DOE/MC/29470--2

DE-AC21-92MC29470--2

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Surface Acoustic Wave Sensors/Gas Chromatography
Low Quality Natural Gas Sulfur Removal and Recovery
CNG Claus Sulfur Recovery Process

Topical Report
December 1997

By:
Bruce W. Klint
Peter R. Dale
Charles Stephenson

Work Performed Under Contract No.: DE-AC21-92MC29470

For
U.S. Department of Energy
Office of Fossil Energy
Federal Energy Technology Center
P.O. Box 880
Morgantown, West Virginia 26507-0880

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Surface Acoustic Wave Sensors/Gas Chromatography
Electronic Sensor Technology
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A: BASIC

GETE ID:	17
Date:	5/12/97
Technology:	Model 4100 Vapor Detector and Analyzer
IP Holder:	Electronic Sensor Technology
Interviewer:	Tory Failmezger
Affiliation:	G.E.T.F./G.E.T.E.
1. What is the the relative importance, priority or significance of the technology to the focus area?	
2. Is there a private sector company already involved with the technology? (If the answer is yes then proceed; if the answer is no, then the focus area must decide whether there is sufficient interest to warrant the search for a partner, and if so with what urgency.)	Yes, Electronic Sensor Technology.
3. Is the technology nearing commercial readiness? (This requires a definition of the status of the technology as defined by the DOE (EM-50) Technology Decision Model. The CAC can become involved early or late in the process depending on EM's needs for a particular technology).	Yes, the technology is available for use.
4. Is the company bringing the technology to market one that requires no assistance? (This requires a decision whether the commercial partner requires assistance in the commercialization process. If there is a strong commercial partner for example, fewer commercialization activities are likely to be required. Some of the considerations for this evaluation are as follows).	The commercialization effort is being aided by a partnership between Amerasia, the managing limited partnership company of Electronic Sensor Technology, Inc., and Federal Energy Technology Center (Morgantown).
a) What size is the company?	
Employees:	
Offices:	Westlake Village, California
b) Is it publicly or privately owned?	Private
c) Is an annual report available?	
d) What are the annual revenues?	
e) What relevant experience does the company possess?	
f) What is the company's capability and interest in commercializing the technology?	
g) Has the company a previous track record commercializing environmental technologies?	

h) Has the company a track record working in the DOE complex?

If yes, explain

Open Next Form

B: TECHNOLOGY

GETE ID: 17

Technology: Model 4100 Vapor Detector and Analyzer

1. What is the technology? (Defined and described in simple non-scientific terms.)	The 4100 uses a Surface Acoustic Wave (SAW) detector to determine the mass density of a vapor or particle sample. Initially, the sample is collected in a cryo-focus chamber and passed through a gas chromatograph. The SAW detector reports the results to a database in a 486 laptop computer. The sample is compared with a library of chemical signatures on file and a Windows based program calculates the mass. All of the components are shock mounted in a rugged field-portable fiberglass carrying case.
2. What primary problem areas does this technology address?	The technology address the problem of measuring emissions from various sources in a small amount of time.
3. Have realistic potential secondary applications been identified?(List)	Detection of contraband and explosives.
4. Is data available that supports the developers' claims regarding the effectiveness of the technology? Can that data be readily obtained?	The technology was successfully field tested at the Savannah River Site.
5. Is adequate information available about the technology in order to allow the investigator to define the market(s) into which the technology can be sold?	Yes
6. Are test/demonstration results available?	Yes.
7. In what stage of development is the technology?	The technology is available for use.
8. Is the technology ready for examination and review by potential licensees/users?	Yes
9. How does new technology compare to current technologies doing the same job? (Quantify)	The advantages of the technology are portabillity, accuracy, and speed.
10. How readily can it be modified to address additional markets?	
11. What problems have occurred in the development cycle? How were they addressed?	
12. What are the limitations of the technology?	
13. How else is this technology unique?	
14. How difficult is it to "try before you buy"?	
15. Where can it be viewed?	

[illegible] Form

C: INTELLECTUAL PROPERTY

GETE ID: 17

Technology: Model 4100 Vapor Detector and Analyzer

1. Who holds the Intellectual Property?	Electronic Sensor Technology
2. Is the developer (principal investigator) of the technology easily identifiable?	Yes, the prinicpal investigator is Edward J. Staples.
3. Who are the other team members/partners government, commercial or otherwise?	Federal Energy Technology Center (Morgantown) has partnered with Electronic Sensor Technology.
4. What is the attitude of the intellectual property holder(s) towards commercialization?	The intellectual property holder is aggressively initiating commercialization.
5. Is the intellectual property custody trail well-documented and are there no serious impediments apparent to the use/licensing of the patents by commercial for-profit parties?	
6. Is the technology eligible for a patent?	
7. Have patents been applied for (or issued)?	
8. Does DOE have the rights (or can expeditiously obtain the rights) to license (or otherwise transfer) the technology and is a clear process to do so defined?	No.
9. Who has already licensed the technology?	
10. Can the intellectual property team be reached by parties seeking to commercialize the	
11. What are the intellectual property team views on how this product can be privatized?	

[illegible]

e) How big is the market? Total/Each

f) How diversified?

g) What primary forces influence it? (Federal, State, Local, International)

h) How is the market segmented?

i) Is it mature, saturated, fragmented?

j) Where do the specific near term opportunities lie for this technology?

k) What market advantage does this technology have? (Estimate value)

l) Who are the primary competitors? (US, Foreign)

m) Who are "easy competitors (i.e. who has ready access to the market this technology will address)?"

n) What are the market barriers to entry?

9. How can this technology be marketed?

10. Does this technology have any "leverage" in opening new markets?

[redacted]

E: COMPETITIVENESS

GETE ID: 17

Technology: Model 4100 Vapor Detector and Analyzer

1. The technology can be clearly differentiated from existing in-use technology, has a clear competitive advantage over other competition, and the by products/residues and final treatment and disposal costs are significantly lower than existing or competitive technologies.	The technology has advantages that include portability, accuracy, and speed.
a) What are the by products?	N/A
b) What are the residues?	N/A
c) What additional treatment is required?	N/A
d) What is the final disposal cost?	N/A
2. What are the specific competitive advantages of this product?	The advantages of the product include portability, accuracy, and speed.
a) Cost	The cost savings over current technologies are estimated to be more than $50,000 per month.
b) Simplicity	
c) Pollution prevention features	
d) Risk	
e) Maintenance	
f) Waste stream quality (amount)	
g) Remediation properties	
h) Safety/Health (any obvious deal breakers)	
3. DOE is not supporting other directly competitive approaches to this technology's targeted applications. A technology selected by the commercialization activity should have a clear market niche and be supported by DOE.	
4. The cost effectiveness of the technology (cost vs. application) is defined and reasonable, while other cost profiles for acquiring, building, operating, maintaining and dismantling the technology are reasonable in view of the potential market	
5. These cost drivers are understood by the principal investigators (PIs) and documented in the data.	

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F: COMMITMENT

GETE ID: 17

Technology: Model 4100 Vapor Detector and Analyzer

1. The principal investigator and team (to include commercial and other partners) remain committed to commercializing the technology and will support/ cooperate with innovative commercialization approaches.	Yes.
2. The technology has an active supporter within the government establishment.	Yes, Mr. Eddie Christy, the METC contracting officer is supporting this technology.
3. The intellectual property holder is supportive and committed to commercializing the technology?	Yes

4. Who is sponsoring/supporting the technology (By Name)?

a) EM50	
b) EM30	
c) EM40	
d) DP	
e) LAB	
f) M and O	
g) DOE Regional Office	
h) University	
i) Industrial Partner	
j) Small companies	
k) Foreign partners	
l) Other government	

5. What is the committed future funding line for this technology? If any, by year?

6. The investment profile of the technology is documented. Is the investment significant, relative to the size of the overall market, and the problem(s) to be solved?

7. The resources and financial ability of the "supporters" relative to this technology are known and documented and indicate an ability to follow through on a commercialization initiative?

8. What are the resources of any proposed "commercializer" of the technology and are they financially capable of following through on their commitments?

9. What is the initial non-government investment in this technology, if any?

To Next Form

H: COST INFORMATION

GETE ID: 17 **Technology:** Model 4100 Vapor Detector and Analyzer

1. What information is available on cost effectiveness?

2. What are the cost profiles?

a) To acquire intellectual property rights?

b) To build?

c) To operate? (utilities, equipment, disposable supplies, time, etc.)

d) To train personnel?

e) To maintain?

f) What unique materials are required for the design?

g) What unique equipment is required for the design?

h) What is product's projected operating lifetime? Projected technology lifetime?

3. What are the cost drivers for use of this technology?

a) Materials?

b) Equipment?

c) Software?

d) Hardware?

e) Labor?

f) Time?

g) Oand M?

h) Digging/Drilling/Fabrication/Installation? N/A

i) Disposal? N/A

j) Regulations? N/A

4. What are the resources of the "partners" sponsoring this technology (i.e. company, government agency, financiers)?

5. How rapidly is assistance required?

6. What defines a "good" result in the target market (minimum standards)?

7. How is the company "getting by" in the interim period (before full financing)?

8. Are there significant stockholder concerns about the product/procedures? Should there be?

[illegible]

I: FUTURE PLANNED ACTIVITIES

GETE ID: 17 **Technology:** Model 4100 Vapor Detector and Analyzer

1. What further demonstrations are planned or necessary?	
2. What is needed before the technology is available in the field as an off-the-shelf item or service?	The technology is currently available.
3. Is the technology dependent on any seperate developing technology prior to implementation?	No.
4. Are there any other critical issues that remain to be addressed (science, technology, scale-up, environmental, other)?	

END

2

Draft

Surface Acoustic Wave/ Gas Chromatography

INNOVATIVE TECHNOLOGY SUMMARY REPORT

demonstrated at

U.S. Department of Energy Sites:
Savannah River Site, SC
Idaho National Engineering and
Environmental Laboratory, ID
Hanford Site, WA

prepared for

U.S. Department of Energy
Office of Environmental Management
Office of Science and Technology

April 1997

U.S. Department of Energy

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APPENDIX

SECTION 1

SUMMARY

Technology Description

Surface Acoustic Wave/Gas Chromatography (SAW/GC) provides a cost-effective system for collecting real-time field screening data for characterization of vapor streams contaminated with volatile organic compounds (VOCs). The technology was developed by the Amerasia Technology Corporation supported by the U.S. Department of Energy (DOE) Office of Science and Technology (OST). A new company, Electronic Sensor Technology (EST), was incorporated to manufacture, distribute, and market the Model 4100 SAW/GC.

The Model 4100 can be used in a field screening mode to produce chromatograms in 10 seconds. This capability will allow a project manager to make immediate decisions and to avoid the long delays and high costs associated with analysis by off-site analytical laboratories. The Model 4100 will not replace a reference laboratory but instead provides a cost-effective means of quickly analyzing a large number of samples during site characterization and minimizes the number of 'clean samples', i.e. those below threshold contamination levels, that need to be analyzed.

The Model 4100 SAW/GC:

- is based on gas chromatography (GC) and surface acoustic wave (SAW) technologies;
- consists of the following two components connected with an umbilical cord:
  - ◊ a handheld module containing a piezoelectric surface acoustic wave sensor, a capillary gas chromatograph, an air pump and a six-way GC valve;
  - ◊ a support module, which supplies the helium gas and the electrical power and incorporates a laptop computer;

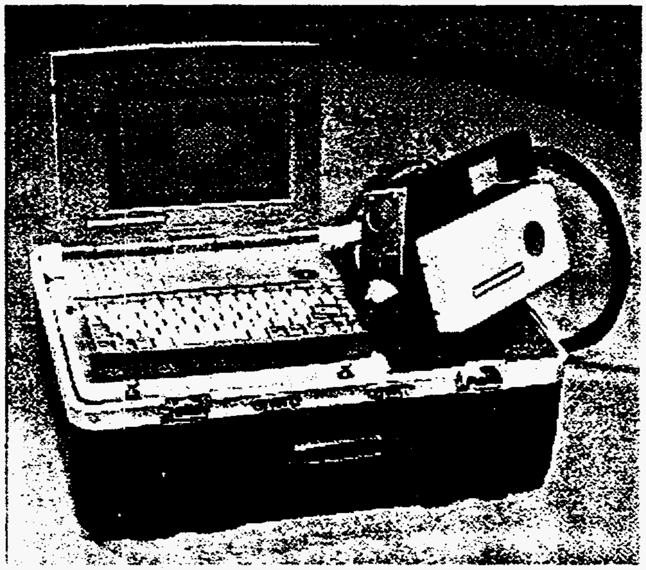
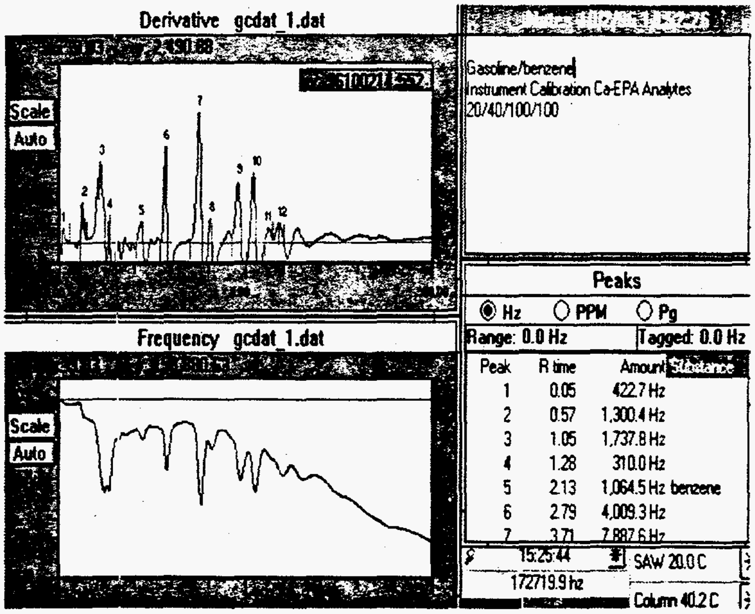


Figure 1. Model 4100 SAW/GC

- can be used for sampling vapor streams from environmental characterization and monitoring applications, remediation waste streams, processing applications that include food and medical analyses, and other monitoring applications such as workplace monitoring;
- specific environmental applications include air monitoring, stack emissions monitoring, underground storage tank monitoring, soil and ground water characterization and screening of hazardous workplaces;
- is fast, portable, rugged, and can detect compounds at the parts per billion level using extremely low-volume samples.

- should be considered for field monitoring and characterization if there is a need:
    - ◊ to make in-situ measurements;
    - ◊ to make decisions in real time, i.e. 8 hours or less;
    - ◊ to reduce fixed laboratory costs;
    - ◊ to identify and characterize contaminant hot spots instead of sampling a complete site.



Gasoline/benzene
Instrument Calibration Ca-EPA Analytes
20/40/100/100

Range: 0.0 Hz
Tagged: 0.0 Hz

Peak	R time	Amount	Substance
1	0.05	422.7 Hz	
2	0.57	1,300.4 Hz	
3	1.05	1,737.8 Hz	
4	1.28	310.0 Hz	
5	2.13	1,064.5 Hz	benzene
6	2.79	4,009.3 Hz	
7	3.71	7,887.6 Hz	

15:25:44
172719.9 hz
SAW 20.0 C
Column 40.2 C

Figure 2. Representative chromatogram showing the use of Model 4100 in field mode to analyze soils near a leaking underground fuel tank (LUFT).

Technology Status

The Model 4100 SAW/GC has been demonstrated and evaluated at a number of DOE sites (Savannah River Site [SRS], Lawrence Berkeley National Laboratory [LBNL], Hanford, and Idaho National Engineering and Environmental Laboratory [INEEL]) to verify its performance under a number of different applications.

The Model 4100 SAW/GC is currently under evaluation by the California Environmental Protection Agency (Cal EPA) Technology Certification Program. This process requires the review of all quality control aspects in the field, operational and manufacture. Initial certification focuses upon the following compounds:

cis- dichloroethylene [cis-DCE]
chloroform [CF]
carbon tetrachloride [CT]
trichloroethylene [TCE]
tetrachloroethylene [PCE]
tetrachloroethane [1,1,2,2 TCA]
benzene
ethylbenzene
toluene
o-xylene

Specific and defensible performance claims will be available when certification is completed. Cal EPA certification is expected in the Second Quarter of 1997. Cal EPA certification is accepted currently by all state, county, and municipal agencies in California and by five other states: New York, Massachusetts, New Jersey, Illinois, and Pennsylvania.

- The SAW detector shows a large dynamic range and is linear over a wide range of concentrations.

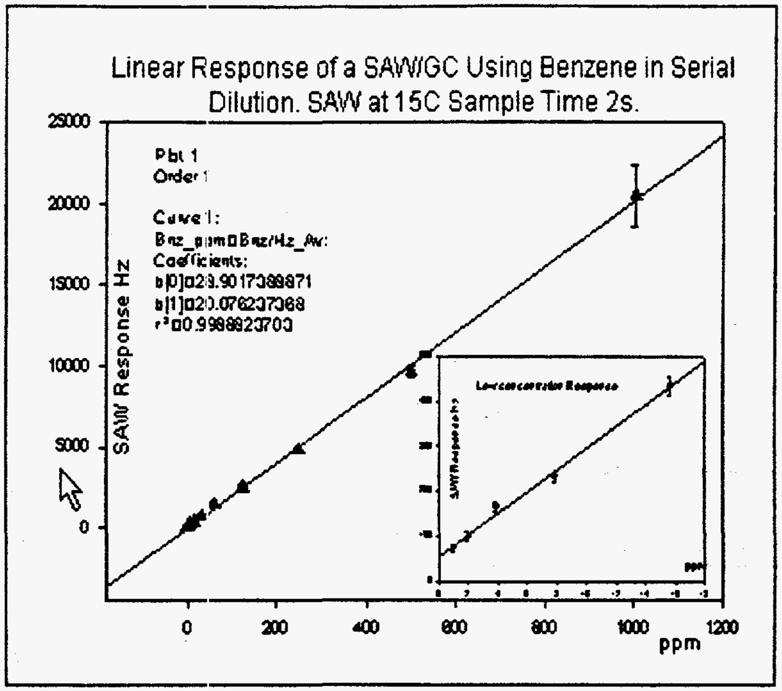


Figure 3. Representative chromatogram of benzene samples generated during CalEPA certification.

◊ The r^2 for this calibration curve was found to be 0.9998.

◊ The dynamic range for this calibration at a 2s sample time was 450ppb to 1100ppm.

The Model 4100 SAW/GC is commercially available at this time from Electronic Sensor Technology (EST), which holds a U.S. patent (number 5,289,715) for the technology. The computer programs controlling the Model 4100 are proprietary and restricted. This includes all algorithms for peak detection and signal processing.

CONTACTS

Technical

Edward Staples, Electronic Sensor Technology Inc., (805) 495-9388, homepage: www.estcal.com, e-mail: staples@estcal.com.
George Pappas, Electronic Sensor Technology Inc., (805) 495-9388, homepage: www.estcal.com, e-mail: pappas@estcal.com.

Management

Eric Lightner, DOE Characterization, Monitoring, and Sensor Technology Crosscutting Program, (301) 903-7935,
C. Edward Christy, DOE Federal Energy and Technology Center, (304) 285-4604.
James B. Wright, Subsurface Contaminants Focus Area Implementation Manager, (803) 725-5608.

SECTION 2

TECHNOLOGY DESCRIPTION

Overall System Description

- The Model 4100 SAW/GC instrument couples a piezoelectric surface acoustic wave sensor (SAW) and a capillary gas chromatograph (GC) with a dynamic particle/vapor sampling interface. The instrument is comprised of two parts: a head assembly containing the capillary column, the six port valve, oven trap and SAW detector; and a support chassis containing the helium carrier gas, laptop computer, and the thermoelectric processors. The unit is 14 by 20 by 10 inches and weighs only 35 pounds. The components are housed in a shock-mounted field-portable fiberglass carrying case.

- Samples are introduced to the Model 4100 using a tedlar bag or by direct injection. A needle attached to the nose of the instrument is used to puncture the septum of the tedlar bag and approximately 5 ml of sample is injected for one analysis.

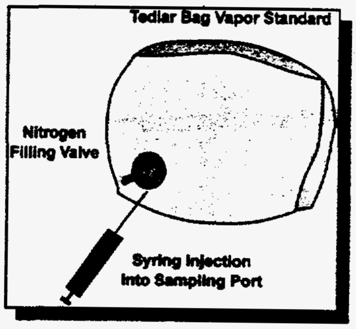


Figure 4. Tedlar Bag

- The instrument is controlled by proprietary software that allows the operator to select or develop a method, to program various component temperatures, and to automatically record the data for future recall.

- The system uses a two-position, six-port GC valve to switch between sampling and injection modes.
  - ◊ In the sample position, headspace vapor is first passed through an inlet preconcentrator or water trap and then through the inlet, valve and loop trap. The loop trap concentrates the suspect compounds. At the same time, helium carrier gas passes through the alternate ports of the valve to a capillary column, impinging on the surface of a temperature-controlled SAW resonator.

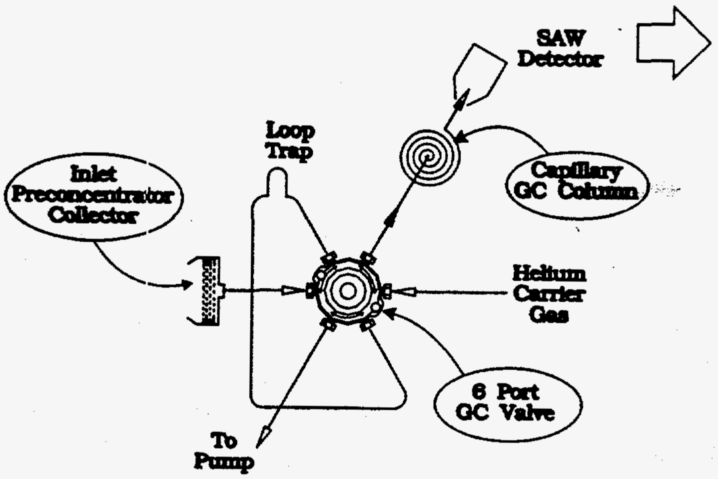
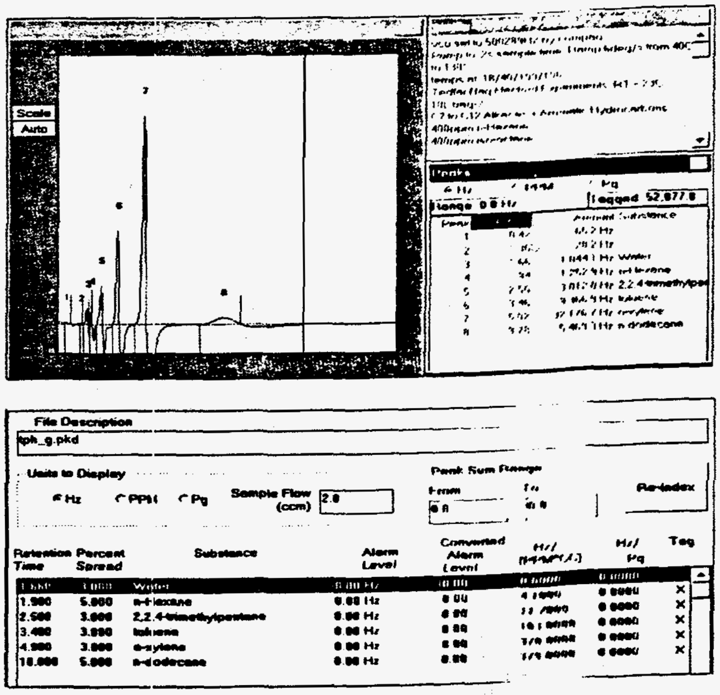


Figure 5. System Diagram

- ◊ When the valve trap is switched into the injection position, the flow of helium carrier gas is reversed through the column, and the loop trap is rapidly heated to 200°C causing the release of the contaminants to the head of the GC column. The temperature of the GC column is linearly ramped to approximately 125°C over 10 seconds causing the VOCs to separate as they travel down the column.

- As the contaminants pass through the GC column, they are separated. As the resolved analytes strike the surface of the resonant SAW sensor, they cause the resonant frequency of the crystal to change. The frequency shift is recorded and the concentration level of the compound is calculated in parts per billion (ppb) or picograms (pg).
  - ◊ The adsorption to the surface of the resonator causes a change in the characteristic frequency of the piezoelectric crystal. The adsorption efficiency for a given compound is dependent on the crystal temperature.
  - ◊ By operating the crystal at different temperatures using the system software, the crystal can be made specific for given materials based on the vapor pressure of the material.
  - ◊ The SAW sensor determines the mass density of the compound and reports it to the database maintained in the 486 laptop computer.
  - ◊ To obtain a conventional chromatogram plot of retention time, the derivative of frequency versus time is calculated. A representative chromatogram with a peak identification table is shown in Figure 6.



File Description: tph_g.pkd

Units to Display: ● Hz ○ PPM ○ Pg

Sample Flow (ccm): 2.0

Peak Sum Range: From 0.0 To 0.0

Retention Time	Percent Spread	Substance	Alarm Level	Converted Alarm Level	Hz/ppm	Hz/Pg	Tag
[illegible]	3.000	Water	0.00 Hz	0.00	0.0000	0.0000	
1.900	5.000	n-Hexane	0.00 Hz	0.00	[illegible]	0.0000	X
2.500	3.000	2,2,4-trimethylpentane	0.00 Hz	0.00	[illegible]	0.0000	X
3.400	3.000	toluene	0.00 Hz	0.00	[illegible]	0.0000	X
4.900	3.000	o-xylene	0.00 Hz	0.00	[illegible]	0.0000	X
10.000	5.000	n-dodecane	0.00 Hz	0.00	[illegible]	0.0000	X

Figure 6. A 20-second chromatogram with a Peak Identification Table

- ◊ In the Leaking Underground Fuel Tank (LUFT) configuration a useful set of standards is the alkane hydrocarbons homologous series from C6-C12.
- ◊ A peak identification table shows alkanes and aromatic hydrocarbons.

