

# Advanced Low-Temperature Fluid Bed Sorbents

Javad Abbasian (abbasian@igt.erg; 847-768-0611)  
Rachid B. Slimane (slimane@igt.erg; 847-768-0606)  
James R. Wangerow (wangerow@igt.erg; 847-768-0610)  
Institute of Gas Technology  
1700 S. Mt. Prospect Rd.  
Des Plaines, IL 60018

## Abstract

The Integrated Gasification Combined Cycle (IGCC) process is emerging as the most promising technology for advanced electric power generation from coal that is likely to replace conventional coal combustion. The commercialization of this technology and realization of its full potential, however, depend to a great extent on the development of **regenerable** sorbents capable of reducing the  $H_2S$  content of **gasifier** fuel gas from several thousand ppmv levels down to a few ppmv at elevated temperatures (i.e.,  $>3500C$ ), over hundreds or even thousands of sulfidation/regeneration cycles.

Over the last two decades, development of high temperature **regenerable** desulfurization sorbents has been pursued, primarily using various combinations of transition metal oxides. The primary focus has been directed toward application above  $550^{\circ}C$ , limiting the choice of metal oxides to those which possess favorable thermodynamic equilibria. These efforts led to the emergence of zinc-based sorbents, such as zinc titanates, as the leading candidates. However, because of problems with sorbent deterioration, further improvement is needed to minimize the overall cost of desulfurization in the IGCC process.

Recent studies on total IGCC system integration have indicated that system components become prohibitively expensive with increasing operating temperature and that the overall process efficiency gains of conducting **desulfurization** at above  $550^{\circ}C$  may not be sufficient to justify operation at such high temperatures. The optimum desulfurization temperature appears to be in the range of  $350^{\circ}$  -  $550^{\circ}C$ , where technical viability and process efficiency result in lower overall process costs. In addition, because of the more favorable thermodynamic equilibria at the lower temperature range, a large number of metal oxides can be considered for coal gas desulfurization, increasing the likelihood of developing suitable sorbents.

The three most advanced reactor configurations for hot gas desulfurization include **fixed-bed**, **moving-bed**, and **fluidized-bed** systems. The fluidized-bed approach offers advantages over the moving-bed and fixed-bed reactors because of its ability to control temperature particularly during the **highly** exothermic regeneration step; however, a more durable, attrition-resistant sorbent is required.

This paper discusses the results obtained in an ongoing study geared towards developing advanced mixed-metal oxide **sorbents** for desulfurization of coal-derived fuel gases in the temperature range of 350° to 550°C. The paper focuses on the study related to the development of durable **sorbents** suitable for **fluidized-bed** application and addresses thermodynamic considerations, **sulfidation** kinetics, regenerability, and the physical and chemical characteristics of a number of novel **sorbents**.

## **Acknowledgments**

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**Javad Abbasian  
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James R. Wangerow**

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**Des Plaines**, Illinois

## **Overall Objective**

Develop **regenerable sorbents** for removal of hydrogen sulfide from **coal-derived fuel gases** in **fluidized** bed reactor in the temperature range of **343-538°C (650-1000 °F)**

# Why Lower Temperature?

- Alkali condensation and removal
- More favorable H<sub>2</sub>S Equilibria
- Greater number of suitable metal oxides
- Lower stress on sorbent
- Higher sorbent stability in long run
- Less expensive system components

## Specific Objectives

- Selection of potential candidate sorbents
- Screening of potential candidate sorbents
- Laboratory evaluation of selected sorbents
- Bench scale testing of superior sorbent formulations
- Development of cost assessment/Market Plan
- Long-term testing of most promising sorbent

# Thermodynamic Calculations

## • H<sub>2</sub>S equilibria calculations

- Pure metal oxides
- Sorbent/H<sub>2</sub>S = 2
- 350-5500C, 20 bar
- Texaco & U-Gas

## • Phase stability diagrams

### SULFIDATION FEED GAS COMPOSITION

<u>Component</u>	<u>MeI%</u>
H <sub>2</sub> S	0.5-2
H <sub>2</sub>	10
CO	20
CO <sub>2</sub>	10
H <sub>2</sub> O	10
N <sub>2</sub>	48. 49. 5

