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Microbially-Enhanced Redox Solution Reoxidation for Sweetening Sour Natural Gas

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CONTRACT INFORMATION

Contract Number:	DE-FG21-94MC31162
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METC Project Manager:	Harold D. Shoemaker
Period of Performance:	June 1, 1994 - May 31, 1996
Schedule and Milestones:	FY'95-96 Program Schedule

Task No. and Activity	By Quarters: Year 1 1	2	3	4	Year 2 1	2	3	4
TASK 1 - Fe^{3+}•EDTA/NTA Oxidation of H_2S and Regeneration	▬▬▬▬▬▬▬▬							
TASK 2 - LO-CAT 310 and LO-CAT 340 Experimentation		▬▬▬▬▬▬▬▬						
TASK 3 - LO-CAT 310/340 and Fe^{3+}•EDTA/NTA Degradation Experimentation			▬▬▬▬▬▬▬▬					

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OBJECTIVES

About twenty five percent of natural gas produced in the United States is sour containing significant volumes of hydrogen sulfide and other contaminants (1, 2). Liquid redox processes remove hydrogen sulfide from natural gas. Aqueous solution of chelated ferric ions oxidize the hydrogen sulfide to elemental sulfur. The reduced iron chelate is then oxidized by contact with air and recycled. This requires expensive equipment for regeneration, costly chemicals and the process is usually energy intensive (3, 4, 5).

Recent studies by Rai et.al (12, 16) show that the ferric ion regeneration rates are substantially enhanced in presence of acidophilic bacteria. The specific objectives of this project are to advance the technology and improve the economics of the commercial iron-based chelate processes utilizing biologically-enhanced reoxidation of the redox solutions used in these processes, such as LO-CAT II and SulFerox.

BACKGROUND INFORMATION

The Liquid Redox Sulfur Recovery Processes absorb hydrogen sulfide from the sour gas stream and produce elemental sulfur. The liquid redox processes may use vanadium, iron or a mixture of iron and quinone as the primary catalysts interacting with hydrogen sulfide. The iron-based processes have been most successful because of their superior performance, simple operation, greater reliability and environmental acceptability (6). However, the process conditions promote the oxidation reactions that accelerate the decomposition of metal-chelate catalysts resulting in high processing costs, and recirculation power requirements. Moreover, in all the commercial liquid redox processes, expensive redox solution is lost via salt formation and inadequate washing of the sulfur cake produced (7).

Redox Process Chemistry:

The iron-based redox processes employ iron in the ferric state (Fe^{3+}) to oxidize hydrogen sulfide to the elemental sulfur (S^o). The ferric ion is reduced to the ferrous state (Fe^{2+}), which is then regenerated to the ferric state by oxidation with air as follows:

$$H_2S_{(l)} + OH^- \leftrightarrow HS^- + H_2O$$
$$HS^- + OH^- \leftrightarrow S^= + H_2O$$
$$S^= + 2\,Fe^{3+} \leftrightarrow S^o + 2Fe^{2+}$$
$$2Fe^{2+} + \tfrac{1}{2}O_2 + H_2O \leftrightarrow 2Fe^{3+} + 2OH^-$$

Typical iron concentrations in the chelated catalysts range from 500 to 2500 ppm as determined by economics involving pumping and chemical costs. The LO-CAT chelated catalysts can handle concentrations of H_2S as high as 100% and there appears to be no lower limits (9).

Neither ferrous nor ferric ions are stable in aqueous solutions at neutral or alkaline pH levels and ordinarily will precipitate either as ferrous or ferric hydroxide. This precipitation is prevented by complexing the iron with organic chelates which are capable of holding both forms of iron in solution. These organic chelates are classified into two groups: Type A chelates such as ethylenediaminetetraacetic acid (EDTA) or nitrilotriaectic acid (NTA) which are powerful chelating agents at low pH's; and the type B chelates, consisting of polyhydroxylated sugars such as sorbitol that are effective at pH above 8. Combination of both types of chelates makes the catalyst stable at any pH from 5 to 9.0.

The selection of a chelant is dependent on the reaction rate of Fe^{3+} - chelate with H_2S, Fe^{3+} - chelate with oxygen and the rate of degradation of the chelate. The chelate degradation occurs through the oxidation of the chelate by Fe^{3+} ion and the free radical induced oxidation (8). Other variables that control the oxidative degradation are: pH, temperature, chelant concentration, chelant to iron ratio, and the type of degradation products formed.

The LO-CAT™ process was originally developed by ARI Technologies, now Wheelabrator Clean Air Systems, Inc., to treat sour gas in the absorber at feed gas pressure, relatively low iron concentrations (1000 to 1500 ppmw) and high circulation rates. This system referred to as conventional LO-CAT works well for many low-pressure plants, however it results in excessive equipment and pumping costs for high pressure applications. The ARI-LO-CAT II process as shown in Figure 1, was developed for the high pressure direct treat applications (9). The process uses

substoichiometric iron chelated catalyst in the absorber and an oxidizer unit that circulates liquid through density differences. The process is described in greater detail in the literature (9, 10). The process also uses a separate sulfur settler vessel. These features reduce the chemical and operating costs.