- Several standard compounds are used for contaminant identification. The Model 4100 identifies the unknown compounds through comparison with the many chemical signatures stored in the database. For screening analyses, benzene is needed to establish retention time reference. For quantitative results at the ppm level in real time, additional standards can be run before field screening to determine a scaling factor for compounds.

- SAW sensors are quartz crystals having patterned electrodes that allow a high frequency (60-1000 Mhz.) surface acoustic wave to be maintained on their surface.

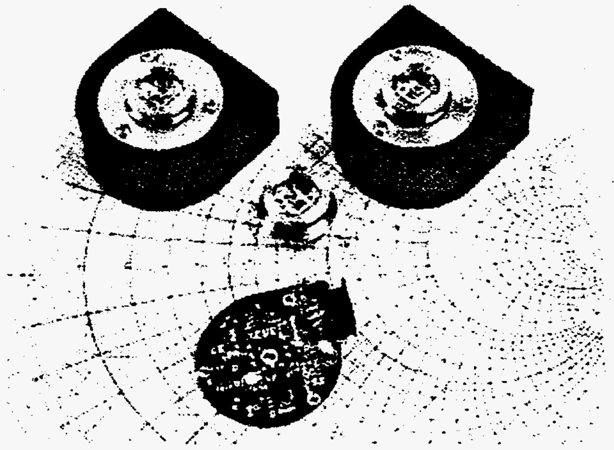


Figure 7. Photo of the SAW Detector

- ◊ The crystal frequency is dependent on the spacing of the electrode pattern, the crystal temperature, and the condition of the surface. Because the electrode pattern of a given SAW crystal is fixed, the frequency of the SAW sensor is dependent on the material adsorbed onto the quartz surface.
- ◊ The innovative feature of the SAW resonator is that the temperature of the SAW sensor can be programmed using a thermoelectric cooling/heating module bonded under the SAW crystal.
- ◊ In this configuration, the detector displays a wide dynamic operational range, up to 6 orders of magnitude. This dynamic range is not found in other detectors.
- ◊ The thermoelectric module is controlled by the computer, maintaining the SAW crystal at temperatures between 20 and 200°C. Lower temperatures can be obtained by cascading thermoelectric coolers. The SAW resonator can be cooled during the analysis time when the materials are eluting from the column, thus insuring that materials will adsorb onto the SAW surface.
- ◊ At the end of the inject cycle, the SAW can be heated to temperatures greater than 150°C to boil off materials from the previous injection, thus cleaning the SAW surface for the subsequent analysis. This feature makes it unique among existing detectors.

- During development of the technology, a quality control plan was implemented. This plan called for the use of UL approved wiring for safety and thermal imaging of the column and oven to optimize performance while minimizing cold spots. The instrument was reduced in size and weight by fifty percent. Significant changes were made to the electronic and mechanical design to reduce power consumption while increasing sensitivity.

- The system is simple to operate and can be used safely by properly trained technicians. Technicians should complete 16 hours of basic training provided by the vendor.

PERFORMANCE

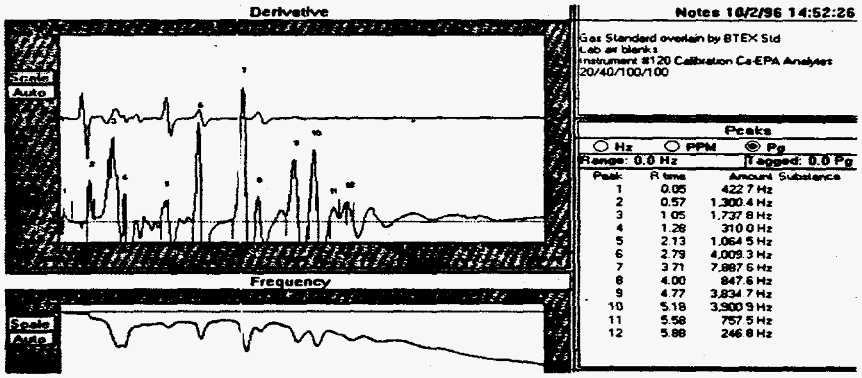
Laboratory Testing

Laboratory testing first established ideal minimum detection levels for selected target compounds (Table 1). Representative compounds typical of those found at hazardous waste sites were tested using two different GC columns.

Table 1. Measurement Range of Model 4100 for Selected Compounds

Analyte	Minimum (ppb)	Maximum (ppm)
Carbon Tetrachloride (CT)	70	100,195
cis-Dichloroethylene (DCE)	110	186,420
Chloroform (CF)	65	182,351
Trichloroethylene (TCE)	10	74,926
Tetrachloroethylene (PCE)	3	17,965
1,1,2,2 Tetrachloroethane (TCA)	1.3	6,256
Benzene (B)	45	106,711
Toluene (T)	4.5	29,276
Ethylbenzene (EB)	2.0	98,263
O-Xylene (o-X)	2.0	6,465

A typical display screen presenting both visual and numerical data results is shown in Figure 8.

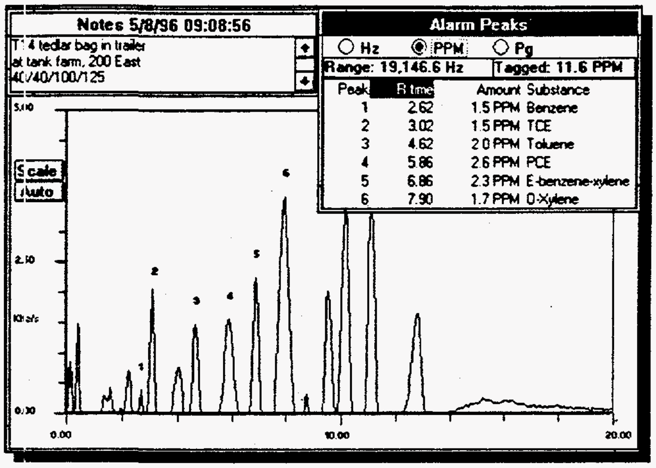


```
Notes 10/2/96 14:52:26
Gas Standard overlain by BTEX Std
Lab air blanks
Instrument #120 Calibration Ca-EPA Analytes
20/40/100/100

Range: 0.0 Hz                Tagged: 0.0 Pg
 Peak    R time       Amount Substance
    1      0.05      422 7 Hz
    2      0.57    1,300.4 Hz
    3      1 05    1,737 8 Hz
    4      1.28      310 0 Hz
    5      2.13    1,064 5 Hz
    6      2.79    4,009.3 Hz
    7      3 71    7,887 6 Hz
    8      4.00      847.6 Hz
    9      4.77    3,834 7 Hz
   10      5.18    3,900 9 Hz
   11      5.58      757 5 Hz
   12      5.88      246 8 Hz
```

Figure 8. Chromatogram displaying screen presenting visual and numerical data results

The instrument was able to characterize and separate a calibrated mixture of TO-14 compounds in 20 seconds. Figure 9 displa the result of such an analysis.



Notes 5/8/96 09:08:56

T 4 tedlar bag in trailer
at tank farm, 200 East
40/40/100/125

Range: 19,146.6 Hz

Tagged: 11.6 PPM

Peak	R time	Amount	Substance
1	2.62	1.5 PPM	Benzene
2	3.02	1.5 PPM	TCE
3	4.62	2.0 PPM	Toluene
4	5.86	2.6 PPM	PCE
5	6.86	2.3 PPM	E-benzene-xylene
6	7.90	1.7 PPM	O-Xylene

Figure 9. TO-14 analysis with optional peak tagging

Demonstrations and Evaluations

- Field testing of the SAW/GC was first performed in M-Area at SRS in 1995. Testing was done on water, soil, and gas samples. The performance of the SAW/GC prototype was validated with results obtained with an on-site Hewlett Packard ga chromatograph. The comparison showed that results agreed within 20%. This evaluation documented that the Model 4100 could identify and quantify the presence of VOCs, specifically TCE and PCE. Figure 10 shows results of analysis of gas samples from the headspace of contaminated wells.

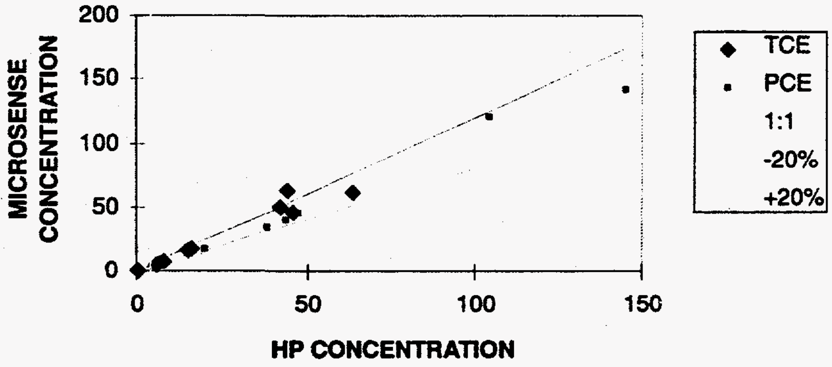


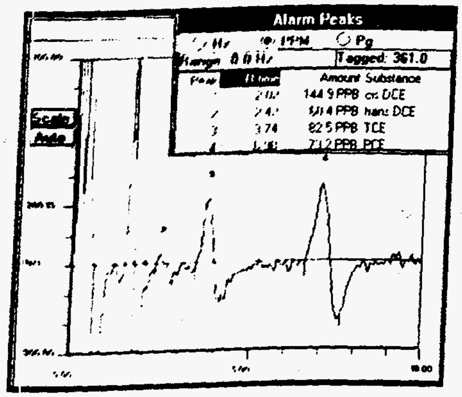
Figure 10. Verification of the SAW/GC with the SRS gas chromatograph. Measurement units are ppm.

- At LBNL in February 1996, the Model 4100 was used for characterization of soil gas and ground water with PCE and TCE at the ppb levels. Samples were collected from wells fitted with septum lids designed to accumulate soil gas. The Model 4100 demonstrated its ability to speciate contaminants in real-time. Instrument calibration and compound identification was provided by preparing a one-liter tedlar bag with the analytes at a one ppm concentration level.

- In April 1996, the Model 4100 was demonstrated at DOE's INEEL to perform vapor monitoring in wells surrounding the Radioactive Waste Management Complex (RWMC) and ground water monitoring at the Test Area North (TAN).
  - ◊ At the first site, the Central facility in the RWMC, the Model 4100 was evaluated as a well headspace monitor. Forty tedlar bags from twenty wells (samples were collected at different depths within a well) were analyzed in approximately one hour for carbon tetrachloride, chloroform, and TCE. Accuracy over the range of 20-500 ppm was validated by more than ten calibration runs. Samples were also collected directly from the sampling port at the wellhead for field analysis, thus removing the need and cost of tedlar bags.

Figure 11. Sampling a well head at INEEL, Idaho.

◊ At the second site, the Ground Water Treatment Facility (GWTF) at TAN, the Model 4100 successfully detected DCE, TCE and PCE at the ppb level. Figure 12 displays a chromatogram of the analytes of interest at the GWTF.



Alarm Peaks

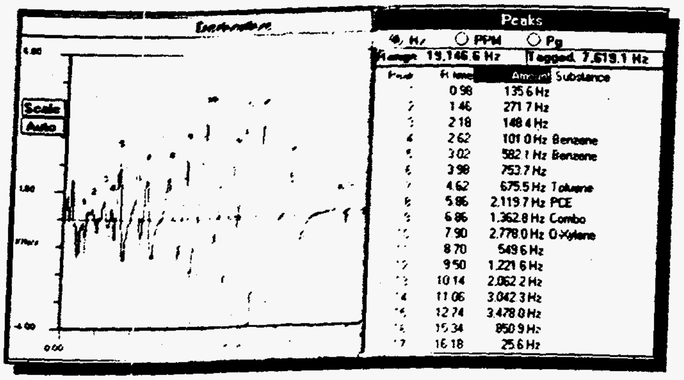
○ Hz ● PPM ○ Pg

Range 0.0 Hz | Tagged: 361.0

Peak	R time	Amount	Substance
1	2.0[illegible]	144.9 PPB	cis DCE
2	2.4[illegible]	[illegible]14 PPB	trans DCE
3	3.74	82.5 PPB	TCE
4	[illegible]	71.2 PPB	PCE

Figure 12. Chromatogram of GWTF analytes of interest

- During May 1996, demonstrations were conducted at the DOE Hanford Site, both the 200 Area West and the 200 Area East. Samples were collected from the headspace of tanks containing mixed wastes. Both tedlar bags and summa canisters were used for sample collection. Two instruments with different columns were demonstrated first in the laboratory and then in the field.
  - ◊ Typically, the samples are collected and sent to a laboratory for analysis of hydrocarbons by GC-MS using an EPA method for TO-14 compounds.
  - ◊ For this demonstration, the samples were analyzed using standards supplied by the laboratory. Total hydrocarbon content was determined by summation of the individual components. The Model 4100 reduced the time required for an individual sample analysis from 50 minutes using a GC/MS in the lab to two minutes using the Model 4100 in the field.



Peaks

● Hz ○ PPM ○ Pg

Range 19,146.6 Hz | Tagged 7,619.1 Hz

Peak	R time	Amount	Substance
1	0.98	135.6 Hz	
2	1.46	271.7 Hz	
3	2.18	148.4 Hz	
4	2.62	101.0 Hz	Benzene
5	3.02	582.1 Hz	Benzene
6	3.98	753.7 Hz	
7	4.62	675.5 Hz	Toluene
8	5.86	2,119.7 Hz	PCE
9	6.86	1,362.8 Hz	Combo
10	7.90	2,778.0 Hz	O-Xylene
11	8.70	549.6 Hz	
12	9.50	1,221.6 Hz	
13	10.14	2,062.2 Hz	
14	11.06	3,042.3 Hz	
15	12.74	3,478.0 Hz	
16	15.34	850.9 Hz	
17	16.18	25.6 Hz	

Figure 13. Chromatogram of tank farm analytes of interest

- The Model 4100 SAW/GC was tested at the Cal EPA Hazardous Materials Laboratory, Berkeley, CA, for specificity and sensitivity toward dioxins, furans, and PCB's. Because these compounds have vapor pressures of 5-12 orders of magnitude less than VOCs, the SAW was modified to operate at significantly higher temperatures to ensure uniform evaporation. Target detection limits were established at the ppm levels by using calibrated amounts of the subject compounds (EPA-prepared solvent solutions with contaminant concentrations ranging from 0.1 ppm to 50 ppm).
  - ◊ The instrument was able to detect these compounds at the 0.1 ppm level. Subsequent experiments using real samples of fly ash containing dioxins and PCB's showed that the instrument was sensitive to five picograms over a sampling time of 10 seconds.

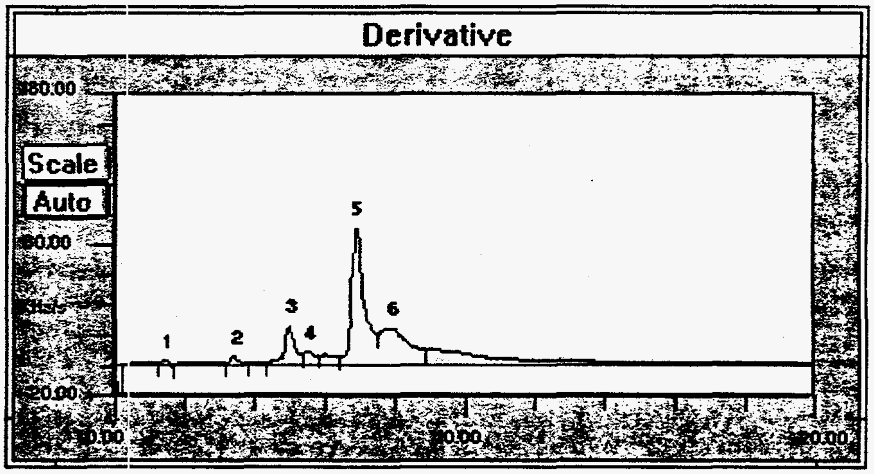


Figure 14. Response to dioxin-dibenzofurans mixture

- The Model 4100 SAW/GC was demonstrated at a Chicago Refinery to identify and quantify carbon scrubber efficiency in the containment of VOCs. The Model 4100 accurately detected benzene and toluene at the inlet and outlet to the scrubber.

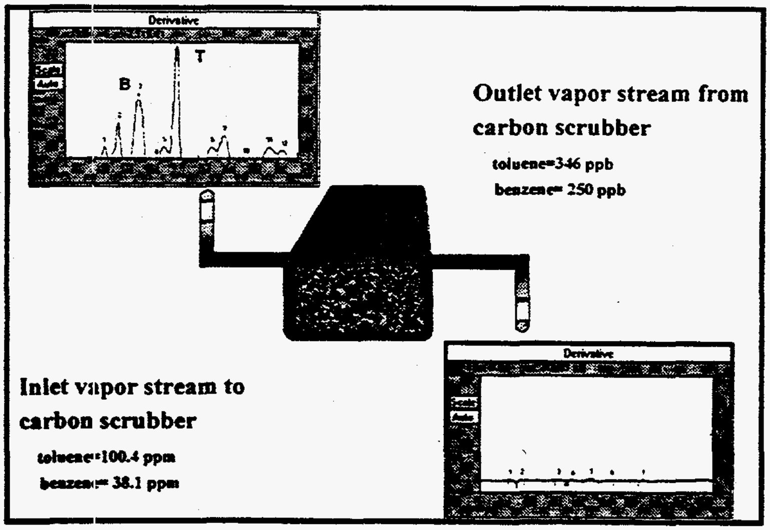


Figure 15. VOCs detected at the inlet and outlet of a carbon scrubber

SECTION 4

TECHNOLOGY APPLICABILITY AND ALTERNATIVE TECHNOLOGIES

Technology Applicability

- The specific problem targeted for this technology is quantitative field analysis of non-polar compounds, generally solvents and PCBs, at hazardous waste sites. The technology is especially effective at waste sites where historical information is available on the expected contaminant suite and concentration. SAWS/GC can be used for site characterization and monitoring, and remediation waste stream (e.g. offgas) monitoring.

- The SAWS/GC technology can also be used for other applications such as:
  - ◊ environmental monitoring of chemical processes, fugitive emissions and OSHA/CAA materials;
  - ◊ industrial monitoring including continuous emissions monitoring (CEM) of stack emissions, particle chemical processes, and other processes;
  - ◊ detection of contraband, drugs, explosives, and lethal chemicals for law enforcement and the military;
  - ◊ workplace monitoring for environmental health and safety.

- It is not expected that field screening with the Model 4100 will replace laboratory analysis but can be used to significantly reduce the number of samples sent offsite for more expensive laboratory analysis.

- Field screening with the Model 4100 allows collection of large data sets because of the relatively low cost of analyzing additional samples. Collection of a large number of replicate quantitative measurements at a low cost allows for a more robust statistical evaluation of the analytical results.

Competing Technologies

- In the Cost Section (#5), the technology is compared with two baseline scenarios:

  - ◊ The first comparison is to a stationary analytical laboratory using RCRA protocols.

    The standard method for analysis of soil and ground water samples is to package and document the sample according to EPA handling and chain-of-custody requirements and to ship the samples to a commercial laboratory for analysis. Laboratory analyses are done using procedures defined by the EPA in document SW-846.

  - ◊ The second comparison is with other portable analytical instruments including GC and GC/MS.

- Analyses by a commercial laboratory on a per sample basis are significantly more expensive than field screening.

- Field analysis of samples minimizes problems associated with potential loss of VOCs during sample handling, transport and holding.

- Field analysis of samples eliminates problems associated with holding time requirements.

- Many other portable field instruments are commercially available but most are not comparable because either they do not adequately speciate contaminants or they do not have adequate sensitivity (e.g., FTIR, fluorescence, Raman, simple surface acoustic wave spectroscopy, solid state sensors on a chip, photoionization detectors, electron capture detectors, flame ionization detectors, immunoassay test kits).

- The performance of field portable GC/MS instruments is comparable with the Model 4100 but capital costs are approximately three times as expensive.

SECTION 5

COST

Introduction

In 1992, the Los Alamos National Laboratory (LANL) compared the cost of analysis for VOCs in soil, water and gas samples by commercial laboratory and by six different field instruments (Henricksen and Booth, 1992). The study concluded that field sampling and analysis of VOCs offers substantial savings above a certain threshold number of samples per year (the order of 100 samples per year). They documented a factor of five reduction in cost per sample using field screening methods over commercial laboratory analysis. They compared six field screening instruments several of which are comparable in performance to the Model 4100. The cost decision for selection of the field screening devices was driven by capital cost of the instrument and supporting equipment, because the annual operating costs of the six methods was relatively constant. The capital costs, in 199 dollars, ranged from $42K to $166K while the estimate of annual operating expenses ranged from $147K TO $159K.

Discussion

A direct comparison of the Model 4100 with the results obtained from the LANL study is not possible for the following reason First, due to lack of operating experience, reliable estimates of the operating cost for the EST Surface Acoustic Wave/GC (SAW/GC) are not available. A rough estimate was obtained from the vendor and is included below. Second the LANL report was written in 1992 and is priced in 1992 dollars. While it is reasonable to escalate operating costs to 1997 dollars, it is not reasonable to escalate hardware prices because they typically do not increase. Most often, the equipment manufacturer releases a new version of the instrument with improved capabilities and at an increased price.

The following analysis summarizes capital and operating cost estimates for the Model 4100 instrument. It is very important to note that a rather robust operational scenario for the equipment was used. This includes the cost of two technicians and a vehic to support the use of the instrument. In most DOE applications, personnel and vehicles are available and are currently assigned to sampling and analysis tasks. The cost of implementation in this case is much less than estimated here. This scenario was chosen so that the operational costs can be compared with the other instruments in the referenced LANL report. In fact, the report showed that operational costs for the selected field screening instruments are very similar. Because a price was not available from the vendor, the estimated capital price of $44,000 is based on similar technology used in the security industry.

MODEL 4100 CAPITAL COST ESTIMATE 1997 dollars

Instrument	$44,000
Training and User Support Kit	$2,000
Vehicle**	$20,000
Total	$66,000

SAW/GC OPERATING COST ESTIMATES 1997 dollars

Two full time technicians**	$139,200
Trap	$350
Helium carrier gas 99.9995%	$740
VOC Offgas Treatment	$1,000
GC Column	$4,000
Maintenance Contract	$2,500
Other Consumables	$2,500
Vehicle Operation**	$3,560
Admin Overhead	$7,420
Total	$161,270

**Sponsoring organizations at SRS, INEEL, and Hanford currently have technicians and vehicles in place within the organization. The cost of the technicians and vehicle is included to allow comparison with the following instruments.

COST continued

The following is a summary of the LANL data for detectors summarized in Table 5 (page 35-36) of the LANL report. The escalation to 1997 dollars is a compounded 3.1% per year and no attempt was made to reprice the instruments evaluated in the report.

Table 2: LANL ESTIMATES OF CAPITAL AND OPERATING COSTS FOR SIX METHODS OF VOC ANALYSIS IN THE FIELD

INSTRUMENT	CAPITAL COST 1992 dollars	OPERATING COST 1992 dollars	OPERATING COST 1997 dollars
Direct Sampling Ion Trap Mass Spectrometer (ORNL)	$111,525	$148,674	$172,460
LANL GC/MS w/ITMS	$142,725	$149,571	$173,500
Hewlett Packard 5890 GC w/5971A MSD	$78,947	$151,170	$175,357
VIKING SPECTRA TRAK 620	$165,575	$158,541	$183,910
PHOTOVAC GC w/PID	$42,010	$146,823	$170,315
SENTEX GC w/AID/ECD	$42,645	$147,862	$171,520

Assumes 3.1% per year escalation rate 1992-1997.

Estimated operating costs for all instruments including the Model 4100 are very similar. A detailed discussion of the performance characteristics of the instruments is contained in the LANL report. In summary, the DSITMS, the GC MS, and the Model 4100 can detect most compounds of interest at the low ppb level in real time. The Sentex instrument requires preconcentration to routinely detect at the ppb level and in real time can only detect at the ppm level. The Photovac can detect from ppm to low ppb for some compounds but in real time only at the ppm level.

Conclusions

- The LANL study clearly documents a significant cost savings (a factor of five) when field screening instruments are used for analysis of VOCs in ground water and sediments, reducing the number of samples sent to comemrcial labs for analysis.
- The LANL study shows that field screening technology selection decisions are driven by capital cost of the instrument, because annual operating costs are relatively similar for different instruments.
- The only commercially available analytical instruments that provide unambiguous compound identification with ppb levels of sensitivity utilize mass spectrometric techniques.
- The cost comparison of the Model 4100 to mass spectrometric techniques is very favorable (capital costs of Model 4100 ≈30% of mass spectrometer).

SECTION 6

REGULATORY/POLICY ISSUES

Regulatory Considerations

- Although field screening methods, such as the Model 4100 SAW/GC, generally provide rapid, high quality, compound-specific data with minimal instrument maintenance and operating cost, procedures and application for their use are not generally as well documented as the EPA reference methods.
- A significant effort must be made by technology developers to acquire regulatory acceptance for new field methods.
- Secondary waste stream generation is the same or slightly reduced with the use of the Model 4100 over baseline methods.
- Field analysis of samples minimizes problems associated with potential loss of VOCs during sample handling, transport and holding.
- Field analysis of samples eliminates problems associated with holding time requirements
- Chain of custody requirements do not apply because samples are not transported.

Safety, Risks, Benefits and Community Reaction

Worker Safety

- The system is simple to operate and can be used safely by properly trained technicians. Technicians are required to complete sixteen hours of basic training.

Community Safety

- Field analysis of samples minimizes risks posed by sample handling, transport and holding.
- Use of field screening methods can significantly reduce the time necessary for site characterization.

Environmental Impact

- None

Socioeconomic Impacts and Community Perception

- Use of the technology will have minimal impact on the labor force and the economy of the region.