# METAL OXIDE SCREENING

## Function

## Metals Chosen

Primary Desulfurization

Cu, Fe, Zn, Mn, Ce

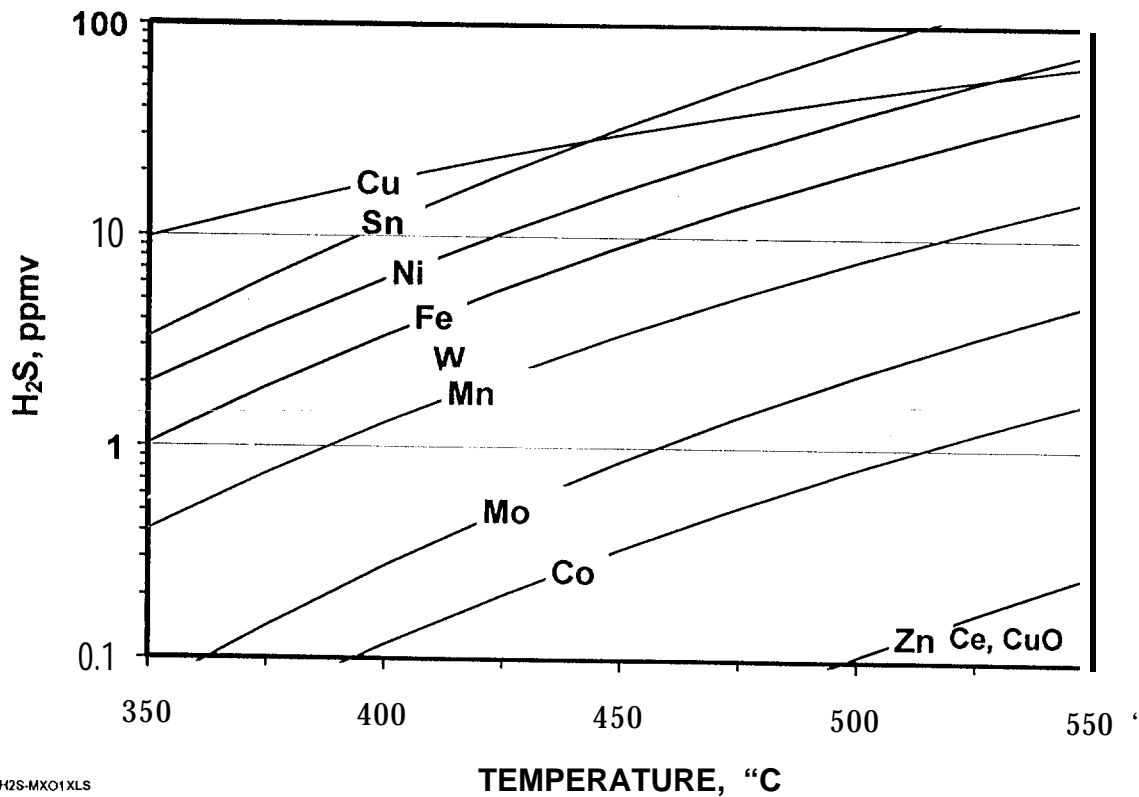
Secondary Additives

Cu, Fe, Mn, Cr, Ce, Co, Mo

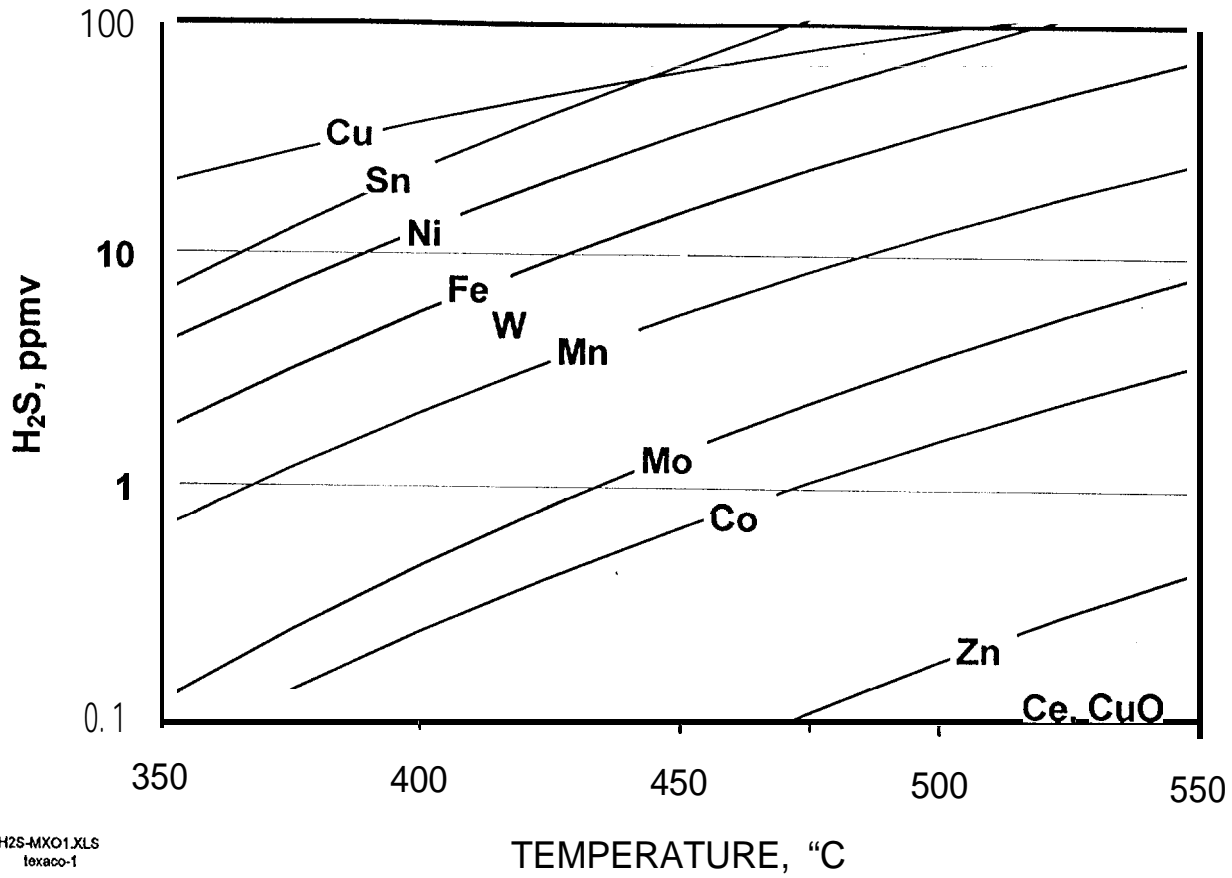