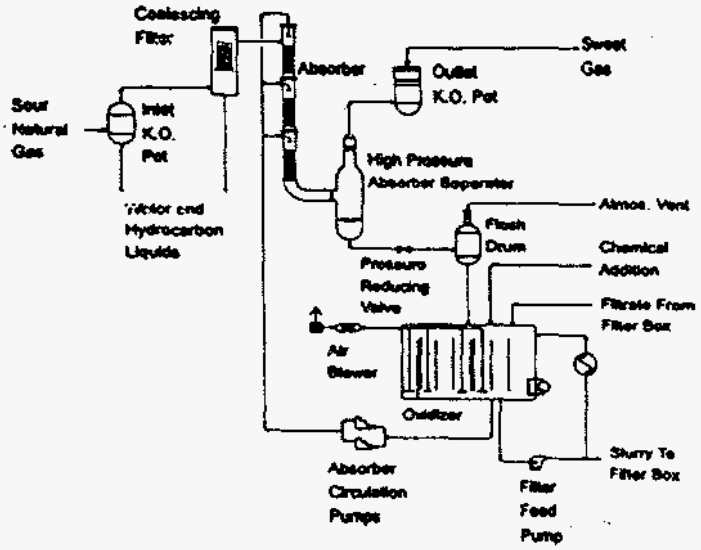

Figure 1. Process Flow Diagram for the ARI-LO-CAT II System

PROJECT DESCRIPTION

The iron-oxidizing bacteria are capable of oxidizing ferrous ions to the ferric state at low pH. According to the literature references these microbes are capable of oxidizing Fe^{2+} to Fe^{3+} state at 500,000 times faster rate than the purely chemical oxidation process in the absence of bacteria (11). The regeneration of Fe^{3+} chelate in the presence of acidophilic microbes under mild conditions at 25-45°C, and atmospheric pressure would minimize the chelate degradation process and thus help in improving the economics of hydrogen sulfide oxidation in the natural gas sweetening process. The Fe^{3+}-chelates are also capable of oxidizing the mercaptans to the insoluble disulfides (12). It is proposed to use these microbes for achieving enhanced ferric ion reoxidation rates in ARI-LO-CAT II process thereby improving the overall sour gas processing economics.

The basic objective of this study, jointly sponsored by U.S Department of Energy and Gas Research Institute, is to develop information and technology to improve the economics of the commercial iron-based redox processes such as ARI-LO-CAT II and SulFerox, with emphasis on the biologically-enhanced reoxidation of the redox solution used in these processes. In this study, a mixed culture of iron oxidizing bacteria are used to regenerate the commercially used iron chelates for reoxidation of reduced redox solutions. There are more than forty gas processing units nationwide using liquid redox technology. These gas processing plants could use the new technology being developed in this project and thus lower the gas processing costs of sub-quality sour natural gas substantially.

Mechanism of Microbial Oxidation of Ferrous Iron:

The iron oxidizing bacteria derive the energy required for their growth from the oxidation of reduced sulfur compounds and from the oxidation of Fe^{2+} to Fe^{3+} ions, using air as an oxidant. The major electron transfer components of the respiratory chain of the iron oxidizing bacteria have been postulated by Ingledew et.al (13) and Cox et.al (14). These components are organized in the cytoplasmic membrane in such a way as to couple Fe^{2+} oxidation to generate a transmembrane proton electrochemical potential. This potential is the main driving force for electron transfer. A diagrammatic representation of electron transfer mechanism is shown in Figure 2. The major electron transfer components of the respiratory system of the bacteria are comprised of: a cytochrome oxidase, cytochrome c, cytochrome a and a blue colored copper protein, rusticyanin (15). Ferrous ion oxidation takes place at the cell wall and generates a transmembrane electrochemical potential of 250 mV. The reduction of molecular oxygen is catalyzed by a cytochrome oxidase at a pH of 6.5 on the inside of the cytoplasmic membrane (16).

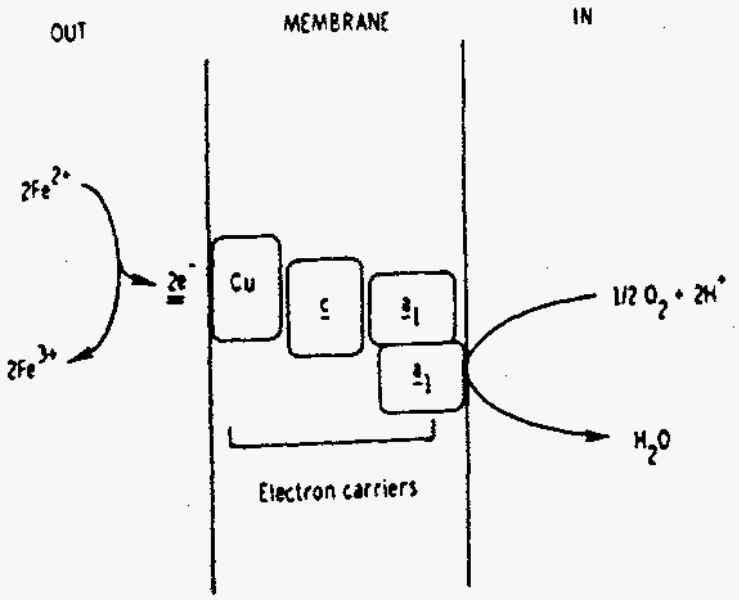

Figure 2. Components of the iron oxidase are identified by their prosthetic groups and are arranged from left to right in order of increasing redox potential (Cobley and Haddock, 1975; Ingledew and Cobley, 1980). "Out" and "in" refer to the bulk phase and cytoplasm, respectively.