SECTION 7

LESSONS LEARNED

Implementation Considerations

- The site manager must work with the regulator to assure acceptance of the data collected. The pending Cal EPA Certification will assist in this acceptance.
- Field screening is not expected to replace laboratory analysis but can be used to significantly reduce the number of samples sent offsite.

Technology Limitations/Needs for Future Development

- The Model 4100 will not distinguish constituents that elute from the GC column at the same time. Careful choice of GC columns taking into consideration anticipated constituents will eliminate this problem.
- The Model 4100 is an excellent choice for robust field screening of non-polar compounds. Baseline GC-MS may provide better speciation in some settings, but capital costs are approximately three times as much.

Technology Selection Considerations

- Field screening methods, such as the Model 4100 SAW/GC, have the potential to provide rapid, high quality, compound-specific data with minimal instrument maintenance and operating cost when compared to EPA reference methods. These methods should be used in conjunction with laboratory analysis of a subset of samples for verification of the technology's performance.
- The accuracy of the Model 4100 SAW/GC usually derives from the fact that an expected suite and concentration of contaminants at DOE sites are generally known. Field screening using the Model 4100 can provide very good data where there is historical information to serve as a guide.
- Field screening methods allow collection of a large data set at a low cost, consistent with the well documented statistical approaches in exploration geochemistry.

APPENDIX A

REFERENCES

Anne D. Henriksen and Steven R. Booth, 1992, Cost Effectiveness of an Innovative Technology for VOC Detection: The Direct Sampling Ion Trap Mass Spectrometer", Los Alamos National Laboratory, LA-UR 92-3527.

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Lockheed Martin Energy Systems

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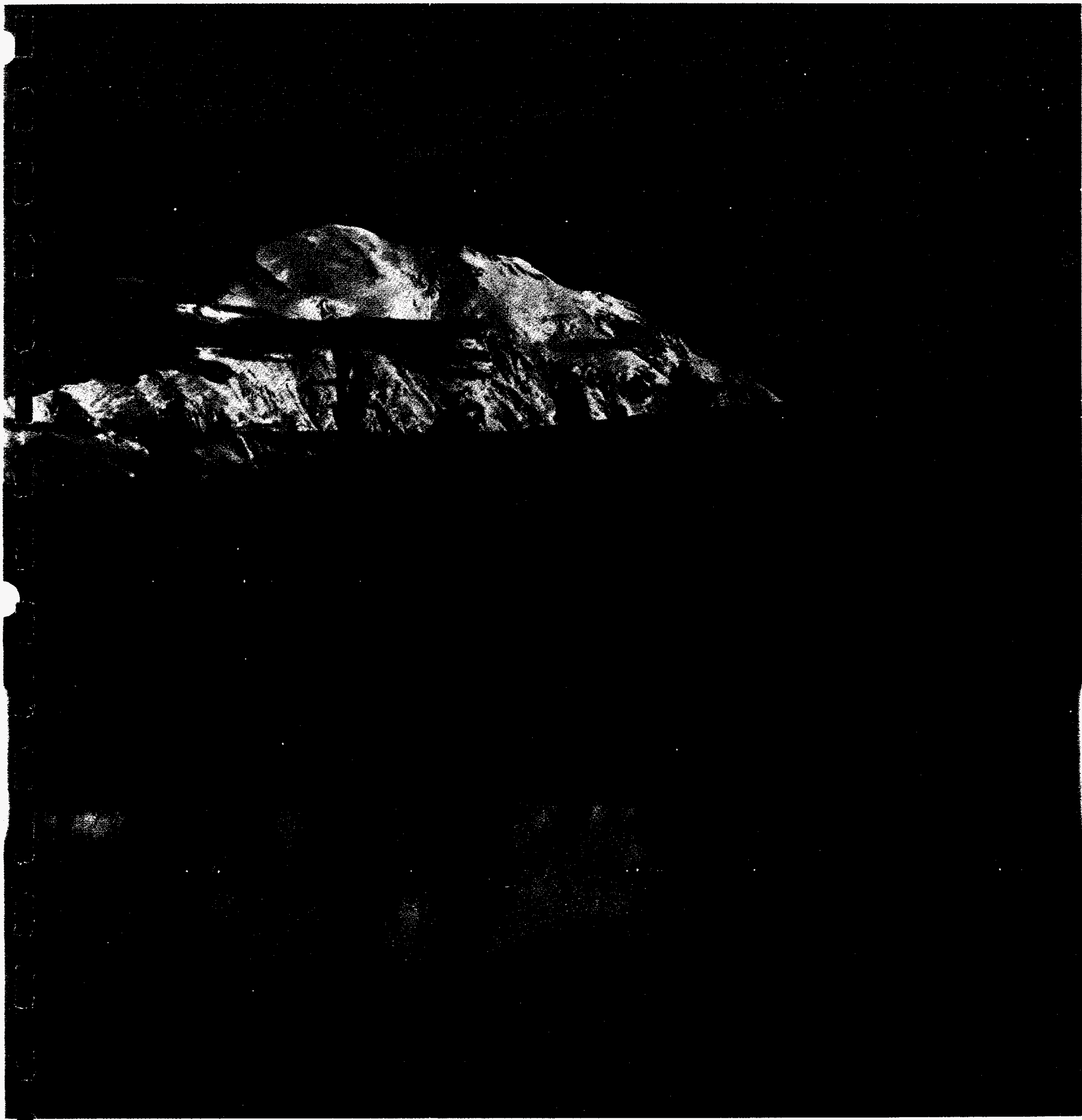
(423) 435-3470

Assistance was provided by

Amerasia Technology Corporation

3

TEST THE AIR ANYWHERE!



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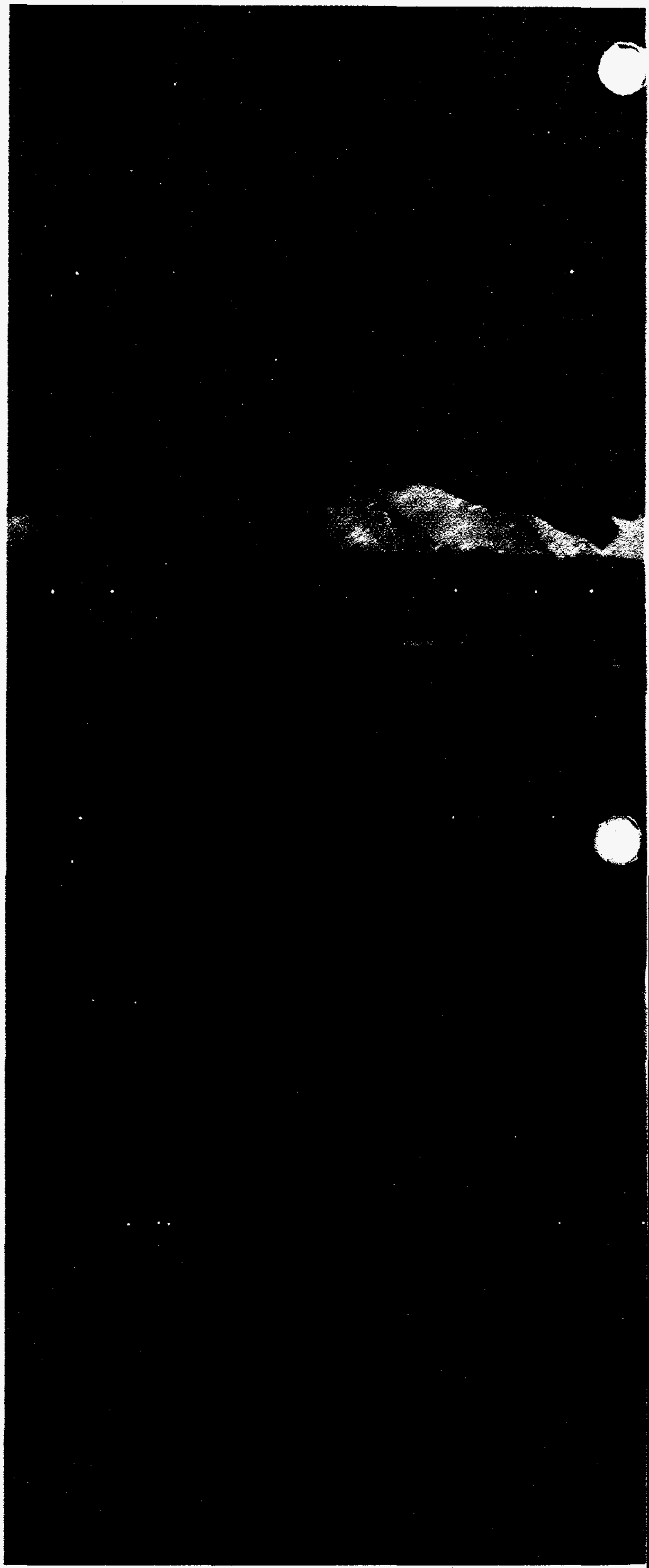


2301 Townsgate Road
Westlake Village, CA 91361
Phone (805) 495-9388
FAX (805) 495-1550

Model 4100 Vapor Detector and Analyzer

Description

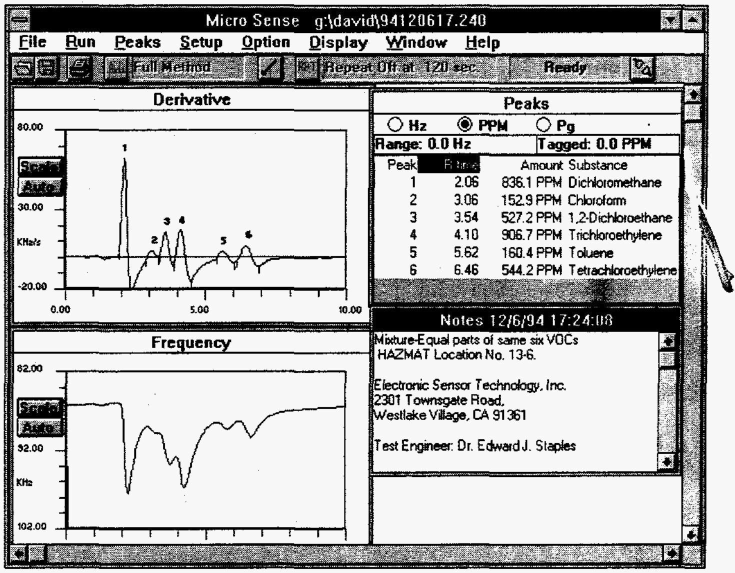
The 4100 consists of a portable Gas Chromatograph (GC), a SAW sensor and a dynamic particle/vapor sampling head. The components are shock mounted in a rugged field-portable fiberglass carrying case.

The solid-state resonator sensor has excellent recovery characteristics and provides sensitivity to picogram levels and spans, in dynamic range, more than five orders of magnitude.

Operation

The 4100 captures a vapor or particle sample in a cryo-focus chamber. After passing through a GC column, the suspect sample is identified and deposited on the SAW sensor. The SAW sensor determines the mass density and reports it to the database maintained in a 486 laptop computer.

The database identifies the suspect sample by comparison with a library of chemical signatures on file and the Windows® based program calculates the mass. The analysis is displayed on a multiple window screen.



Range: 0.0 Hz
Tagged: 0.0 PPM

Peak	R time	Amount	Substance
1	2.06	836.1 PPM	Dichloromethane
2	3.06	152.9 PPM	Chloroform
3	3.54	527.2 PPM	1,2-Dichloroethane
4	4.10	906.7 PPM	Trichloroethylene
5	5.62	160.4 PPM	Toluene
6	6.46	544.2 PPM	Tetrachloroethylene

Notes 12/6/94 17:24:08

Mixture-Equal parts of same six VOCs
HAZMAT Location No. 13-6.

Electronic Sensor Technology, Inc.
2301 Townsgate Road,
Westlake Village, CA 91361

Test Engineer: Dr. Edward J. Staples

Software Selectable Parameters

- Peak detection values
- Peak alarm values
- Printer drivers
- RS-232 communication parameters
- Remote operation with modem

Specifications

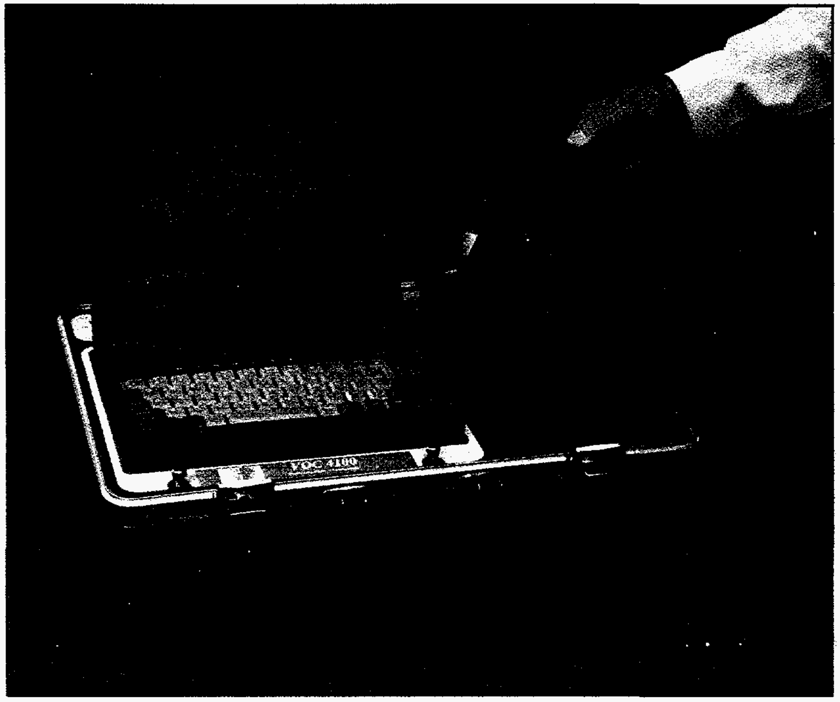
- 14 x 20 x 10 inches
- 35 pounds (15.9 Kg)
- High sensitivity (50 pico grams, or parts per billion)
- Wide dynamic signal range (20,000 minimum)
- Variable sampling time from 10 seconds to 2 minutes
- Analysis time from 5 seconds to 2 minutes
- Low detection threshold (parts per trillion)
- Fast column settling time (30 seconds or less)

Electronic
Sensor
Technology

2301 Townsgate Road
Westlake Village, CA 91361
Phone (805) 495-9388
FAX (805) 495-1550

Please feel free to contact us regarding your specific needs.

Model 4100 Vapor Detector and Analyzer



Portable, Fast, Highly Sensitive

Within 10 seconds, the 4100 identifies vapor concentrations as low as 100 parts per billion in amounts less than 50 pico grams.

The 4100 has demonstrated its speed, portability and sensitivity in varied and challenging screening operations. With its proprietary Surface Acoustic Wave (SAW) detector, it has sensitivity and dynamic range previously unattainable in a portable, low-cost package.

Easy to Use

Windows® based software and extensive on-line help make the 4100 easy to learn and easy to operate. The user interaction mode is selectable for non-technical field use as well as detailed lab analysis.

A range of operator levels can be displayed from simple sunlight legible LCD and audio detection indicators to complete multiple field portable chromatographs.

Electronic
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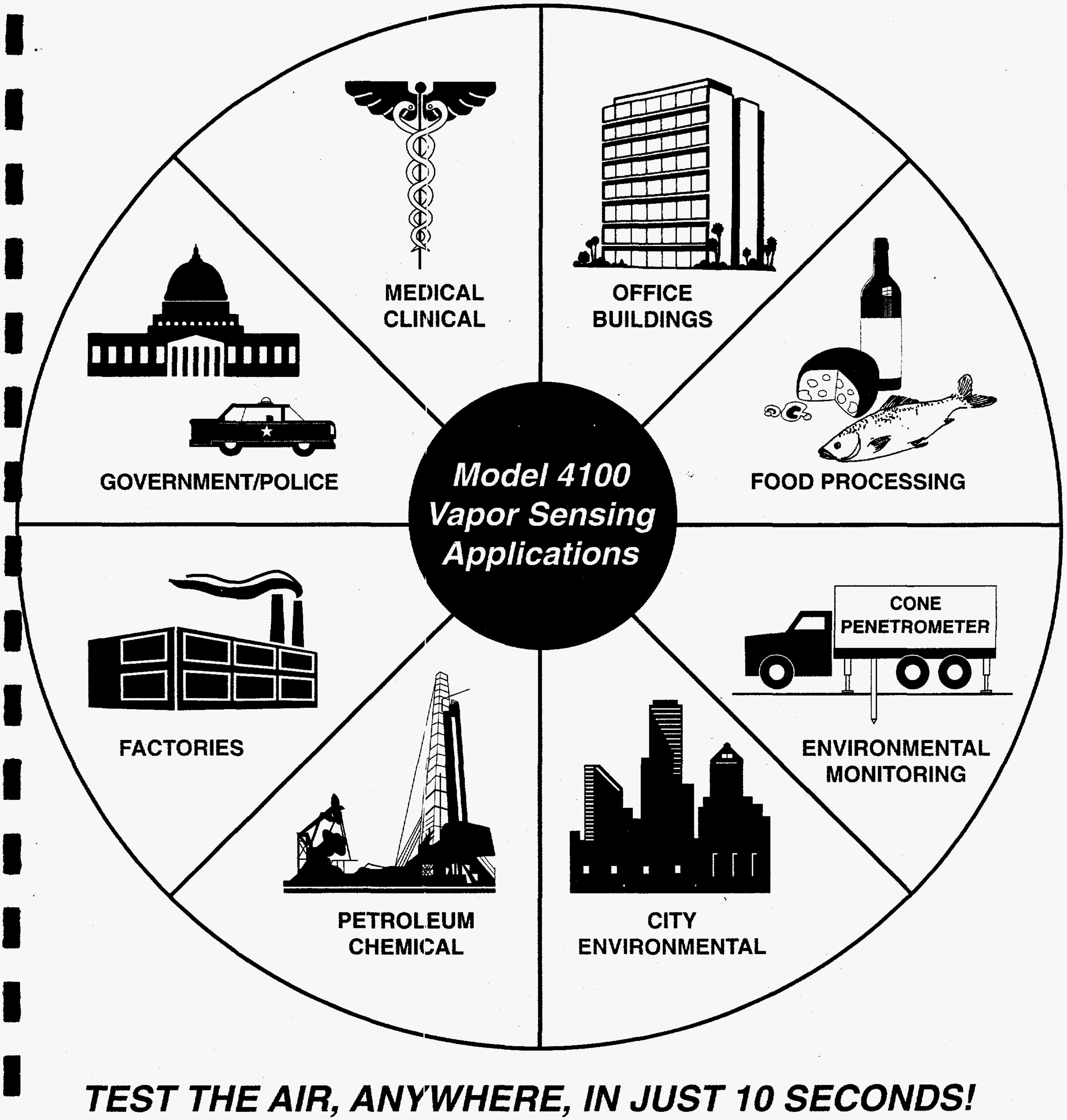
Model 4100 Vapor Detector and Analyzer



TEST THE AIR, ANYWHERE, IN JUST 10 SECONDS!

(More detailed listing on reverse side)

Model 4100 Applications

Configuration

- Process Control
- Food Processing
- Medical Laboratories
- Volatile Organic Compounds
- Military
  Chemical/Biological Warfare (CBW) Detection Systems

Environmental

PRINCIPAL APPLICATIONS

- Stack Emissions Monitoring
- Ambient Air Hazard and Emissions Monitoring
- Underground Tank and Water Contamination
- Fugitive Emissions
- Clean Air Compliance
- Industrial Hygiene-Related Air Monitoring

SELECTED VOC SIGNATURES

- Trichloroethylene
- Gasoline / Diesel
- Carbon Tetrachloride
- Benzene
- Toluene

Contraband Detection, Drugs of Abuse

SELECTED CHEMICAL SIGNATURES

- Heroin
- Cocaine
- Meth-Amphetamines
- Marijuana
- PCP

Explosive Detection, Security Systems

SELECTED CHEMICAL SIGNATURES

- Plastic Explosive
- Semtex
- Nitroglycerin
- RDX
- PETN
- TNT
- Dynamite
- C-4
- Taggents

Clinical Applications

- Hospital Floor Monitoring and Analysis
- Ethanol Alcohol Testing

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Please feel free to contact us regarding your specific needs.

TechKnow™
Reference Guide

Global Environment &
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Fax (703) 750-6506
E-mail: gnet@getf.org

www.earthvision.net
www.gnet.org
www.iso14000.net

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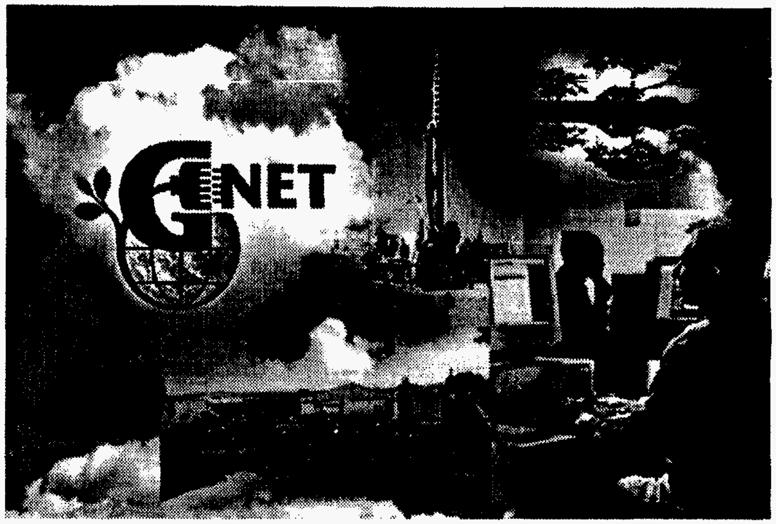


The New Technology Database for Environmental Business

Version 1.0

Background

What is TechKnow™?

TechKnow™ is not just a database on the World Wide Web. It is a free, easy-to-use tool that can help you learn more – or share more – about innovative environmental technologies.

A secure, password-protected database, TechKnow™ allows you to access, add, and update data highlighting environmental technologies over the Web.

All you need to enter TechKnow™ is a personal computer, access to the World Wide Web, and a frame-compatible Web browser (Netscape™ or Microsoft Explorer™ version 3.0 or higher). In order to use TechKnow™, you must register and open a free user account.

This reference booklet provides step-by-step instructions to help you use TechKnow™.

ABOUT TECHKNOW™

Access TechKnow™ at www.gnet.org, and click the "About TechKnow™" button!

GNET™ and TechKnow™ are a part of the Global Environmental Technology Enterprise (GETE), an initiative managed by the Global Environment & Technology Foundation and supported, in part, by the U.S. Department of Energy and the Federal Energy Technology Center, under cooperative agreement DE-FC21-94MC31179.

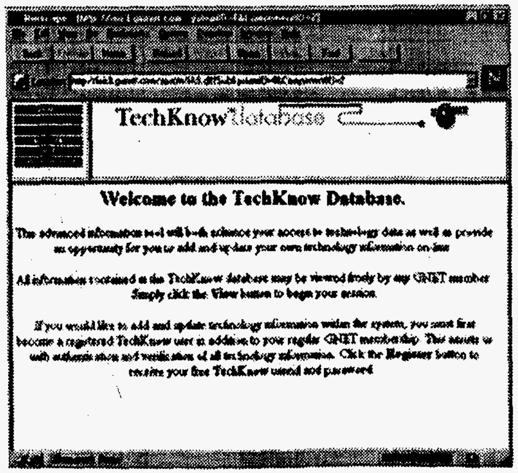
TechKnow™ Reference Guide
Contents

Getting Started

Step 1: Set Up Your TechKnow™ User Account.

1. Go to GNET™ on the World Wide Web at http://www.gnet.org. From this Main Page, click on the "Technology Center", then click on the **TECHKNOW™ DATABASE** button.
2. The TechKnow™ Main Page will appear. Click on the **REGISTER** button.
3. A database entry page will appear. Click on **ENTER TECHKNOW™** button.
4. A window will appear asking you to log in. Type in "techuser" and "guest"*, then hit enter.
5. TechKnow™ will now appear (it produces frames on your screen).
6. In the top, left-hand corner click on the green **REGISTER** button. Fill in all fields (you create your own UserID and Password). **Keep in mind these fields are case-sensitive.**
7. You should receive a confirmation message stating whether your account is approved within 24 hrs.

*The guest UserID and Password may only be used to "View" technology information.



Welcome to the TechKnow Database.

Step 2: Check to See if Your Technology is Already Listed.

1. While inside the system, click on the green **VIEW** button. Eight buttons will appear at the top of your screen.
2. Click on the purple **NAME** button. The alphabet will appear on the left side of your screen. You may either click on the letter that your technology begins with, or click on "Display All" to browse for your technology.
3. If you find your technology in the system, click on it to view the full profile. Check the "Editor" field (on the right side, third from the top) to see who currently owns the profile.
4. Each technology profile in the system is linked to one TechKnow™ account. If you are unfamiliar with the "Editor" you will need to send us a request via e-mail to gnet@getf.org so we can assign the profile to you. **The profile must be assigned to your account before it can be updated.**

ADD

Step 3: Add Your Technology to TechKnow™.

1. After your account has been approved, go to TechKnow™ and log in with your UserID and Password (*Required*).
2. Once inside, click on the green **ADD/UPDATE/DELETE** button in the top, left corner. Three buttons will appear at the top of your screen.
3. Click on the purple **ADD** button. Eight buttons will appear on the left side of your screen.
4. Click on the purple **GENERAL INFO** button to begin creating your profile. Fill in all applicable fields. When finished, click the **SAVE** button.
5. Once you are satisfied with the information, click the green **SEND TO MODERATOR** button. This will send all of your information for inclusion to the public viewing area of TechKnow™. You will receive a confirmation message of its receipt within 48 hrs.*

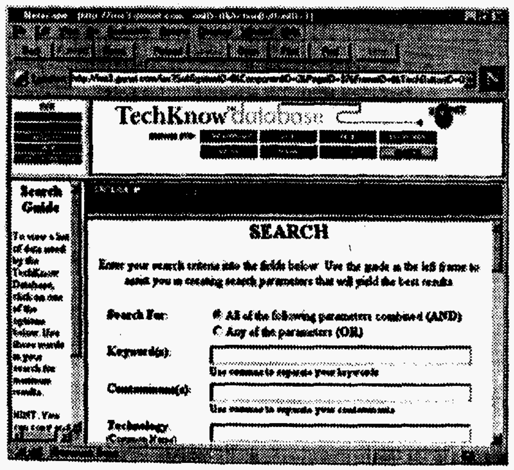
*After your technology has been reviewed by the moderator, you will receive a second notification indicating its "approval" status.