Support/Stabilizers

TiO<sub>2</sub>, ZrO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Binders

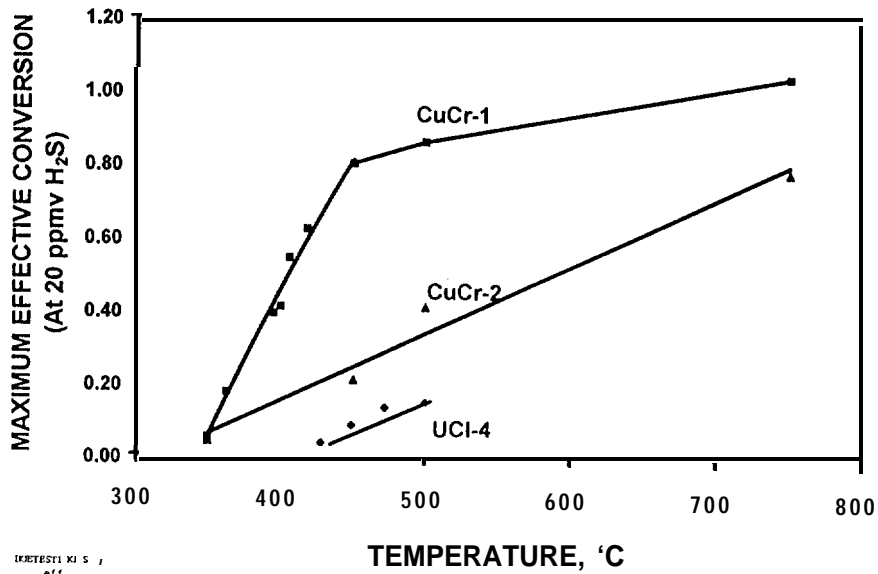
## H<sub>2</sub>S Equilibrium Concentration: U-GAS



