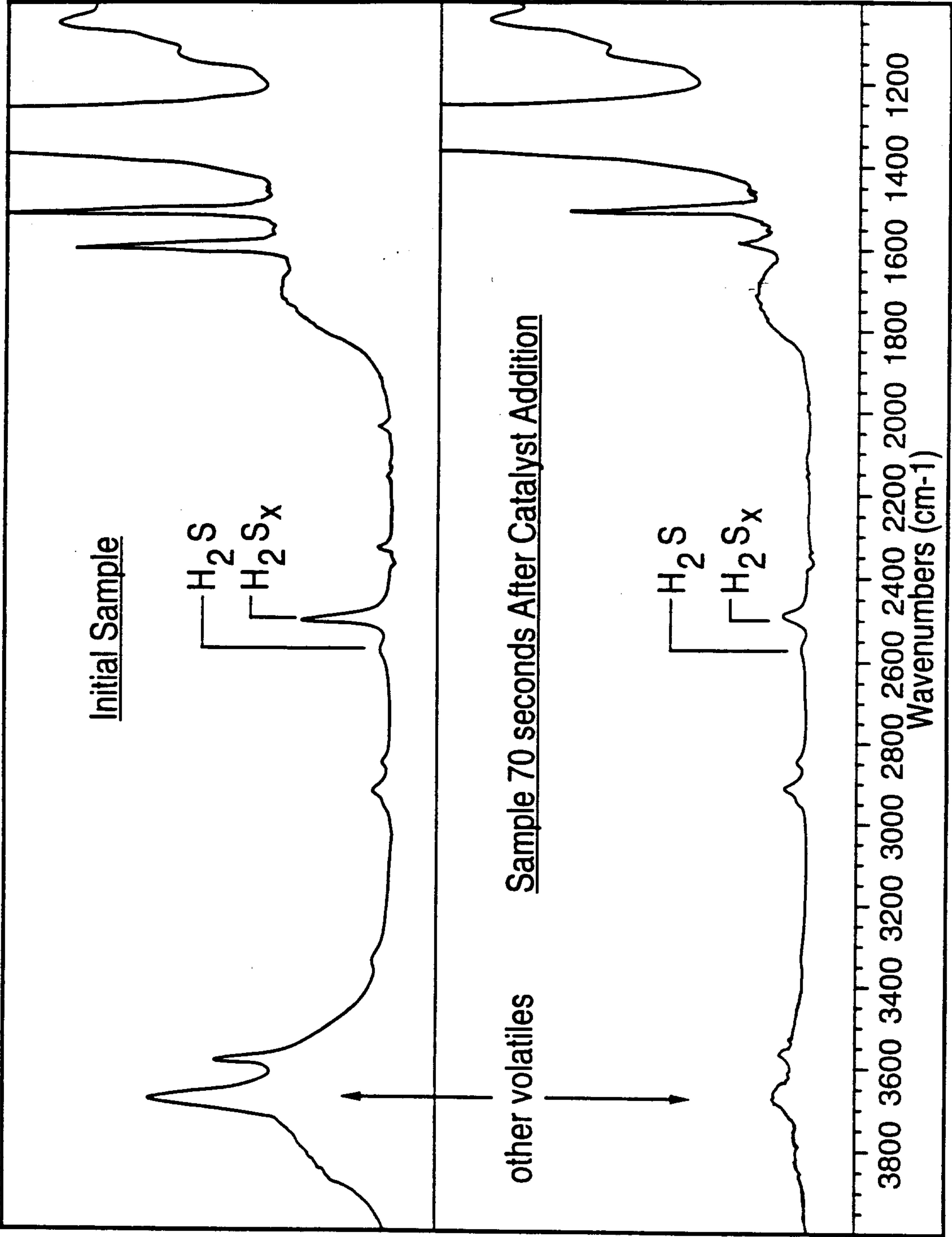


FIG. 7.