Searching for Information

Option 1: Use the Search Feature. **SEARCH**

1. Go to the GNET™ Technology Center and log into TechKnow™ with your UserID and Password or the guest account provided. If you do not have a TechKnow™ user account, refer to the "Getting Started" section of this booklet.
2. Once inside TechKnow™, click on the green **VIEW** button located in the top-left corner of your screen. Eight buttons will appear at the top of your screen.
3. Click on the grey **SEARCH*** button. Search fields and a search guide will appear at the bottom of your screen.
4. Enter your criteria into desired fields. Click on the **SUBMIT QUERY** button at the bottom when finished. An alphabet will appear in the bottom-left of your screen.
5. Click on either the desired letter or "Display All" option. A list of technology profiles will appear in the main window.
6. Scroll down through the list and click on the desired technology to view its entire profile.

* You may click on the **SEARCH** button at any time to restart.



Search Guide

To view a list of data used by the TechKnow Database, click on one of the options below. Use these words in your search for maximum results.

HINT: You

SEARCH

Enter your search criteria into the fields below. Use the guide in the left frame to assist you in creating search parameters that will yield the best results.

Search For: ● All of the following parameters combined (AND)
○ Any of the parameters (OR)

Keyword(s):
Use commas to separate your keywords

Contaminant(s):
Use commas to separate your contaminants

Technology (Common Name):

Option 2: Browse by Keyword. **KEYWORDS**

1. While inside the TechKnow™ "View" mode, click on the purple **KEYWORDS** button. An alphabet will appear in the bottom-left corner of your screen.
2. You may either click on the desired letter, or the "Display All" (which will take a little longer to download). A list of keywords will appear in the bottom-left corner of your screen.
3. Scroll down the list and click on the desired keyword. A list of technologies which match your criteria will appear in the center of your screen (if no matches are found or you wish to try another keyword, just click on another from the list provided).
4. Click on the desired technology to view its full profile.

Browse by

CONTAMINANT	DOE	FRTR
MEDIA	NAME	TSF

Option 3: Browse by Classification.

1. While inside the TechKnow™ "View" mode, click on any of the following purple buttons: **CONTAMINANT**, **MEDIA**, **DOE**, **NAME**, **FRTR**, or **TSF**.* A list of classifications will appear in the bottom-left corner of your screen.
2. Click on the desired classification. A list of applicable technologies will appear in the main portion of your screen.
3. You may scroll through the list of technologies, or click on a letter from the alphabet menu located on the bottom-left corner of your screen.
4. Click on the desired technology to view its full profile.

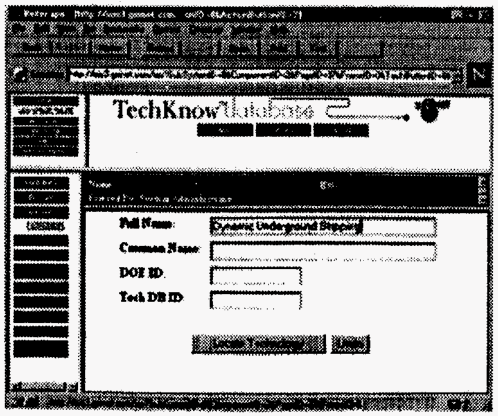
* These classifications are further described on page 8 of this reference guide.

Updating Your Technology

Step 1:

Locate your technology profile.

1. Enter GNET™ (http://www.gnet.org), click first on the "Technology Center," then click on the **TECHKNOW™ DATABASE** button.
2. The TechKnow™ Main Page will appear. Click on the **ENTER TECHKNOW™** button.
3. A window will appear asking you to log in. Type in your approved UserID and Password (**remember this is case sensitive, it must exactly match**) and hit enter.
4. Once inside TechKnow™ , click on the green **ADD/UPDATE/DELETE** button, located in the top-left corner of your screen. Three buttons will appear at the top of your screen.
5. Click on the purple **UPDATE** button. A group of buttons will appear in the bottom-left corner of your screen.
6. Click on the purple **TECH LIST** button. A listing of your technology profile with keywords will appear. If not, go to the "View" area (button at top-right of screen), locate the technology by name, and check the profile's editor field to see who is listed.



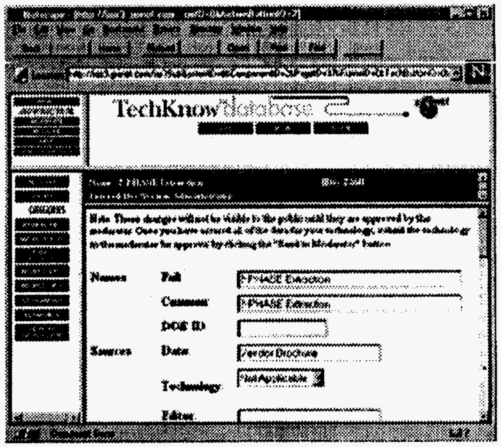
*If someone you are unfamiliar with is named as editor, you will need to notify GNET™ via e-mail to re-assign the profile to your account.

Step 2:

Update Your Technology Profile.

1. Once you have located your technology, click on it to view the entire profile. The buttons located on the bottom-left of your screen will become active.
2. Choosing from these buttons, click on the desired category*. The center of your screen will change and provide you with options on changing the specific fields.
3. Make changes as desired. At the end of each section you will be asked to save or clear your work.

* If you wish to add a keyword or category that is not included among the TechKnow™ menu options, send your request via e-mail to GNET™ for consideration.



Step 3:

Send Your Updated Technology Profile.

1. When you are finished updating your technology profile, review all changes for clerical errors and accuracy (e.g. URL addresses that work).
2. If you are satisfied that your updated profile is ready for public release, click the green **SEND TO MODERATOR** button located at the bottom-left of your screen.
3. A "Send to Moderator" page will appear asking if you are sure you want to upload the updated profile to the public area of TechKnow™. Click the **SUBMIT TECHNOLOGY** button to upload the profile.
4. You should receive an e-mail confirmation* within 48 hrs.

*After your update has been reviewed, you should receive a second notification stating if the updated profile was approved.

Understanding Keywords and Classifications

Keywords

TechKnow™ contains a keyword index of 756 words and phrases which are organized alphabetically. If a keyword is missing that you feel should be included in our index, you may request for its addition via e-mail.

Contaminant

TechKnow™ utilizes 16 of the most commonly used contaminant fields. You may access them through the "Contaminant" button while in the "View" mode.

Media

TechKnow™ allows you to create simple searches based solely on the type of affected media. These basic media categories generate a high-level sort of all technologies which are applicable to a certain field such as soil, or water.

DOE

TechKnow™ identifies eight Focus and Crosscutting Areas in which the U.S. Department of Energy (DOE) targets their technology development programs. This system provides additional background on how potential technology projects can relate to DOE. Definitions for each Area maybe found in the "Help" section of TechKnow™.

FRTR

TechKnow™ denotes the 20 work groups of the Federal Remediation Technology Roundtable (FRTR). Definitions are available in the "Help" section of TechKnow™.

TSF

TechKnow™ offers the White House's taxonomy for technology development from the Technology for a Sustainable Future (TSF) initiative.

Additional Tips

Printing Profiles and Lists

TechKnow™ allows you to print out information from any portion of the system. When you select the "print" icon, your Web browser will spawn a new browser page. Click the above print button within this new browser to print the screen. When finished, close this browser to get back to your original TechKnow™ screen.

Changing Screen Size

You may adjust the size of each frame within TechKnow™. Simply move your mouse over the frames border until your cursor changes to "double arrows." Click and drag the frame across your screen to the desired position.

Clicking on Related URLs

TechKnow™ includes hyperlinks to outside white papers, photographs, and other related Web sites. When you click on one of these links, your Web browser will spawn a new browser page containing the requested information. When finished, close this browser to get back to your original TechKnow™ screen.

Using "Display All"

TechKnow™ contains thousands of records in its system. Each time you click for information, the database is generating a custom page in response to your request. Therefore, choosing the "Display All" option may take some time to complete if you are using a modem.

Low Quality Natural Gas Sulfur Removal and Recovery
CNG Claus Sulfur Recovery Process

Pilot Plant Test Program

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Abstract

Large, non-producing reserves of Low Quality Natural Gas (LQNG) contaminated with hydrogen sulfide (H_2S), carbon dioxide (CO_2) and nitrogen (N_2) exist in identified gas reservoirs in the United States. However, these gas reserves are not suitable for treatment and production using current conventional gas treating methods due to both economic and environmental constraints.

A group of three technologies has been identified as candidates for an integrated system which would allow for economical processing of these LQNG reserves. The three processes which make up the integrated system are the **1) Controlled Freeze Zone (CFZ)**(1) process for hydrocarbon/acid gas separation; **2) Triple Point Crystallizer (TPC)**(2) process for H_2S/CO_2 separation and the **3) CNG Claus process** (3) for conversion and recovery of elemental sulfur from H_2S. The combined CFZ/TPC/CNG Claus group of processes is one program aimed at developing an alternative gas treating technology which is both economically and environmentally suitable for developing these low quality natural gas reserves.

Both of the CFZ and TPC technologies have been proven technically feasible during prior plant studies(4,5). However, the CNG Claus process had only been proven on a technical feasibility basis prior to 1996. This report describes the design, construction and operation of a pilot scale plant built to demonstrate the technical feasibility of the CNG Claus process which was designed to take advantage of the unique process conditions available for the integrated process system.

Acknowledgment

Funds for this project were provided by the United States Department of Energy, Morgantown Energy Technology Center and the Consolidated Natural Gas Company, Pittsburgh. This support is acknowledged with thanks and appreciation. Global Sulphur Experts Inc. (formerly of BOVAR Corp. and BOVAR Western Research) would also like to acknowledge Acrion Technologies, Cleveland, Ohio and the CNG Producing Company, Ardmore, Oklahoma for their support in completing this work.

1.0 Introduction

Increased use of natural gas (methane) in the domestic energy market will force the development of large non-producing gas reserves now considered to be low quality. Large reserves of low quality natural gas (LQNG) contaminated with hydrogen sulfide (H_2S), carbon dioxide (CO_2) and nitrogen (N_2) are available but not suitable for treatment using current conventional gas treating methods due to economic and environmental constraints.

A group of three technologies have been integrated to allow for processing of these LQNG reserves; the Controlled Freeze Zone (CFZ)(1) process for hydrocarbon / acid gas separation; the Triple Point Crystallizer (TPC) (2) process for H_2S / CO_2 separation and the CNG Claus process (3) for recovery of elemental sulfur from H_2S. The combined CFZ/TPC/CNG Claus group of processes is one program aimed at developing an alternative gas treating technology which is both economically and environmentally suitable for developing these low quality natural gas reserves.

The CFZ/TPC/CNG Claus process (4,5) is capable of treating low quality natural gas containing >10% CO_2 and measurable levels of H_2S and N_2 to pipeline specifications. The integrated CFZ / CNG Claus Process or the stand-alone CNG Claus Process has a number of attractive features for treating LQNG. The processes are capable of treating raw gas with a variety of trace contaminant components. The processes can also accommodate large changes in raw gas composition and flow rates. The combined processes are capable of achieving virtually undetectable levels of H_2S and significantly less than 2% CO_2 in the product methane.

The separation processes operate at pressure and deliver a high pressure (ca. 100 psia) acid gas (H_2S) stream for processing in the CNG Claus unit. This allows for substantial reductions in plant vessel size as compared to conventional modified Claus / Tail gas treating technologies. A close integration of the components of the CNG Claus process also allow use of the methane/H_2S separation unit as a Claus tail gas treating unit by recycling the CNG Claus tail gas stream. This allows for virtually 100 percent sulfur recovery efficiency (virtually zero SO_2 emissions) by recycling the sulfur laden tail gas to extinction. The use of the tail gas recycle scheme also de-emphasizes the conventional requirement in Claus units to have high unit conversion efficiency and thereby make the operation much less affected by process upsets and feed gas composition changes.

The development of these technologies has been ongoing for many years and both the CFZ and the TPC processes have been demonstrated at large pilot plant scales. On the other hand, prior to this project, the CNG Claus process had not been proven at any scale. Therefore, the primary objective of this portion of the program was to design, build and operate a pilot scale CNG Claus unit and demonstrate the required fundamental reaction chemistry and also demonstrate the viability of a reasonably sized working unit.

The decision to design, construct and evaluate a field scale pilot plant the CNG Claus process was based on a successful theoretical study(6) of the process conducted by BOVAR Western Research under contract to CNG and the DOE in the Phase 2 portion of this program.

1.1 CNG Claus Program Objectives

In the initial phase of this program, the technical viability of the CNG Claus process was evaluated with respect to the fundamental question:

Would the required Claus reaction chemistry (shown below) proceed to an acceptable level (in terms of unit sulfur conversion) under the unique operating conditions required by the CNG Claus process?

$$2\ H_2S + O_2 \rightleftharpoons S_2 + 2\ H_2O$$

During this phase of the program it was determined through reaction modeling systems that the H_2S oxidation reaction should successfully proceed with an adequate yield to sulfur under the desired operating conditions (6).

After completion of this phase of the work the next fundamental question was presented:

Can a pilot plant be successfully designed, built and operated for these unique conditions?

Through review of existing Claus sulfur recovery technology and equipment specification, it was determined that an operating unit could be designed and built successfully. It was determined that much of the commercially proven hardware components used in a conventional modified Claus plant would have equal applicability to the unique operating conditions presented by the CNG Claus process.

Therefore, the primary objective of this portion of the program was to design, build and operate a pilot scale CNG Claus unit, demonstrate the required fundamental reaction chemistry and demonstrate the operational viability of a reasonably sized working unit.

1.2 Pilot Plant Program Objectives

The pilot plant phase of the program was designed to realize the following specific objectives:

1. Design and construct a viable pilot scale unit.

2. Demonstrate the operation of the unit under the unique CNG Claus conditions in order to verify the applicability of the pilot plant unit equipment.

3. Demonstrate the operation of the unit under the unique CNG Claus conditions in order to verify the desired reaction chemistry and acceptable conversion efficiency of H_2S to sulfur.

4. Identify technical barriers to the design and operation of a commercial CNG Claus sulfur recovery unit.

2.0 Conclusions

2.1 CNG Claus Chemistry

1. The Claus reaction (H_2S oxidation to elemental sulfur) proceeds successfully in a free flame reactor under all of the tested conditions associated with the CNG Claus Process.

2. The H_2S oxidation reaction is stable and sustainable in the "free flame" reaction regime.

3. The H_2S oxidation reaction continues successfully at the very low oxidation reaction stoichiometries present in the CNG Claus process. The typical reaction stoichiometries were at 1/6 of the full oxidation stoichiometry as compared to 1/3 reaction stoichiometry which is employed in traditional Modified Claus process schemes.

4. The Claus reaction proceeds successfully at pressures of 50 psia and greater. The tests conducted at higher reaction pressure did not indicate any measurable negative effects.

5. The extent of H_2S conversion measured during the test runs was consistent with the minimum required value of 50 percent as set out in the test program objectives.

6. The H_2S to sulfur conversion yields at the tested conditions closely matched the predicted results as determined by thermodynamic equilibrium calculations for all of the tested conditions. The measured conversion efficiencies ranged between 42 and 68 percent as compared to the predicted efficiencies between 44 to 70 percent. This implies that the H_2S oxidation reaction is not unusually hindered by reaction kinetics.

7. The measured process gas compositions in the CNG furnace effluent closely matched the predicted values based on computer simulations. In particular the SO_2 concentration was consistent with (and typically less than) the maximum allowed for in the original program objectives. This will ensure successful operation of the downstream tail gas treating section of the process.

8. The measured reaction furnace temperatures (1500 - 2200°F) were well within the acceptable range and the measured test run values closely matched the predicted values.

9. The extent of side reactions in the pilot plant indicate that kinetic parameters do affect the extent of formation of the minor reaction product species; H_2, CO, COS and CS_2. The concentrations of these compounds in the system are not considered detrimental to the overall successful operation of an integrated system.

2.2 Pilot Plant Operation

1. The pilot plant design and equipment was taken directly from conventional technology and performed essentially as planned.

2. The conventional burner and reaction furnace system allowed for successful completion of the H_2S oxidation reaction under the required conditions.

3. The measured reaction furnace temperatures were consistently less than the allowable 2500°F maximum which ensures that conventional furnace refractory design can be employed.

4. The equipment failure in the pilot plant (leaking wasteheat exchanger) was probably a result of conventional corrosion and was not an artifact of the unconventional CNG Claus operating conditions.

5. The pilot plant was designed with entirely conventional metallurgy and did not suffer any unusual failures due to the CNG Claus operating conditions.

3.0 CNG Claus Process and Pilot Plant

The pilot plant was designed to demonstrate the successful operation of the most critical portion of the CNG Claus unit; the Free Flame Reaction Furnace (RF). It is in this unit that the H_2S and O_2 react vigorously to produce the desired product, elemental sulfur and other reaction by-products. The subsequent treating of the reaction by-products would be completed using conventional tail gas treating technology. Since all components of the tail gas treating technology have been in wide commercial use for many years, these components were not included in the CNG Claus pilot plant unit.

3.1 CNG Claus Process Description

The Sulfur Recovery Section of the CNG Claus process is based on a single stage, free flame reactor system. In this unit the acid gas feed is combusted with an oxygen bearing stream (ambient air or high concentration O_2 stream as required) in a <u>reaction furnace</u> unit. This results in the direct oxidation and conversion of H_2S to elemental sulfur. The elemental sulfur is then recovered via condensation in a conventional shell and tube heat exchange <u>sulfur condenser unit</u>.

In the Tail Gas Treating Section the CNG Claus tail gas is processed in a conventional <u>catalytic hydrogenation reactor</u> in order to react all remaining sulfur compounds to H_2S. This stream is then further treated to cool the process gas and remove most of the residual water in a <u>direct contact water contactor</u> and a <u>conventional dehydration unit</u>. The cooled, dehydrated tail gas is then re-compressed to be recycled to the TPC separation system.

The full process flow diagram is given in Figures 3.1-1 and 3.1-2. The primary operating units, their functions and the most important unit details are as follows:

3.1.1 Sulfur Recovery Section

Reaction Furnace

The H_2S oxidation reaction occurs in a free flame, single phase, open volume reactor modeled after a conventional modified Claus reaction furnace.

In order to integrate efficiently with the upstream TPC separation unit several novel operation conditions were proposed for the reaction furnace.

1. Oxygen Composition - In order to minimize the flow of unwanted inert components through the system, it was proposed to use a highly enriched air stream as the oxidant in the reaction furnace. For the purpose of the pilot plant design a 90 percent oxygen stream was used. This effectively decreased the total process gas volumes and thereby decreased the required size of all of the processing equipment.

Figure 3.1-1
CNG Claus Sulfur Recovery Unit
Claus Section

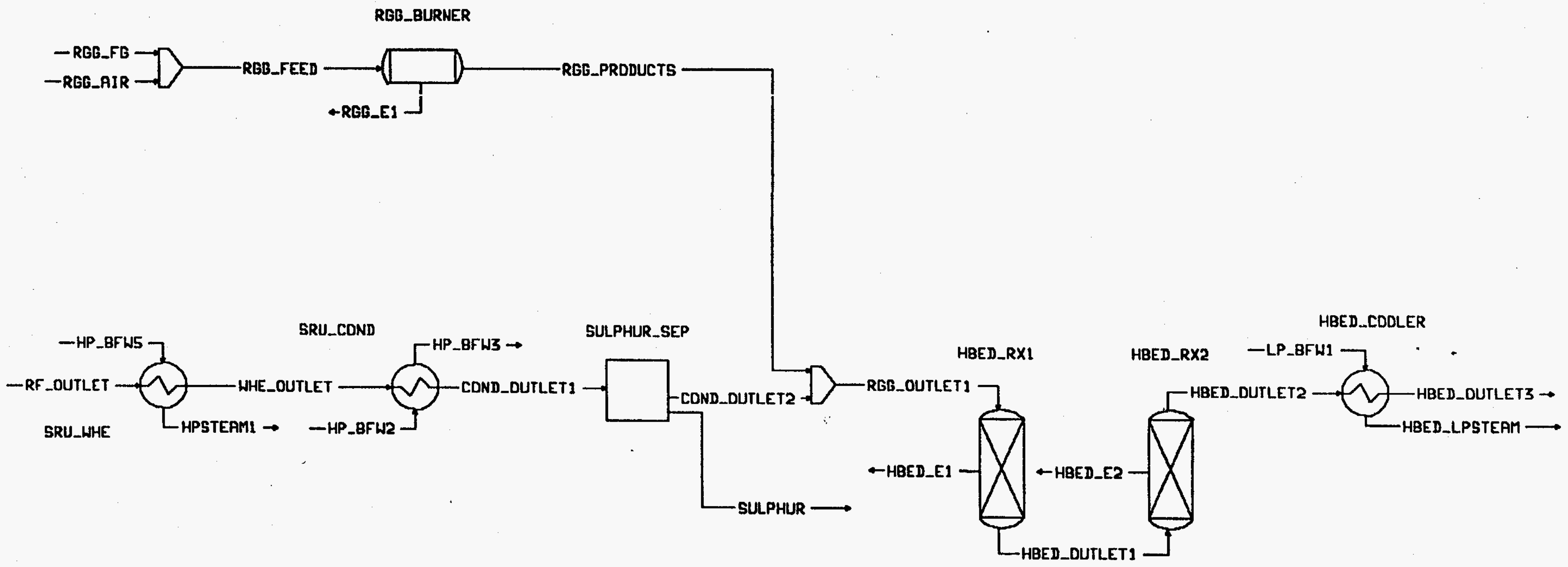
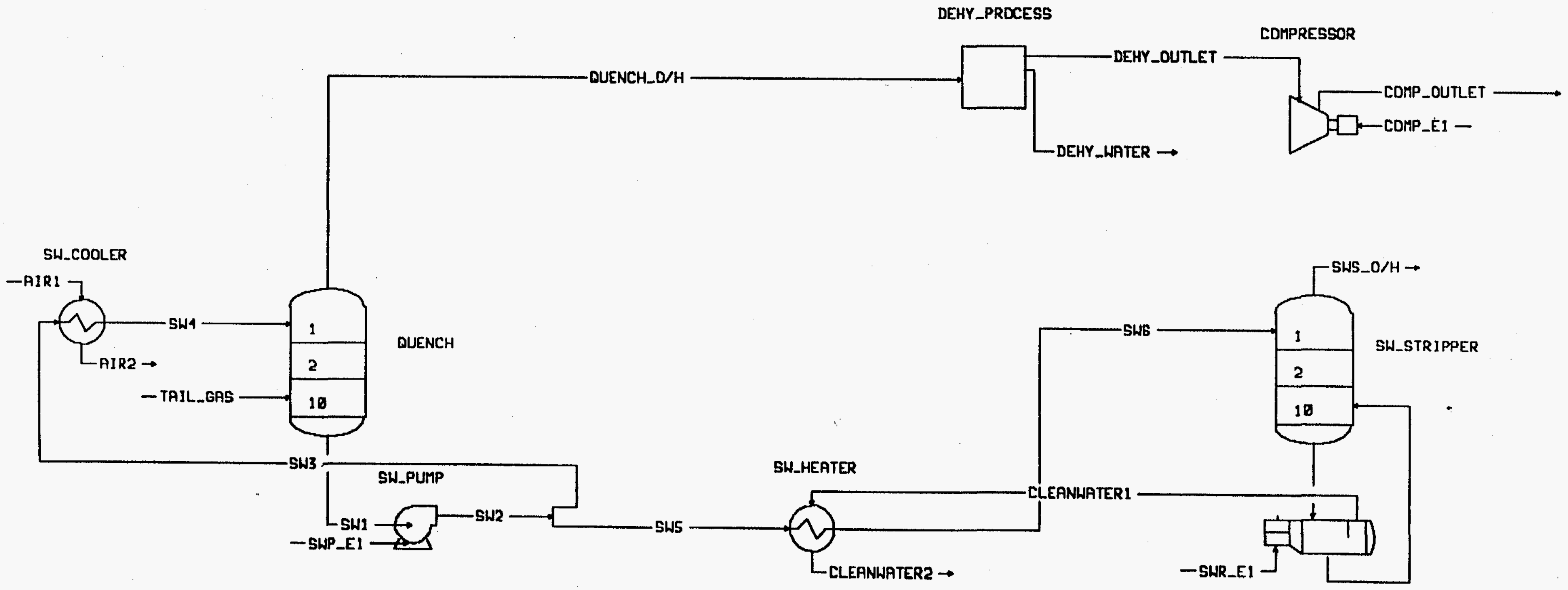


Figure 3.1-2
CNG Claus Sulfur Recovery Unit
Tail Gas Treating Section



2. Reaction Stoichiometry - A traditional modified Claus system calls for a 1/3 oxidation of the H_2S to SO_2. The product SO_2 then reacts with the remaining H_2S to form elemental sulfur. In a traditional modified Claus free flame reactor the typical unit conversion (yield) efficiency is 65 to 75 percent.

   For the CNG Claus system there was a need to minimize the SO_2 production and to control the potentially high reaction temperatures resulting from the use of enriched air as the oxidant. Therefore, it was proposed to operate the CNG Claus reaction furnace with a significantly lower reaction stoichiometry. For the design case, an oxygen deficient operation of approximately 50 percent (of traditional modified Claus stoichiometry) was used. This resulted in a decrease in the predicted unit conversion efficiency to 50 percent and a predicted reaction temperature less than 2000°F. This also resulted in an SO_2 concentration of approximately 1.0 mole percent in the reaction furnace effluent which is appropriate for the subsequent treating in the hydrogenation reactor.