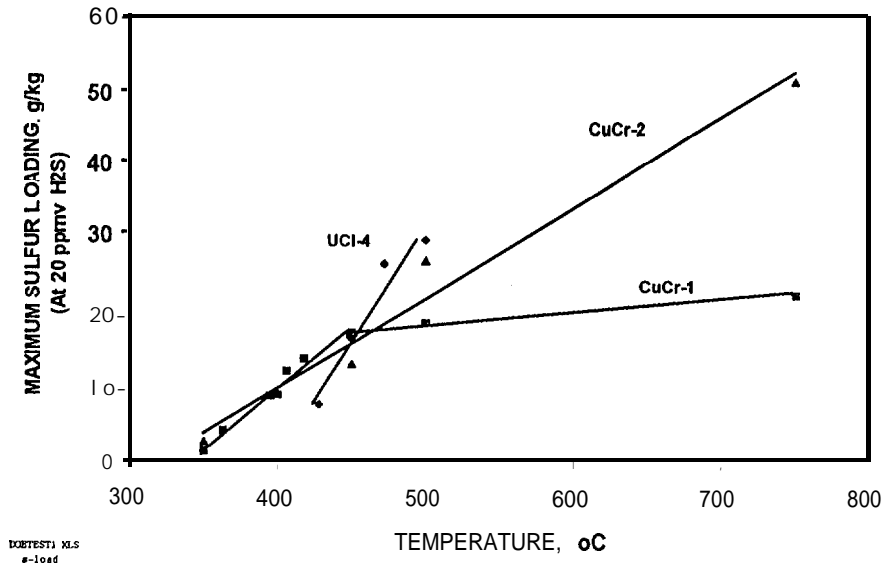
# H<sub>2</sub>S Equilibrium Concentration: TEXACO