Materials and Methods

a.Growth Characteristic of Iron Oxidizing Bacteria

The proprietary iron oxidizing bacteria used in this study were maintained in basal salt solutions at a low pH prior to their use in these experiments. One bacteria (Bacteria A) was grown in 9K media and the other bacteria (Bacteria B) was grown in a high pH nutrient media. These bacteria were also grown in a redox solution system for three to five days prior to use in a high pH media maintained at 25° to 45°C in a controlled temperature shaker bath. The composition of nutrient media is shown in Table I. The iron oxidizing bacterial mixed cultures used in this study were initially obtained from American Type Culture Collection (ATCC), however, they were cultivated either in a high pH media or grown in the redox solution used for the hydrogen sulfide oxidation studies. The cultures were grown separately and then mixed and also were grown in the same media. The maximum cell growth typically occurred in 25 to 50 hours resulting in a cell density of 1.5×10^{11} cells/l in high pH media. The cell densities of 1.0 to 2.0×10^{11} cells/l were achieved in the redox system solutions. Cell densities of $(1.0\text{-}1.5) \times 10^{9}$ cells/l were used in the experiments carried out in the presence of the bacteria. Bacterial cell counts were determined using a Petroff-Hauser bacterial cell counter under a phase contrast microscope.

Table I

High pH Medium for Iron Oxidizing Bacteria

Composition per liter:

$Na_2S_2O_3 \cdot 5H_2O$	10.0g
$Na_2HPO_4 \cdot 7H_2O$	7.9g
Sodium formate	6.8g
Glucose	3.6g
KH_2PO_4	1.5g
NH_4Cl	0.3g
$MgSO_4 \cdot 7H_2O$	0.1g
Trace metals solution	5.0mL

pH 7.6-8.5 at 25°C

b. Gas Samples and Chemicals:

Synthetic sour gas samples used in this study were blended by Alphagaz Inc. of LaPorte, Texas. The synthetic sour gas had the following composition:

H_2S - 0.5 (v/v)
CO_2 - 5% (v/v)
N_2 - 94.5%

Two types of commercial catalysts, Catalyst A and Catalyst B, were used in this study. These catalysts contain chelated ferric ion complexes. Precipitation of ferric hydroxide is prevented by chelating the ferric ions with organic chelates. Two types of chelates: type A, such as ethylene diaminetetraacetic acid (EDTA) or nitrilotriacetic acid (NTA) and type B, such as polyhydroxylated sugars keep the catalyst stable at any pH and were used in the commercial chelated catalyst formulations. This paper presents data using catalyst A only. All other chemicals used were obtained from Sigma Chemical Company.

c. Experimental Procedure:

The oxidation of hydrogen sulfide present in the synthetic sour gas blend was studied in a two-liter Virtis Omni Culture Bioreactor shown in Figure 3 (17, 18). The hydrogen sulfide is readily oxidized by the chelated ferric ions present in the commercial catalyst A used in this study. The ferric ions (Fe^{3+}) are reduced to the ferrous state (Fe^{2+}) and hydrogen sulfide is oxidized to elemental sulfur. The elemental sulfur is removed by filtration or centrifuging and the ferric ion is regenerated by bubbling air through the reduced redox solution under controlled experimental conditions. The rate of hydrogen sulfide oxidation is a function of the pH, temperature, concentration of Fe^{3+} chelate, the gas/liquid ratio and the degree of agitation. These variables were carefully controlled and optimized. Likewise, the rate of ferric ion regeneration is a function of the pH of the redox solution, the temperature, the concentration of chelated iron, air to redox solution ratio and the degree of agitation. The progress of the reaction was monitored by measuring the concentration of Fe^{2+}, Fe^{3+}, pH, temperature, and redox potential of the reaction medium in the Virtis Omni Culture Bioreactor. Two sets of experiments were conducted in each case, one in absence of bacteria (blank) and the other one in presence of a single bacteria or a mixed culture.

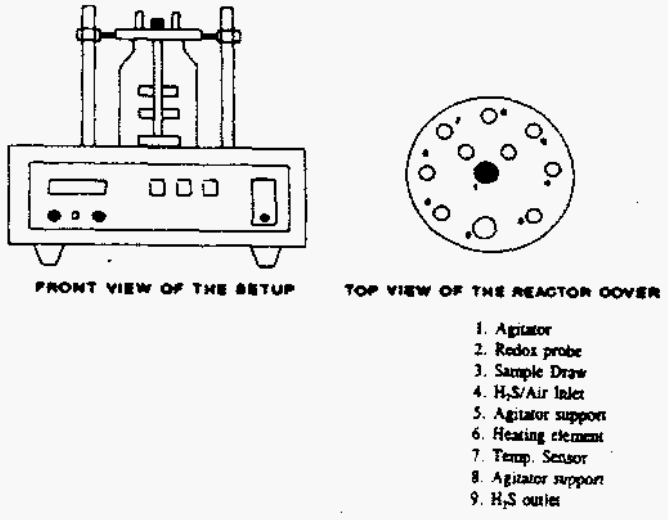

Figure 3. Omni Culture Bio-Reactor

1. One-Cycle Experiments Using Commercial Chelated Catalysts. (Absence of Bacteria) -Baseline.

A set of experiments was conducted at 30°C to 45°C and a pH varying from 3, 5 and 7.6 using 1000 ppm solution of commercial iron-chelate Catalyst A in absence of iron oxidizing bacteria (baseline). A cycle consists of oxidation of hydrogen sulfide by bubbling it through redox solution, filtration of elemental sulfur followed by reoxidation of ferrous ions with air. In a typical experiment, hydrogen sulfide was oxidized by passing the synthetic sour gas mixture through one liter of redox solution in Virtis Omni-Culture Bioreactor, the redox solution was regenerated by bubbling air through it in absence of bacterial cells (blank run) and elemental sulfur was centrifuged after each cycle. The data on these experiments is shown in Figures 4 to 6. The redox solution regeneration rates were fairly constant, for a specific pH, temperature and gas to liquid ratio in the control (baseline) experiments and the quantity of elemental sulfur recovered ranged from 35 to 50% of the theoretical amount.