Wasteheat Exchanger

The partial oxidation of the H_2S in the reaction furnace is an extremely exothermic reaction which results in a very large energy release and high process temperatures in the reaction furnace. In order to recover the produced elemental sulfur it is necessary to cool the process gas significantly. This also allows the opportunity to recover a large amount of high quality energy usually in the form of high pressure steam.

The wasteheat exchanger is used to remove the bulk of that energy and cool the process gas to a lower intermediate temperature. This unit is a conventional shell and tube thermosyphon or kettle type boiler which will produce high pressure (600 psia) steam.

Sulfur Condenser / Collection Vessel

The sulfur condenser is used to further cool the process stream to a temperature (300°F) where essentially all of the sulfur vapor is condensed to liquid. The sulfur condenser heat exchanger has a traditional shell and tube configuration with the process gas on the tube side. This unit also recovers a significant amount of waste heat by producing steam.

The sulfur condenser vessel was equipped with a disengagement section on the outlet end in order to allow for efficient separation of the product liquid sulfur from the process gas. A collection vessel equipped with continuous level control was used to store and remove the product sulfur from the process. This is effectively the final step in the true sulfur recovery portion of the plant. All of the subsequent units are used to treat the sulfur plant tail gas for recycle back to the TPC process.

3.1.2 Tail Gas Treating Section

In order to allow for a 100 percent recycle of the tail gas stream to the TPC system, it will be necessary to further treat the CNG Claus tail gas. The tail gas stream must meet some critical guidelines in terms of stream content to allow efficient processing in the TPC.

1. SO_2 Content - The SO_2 content in the tail gas must be essentially zero so that it does not react with H_2S to form sulfur in the TPC or its auxiliary systems.

2. Water Content - The tail gas must be dried to a very low dewpoint temperature to ensure that water does not freeze in the TPC system.

3. Process pressure - In order to return the treated tail gas to the TPC it will be necessary to re-compress the process gas back up to ca. 100 psia.

The tail gas treating section was designed to perform the following specific process steps :

1. Heat the process gas and introduce reducing compounds into the process.

2. React all of the non-H_2S sulfur components back to H_2S via various reduction reactions.

3. Cool and remove the excess water.

4. Re-compress the cold, water free process gas which is primarily H_2S and CO_2 for recycle to the TPC separation process.

All of these functions are performed in units which have been used extensively in existing gas treating processes. Therefore, the design philosophy in general is based completely on existing technology.

Reducing Gas Generator

This unit performs two functions simultaneously; heating the process gas and introducing reducing compounds (H_2 and CO) into the process stream. The primary process unit in the reducing gas generator is a direct fired hydrocarbon fuel gas burner. The burn products from this burner are mixed directly with the process gas to raise the process temperature to the desired value.

This burner system utilizes a controlled oxygen source (enriched air) and the burn stoichiometry is controlled such that the unit is 5 to 15 percent deficient in oxygen. This has the result of producing significant amounts of H_2 and CO in the burner which is needed in the subsequent catalytic processing step. The base design calls for a typical mixed outlet temperature of 500°F.

Hydrogenation / Hydrolysis Reactor

The next tail gas treating step is to convert all of the SO_2, COS, CS_2 and sulfur vapor to H_2S. This is completed in a fixed bed reactor over a cobalt - molybdenum catalyst. The four main reactions which occur in this converter are :

1. SO_2 conversion: $SO_2 + 2\,H_2 \rightleftharpoons H_2S + H_2O$
2. CS_2 conversion: $CS_2 + 2\,H_2O \rightleftharpoons 2\,H_2S + CO_2$
3. COS conversion: $COS + H_2O \rightleftharpoons H_2S + CO_2$
4. Sulfur conversion: $S_2 + 2\,H_2 \rightleftharpoons 2\,H_2S$

This reaction system is utilized extensively in existing gas treating technology and all of the reactions go to completion to ensure no SO_2, COS, CS_2 or S_2 residual in the reactor effluent. The only sulfur bearing compound in the reactor effluent stream is H_2S. The typical reaction temperature in this system is 650 to 750°F.

Hydrogenation Reactor Cooler

In order to meet the water specification for the recycle stream it is necessary to remove all of the process water. As a preliminary step in the water removal process, the tail gas is cooled significantly from the 700°F+ hydrogenation reactor temperature to approximately 300°F. The initial cooling step is performed by a standard shell and tube heat exchanger with the process gas on the tube side. In a conventional design it is convenient to remove the excess energy in the form of steam.

Direct Water Quench / Water Removal

The next step in the water removal process is a direct contact water quench tower. In this unit the cooled tail gas is contacted directly with cooling water. The quench tower overhead gas is effectively cooled to 100°F and the water content is dropped to the saturated value at that temperature. A typical water content of the quench overhead gas is 2 to 5 percent as compared to 25 to 35 percent in the inlet. This process produces a substantial mass of excess water which has a small sour component. This "sour water" must undergo additional treatment in order to allow for safe disposal. The sour water treatment system is described in a later section.

Tail Gas Dehydration

The final dehydration process must ensure that the water content in the recycled tail gas does not exceed 30 ppmv which translates to a -50°F water dewpoint temperature. This step is completed in a dedicated molecular sieve dehydration system of conventional design.

Tail Gas Recompression

It is expected that the sulfur recovery and tail gas treatment processes will result in a system pressure loss between 5 and 10 psi. Therefore, the treated tail gas must be re-compressed to allow re-introduction back into the upstream TPC process. Based on the expected operating conditions of the system, the recompression ratio in this unit will have to be approximately 1.1 to 1.

3.2 Sulfur Recovery Unit Pilot Plant

The original design concept of the CNG Claus sulfur recovery process is based extensively on existing conventional Claus technology. However, the operating conditions will be substantially different from conventional Claus operations in the following areas :

1. Oxygen source - The primary oxygen source will be a highly O_2 enriched air stream in order to minimize the total process gas volume.

2. Reaction stoichiometry - The reaction will be completed with an oxygen flow of approximately 50 percent of the flow in a conventional Claus plant. This will ensure more moderate reaction temperatures and a low formation rate of SO_2.

3. Operating pressure - The operating pressure will be ca. 100 psia as compared to a conventional Claus plant which operates at 15 to 25 psia.

3.2.1 Design Basis and Equipment Description

Acid Gas Feed

Table 3.2-1 summarizes a typical acid gas and combustion "air" stream data cases for the pilot plant study. The acid gas composition was based on a preliminary estimated composition from the specified low quality raw gas stream. The "air" composition was chosen as typical for an enriched air stream from a pressure-swing O_2 enrichment plant. This acid gas flow rate and the target 50 percent unit recovery efficiency result in a total sulfur production rate of 10.0 lb/h.

Table 3.2-1
Pilot Plant Base Case Feed Gas Conditions

		Acid Gas	Oxygen
Temperature	(°F)	50.0	50.0
Pressure	(psia)	100.0	100.0
Total Flow	(lbmol/h)	0.891	Variable
Composition	(mole %)		
	H_2S	69.7	
	CO_2	30.0	
	COS	0.3	
	O_2		90.00
	N_2		10.00

The P&ID for the entire pilot plant is presented in Figure 3.2.1-1.

Feed Gas Control System

This section of the pilot plant consists of three parallel and nearly identical reactant feed metering trains. The Hydrogen Sulfide (H_2S), transported and stored for use in a liquid form, is vaporized within the storage container through the use of an electric heater blanket wrapped around its exterior surface. This gas is then regulated from a pressure of 250 psig to a delivery pressure of 120 psig by a forward pressure regulator (PCV-21B). The flow rate of the regulated gas is then metered and controlled with a mass flow controller (FT/FV-21B). A manual (un-metered) bypass around the flow controller is available for situations when the mass flow controller is out of service. The metered stream downstream of the flow controller then passes through a dome loaded back pressure (negative bias) regulator. This device maintains a constant back pressure on the flow controller (and thus constant pressure drop across the control valve) enabling accurate steady flow control even with fluctuating downstream pressures. The next components of this section consists of an air operated shut-off valve, for remote (control panel) operation and a check valve.

The Carbon Dioxide (CO_2) feed metering section is similar to the H_2S feed section with the exception that the liquid storage container does not require heating to vaporize the reactant, the mass flow controller (FT/FV-21C) is sized for a lower flow range and the pressure regulators (PCV-21C) have correspondingly lower flow coefficients.

Both feed lines have sample connections on the low pressure side of the feed regulator to allow for controlled flow sampling for reactant feed purity analysis. Block and precision metering valves are supplied at all sample points to provide for pressure let down and safe sampling.

The H_2S and CO_2 feed streams are each individually metered to allow custom blending of the two streams to any relative amount (within the flow ranges of the mass flow controllers) and are then mixed within a specially fabricated mixing tee (MX-1). This tee ensures that the two streams create a thoroughly mixed acid gas stream before they are sent to the burner for combustion.

The Oxygen stream as supplied (90% O_2, 10% N_2) is regulated from bottle pressure to a supply pressure of 110 psig and metered in a similar fashion to the H_2S and CO_2 streams (FT/FV-21A and FCV-21B). Provisions have been made to allow for an external source of nitrogen to be added to this stream if dilution becomes necessary. Also the nitrogen serves as an inert safety gas during system leak testing and emergency shutdowns (oxygen block-in).

After metering, the oxygen flows in one of two directions. During start-up, the stream is diverted (AV-22A on AV-21A) to mix with the fuel gas (methane) for burner ignition. After system warm-up and pressure control is established, the acid gas mixture can be introduced into the system through its metering system. The oxygen stream can then be split between the fuel gas and the acid gas streams (AV-21A on, AV-21B on) to establish combustion of the acid gas. The oxygen and acid gas streams are thoroughly mixed in a second mixing tee (MX-2) and then sent to the burner.

At this point the fuel gas can be completely shut off and the complete oxygen stream mixed with the acid gas (AV-21A on, AV-22A off) for combustion in the reaction section. To prevent reaction of the acid gas and oxygen stream prior to the burner, both mixing tees as well as the fuel gas metering system will be physically mounted as close as possible to the reaction furnace inlet. This will minimize the length of the inlet process piping. Figure 3.2.1-2 presents the detailed P&ID for the feed gas control section of the pilot plant.

Reaction Furnace

The reaction furnace is designed to allow for intimate mixing of the process reactants, initiate a free flame reaction zone and allow continued reaction in an open volume reaction section. This unit is made up of two primary components; 1) the furnace burner unit and 2) the main combustion/reaction chamber.

The furnace burner has a concentric pipe design with a direct mixing flame retaining burner nozzle where the acid gas and oxygen streams are intimately mixed and ignited. The center pipe contains the fuel gas/oxygen mixture required for start-up, while the outer annulus processes the acid gas/oxygen combustion mixture after the fuel gas is shut in. The burner nozzle was of conventional design and metallurgy in order to imitate conventional equipment specifications. The burner was located at the center of the combustion end of the furnace vessel and included a gas pilot burner and electric igniter system.

The reaction furnace vessel is a stainless steel vessel with an inside working diameter of 6 inches and a working length of approximately 30 inches. The vessel design allows for a maximum working pressure of 130 psia at the expected reaction temperatures. The furnace vessel has an effective reaction volume of 0.49 ft^3 which allows for reaction residence times between 1 and 4 seconds. The vessel is lined with a custom molded refractory material which allows for working reaction furnaces temperatures up to 3000°F.

The reaction furnace is equipped with five internal thermocouples, a rear chamber water cooled sample probe and a vessel outer skin high temperature alarm. The internal temperatures are displayed at the control panel on a multi-channel temperature scanner for temperature profile observation. The sample probe will enable capturing of rear chamber reaction furnace samples prior to entering the waste heat exchanger section.

Overall system over-pressure protection is provided by means of a rupture disk mounted on the acid gas feed line to the reaction furnace. This device will protect all downstream equipment from excess pressure in the event of blocked in vents or fire.

Figures 3.2.1-3 and 3.2.1-4 show a working schematic and the detailed equipment drawings of the reaction furnace burner and vessel.

Waste Heat Exchanger

The waste heat exchanger initiates the first stage of process gas cooling in the system. The unit is close coupled to the outlet nozzle of the reaction furnace (mounted in a vertical position) and cools the effluent from the furnace temperature to approximately 900°F. A standard shell and tube design is employed, with four ½-inch tubes providing the necessary heat transfer area.

The inlet nozzle contains a pseudo ceramic checker wall for reflecting radiant heat back towards the combustion chamber, also ceramic ferrules at the tube inlets provide additional tube sheet weld protection from the high operating temperatures. The cooling water flows on the shell side of the exchanger. The cooling water is supplied at 60°F and the flow rate is controlled through a manual valve located on the shell side outlet nozzle and fine tuned by observation of a local return water temperature indicator.

Figure 3.2.1-5 shows a detailed equipment drawing of the wasteheat exchanger vessel.

Sulfur Condenser and Collector

After the process gas exits the wasteheat exchanger it passes through a short section of 1 inch tubing, and then enters the sulfur condenser. This vessel is designed to cool the process gas to a temperature less than the sulfur condensation (dewpoint) to condense the sulfur vapor and separate the liquid sulfur from the gas phase onto the tube surface, and gravity drain the sulfur for collection. This vessel is mounted one percent off horizontal, sloping downward from inlet to outlet and is also of tube and shell design. The process gas flows on the tube side and four ½-inch tubes provide heat exchange area. An enlarged disengagement zone and mist extractor were built into the outlet end of the condenser to remove any entrained sulfur from the exiting vapor. The produced liquid sulfur is collected and drained into a pressurized sulfur collection vessel. Cooling water flows on the shell side and acts as the cooling medium. The temperature control on this unit is similar to the temperature control on the waste heat exchanger.

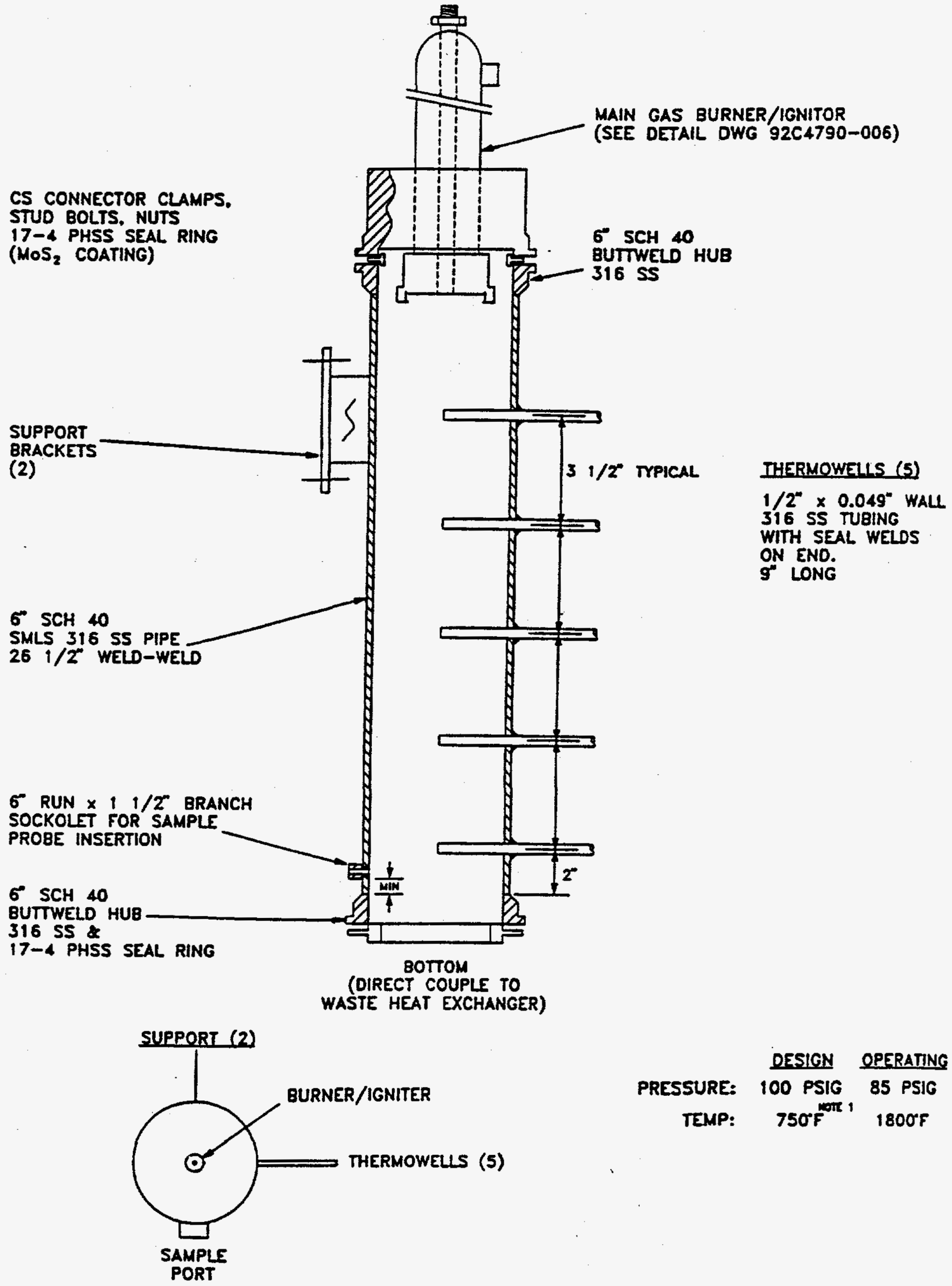
Both the waste heat exchanger and the sulfur condenser are equipped with removable flanged end caps to allow for tube cleaning and inspection.

The condensed sulfur is drained from the disengagement section through a heated 1-inch flexible stainless steel line and into the sulfur collection vessel. A full 1-inch piping cross is provided at a direction transition to allow for rodding of lines. Directly below the cross the sulfur drains into the collection vessel at pressure. This vessel (V-2) has capacity (nominal 6 gallons) to provide for up to six hours worth of sulfur production at design rates. It stands on a load cell transducer (100 lbs. maximum) which instantaneously measures the sulfur production throughout the run. A ½-inch balance line provides means for the gas to vent to the pressure control module.

After a test run is completed and all sulfur produced is recovered, the collection vessel is isolated from the system through hand valves and the liquid sulfur drained under a nitrogen pressure head to a suitable container. Local pressure indication and relief are provided on the vessel for safety.

The vapor exiting from the sulfur condenser passes through a heated ½-inch line with a high point and into a surge vessel/knock out drum (V-1) by means of a dip tube. This vessel has several purposes; first it serves to knock out any residual entrained sulfur in the vapor and drains it to the sulfur collection, second it serves as a pressure balance vessel for the sulfur collection vessel, and third it dampens any flow surges to the downstream system pressure control loop. This vessel as well as all sulfur lines are heat traced (260°F) and insulated to prevent sulfur solidification and plugging. This portion of the system was modified extensively to ensure that there was no plugging of the lines with elemental sulfur. Figures 3.2.1-6 and 3.2.1-7 show the sulfur condenser and sulfur knock-out / collection vessels respectively.

Figure 3.2.1-3
Reaction Furnace

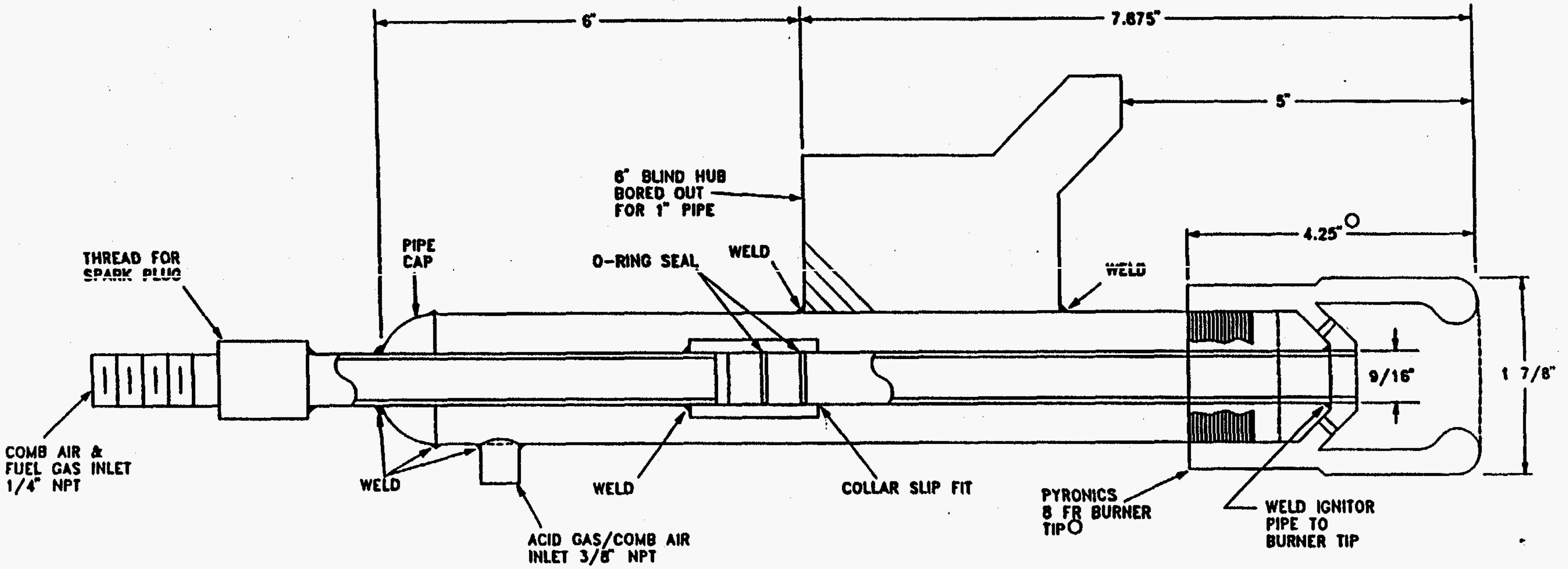


	DESIGN	OPERATING
PRESSURE:	100 PSIG	85 PSIG
TEMP:	750°F NOTE 1	1800°F

TE:

1) CERAMIC/REFRACTORY LINING TO ALLOW FOR 750°F DESIGN WALL TEMPERATURE
2) APPROXIMATE TOTAL WEIGHT IS 180 lbs (EMPTY)

Figure 3.2.1-4
Reaction Furnace Burner



<u>MATLS</u>

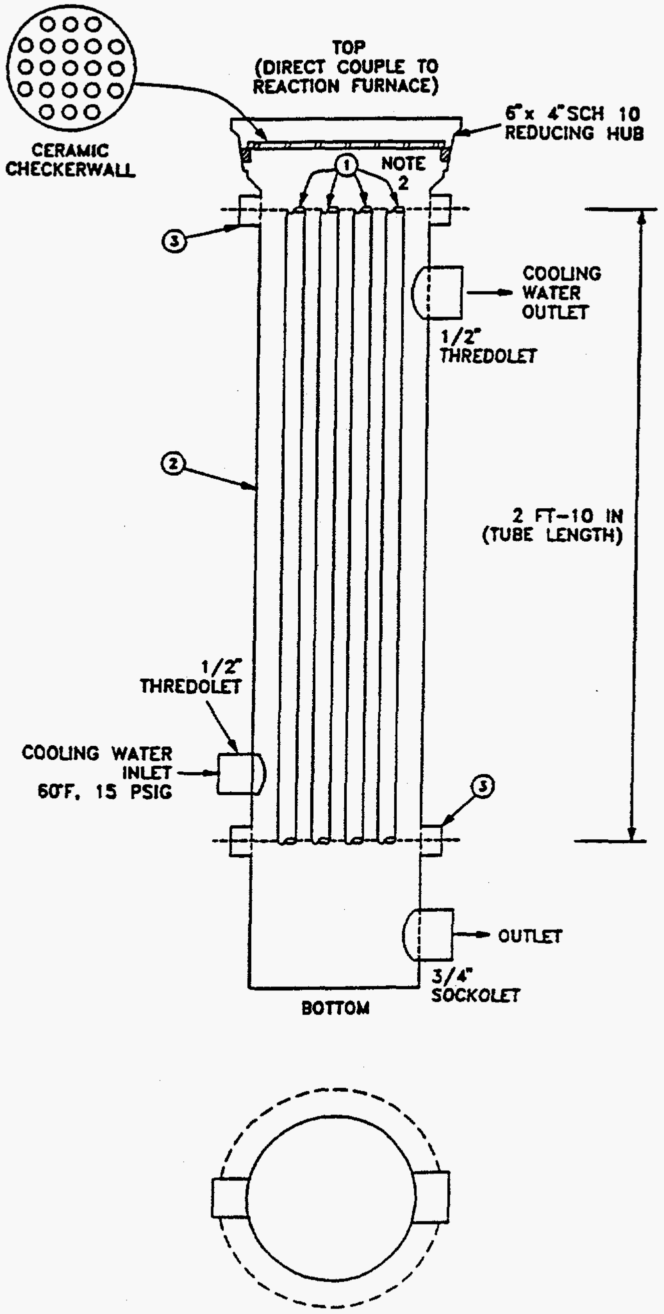
PIPE: 316 SS, OUTER: 1" SCH 10, INNER: 1/4" SCH 10
BURNER TIP: 446 SS
O-RINGS: VITON
HUBS: 316 SS
SEAL RINGS: 17-4 PHSS WITH MoS_2 COATING

<u>NOTE:</u>

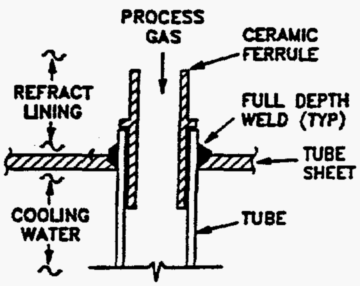
2) CLEARANCE FOR WRENCH FLATS ON THREADED BURNER TIP REQUIRED
1) 6 PORTS STANDARD - DRILL 3 ADDITIONAL #45 MTD (0.082") PORTS EQUALLY SPACED