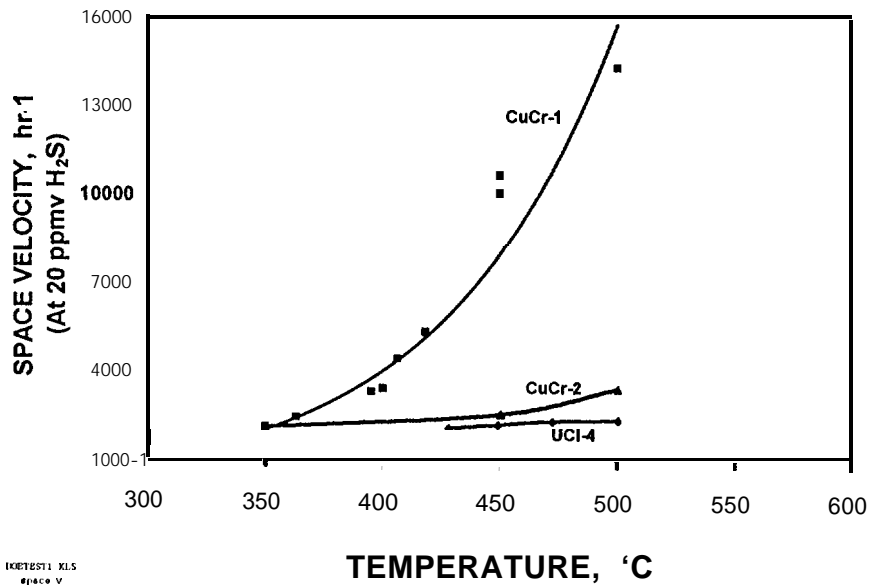
## Comparison of effective conversion



# Comparison of Sulfur Loading

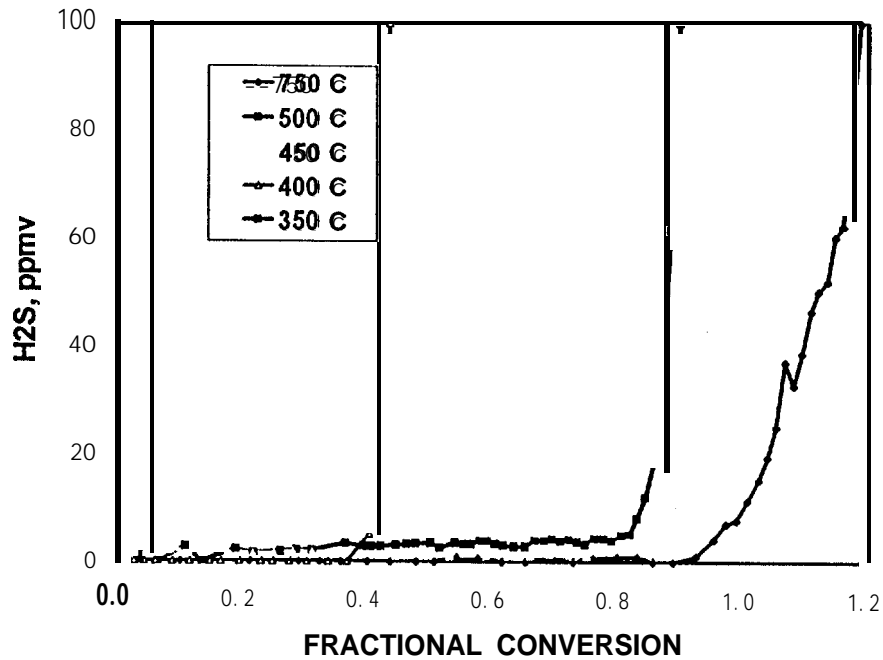


# Sorbent Performance at different Space Velocity



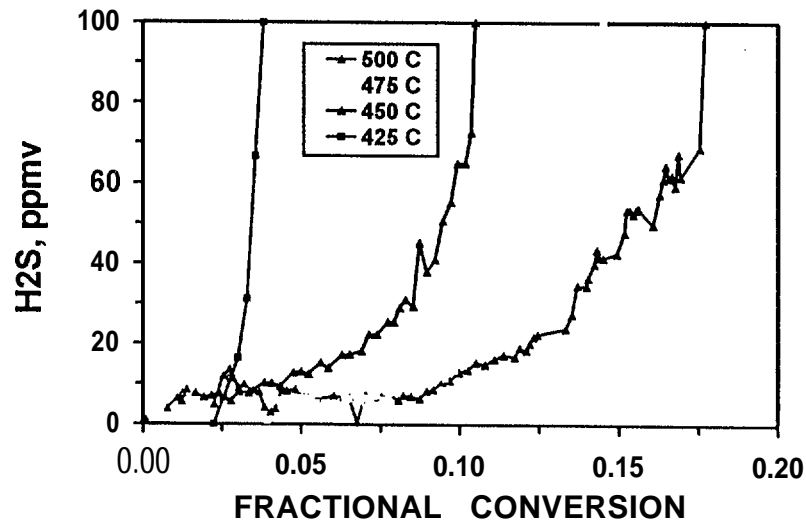


# Performance of CuCr-1 at Different



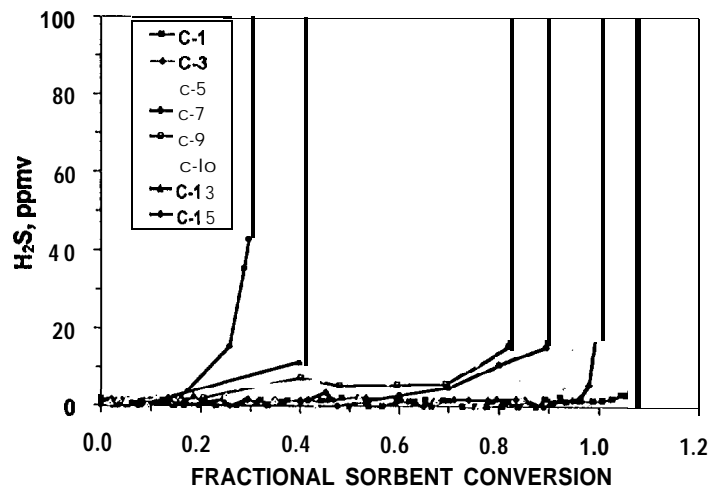
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# Performance of Zinc Titanate at Different Temperatures