2. One Cycle Experiments Using Commercial Chelated Catalyst A in Presence of Iron Oxidizing Bacteria.

In this set of one-cycle experiments the iron oxidizing bacterial cells of bacteria A, B or a mixed culture were used in the redox solution containing 1000 ppm of commercial chelated Catalyst A at 30°C to 45°C and a pH of 3.0 or 7.5. In a typical experiment, hydrogen sulfide was oxidized by bubbling the synthetic sour gas mixture through one liter of redox solution contained in the Virtis Omni-Culture Bioreactor, the redox solution was regenerated by bubbling air through the solution containing iron oxidizing bacteria or a mixed culture at a cell concentration of 1 to 7.5 x 10^9 cells/liter and elemental sulfur produced was filtered (not centrifuged) after each cycle. The data are presented in Figures 4, 5 and 6 and compared with the baseline experimental data obtained in absence of bacteria. The data show a ferric ion regeneration rate enhancement of 50 to 150% and an increased production of elemental sulfur, 80 to 98% recovery as compared to 35 to 50% recovery in absence of bacteria. The data on rate

enhancement is shown in Figures 7 and 8 and the data on sulfur recovery is given in Figures 9 and 10.

RESULTS

The oxidation of hydrogen sulfide present in the synthetic sour gas mixture blended by Alphagaz of LaPorte, Texas has been studied using a commercial chelated iron catalyst in a two-liter Virtis Omni-Culture Bioreactor. The rate of hydrogen sulfide oxidation was found to be primarily influenced by the pH, temperature, gas/liquid ratio and the concentration of iron chelate in the redox solution. Essentially all hydrogen sulfide was oxidized to elemental sulfur in the presence of commercial iron-chelate catalysts at a pH of 7.5 and 30 to 45°C. There was a 20% rate enhancement in hydrogen sulfide oxidation in the presence of mixed cultures.

The regeneration of the ferric ions in the chelated catalysts could be accomplished by bubbling air through the reduced chelated catalyst in the bioreactor. The air regeneration of the chelated ferric ions was dependent on the pH, temperature, air/redox solution ratio and the bacterial cell concentration. Single cycle experiments were carried out both in absence of iron oxidizing bacteria (blank), as well in presence of the bacterial cells. The ferric ion regeneration rates in the reduced redox solution were found to be 50% to 150% higher in presence of bacterial cells at typical cell density of 1 to 5 x 10^9 cells/l under optimum operating conditions. The data are presented in Figures 7 and 8 with a commercial chelated catalyst in one-cycle experiments.

The sulfur recovery was also studied in single cycle experiments. Invariably, 35 to 50% sulfur was recovered by centrifuging in the controlled blank runs, whereas in presence of mixed cultures of iron oxidizing bacteria the sulfur recovery ranged from 80 to 100% of the theoretical values. It was observed that filtration was preferred technique for sulfur recovery in presence of iron-oxidizing bacteria, since centrifuging affected the bacterial cell densities in the redox system. The sulfur recovery data for one-cycle experiments are shown in Figures 9 and 10.

FUTURE WORK

One of the bacterial strain (Bacteria A) was readily grown in 9K media and the other iron-oxidizing bacteria (Bacteria B) used in this study was grown in a high pH medium containing trace metals. Cell densities as high as 2 x 10^{11} cells/l could be achieved in twenty to fifty hours. Moreover, the high pH medium could be easily replaced by the used redox solution of the commercial catalyst evaluated in this study without adversely affecting the growth characteristics and the bacterial cell densities of the iron-oxidizing bacteria and the mixed cultures. The conditions for maximizing bacterial cell densities in the redox solutions will be studied and optimal parameters will be investigated.

These experiments conclusively show that in the presence of iron oxidizing bacteria, Bacteria A, or B or the mixed cultures, the rates of hydrogen sulfide oxidation are enhanced by about 20%, the ferric ion reoxidation rates in the redox system of the commercial chelated redox catalyst are enhanced by 50% to 150% as compared to blank runs in absence of bacterial cells at an operating pH of 7.5 and the redox solution temperature varying from 30° to 45°C. Moreover, the iron oxidizing bacteria also induce higher elemental sulfur recoveries ranging from 80 to 100% of theoretical as compared to 35 to 50% in absence of bacteria. The mechanism of bacterial action on sulfur recoveries will be investigated.

During processing of sour natural gas there is excessive degradation of the aminopolycarboxylic acid chelating agents such as Fe^{3+}•EDTA, Fe^{3+}•NTA and the commercial chelants used in the industry. Chelate degradation occurs through the oxidation of the chelate by Fe^{3+} ions, and the free radical induced oxidation (8). During the regeneration of Fe^{3+} chelate, hydroxy radicals are formed that degrade the Fe^{3+} chelate. Attempts will be made to determine the mechanism of chelate degradation more precisely and the role of bacterial cells in preventing such degradation. It has been observed in our preliminary laboratory study that the degree of chelate degradation is minimal in presence of bacterial cells (22).