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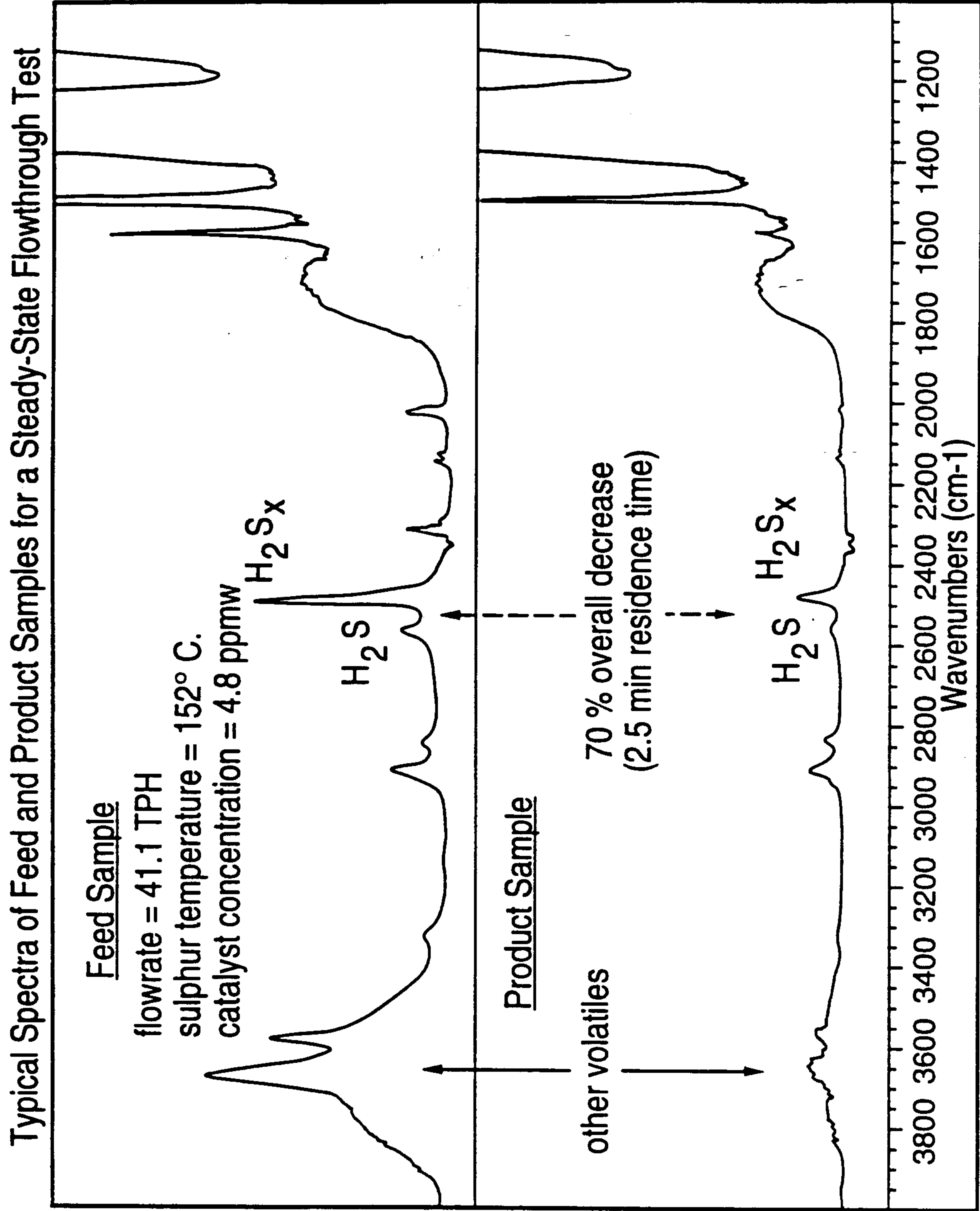


FIG.8.