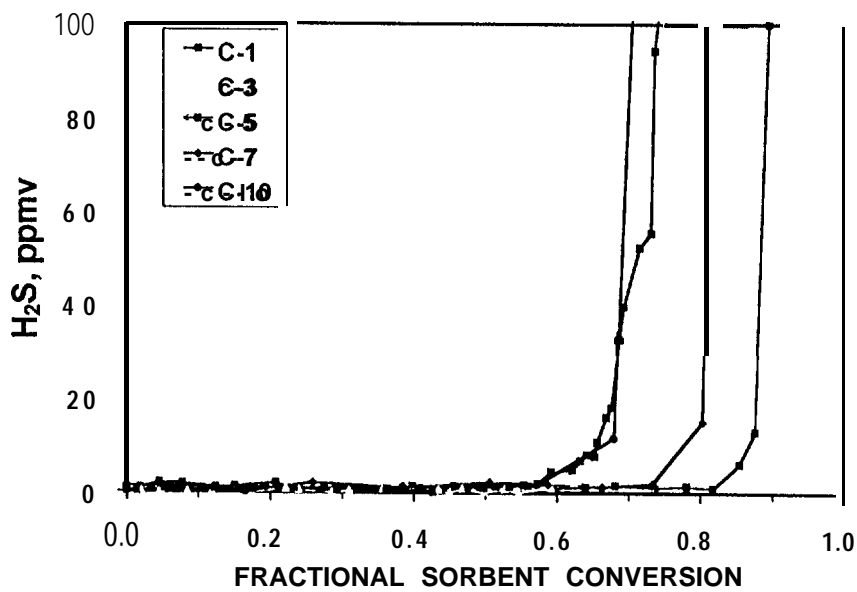
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# Performance of IGTSS-1 at 350°C



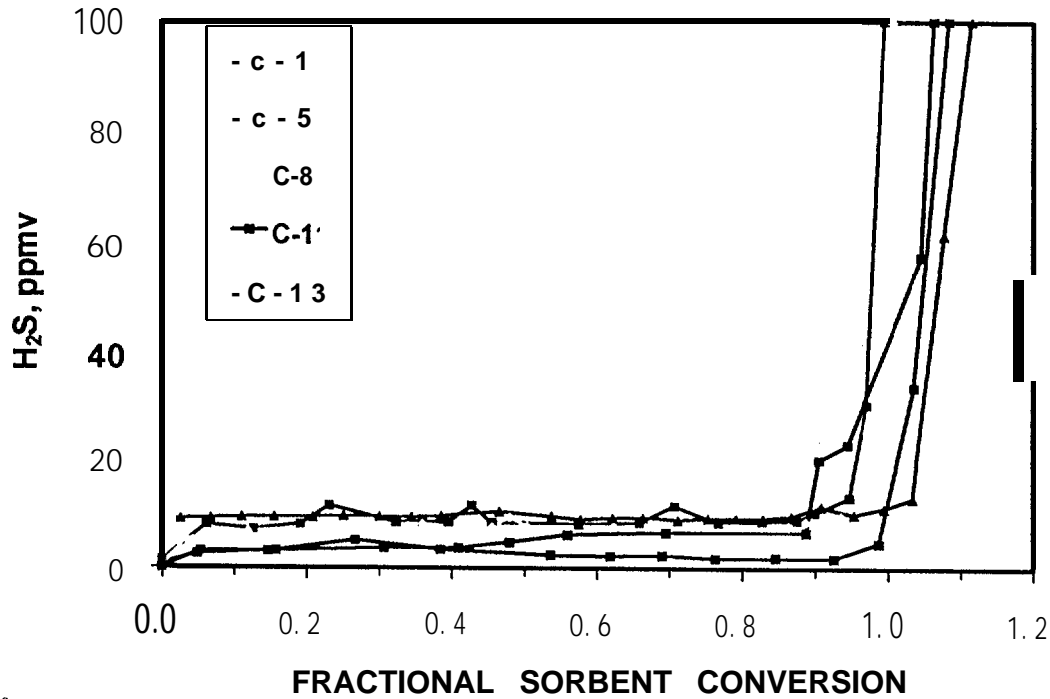
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# Performance of IGTSS-6 at 350°C