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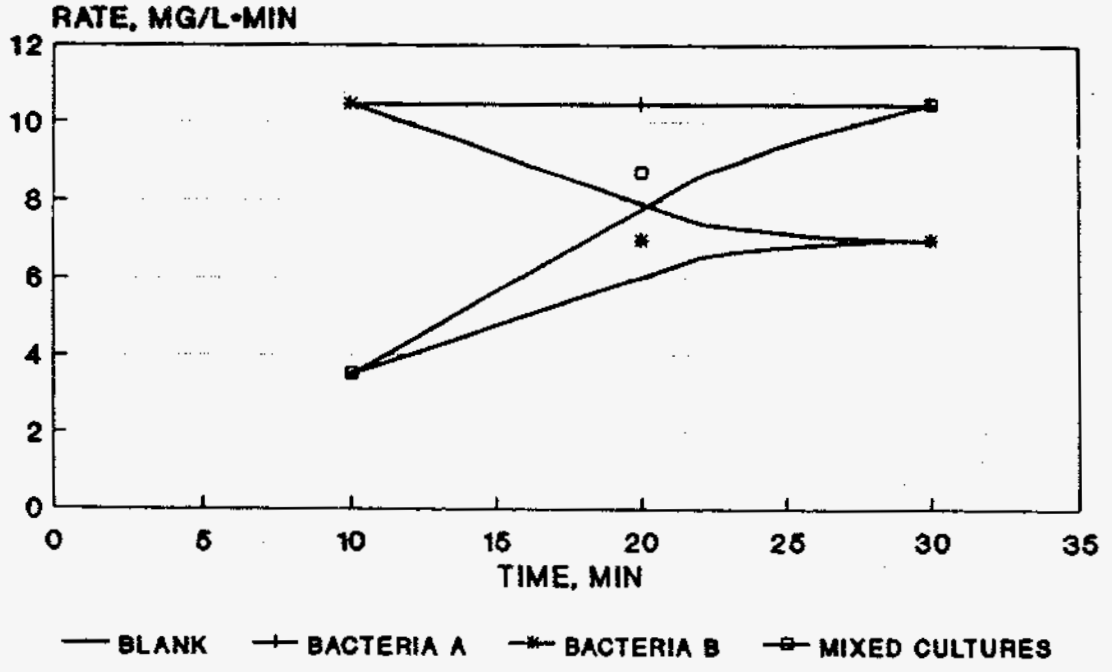


Figure 4A. Comparison of H_2S Oxidation Rates With and Without Bacteria at 30°C and pH, 3.0

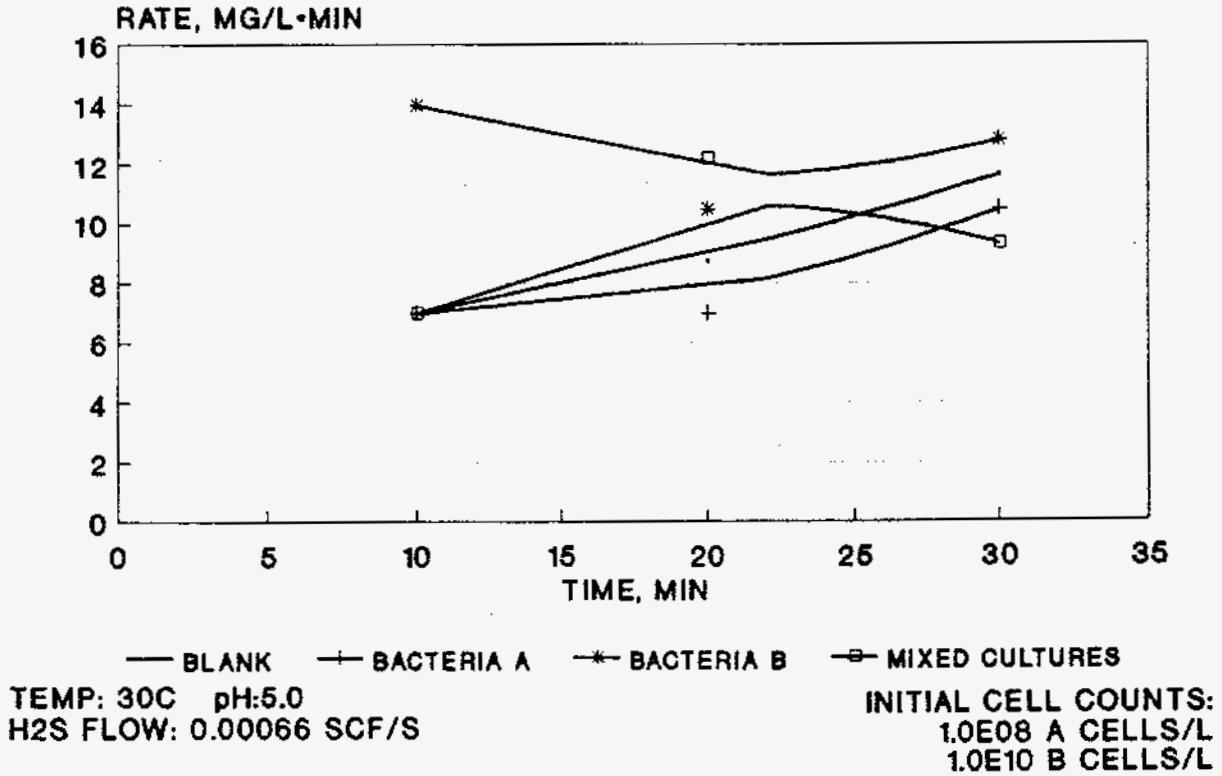


Figure 4B. Comparison of H_2S Oxidation Rates With and Without Bacteria at 30°C and pH, 5.0