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H_2S_x Half-life vs Catalyst Concentration
Pilot-scale Contactor, Continuous Flow Through Tests

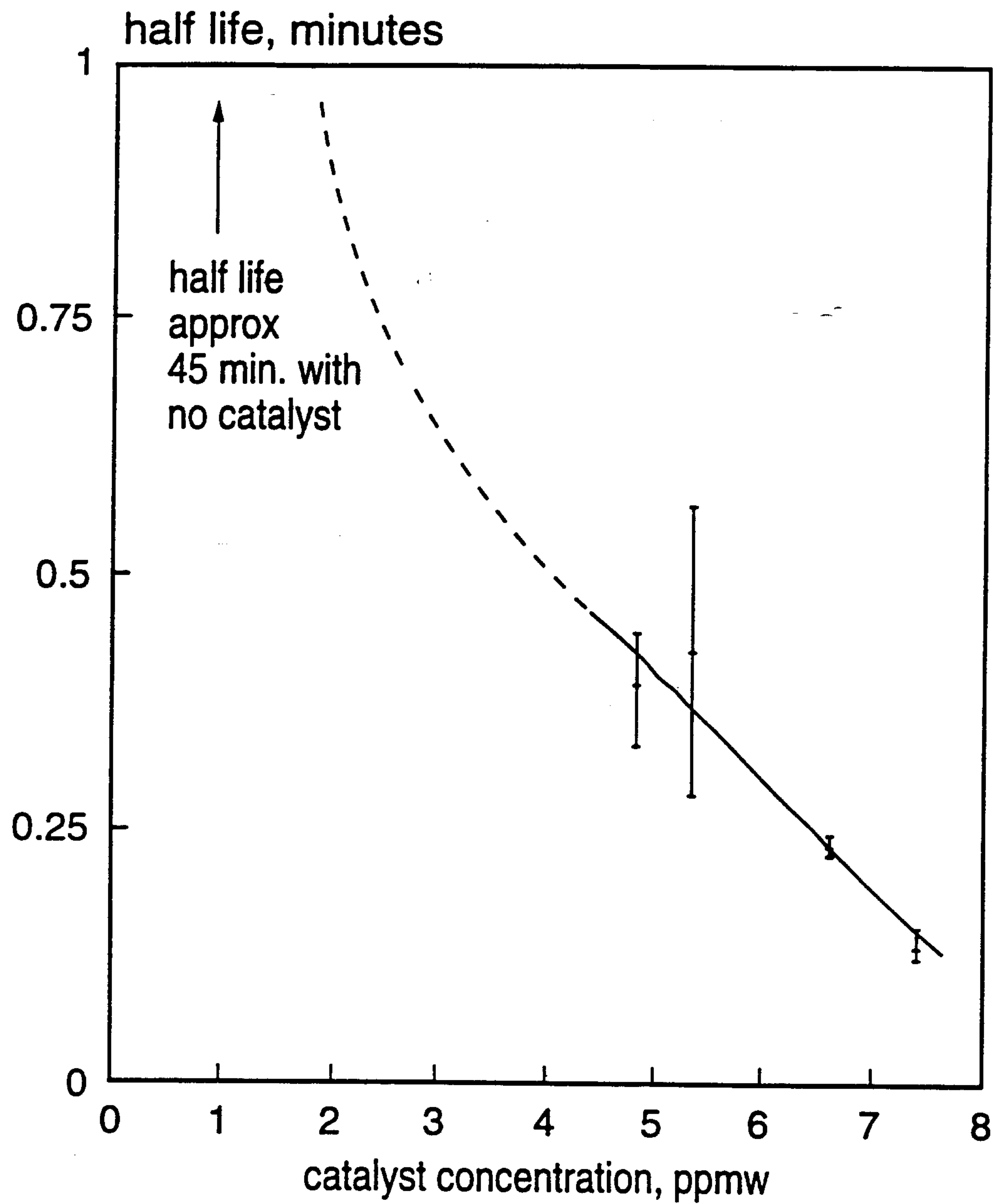


FIG.9.