Figure 3.2.1-5
Wasteheat Exchanger

ITEM	QUAN	DESCRIPTION
①	4	1/2"-16 BWG TUBES (0.065" WALL) 316 SS
②	1	4" SCH 10, 316 SS, SHELL
③	4	4"-150# S.O. FLANGE, 316 SS
④	1	6"x4" SCH 10 REDUCING HUB, 316 SS (OTECO TYPE)



FERRULE DETAIL



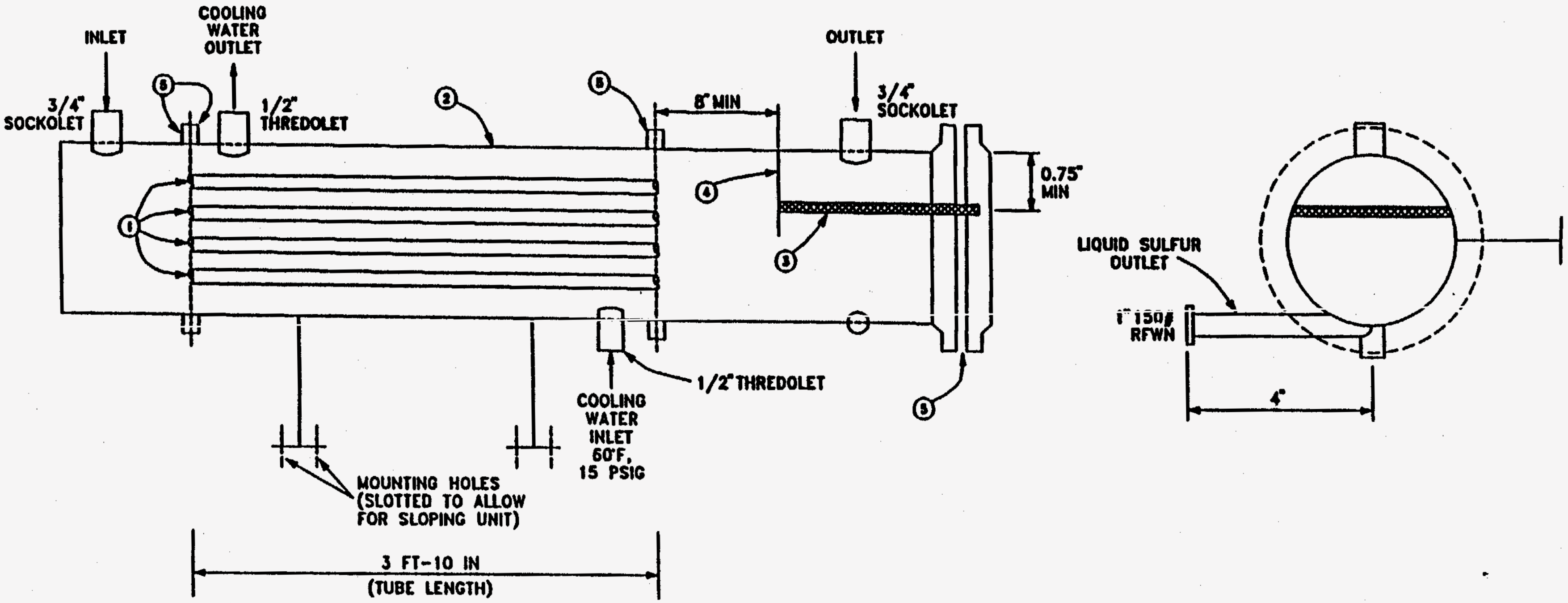
DESIGN

TUBES: 125 PSIG @ 2000°F
SHELL: 100 PSIG @ 750°F

NOTE:

3) SHELL BAFFLES AS REQUIRED
2) TUBE INLETS WILL REQUIRE REFRACTORY LINING AND CERAMIC FERRULES FOR HIGH TEMP. PROTECTION (SEE DETAIL)
1) UNIT WILL BE INSTALLED VERTICALLY

Figure 3.2.1-6
Condenser



ITEM	QUAN	DESCRIPTION
①	4	1/2"-16 BWG TUBES (0.065" WALL THKNESS), 316 SS
②	1	4" SCH 10, 316 SS, SHELL (ASME SECT I)(4.26" I.D.)
③	1	YORK STYLE 326 OR EQUIVALENT, 316 SS (REMOVABLE)(98% VOID)(1/2" SURFACE AREA MINIMUM)
④	1	IMPINGEMENT BAFFLE
⑤	6	4"-150# S.O. FLANGE, 316 SS

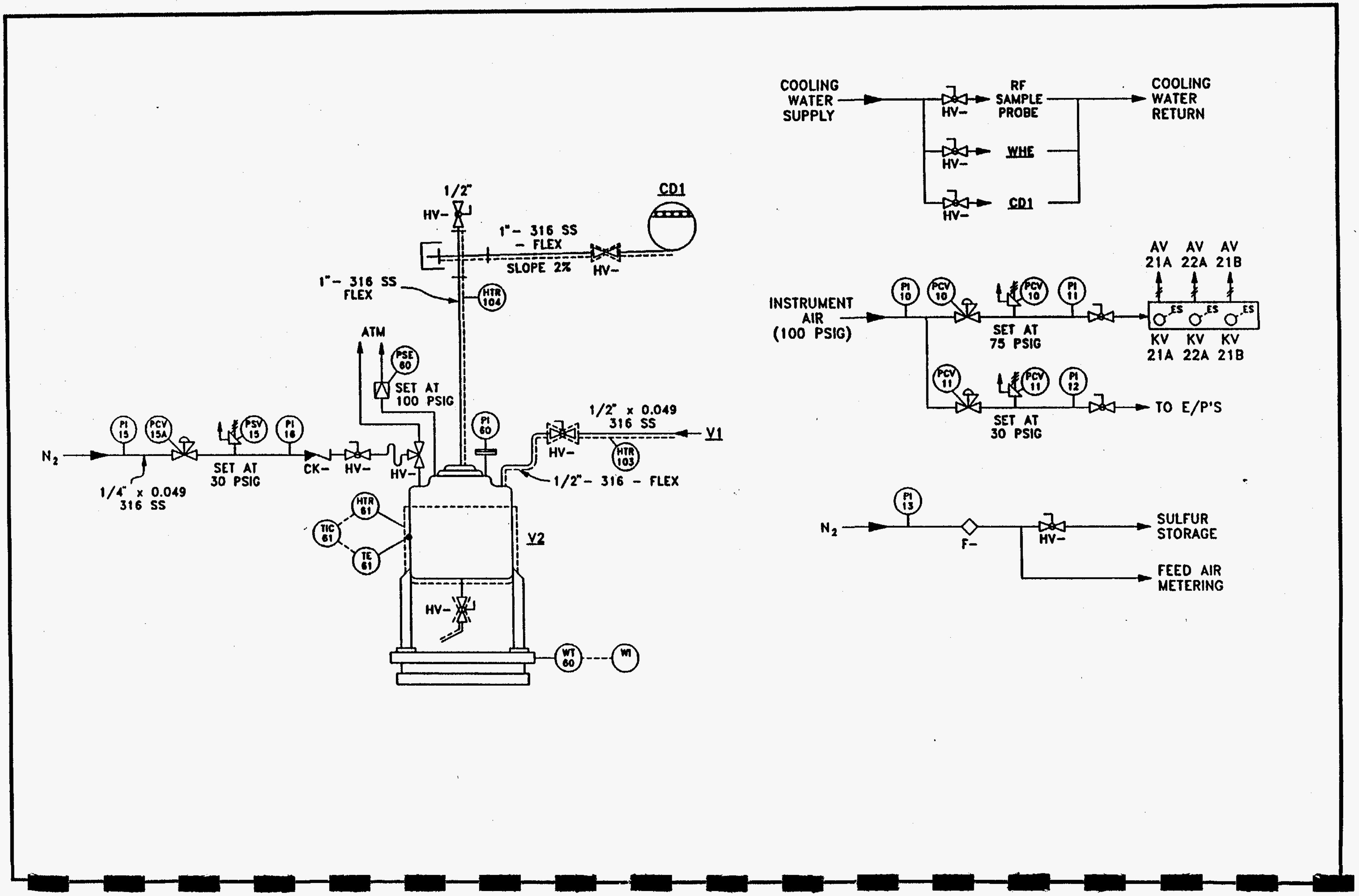
DESIGN

TUBES: 125 PSIG @ 1000°F
SHELL: 100 PSIG @ 500°F

NOTE:

3) SHELL BAFFLES AS NECESSARY
2) GENERAL ORIENTATION OF UNIT IS HORIZONTAL
1) SLOPE UNIT 1% INLET TO OUTLET WITH MOUNTING BRACKETS

Figure 3.2.1-7
Sulfur Knock-Out and Collector



3.2.2 Monitoring Equipment Description

The fundamental issues to be addressed in the pilot plant program were related to the H_2S oxidation reaction chemistry. Therefore, the data gathering requirements were related primarily to accurate measurement of the temperature, pressure, flow rate and composition of the process streams in question. The data gathered during the test runs were expected to be similar to data typically generated from the operation of a traditional modified Claus process. Therefore, the monitoring systems used in the CNG Claus pilot plant were patterned after conventional data monitoring systems.

Pressure and Temperature Monitoring Systems

One of the primary objectives of the pilot plant project was to operate the H_2S oxidation reactions at somewhat elevated pressures as compared to the conventional modified Claus process. In the modified Claus process the typical operating pressures in the system are 15 to 25 psia. The test program for the CNG Claus project called for system pressures up to 100 psia.

The pressures expected for the test runs fall well within the pressures which can be adequately handled by conventional pressure measuring devices and the pilot plant was well equipped with continuous pressure measurement at key process locations.

Similarly, the reaction furnace temperatures were expected to be consistent with conventional Claus furnace conditions. However, extensive experience with conventional Claus furnaces has shown that standard in situ thermocouples do not have acceptable longevity. Therefore, non-invasive (infrared) types of temperature detection have become the norm. This was considered for the CNG system however, space and size limitations precluded the use of these devices.

In response to this, a modified air purged thermocouple system was installed in the critical flame zone of the reaction furnace. These devices gave good performance during the test runs. The P&I diagrams in Section 3.2.1 indicate the location and type of pressure and temperature monitoring devices installed on the CNG Claus pilot plant.

Gas Analysis Equipment

The other primary required data from the pilot plant was the composition of the process gas streams at the various points in the process. This data allowed for completion of the detailed heat and material balances which were required for evaluating all of the performance parameters in the system.

The modeling work conducted in the earlier phases of this project indicated that the expected process gas compositions would be consistent with those often observed in traditional modified Claus processes. Therefore, the gas analysis equipment and methodologies were adopted directly from the existing procedures used in sour gas systems.

Of primary interest was the accurate and reliable measurement of the sulfur bearing components expected in the system; H_2S, SO_2, COS and CS_2. In order to complete rigorous material balances on this system however, additional compounds were also measured: N_2, Ar, O_2, CO_2, CO, H_2 and an array of light hydrocarbon compounds. For this application, gas chromatography was the analytical system of choice. A full description of the analytical methods employed for this work are included in Appendix A.

3.2.3 Commissioning and Operation

The pilot plant was designed to be assembled as a transportable skid mounted unit. After fabrication of the major components the pilot plant was assembled at a fabrication shop in Houston, Texas. The fully assembled unit underwent extensive functionality testing in the shop under controlled conditions.

After initial functionality testing in the shop, the unit was shipped to the pilot plant operations site.

Test Site and Site Selection

During the Phase 2 work a review of possible test sites was completed. The primary criterion for selecting the site were as follows:

1. Existing experience with handling sour gas processes.
2. Available utilities (power, cooling water).
3. Capability to allow for (via an existing flare system) the disposal of the tail gas waste stream.
4. Reasonable access to the support trades (welding, electricians, etc.)

During the initial Phase 2(5) portion of the program, an extensive site selection procedure was used to select an acceptable site. The final choice for the pilot plant site was an existing CNG Producing oil and gas processing plant in Davis, Oklahoma. The site met all of the designated criteria and also had the advantage of already dealing with sour gas streams. A full description of the site selection process and all of the associated operating considerations is dealt with in the National Environmental Policy Act (NEPA) documentation provided in the Phase 2 report(5).

Photographs and maps showing the pilot plant site are included in Appendix D. Additional photographs of the pilot plant equipment are presented in Appendix E.

Pilot Plant Installation

After siting the pilot plant unit, all of the plant support facilities were also sited and connected to the pilot plant. These facilities included:

- feed gas supply; liquid H_2S, liquid CO_2, compressed O_2, N_2 and methane.
- exchanger cooling water system
- electrical supply

Prior to starting the full Claus test runs, the plant was started up and run on fuel gas (methane). This was done to allow for complete functionality tests on all of the major and associated equipment. The unit was operated with a fuel gas combustion in order to ensure proper operation of the following systems:

- feed gas delivery systems
- plant pressure control system
- furnace and heat exchanger temperature measurement systems
- sample ports and probes
- exchanger cooling systems
- tail gas disposal to the flare

These initial test runs revealed many problems with the unit which had to be rectified prior to operating the unit with H_2S. Most of these problems were related to excessive heat losses in the vessels and the associated problems with maintaining proper temperature profiles through the process units. There were also significant problems with excessive system pressure drop in the feed gas delivery systems. Various minor field modifications were made on the unit to overcome these problems. The P&ID diagrams presented in Section 3.2.1 represent final configuration of the pilot plant as it entered the test phase.

4.0 Test Program

4.1 Experimental Test Plan and Test Matrix

The fundamental questions to be answered by the experimental program were whether a viable pilot scale plant could be designed, built and operated successfully and whether the Claus H_2S oxidation reactions would proceed to a sufficient degree under the proposed operating conditions.

The pilot plant design was taken largely from existing industrial technology and as such no significant technical barriers were anticipated. The final plant design utilized completely conventional unit equipment, properly scaled for the proposed feed gas flow rates.

For the experimental test plan, a full review of conventional modified Claus chemistry was used as a base line reference. It is well known from the operation of conventional Claus Process Reaction Furnaces, at or near one atmosphere of pressure (15 psia), that one-third of the H_2S can be oxidized within 1 to 2 seconds at temperatures above 1700°F with acceptable yields according to the H_2S oxidation reaction:

$$2\,H_2S + O_2 \leftrightharpoons S_2 + H_2O$$

This highly exothermic reaction is fully sustainable and controllable in a free flame reactor.

However, it is also known that although all the O_2 is consumed in the reaction furnace, the equilibrium yield of sulfur is not always achieved, particularly at lower temperatures. This indicates that the reaction is to some extent, kinetically limited, usually as a result of the mechanical aspects of the furnace design (mixing and residence time).

Since the proposed strategy for the CNG Claus process is to oxidize only one-sixth of the H_2S at a higher system pressure (*ca.* 100 psia or 6 atmospheres) and a lower temperature (*ca.* 1500 to 2000°F) the experiments were designed to determine if, indeed, the proposed strategy was practicable.

The reaction furnace operation has been devised to allow for stable operation while meeting the following criteria to ensure that a conventional furnace design (metallurgy etc) is adequate and that the downstream tail gas treating section can adequately handle the hydrogenation of SO_2 in a conventional manner.

1. The adiabatic reaction temperature must be maintained at or below 2500°F.
2. The overall conversion efficiency must be maintained at 50+ percent.
3. The SO_2 in the furnace outlet must be maintained at less than 2.0 mole percent.
4. The system pressure would be at approximately 100 psia.

Critical Operating Variables

The critical operating parameters are related to two well-defined concepts; the chemical equilibria of the reaction system and the kinetic effects of the mechanical system. For the pilot plant test program it was necessary to determine the impact of these parameters on the key reaction.

Chemical Equilibrium Effects

The chemical equilibrium of the reaction system is affected and determined by :
- reaction pressure
- reaction temperature
- reactant composition

The reaction pressure was determined by the system pressure as defined in the process design. While it may be possible to determine the effect of reaction pressure on the reaction equilibrium, this parameter has not been identified as critical to the successful operation of the process.

The pilot plant was designed for adiabatic operation in the reaction furnace (while heat losses to some extent were fully expected). Therefore the reaction temperature is set by the reactant composition and the extent of reaction of those reactants. This leaves the reactant composition as the most significant process variable and was the focus of the pilot plant test plan. The information in the Test Plan Matrix discusses the composition variables which will be evaluated and presents the results from an array of simulated reaction runs which were completed using an equilibrium model based on a Gibbs free energy calculation technique.

Kinetic Reaction Effects

The reaction equilibrium can also be significantly affected by kinetic effects in the thermal reactor. The kinetics of the reaction system can be affected by the following physical conditions:
- reaction residence time
- reaction temperature
- extent of mixing in the reaction section

It is assumed that the kinetic effects could have a measurable impact on the outcome of the reaction. It is expected that the effect of some kinetic limitation on the H_2S oxidation will not significantly affect the overall feasibility of the process, however, the test plan included some studies to determine the kinetics of the new operating conditions.

The furnace design allowed for a maximum residence time of 4 seconds which is significantly longer than in conventional Claus furnace design. The design also allows for decreasing the residence time substantially by altering the reactant flow rates which would allow for some evaluation of residence time on the overall reaction. The mixing characteristics of the burner were identical for all of the tests, therefore it was possible to determine the effects of mixing and turbulence on the reaction.

Pilot Plant Test Matrix

The work completed in the original technical feasibility study(6) was used to evaluate the appropriate test variable matrix for the pilot plant tests. Those simulation results define the range of potential reactant compositions.

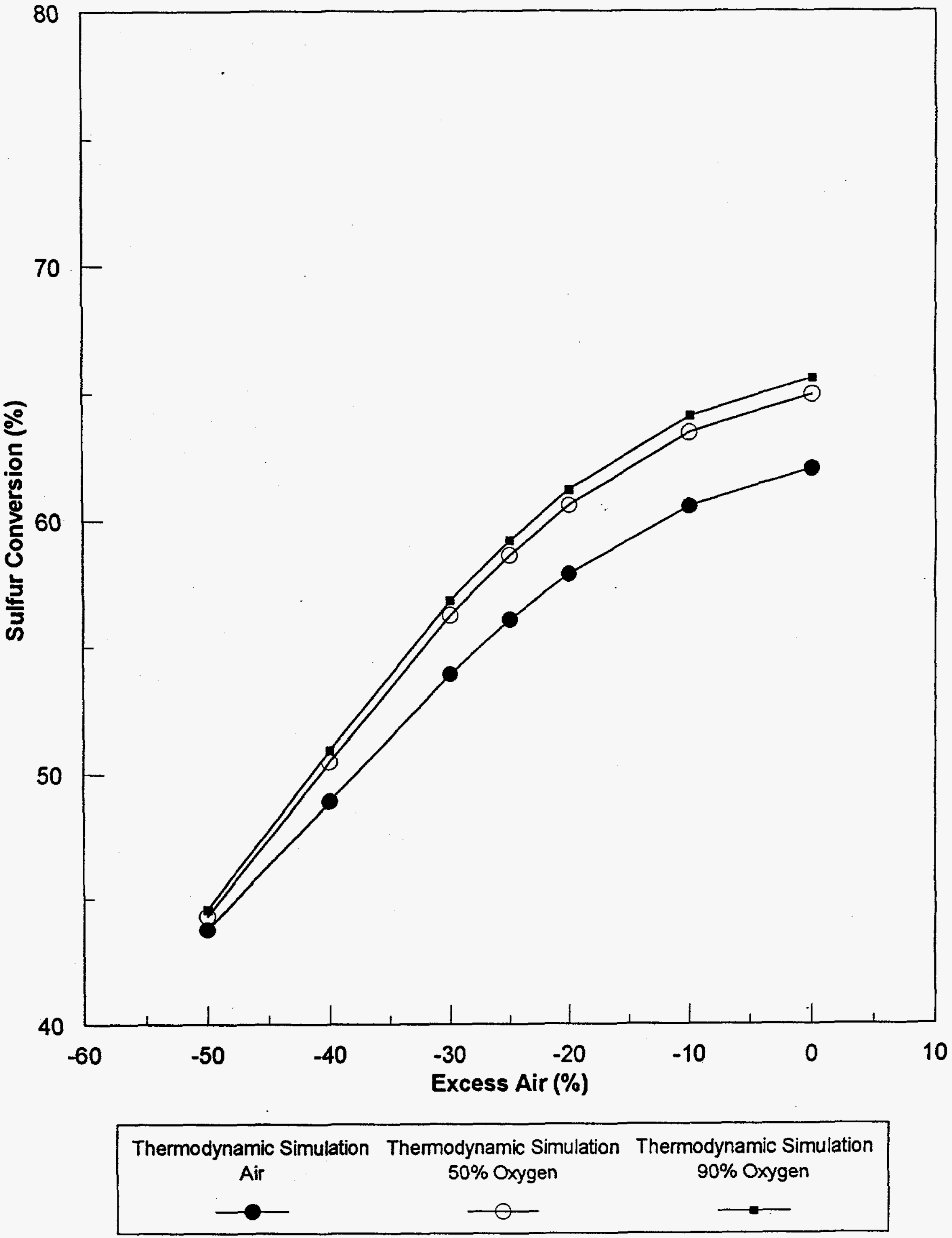
The most significant reactant components are H_2S and O_2. There will also be CO_2 and N_2 in the feed gas which may have an effect on the reaction equilibrium. The original test matrix allowed for "acid gas" feed compositions varying in H_2S content from 50 to 90 mole percent. The balance of the acid gas is CO_2. The test matrix also allowed for "air" compositions varying in O_2 content between 21 and 90 mole percent. The balance of the combustion air was N_2.

Table 4.1-1 on the following pages summarizes the extensive set of equilibrium calculations completed for a range of inlet reactant compositions. This simulation matrix encompasses the full range of potential operating conditions proposed for the acid gas and air compositions for the experimental test runs. Figures 4.1-1 through 4.1-6 illustrate in graphical form the range of operating conditions and expected results for the most critical performance parameters.

Table 4.1-1
Experimental Test Plan - Original Simulation Results Summary

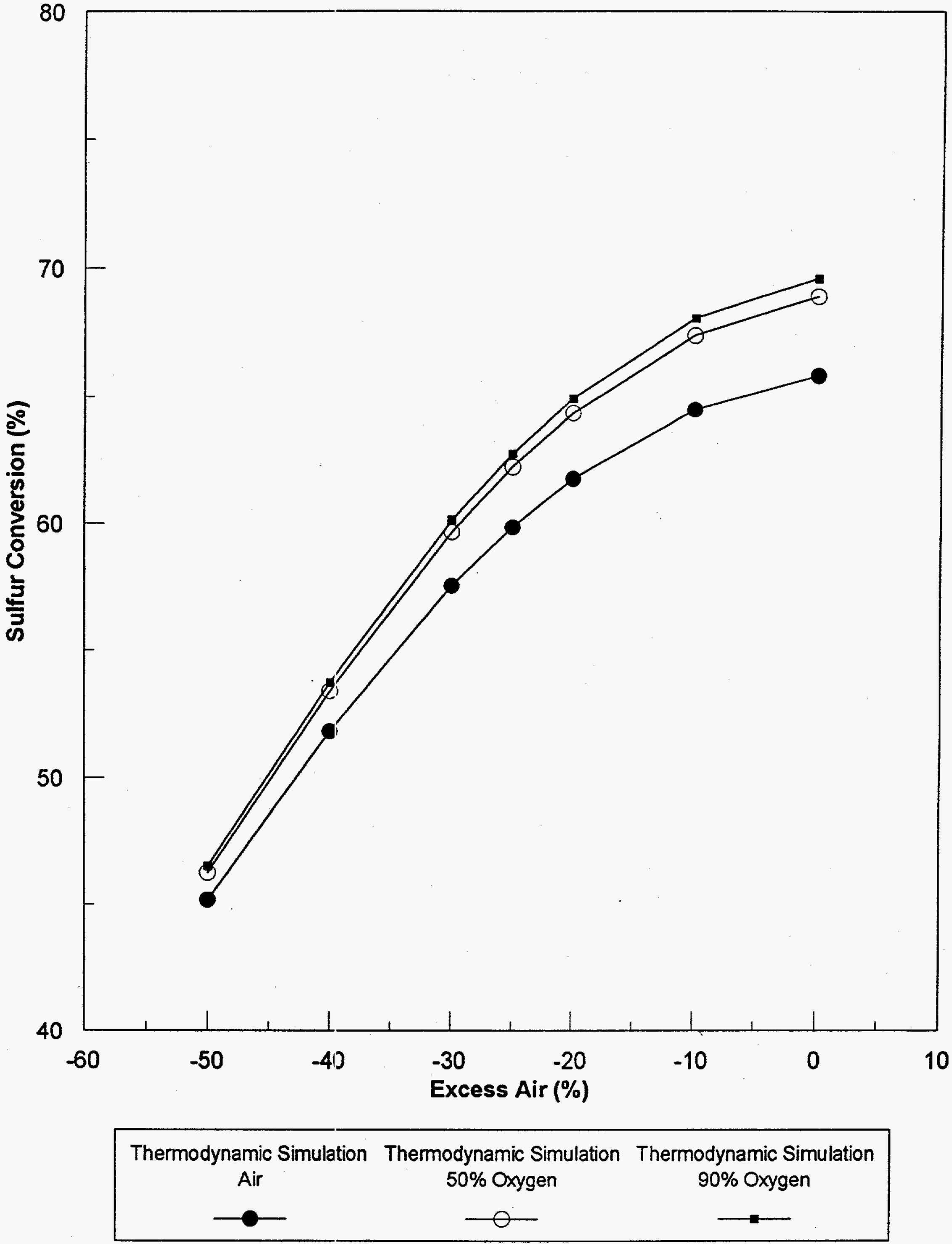
Acid Gas Composition H_2S (mole %)	Air Composition O_2 (mole %)	H_2S / O_2 Flow Ratio	Excess Air (%)	H_2S Equilibrium Conversion (%)	Reaction Temp. (°F)	SO_2 Residual (mole %)
50	21	2.11	0	62.0	1719	3.21
50	21	2.77	-25	56.1	1498	1.79
50	21	4.06	-50	43.8	1241	0.70
50	50	2.28	0	64.9	1987	4.53
50	50	2.92	-25	58.6	1710	2.20
50	50	4.14	-50	44.3	1382	0.84
50	90	2.36	0	65.6	2070	5.26
50	90	3.00	-25	59.2	1776	2.48
50	90	4.19	-50	44.6	1432	0.88
70	21	2.16	0	65.8	1975	3.45
70	21	2.84	-25	59.8	1743	1.77
70	21	4.14	-50	45.2	1456	0.68
70	50	2.40	0	68.9	2335	5.30
70	50	3.10	-25	62.2	2009	2.33
70	50	4.38	-50	46.2	1649	0.75
70	90	2.51	0	69.6	2454	6.42
70	90	3.22	-25	62.7	2091	2.67
70	90	4.49	-50	46.5	1708	0.80
90	21	2.18	0	68.7	2195	3.53
90	21	2.89	-25	62.4	1945	1.68
90	21	4.24	-50	46.4	1642	0.57
90	50	2.45	0	72.6	2714	5.66
90	50	3.21	-25	65.2	2305	2.23
90	50	4.59	-50	47.4	1883	0.62
90	90	2.58	0	73.7	2905	7.05
90	90	3.36	-25	65.9	2424	2.66
90	90	4.74	-50	47.7	1956	0.66