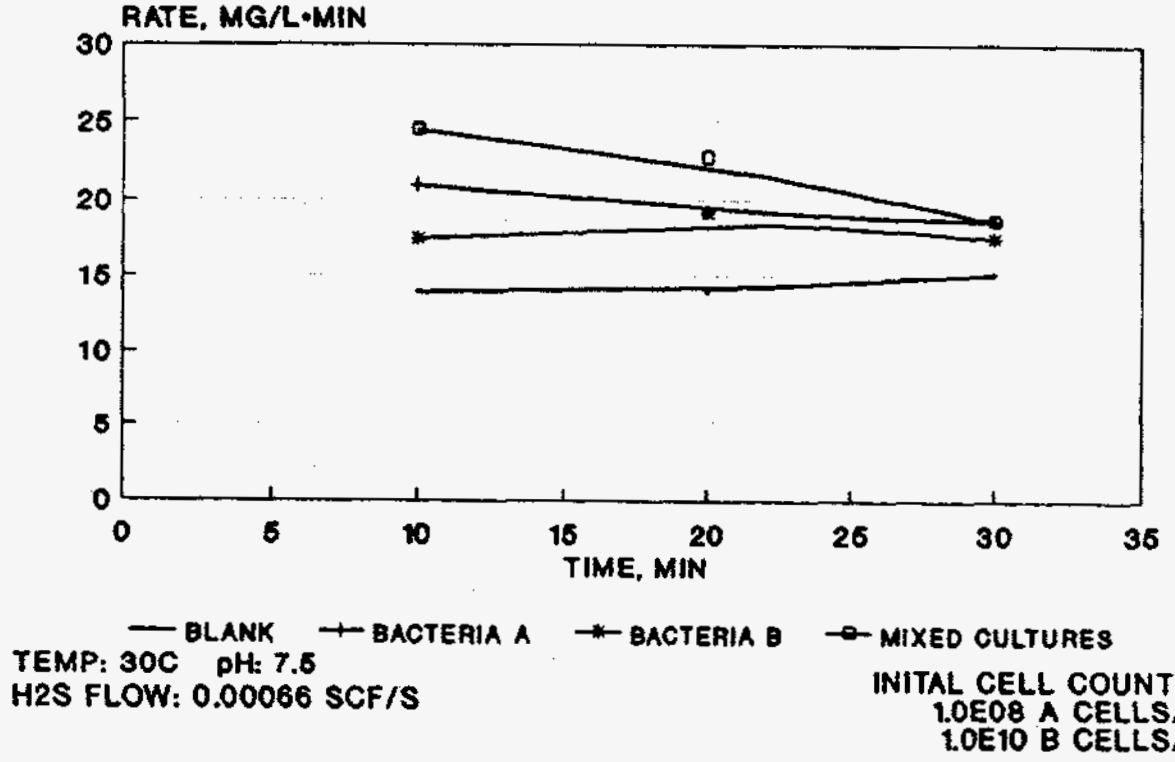


Figure 5. Comparison of H_2S Oxidation Rates With and Without Bacteria at 30°C and pH, 7.5

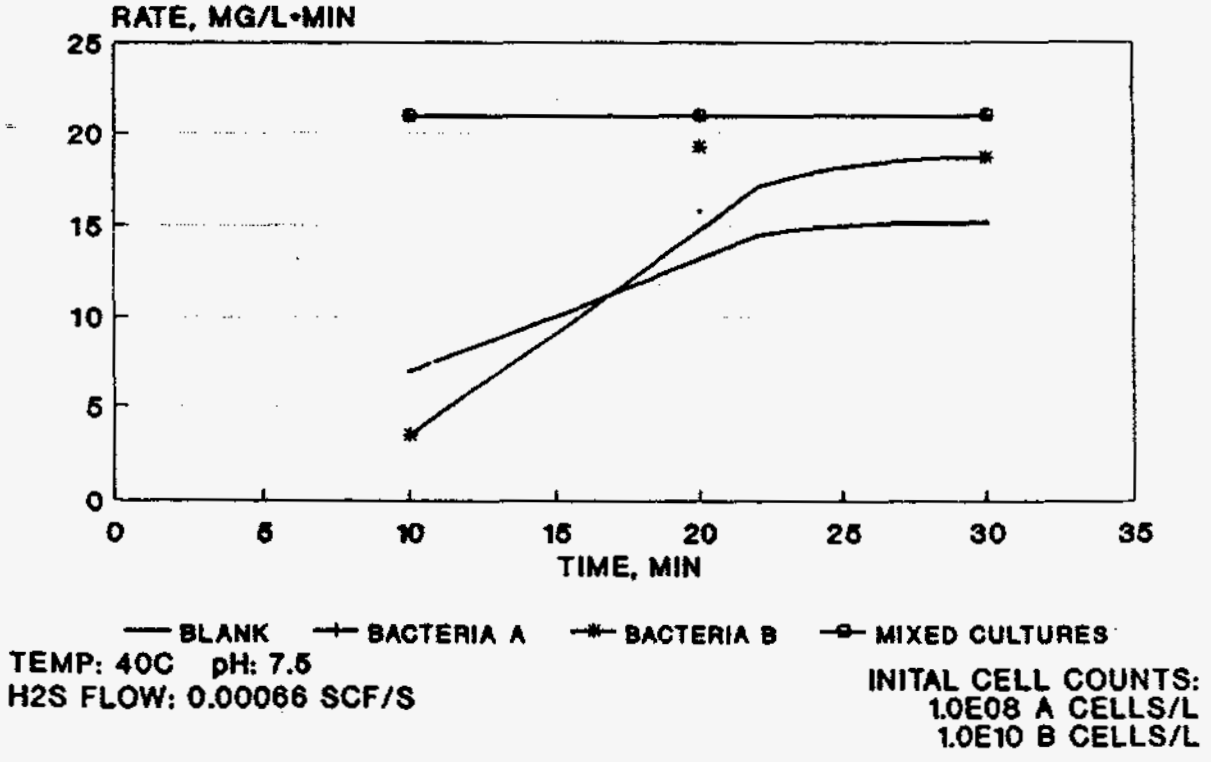


Figure 6. Comparison of H_2S Oxidation Rates With and Without Bacteria at 40°C and pH, 7.5