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Spectra of Feed and Well Degassed Product

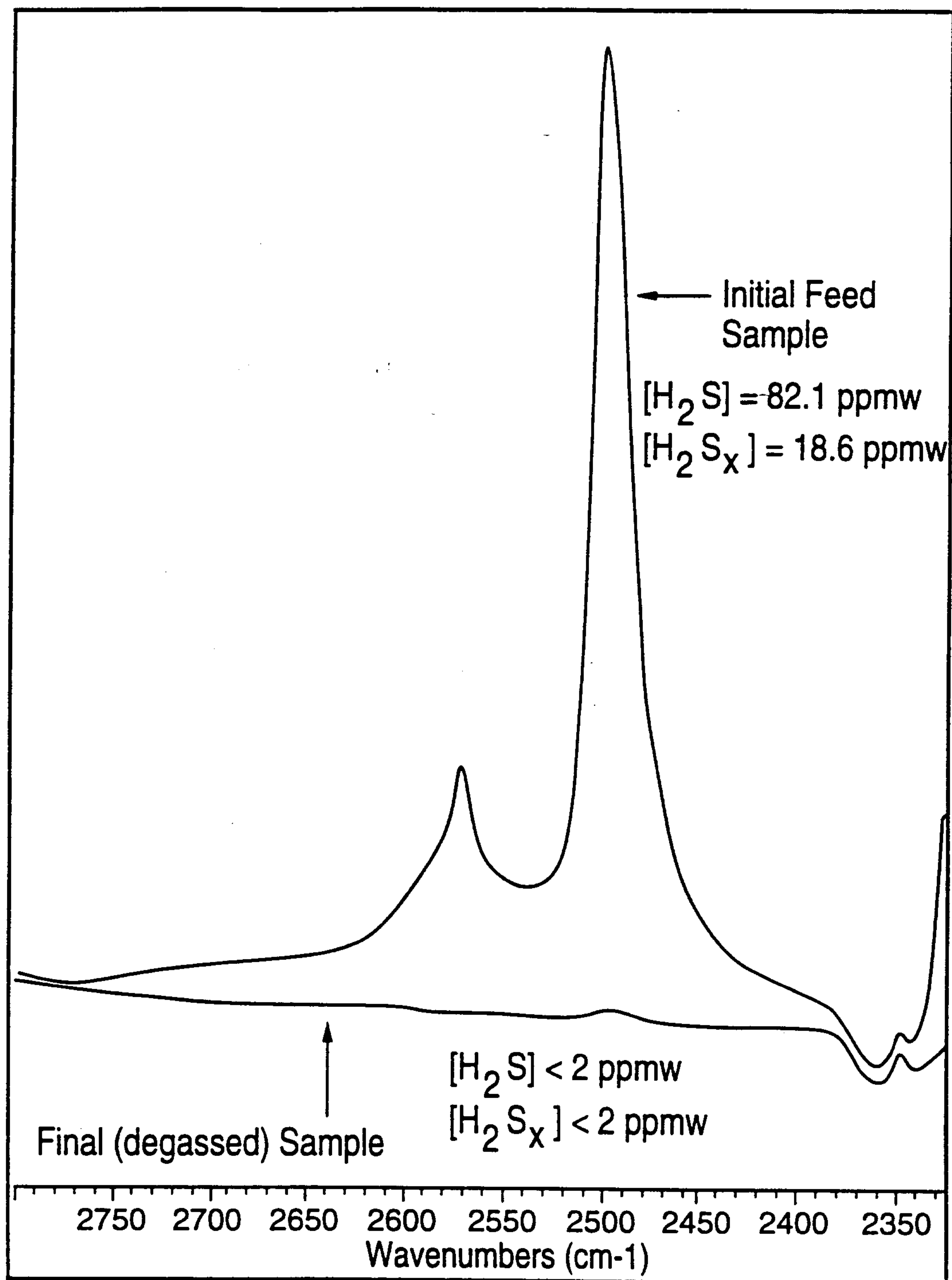


FIG.10.