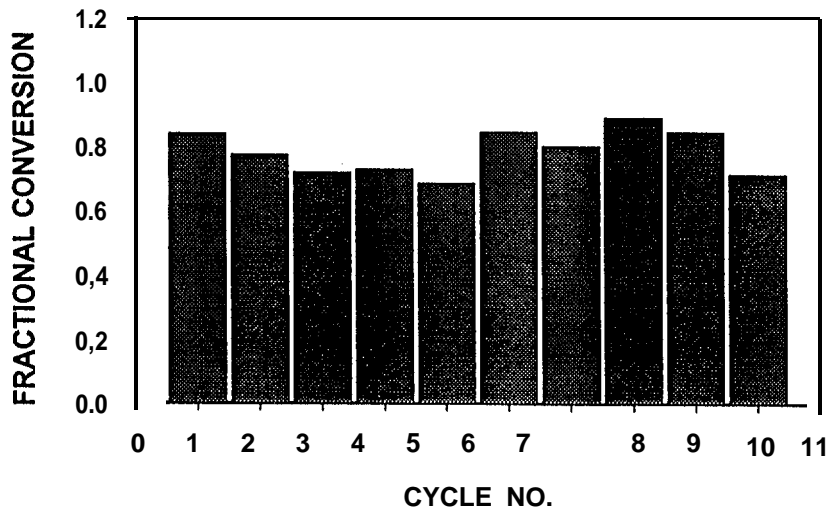
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# Performance of IGTSS-11 at 450°C



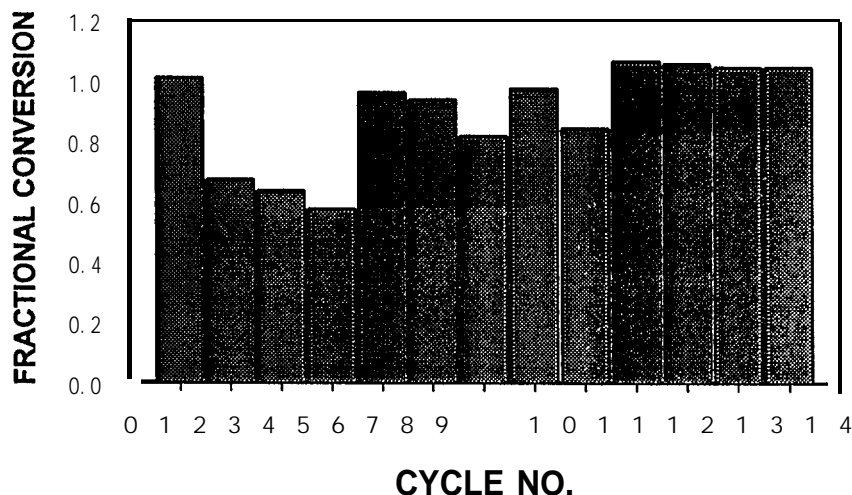
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# Breakthrough Conversion of IGTSS-6 at 350 °C



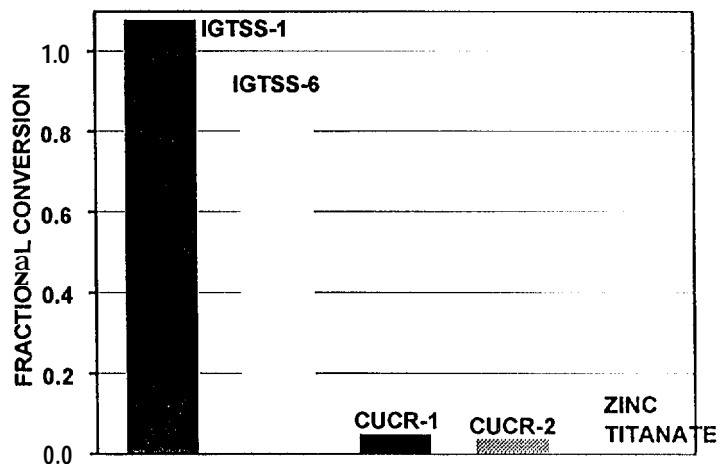
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# Breakthrough Conversion of IGTSS-11 at 450 \*C



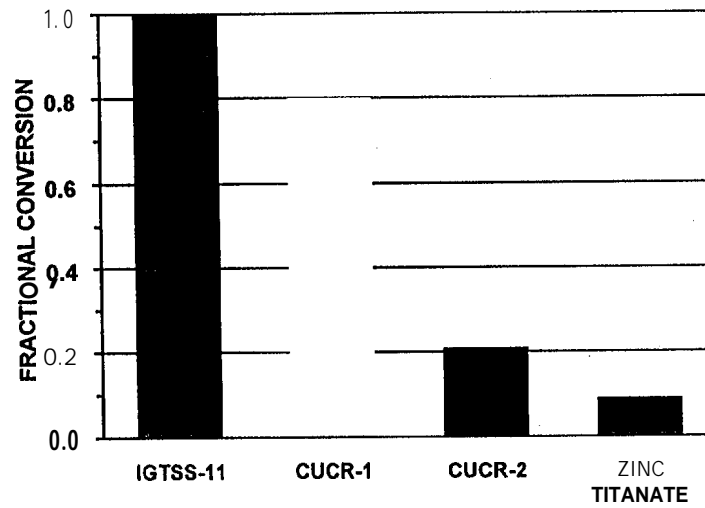
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# Comparison of Sorbent Conversion at 350 \*C