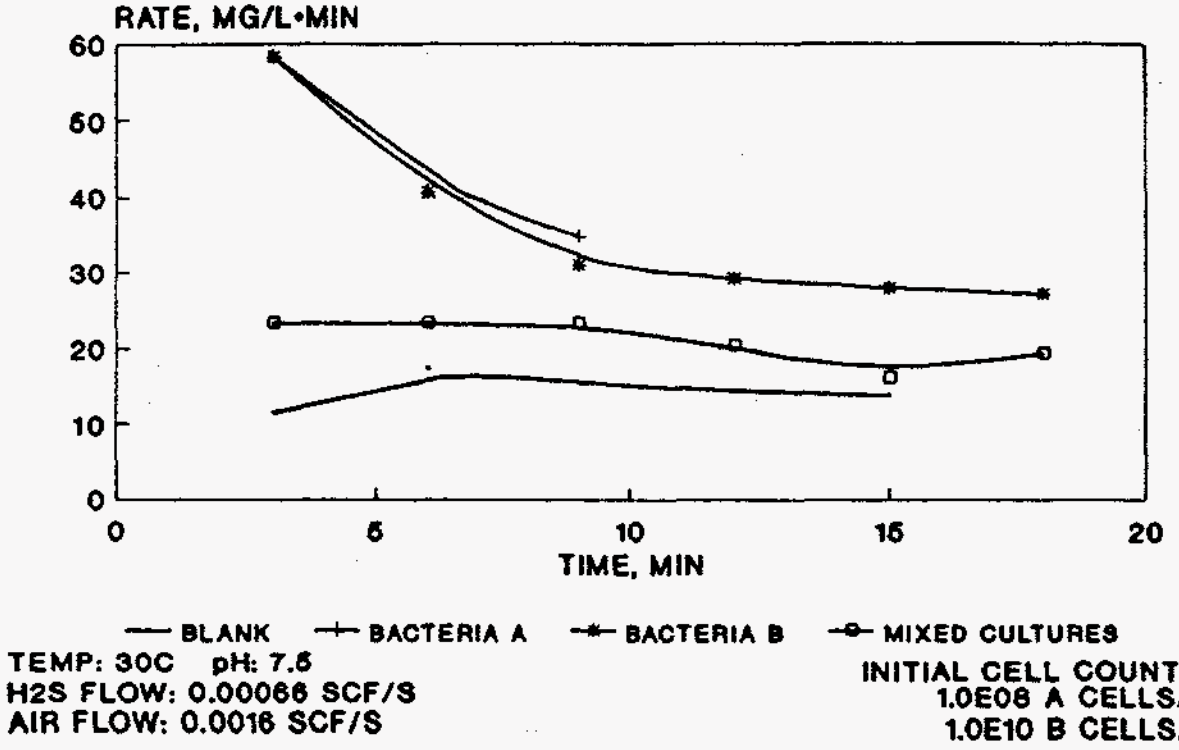


Figure 7. Comparison of Regeneration Rates With and Without Bacteria at 30°C and pH, 7.5