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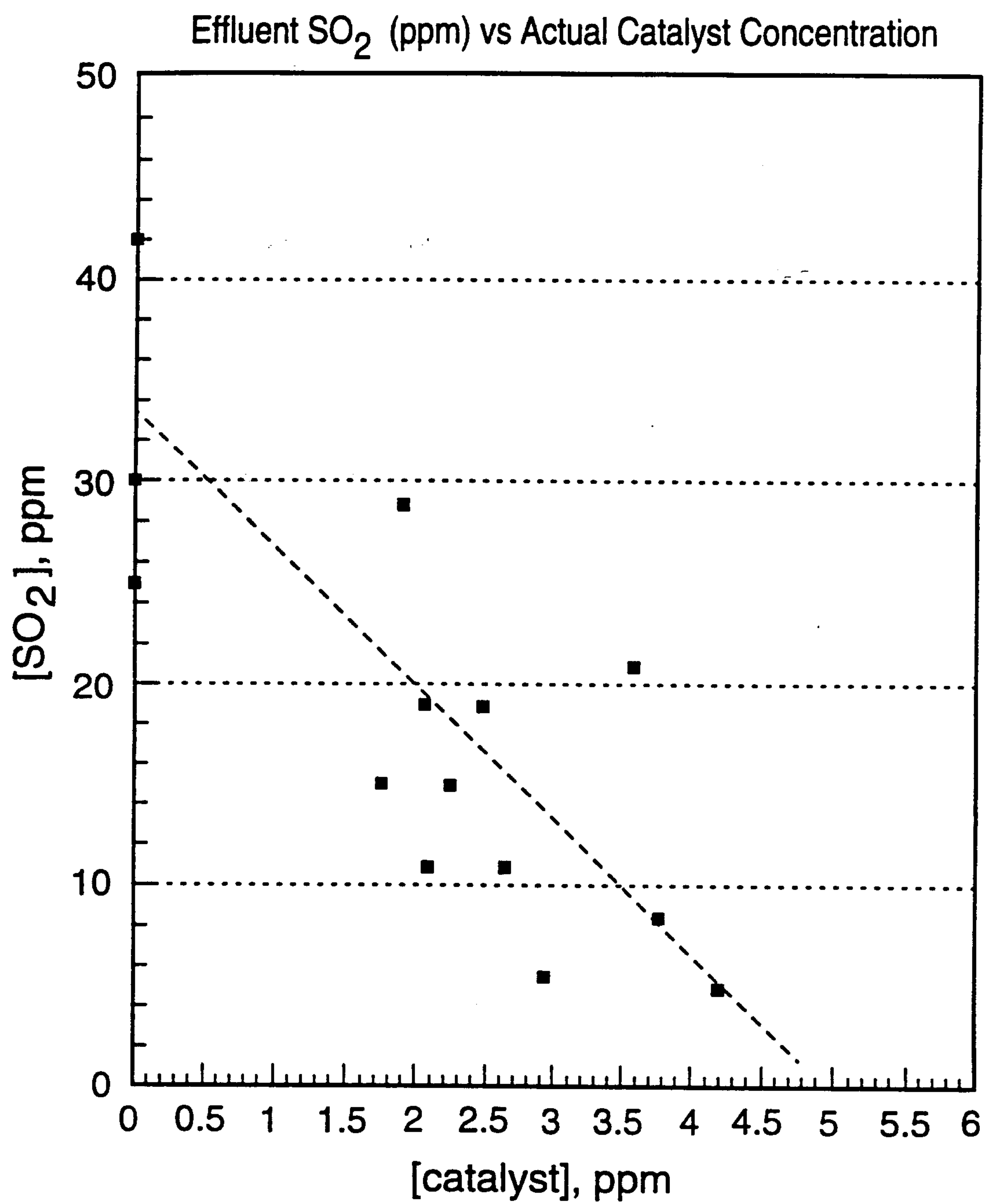


FIG.11.

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