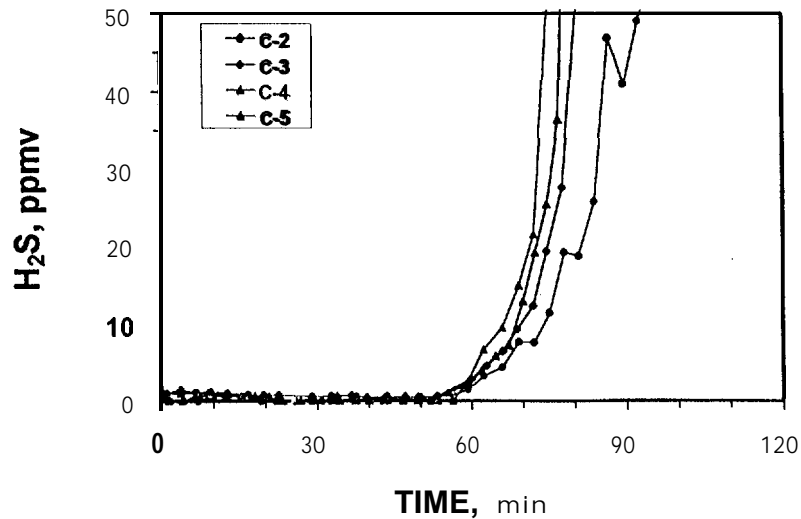
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# Comparison of Sorbent Conversion at 450°C



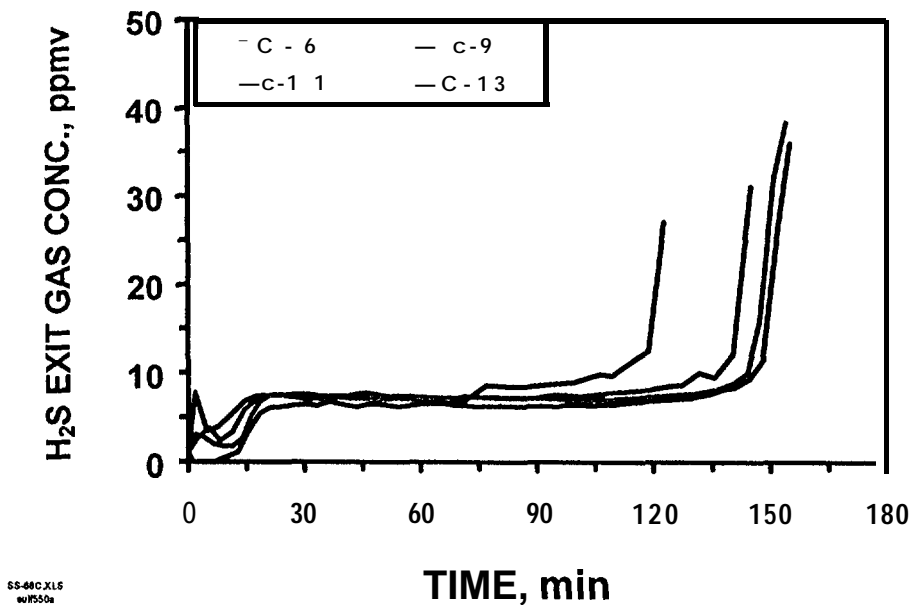
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# Performance of IGSS-57 at 450°C



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11/17/97-45

# Performance of IGTSS-68 at 550°C



## Conclusions

- Three promising sorbents have been developed for temperature range of 350°-550°C
- The sorbents reactivities and sulfur capacity appear to be stable or improving during the first 10 to 15 cycles
- The attrition resistance of the sorbent are better than UCI-4169 Zinc Titanate sorbent.