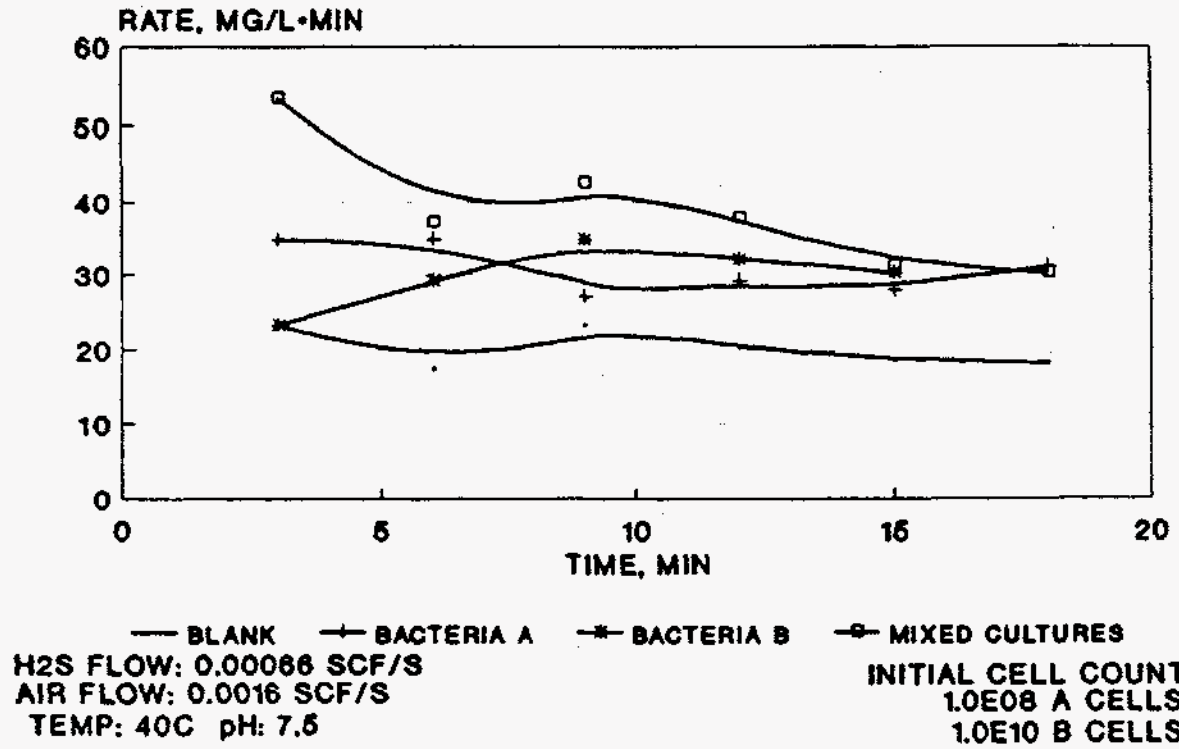
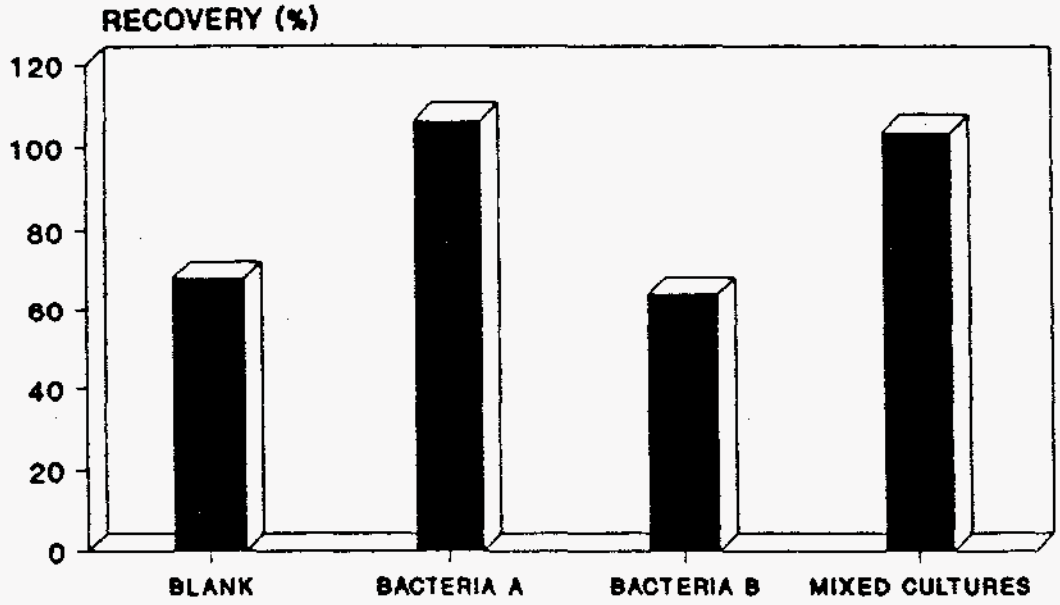


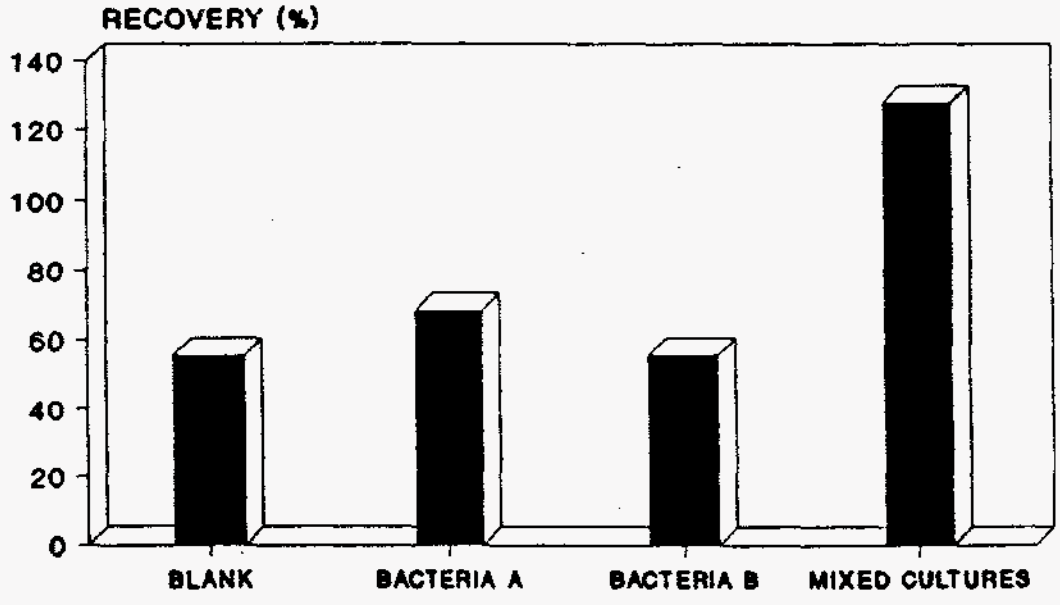
Figure 8. Comparison of Regeneration Rates With and Without Bacteria at 40°C and pH, 7.5



TEMP: 30C pH: 7.5
H2S FLOW: 0.00066 SCF/S
AIR FLOW: 0.0016 SCF/S

INITIAL CELL COUNTS:
1.0E08 A CELLS/L
1.0E10 B CELLS/L

Figure 9. Comparison of Sulfur Recovery With and Without Bacteria at 30°C and pH, 7.5



TEMP: 40C pH: 7.5
H2S FLOW: 0.00066 SCF/S
AIR FLOW: 0.0016 SCF/S

INITIAL CELL COUNTS:
1.0E08 A CELLS/L
1.0E10 B CELLS/L

Figure 10. Comparison of Sulfur Recovery With and Without Bacteria at 40°C and pH, 7.5