Figure 4.1-1
Test Plan Simulation Study
Sulfur Conversion vs. Reaction Stoichiometry



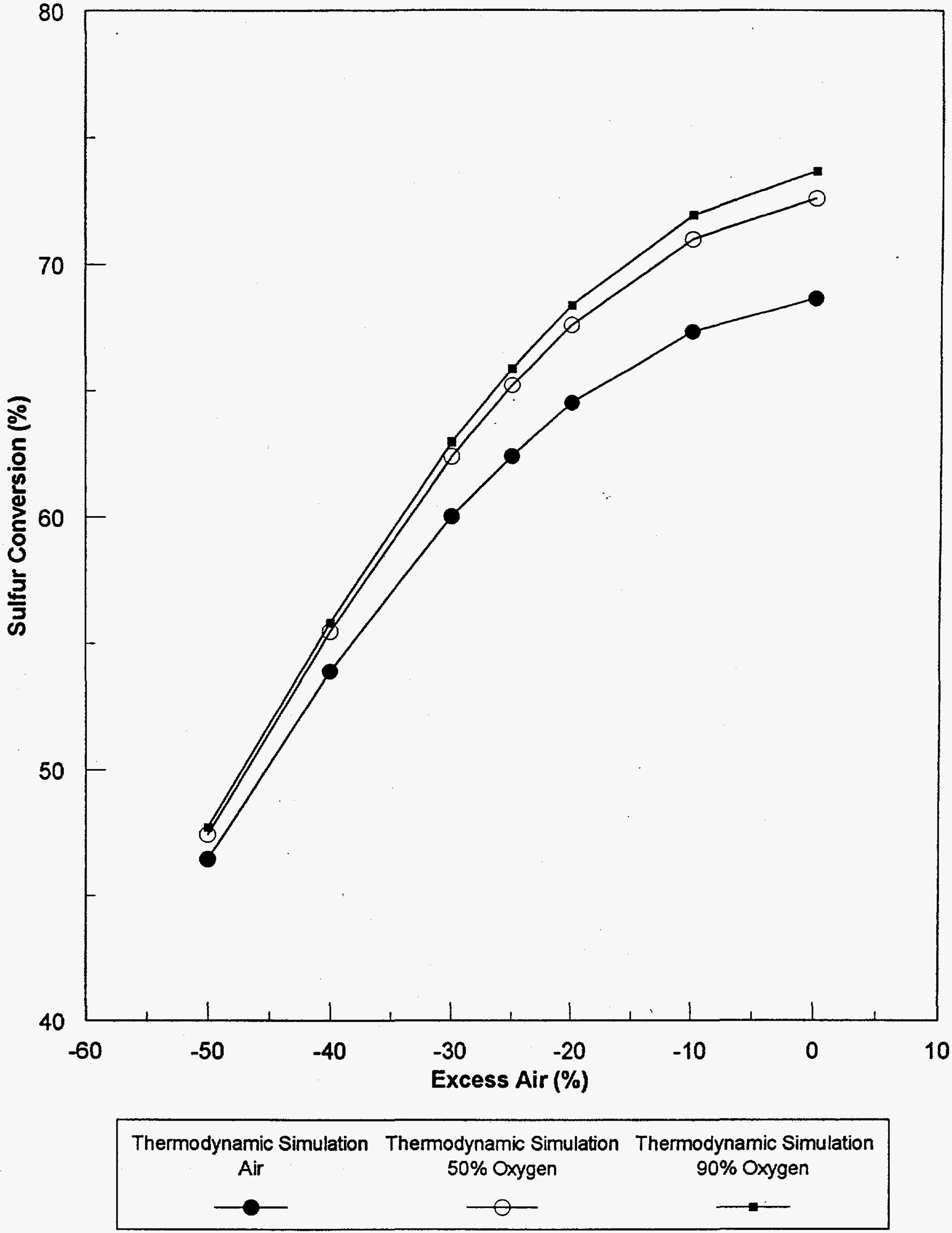
1. Feed gas : 50% H2S
2. Various "Air" Compositions

Figure 4.1-2
Test Plan Simulation Study
Sulfur Conversion vs. Reaction Stoichiometry



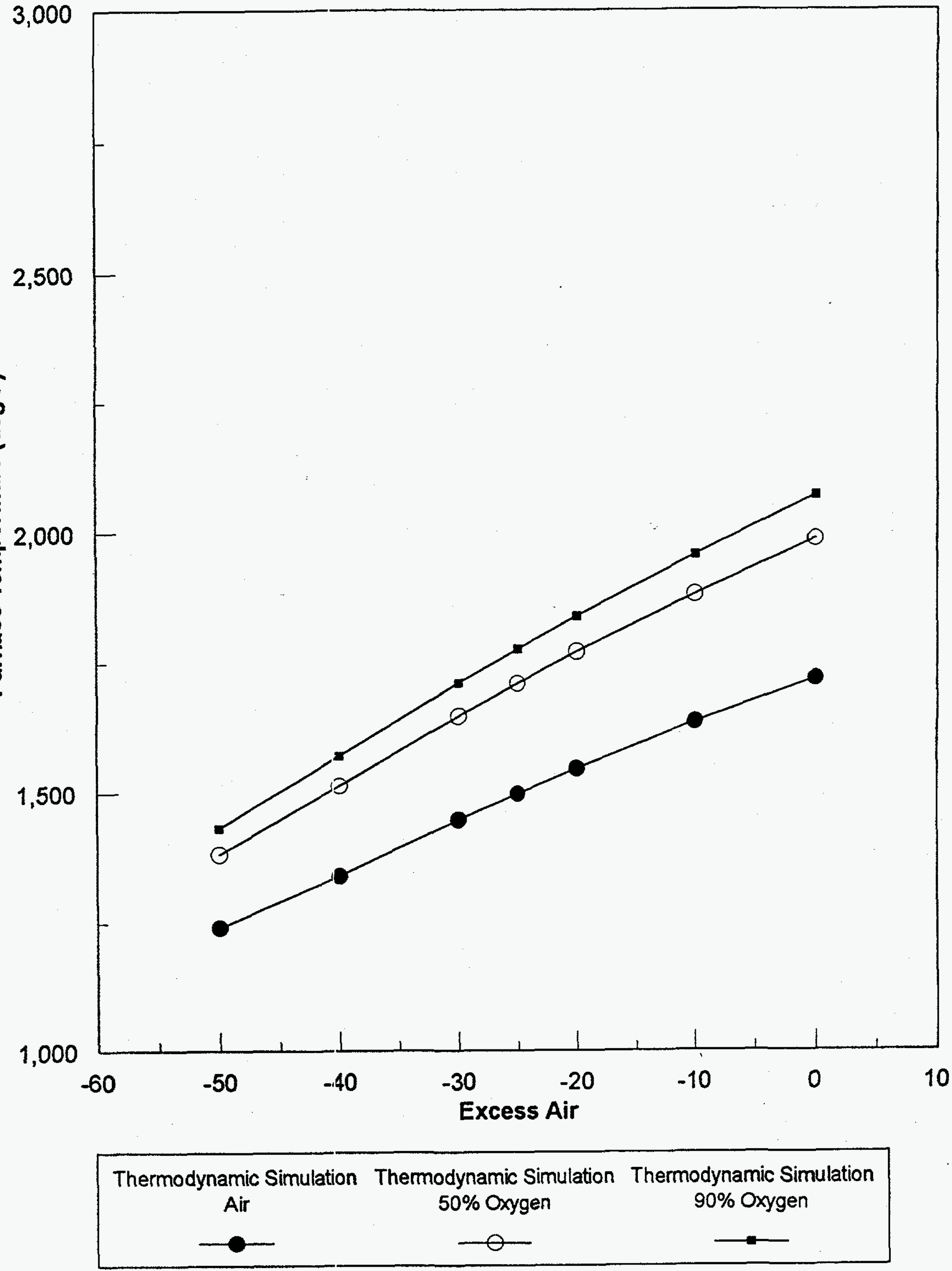
1. Feed gas : 70% H2S
2. Various "Air" Compositions

Figure 4.1-3
Test Plan Simulation Study
Sulfur Conversion vs. Reaction Stoichiometry



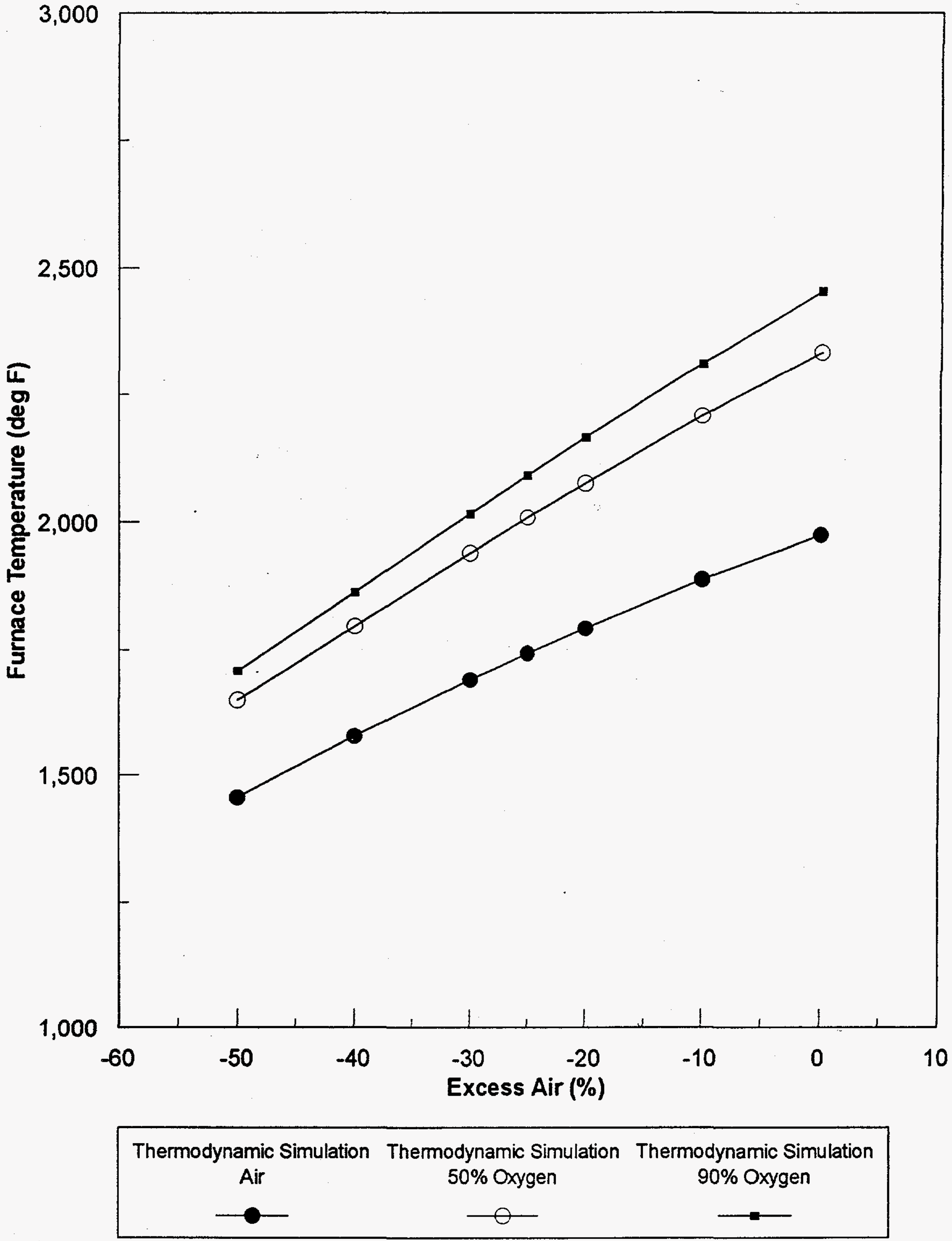
1. Feed gas : 90% H2S
2. Various "Air" Compositions

Figure 4.1-4
Test Plan Simulation Summary
Reaction Furnace Temperature vs. Reaction Stoichiometry



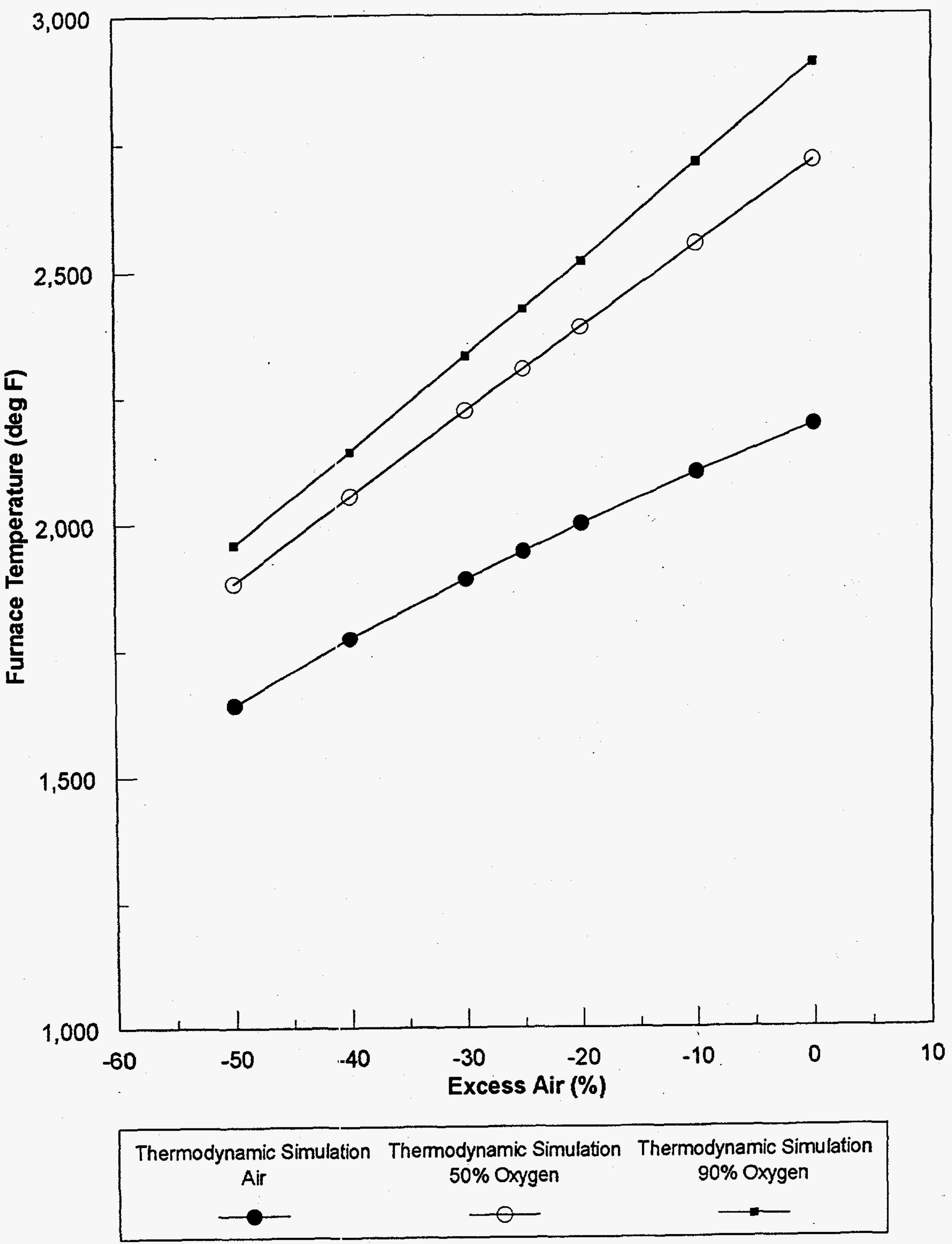
1. Feed gas : 50% H_2S
2. Various "Air" Compostions

Figure 4.1-5
Test Plan Simulation Summary
Reaction Furnace Temperature vs. Reaction Stoichiometry



1. Feed gas : 70% H2S
2. Various "Air" Compostions

Figure 4.1-6
Test Plan Simulation Summary
Reaction Furnace Temperature vs. Reaction Stoichiometry



1. Feed gas : 90% H2S
2. Various "Air" Compositions

Test Run Procedure

Chemical Equilibrium Tests

The pilot unit will be started up using sufficient O_2 to oxidize between one-third and one-half of the H_2S with a target residence time in the reaction furnace of 2 seconds. The temperature will be monitored to ensure that the Reaction furnace does not become overheated. If necessary, the flow rates of the Acid Gas and O_2/Air Feeds, and/or their ratio, will be adjusted to prevent overheating. When stable operation is achieved, a routine sample set will be taken and analyzed to confirm the performance of the Reaction furnace.

Once the operability of the pilot unit is established, the ratio of O_2/Air Feed to Acid Gas Feed will be adjusted, step wise, towards oxidation of one-sixth of the H_2S while monitoring the temperature and maintaining the residence time in the Reaction furnace between 2 and 3 seconds. At each step, the unit will be allowed to stabilize (between 20 to 30 minutes should be sufficient) as indicated by stable Reaction furnace temperature(s) and a routine sample set will be taken and analyzed. When the target of one-sixth oxidation of the H_2S is reached (increasing the residence time if necessary),duplicate routine sample sets will be taken.

In addition, for each routine sample set, a set of quenched samples directly from the furnace will be taken and analyzed. The analytical data from the routine sample set will be used to evaluate overall performance while the analytical data from the quenched samples set will be used for comparison with equilibrium-calculation results to determine the degree of approach to equilibrium in the Reaction furnace.

Within the range of operating variables described above, tests will be carried out at the maximum and minimum attainable CO_2/H_2S ratios for the Acid Gas and the maximum and minimum attainable O_2/N_2 ratios for the "Air" Feed Streams. For these tests, the "Air" to Acid Gas ratio will be for one-sixth oxidation of the H_2S and the residence time in the Reaction furnace will be that which in prior tests achieved the closest approach to equilibrium. Both a routine sample set and a quenched samples set will be taken and analyzed for each condition.

Kinetic Reaction Tests

Assuming that the feasibility and practicability of the fundamental operating strategy are established by the foregoing, the "kinetics" of the pilot unit equipment will be characterized by the following series of tests.

Holding the O_2/Air to Acid Gas Feed ratio constant at one-sixth oxidation of the H_2S, the residence time in the Reaction furnace will be varied from 1 to 4 seconds in one-second steps. Routine sample sets and quenched samples sets will be taken and analyzed at each step. If equilibrium is approached at 1 second residence time, a test will be run at 0.5 seconds residence time.

Holding the residence time constant at the value indicated by the foregoing as sufficient to reach equilibrium, the O_2/Air to Acid Gas Feed ratio will be reduced step wise to determine the practical lower limit of operation as indicated by analytical results, the Reaction furnace temperature and stability of operation. Routine sample sets and quenched samples sets will be taken and analyzed at each step.

This set of tests will be repeated at **twice** the residence time indicated as sufficient to reach equilibrium at one-sixth oxidation of the H_2S.

4.2 Test Results - Data, Analysis and Methods

As described earlier, the actual number of test runs completed was significantly less than described in the original test run matrix due to a major mechanical failure in one of the primary units. Fortunately, the small number of test runs completed occurred with a set of operating conditions that covered a significant number of the fundamental parameters. More importantly, the test runs which were completed clearly demonstrated the critical performance of the reaction system.

4.2.1 Field Data

Operating Conditions

The test runs were completed under the following primary conditions:

- Acid Gas with ~90 percent H_2S only.
- Combustion air with ~21 percent O_2 only.
- Excess air ranging between 21 and 55 percent of "normal" modified Claus stoichiometry.
- Furnace residence times ranging between 1.3 and 3.6 seconds.
- Furnace temperatures ranging between 1620 and 1870°F.

This clearly represented only a small fraction of the originally planned test runs. However, after the failure of the wasteheat exchange unit (which would require substantial repair time and costs) a review of the data gathered to that point was completed. This review indicated that the runs completed to that point had substantially demonstrated many of the critical performance parameters and therefore, a decision was made to discontinue further testing pending completion of the full report and a determination whether testing was warranted. Table 4.2-1 on the following page summarizes the primary operating data for the completed test runs.

Process Gas Analysis Results

For each test run set, a detailed set of gas analyses were completed on each of the following sample locations:

- Acid gas furnace feed (combined H_2S and CO_2 feed streams)
- Reaction furnace product
- Wasteheat exchanger process gas

In essence, the reaction furnace products and the wasteheat exchanger process gas are normally considered to be of the same composition. However, experience with industrial modified Claus furnaces has shown that often there is a change in gas composition between these two points due to continued reaction in the exchanger unit. The analysis of the actual reaction products in the furnace was of primary interest with regard to determining the performance of the reaction system. However, the analysis of the wasteheat exchanger process gas allowed for direct measurement of the gas which would be treated further in the tail gas treating section of the CNG Claus process. Analysis of both of these streams allowed for a complete review of the "thermal stage" reaction system. A complete set of gas analysis results is presented in Appendix A.

Table 4.2-1
Pilot Plant Test Data - Session 1

Test			1	2a	2b	3	4
Ambient	Temperature	(°F)	52	52	52	52	52
	Pressure	(psia)	14.3	14.3	14.3	14.3	14.3
Acid Gas	Flow	(lbmol/h)	0.583	0.695	0.695	0.600	0.600
	Flow	(scfm)	3.68	4.386	4.439	3.782	4.810
	Temperature	(°F)	60	60	60	60	60
	Pressure	(psia)	76.3	76.3	76.3	76.3	76.3
	[H_2S]	(mole %)	89.4	89.4	89.4	89.4	89.4
Air	Flow	(lbmol/h)	0.851	1.210	1.192	0.658	0.579
	Flow	(scfm)	5.388	7.661	7.547	4.166	3.666
	Temperature	(°F)	60	60	60	60	60
	Pressure	(psia)	114.3	114.3	114.3	114.3	114.3
	[O_2]	(mole %)	20.81	20.81	20.81	20.81	20.81
Furnace	Flow	(lbmol/h)	1.273	1.672	1.680	1.134	1.091
	Flow	(scfm)	8.060	10.587	10.637	7.180	6.908
	Temperature	(°F)	1570	1644	1873	1618	1620
	Temperature	(°F)	---	1651	1857	1583	---
	Temperature	(°F)	---	1521	1709	1454	---
	Pressure	(psia)	28.8	31.3	63.3	54.3	49.8
Wasteheat Exc.	Temperature	(°F)	541	541	632	555	555
	Pressure	(psia)	19.8	21.3	58.8	50.3	45.3
Condenser	Temperature	(°F)	275	275	289	284	284
	Pressure	(psia)	16.3	16.3	55.3	48.3	44.3

4.3 Data Analysis and Results

4.3.1 Material Balances

The primary method for compiling and reviewing the test run data was to construct detailed multi-component material and heat balances. Compilation of these balances for each and every sample set allow for direct calculation of the following performance parameters:

- H_2S conversion efficiency (reaction yield) and approach to equilibrium
- Reaction stoichiometry (extent of "deficient" oxygen in the reaction system)
- Adiabatic reaction temperature
- Evaluation of the extent of side reactions in the system

Table 4.3-1 illustrates a typical material balance for one of the test run sets. Full material balances for all of the test run sets are presented in Appendix B-1.

Table 4.3-1
Material Balance - Test 1

Process Location Sample No.	**Acid Gas 15**	**Air**	**Reaction Furnace 07**	**Tail Gas 08**
H_2	0.0000	0.0000	0.0259	0.0155
Ar	0.0001	0.0079	0.0080	0.0078
O_2	0.0000	0.1771	0.0030	0.0002
N_2	0.0065	0.6604	0.6666	0.6549
CH_4	0.0000	0.0000	0.0000	0.0000
CO	0.0000	0.0000	0.0022	0.0014
CO_2	0.0530	0.0003	0.0518	0.0539
H_2S	0.5213	0.0000	0.1590	0.1863
COS	trace	0.0000	0.0014	0.0015
SO_2	0.0001	0.0000	0.0021	0.0084
CS_2	0.0000	0.0000	0.0034	0.0019
H_2O	0.0000	0.0057	0.3493	0.3323
C_3^+	0.0018	0.0000	0.0000	0.0000
Total	0.5828	0.8511	1.2727	1.2641
S vapor (as S_1)	----	----	0.3522	0.0008
Temperature (°F)	60	60	1888	271
Pressure (psig)	62.0	100.0	5.5	2.0

4.3.2 Performance Summary

Chemical Equilibrium Results

A thorough review was completed on all of the material balances generated from the February, 1997 test runs. Table 4.3-2 summarizes the most important performance parameters from the test runs.

Table 4.3-2
Test Run Results

Sample	Acid Gas H_2S (mole %)	Combustion Air O_2 (mole %)	Excess Air (%)	H_2S Conversion (%)	SO_2 Residual (mole %)	Temperature Meas. / Theo. (°F)
Results Based on Furnace Samples						
RF09	89.40	20.81	-18.6	79.47	0.013	1644 / 2047
RF07	89.40	20.81	-30.1	67.55	0.163	1770 / 1888
RF11	89.40	20.81	-49.2	49.70	0.157	1620 / 1702
RF13	89.40	20.81	-53.7	42.16	0.670	1618 / 1627
Results Based on Tail Gas Samples						
TG10	89.40	20.81	-21.3	65.17	1.657	1873 / 2127
TG08	89.40	20.81	-33.4	61.64	0.668	1770 / 1946
TG14	89.40	20.81	-48.9	47.65	0.523	1620 / 1764
TG12	89.40	20.81	-55.2	43.72	0.168	1618 / 1652

For base line comparison, each of these test sets was also simulated on **Sulsim®** to determine the expected thermodynamic equilibrium behavior for these particular test runs. The full material balances for these runs are included in Appendix B-2 and are summarized in Table 4.3-3.

Table 4.3-3
Test Run Comparison - Thermodynamic Equilibrium

Test	Acid Gas H_2S (mole %)	Combustion Air O_2 (mole %)	Excess Air (%)	H_2S Conversion (%)	SO_2 Residual (mole %)	Temperature (°F)
2	89.40	20.81	-12.5	70.23	2.096	2042
1	89.40	20.81	-27.2	64.39	1.129	1895
3	89.40	20.81	-47.6	49.08	0.497	1694
4	89.40	20.81	-53.2	44.41	0.367	1624

H_2S Conversion Efficiency

As illustrated in Tables 4.3-2 and 4.3-3 and Figure 4.3.2-1, the conversion of H_2S to elemental sulfur varied between 42 and 68 percent. These results were marginally lower than the equivalent values determined by the thermodynamic equilibrium calculations. These values were however, consistent with the expected results and indicated that the H_2S oxidation reaction proceeded adequately even under these conditions which were severely deficient in oxygen.

The test runs were also conducted at furnace pressures varying between 16 and 55 psia. These tests did not cover the full range of the initial test plan and did not reach the expected maximum test pressure of 100 psia. However, the test runs did indicate that the H_2S oxidation reaction was not measurably affected by system pressure in a negative manner. It is expected that based on these results, further increases in system pressure would not be expected to have any detrimental effects on the reaction system. Indeed, as with most homogeneous gas phase reaction systems, increasing pressure could be expected to improve the overall mixing in the system which would have a net positive effect on the approach to equilibrium in this system.

In general, these conversion efficiencies were also consistent with the target value of 50 percent as stipulated in the original program objectives. The test results demonstrated that the conversion efficiency could be maintained at 50 percent as long as the reaction stoichiometry was maintained at no less than 50 percent of traditional modified Claus stoichiometry.

Therefore, the pilot plant runs demonstrated that the H_2S oxidation reaction will proceed successfully under the operating conditions imposed by the CNG Claus process.

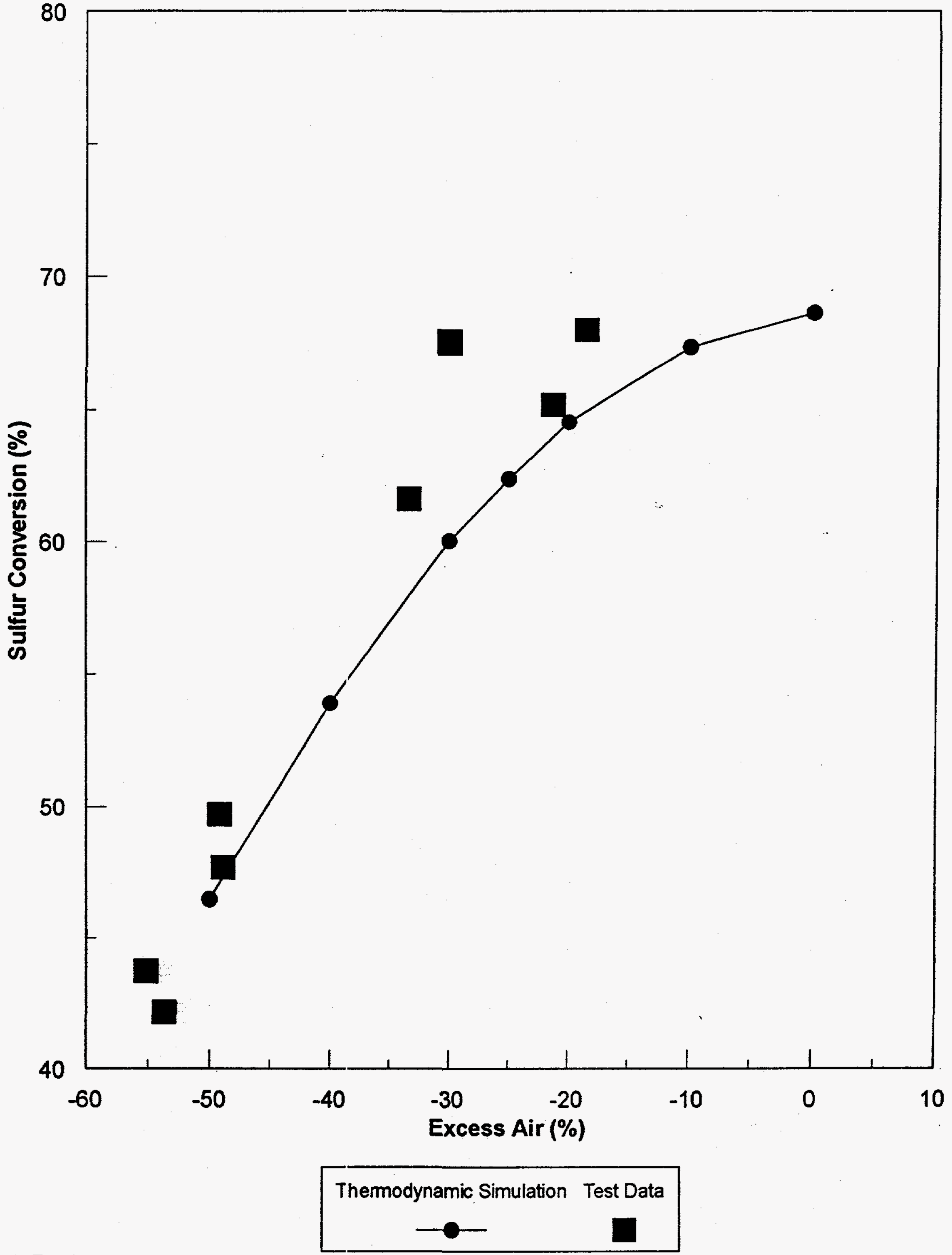
Reaction Temperature

The criterion in the original program objectives required that the reaction system be maintained below 2500°F. This would ensure that the CNG Claus furnace could be of conventional design with commercially available vessel refractory systems.

The test results (Figure 4.3.2-2) clearly indicated that the furnace temperatures were less than the benchmark value and were also remarkably consistent with the predicted equilibrium results. The measured furnace temperatures were actually less than the predicted values largely as a result of heat losses from the furnace vessel. The level of heat loss observed in the pilot plant runs was consistent with what is typically observed in industrial systems.

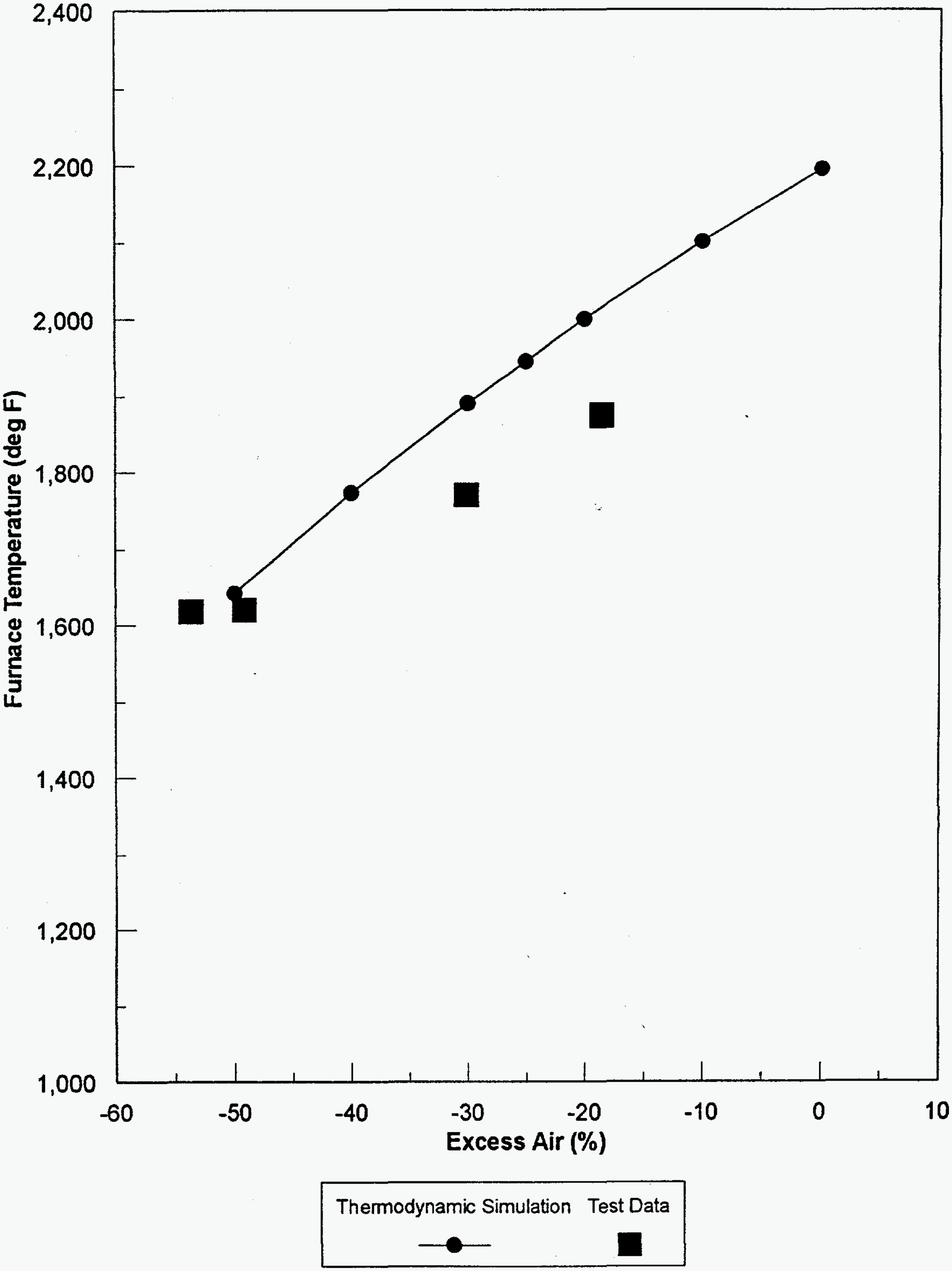
Due to the mechanical failure in the exchanger system, full test runs were not completed with the higher level oxygen concentrations prescribed in the original test run matrix. However, the test runs did demonstrate that the actual furnace temperatures for the completed runs were consistent with the equilibrium values. Therefore, by extrapolation and review of the original simulation studies conducted, it is a reasonable assumption that the furnace temperatures would be consistent with the equilibrium simulation results. The simulated results indicated that the furnace temperature could be maintained below the prescribed 2500°F value even with extremely rich acid gas feeds and very high oxygen concentrations in the combustion air (90 percent H_2S and 90 percent O_2 respectively).

Figure 4.3.2-1
Test Run Data vs Theoretical Model
Sulfur Conversion vs. Reaction Stoichiometry



1. Feed gas : 90% H2S

Figure 4.3.2-2
Test Run Data vs Theoretical Model
Reaction Furnace Temperature vs. Reaction Stoichiometry



1. Feed gas : 90% H2S

Reactant Composition

The test results clearly indicated that the performance of the furnace with respect to H_2S conversion met the program objectives. By inference therefore, the composition of the reaction product stream was also consistent with the expected results. However, there were two additional considerations regarding reaction product composition which were reviewed:

1. SO_2 concentration in the reaction effluent stream
2. Concentration of other sulfur bearing compounds in the reaction effluent stream

As discussed in Section 3.1, a further requirement on the CNG Claus process was that the SO_2 concentration be maintained at a prescribed maximum level. This would ensure successful operation of the downstream tail gas clean-up section of the process. The maximum desired SO_2 concentration was set at 2.0 mole percent to ensure that the downstream hydrogenation reaction would not be overloaded with respect to SO_2. The results shown in Table 4.3-2 indicate that the SO_2 concentrations were less than the prescribed 2 mole percent for all test cases. Even in the cases at relatively high reaction stoichiometry (Test 2) the SO_2 concentration was less than the prescribed level.

The role of side reactions in traditional Claus furnaces is well documented and ultimately can affect the conversion performance of the system in addition to creating new compounds which can affect the downstream operations. These side reactions are generally predicted in thermodynamic equilibrium models and have been shown to be affected by kinetic limitations also.

The most commonly documented products of these side reactions in industrial Claus furnaces are H_2, CO, COS and CS_2. The formation of these compounds is consistently predicted by the equilibrium model however, the extent of formation of these compounds is often affected significantly by kinetic limitations.

Both H_2 and CO exist in fairly high concentrations in these H_2S oxidation systems and the test results confirmed the extent of these side reactions in the pilot plant. Generally, the measured concentrations of these species was less than predicted by thermodynamic equilibrium. This indicated some level of kinetic limitation in the pilot plant furnace which is entirely consistent with industrial Claus furnaces. Generally, low reaction furnace temperatures result in H_2 and CO formation rates less than equilibrium in the furnace. The oxygen deficient conditions used for the pilot plant studies resulted in relatively low furnace temperatures (<2000°F) and comparatively low formation rates also.

The formation of H_2 and CO does not affect the H_2S conversion efficiency and is therefore not important to overall sulfur recovery. However, they do affect the "air demand" of the system and can have some effect on the reaction temperature and material balance through the system. For this system, however, the effects on overall performance of the process were not deemed significant.

Of more interest are those reactants and reactions which lead to other sulfur bearing compounds in the reaction product stream; primarily the carbon-sulfur compounds COS and CS_2. Because these compounds contain sulfur, they can have a measurable effect on the furnace conversion efficiency and also require that the overall process system be designed to ultimately eliminate these compounds from the process stream or risk adding to the sulfur emissions from the process.

COS is formed in a system that contains both sulfur and carbon from any source. In the test system the acid gas stream contained a significant level of CO_2 (~10 mole percent) and therefore, formation of COS was expected. The thermodynamic models predict relatively low levels of COS formation; typically less than 1000 ppm for the pilot plant feed streams. However, kinetic limitations can lead to higher levels as was observed in the pilot plant tests which had COS levels in the 1000 to 3000 ppm range.

The levels of COS formation in the CNG Claus system were in the range observed in industrial Claus furnaces as expected. The tail gas treating system proposed for the CNG Claus process is fully capable of dealing with this level of COS in the hydrogenation reactor. Therefore, no detrimental effects due to COS formation are expected for the CNG Claus process.

CS_2 is formed in a system that contains both sulfur and carbon from hydrocarbon compounds. In the test system it was originally expected that there would be no hydrocarbon in the acid gas. However, analysis of the liquid H_2S indicated nominal levels of propane (approximately 300 ppm) and therefore formation of CS_2 was expected. The thermodynamic model predicts very low levels of CS_2 formation; typically less than 50 ppm for the pilot plant feed streams. However, kinetic limitations can lead to higher levels as was observed in the pilot plant tests which had CS_2 levels in the 1700 to 4300 ppm range.

The levels of CS_2 formation in the pilot plant were in the range observed in industrial Claus furnaces. The tail gas treating system proposed for the CNG Claus process is fully capable of dealing with this level of COS in the hydrogenation reactor. Therefore, no detrimental effects due to CS_2 formation are expected for the CNG Claus process.

Kinetic Limitations

Industrial Claus reaction furnaces systems are known to have some level of kinetic limitations which cause the reaction products to vary from those predicted by thermodynamic equilibrium. These kinetic limitations are directly affected by reaction residence time, reaction temperature and the extent of mixing in the burner and reaction zone.

It was assumed that the pilot plant burner/furnace system would suffer some level of kinetic limitation and a portion of the test matrix was dedicated to measuring the effects of residence time and reaction temperature on the reaction system. Due to the mechanical problems encountered during the pilot plant test runs, these specific test runs were not completed. However, a thorough review of the completed test runs indicated that a range of residence times and reaction temperatures was achieved. Table 4.3-3 on the following page summarizes the effect of the measured kinetic parameters from the completed test runs.

Table 4.3-4
Test Run Results - Kinetic Effects

Sample	Process Flow Rate (acfm)	Residence Time (s)	H_2S Conversion (%)	H_2S Conversion Theo. (%)[1]	Approach to Equilibrium[2] (%)	Temperature Measured (°F)
TG10	22.63	1.30	65.17	70.23	93	1644
RF07	20.06	1.47	67.55	64.39	95	1770
RF11	8.68	3.39	49.70	49.08	100	1620
RF13	8.07	3.64	42.16	47.23	89	1618

[1] Based on thermodynamic equilibrium simulations.
[2] Measured H_2S conversion / Theoretical H_2S conversion.

These results indicated that the pilot plant furnace generally had a very close approach to equilibrium for all of the test cases. It also indicated that the residence time did not have a significant impact on the approach to equilibrium and lower residence times did not have a significant detrimental effect on the conversion efficiency. Similarly, the lower reaction temperatures measured in the very low reaction stoichiometry cases did not adversely affect the approach to equilibrium conversion. This is consistent with observations in industrial furnaces which show a good approach to equilibrium for H_2S conversion regardless of measured kinetic parameters. It is generally agreed in the industry that this lack of kinetic limitations on the H_2S oxidation reaction is largely a function of the extremely fast reaction times associated with this reaction.

As described earlier, the concentrations of H_2, CO, COS and CS_2 were measurably different than the equilibrium predictions which does indicate measurable kinetic limitation with respect to side reactions in the furnace. This is also consistent with observations in industrial furnaces and is attributed to the fact that all of the side reactions in question have considerably slower overall reaction rates and are therefore much more susceptible to kinetic limitation in the reaction zone.

In summary, the following conclusions were drawn for the test run results:

Reaction Considerations

- the H_2S oxidation reaction proceeds successfully at the fundamental CNG Claus conditions; specifically with very low reaction stoichiometry and high reaction pressures.
- the oxidation reaction is stable and sustainable in the "free flame" reaction regime.
- the extent of H_2S conversion to elemental sulfur measured during the test runs was consistent with the required values set out in the test program objectives (>50 percent).
- the extent of H_2S conversion to elemental sulfur measured during the test runs was comparable to the predicted results using thermodynamic equilibrium reaction models. This implies that the H_2S oxidation reaction is not unusually hindered by reaction kinetics at the CNG Claus conditions.
- the increased pressure associated with the CNG Claus process had no discernable effect on the H_2S oxidation reaction. Traditional homogeneous gas phase reaction theory suggests that the reactions would actually be enhanced under higher pressure conditions.
- the concentration of the reaction products (primarily $SO_2 < 2.0$ mole percent) were consistent with the values required for successful operation of a conventional downstream tail gas treating process.
- the measured and calculated reaction temperatures (1500 - 2200°F) were consistent with the values required to ensure that the process can be operated in furnace of conventional design.
- the measured and calculated reaction temperatures were consistent with the predicted results using thermodynamic equilibrium reaction models.
- the extent of side reactions in the pilot plant indicate that kinetic parameters do affect the extent of formation of the minor reaction product species; H_2, CO, COS and CS_2. The concentrations of these compounds in the system are not considered detrimental to the overall successful operation of an integrated system.

Equipment Considerations

- the pilot plant design and equipment was taken directly from conventional technology and performed essentially as planned.

- the conventional burner and reaction furnace system allowed for successful completion of the H_2S oxidation reaction under the required conditions.

- the measured reaction furnace temperatures were consistently less than the allowable 2500°F maximum which ensures that conventional furnace refractory design can be employed.

- the equipment failure in the pilot plant (leaking wasteheat exchanger) was probably a result of conventional corrosion and was not an artifact of the unconventional CNG Claus operating conditions.

- the pilot plant was designed with entirely conventional metallurgy and did not suffer any unusual failures due to the CNG Claus operating conditions.

References

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APPENDIX A

Appendix A

Gas Analysis Results

TABLE A-1

Gas Chromatographic Analyses
(Mole Percent)

Test 1

Consolidated Natural Gas
Davis, Oklahoma

Feb 25, 1997
File Number: 4309

Sample No:	15	7	8
Site:	ACID GAS	WHB	TAIL GAS
Time:	12:41	12:58	12:58
H_2	0.000	2.924	1.728
Ar	0.013	0.850	0.827
O_2	0.000	0.343	0.019
N_2	1.110	71.080	69.116
CH_4	0.000	0.000	0.000
CO	0.000	0.246	0.158
CO_2	9.101	5.845	6.020
C_2H_4	0.000	0.000	0.000
C_2H_6	0.000	0.000	0.000
H_2S	89.454	17.942	20.810
COS	0.005	0.153	0.170
C_3H_8	0.307	0.000	0.000
SO_2	0.010	0.235	0.943
CS_2	0.000	0.382	0.209
iC_4H_{10}	0.000	0.000	0.000
nC_4H_{10}	0.000	0.000	0.000
iC_5H_{12}	0.000	0.000	0.000
nC_5H_{12}	0.000	0.000	0.000
$C_6H_{14}+$	0.000	0.000	0.000
	100.000	100.000	100.000

Zero means not detected.

Sampled water- and
sulfur-free.

Global Sulphur Experts Inc.
Tyler, Texas

TABLE A-2

Gas Chromatographic Analyses
(Mole Percent)

Test 2

Consolidated Natural Gas
Davis, Oklahoma

Feb 25, 1997
File Number: 4309

Sample No:	15	9	10
Site:	ACID GAS	WHB	TAIL GAS
Time:	12:41	13:10	13:36
H_2	0.000	1.629	0.917
Ar	0.013	0.970	0.901
O_2	0.000	0.454	0.017
N_2	1.110	81.115	75.318
CH_4	0.000	0.000	0.000
CO	0.000	0.232	0.164
CO_2	9.101	5.451	5.639
C_2H_4	0.000	0.000	0.000
C_2H_6	0.000	0.000	0.000
H_2S	89.454	9.549	14.543
COS	0.005	0.348	0.196
C_3H_8	0.307	0.000	0.000
SO_2	0.010	0.017	2.206
CS_2	0.000	0.235	0.099
iC_4H_{10}	0.000	0.000	0.000
nC_4H_{10}	0.000	0.000	0.000
iC_5H_{12}	0.000	0.000	0.000
nC_5H_{12}	0.000	0.000	0.000
$C_6H_{14}+$	0.000	0.000	0.000
	100.000	100.000	100.000

Zero means not detected.

Sampled water- and sulfur-free.

Global Sulphur Experts Inc.
Tyler, Texas

TABLE A-3

Gas Chromatographic Analyses
(Mole Percent)

Test 3

Consolidated Natural Gas
Davis, Oklahoma

Feb 25, 1997
File Number: 4309

Sample No:	16	11	12
Site:	ACID GAS	WHB	TAIL GAS
Time:	14:00	14:06	14:06
H_2	0.000	1.634	0.957
Ar	0.000	0.762	0.732
O_2	0.000	0.045	0.000
N_2	0.188	63.700	61.369
CH_4	0.000	0.000	0.000
CO	0.000	0.125	0.081
CO_2	10.094	6.094	6.219
C_2H_4	0.000	0.000	0.000
C_2H_6	0.000	0.000	0.000
H_2S	89.396	26.771	29.947
COS	0.006	0.245	0.227
C_3H_8	0.307	0.000	0.000
SO_2	0.009	0.185	0.207
CS_2	0.000	0.439	0.261
iC_4H_{10}	0.000	0.000	0.000
nC_4H_{10}	0.000	0.000	0.000
iC_5H_{12}	0.000	0.000	0.000
nC_5H_{12}	0.000	0.000	0.000
$C_6H_{14}+$	0.000	0.000	0.000
	100.000	100.000	100.000

Zero means not detected.

Sampled water- and
sulfur-free.

Global Sulphur Experts Inc.
Tyler, Texas

TABLE A-4

Gas Chromatographic Analyses
(Mole Percent)

Test 4

Consolidated Natural Gas
Davis, Oklahoma

Feb 25, 1997
File Number: 4309

Sample No:	16	13	14
Site:	ACID GAS	WHB	TAIL GAS
Time:	14:00	14:08	14:08
H_2	0.000	1.699	0.901
Ar	0.000	0.730	0.755
O_2	0.000	0.016	0.030
N_2	0.188	61.019	63.087
CH_4	0.000	0.000	0.000
CO	0.000	0.115	0.086
CO_2	10.094	5.847	6.308
C_2H_4	0.000	0.000	0.000
C_2H_6	0.000	0.000	0.000
H_2S	89.396	29.169	27.730
COS	0.006	0.202	0.226
C_3H_8	0.307	0.000	0.000
SO_2	0.009	0.732	0.626
CS_2	0.000	0.471	0.251
iC_4H_{10}	0.000	0.000	0.000
nC_4H_{10}	0.000	0.000	0.000
iC_5H_{12}	0.000	0.000	0.000
nC_5H_{12}	0.000	0.000	0.000
$C_6H_{14}+$	0.000	0.000	0.000
	100.000	100.000	100.000

Zero means not detected.

Sampled water- and
sulfur-free.

Global Sulphur Experts Inc.
Tyler, Texas

TABLE A-5

Gas Chromatographic Analyses
(Mole Percent)

Fuel Gas Runs

Consolidated Natural Gas
Davis, Oklahoma

Feb 23, 1997
File Number: 4309

Sample No:	1	2	3
Site:	TAIL GAS	TAIL GAS	TAIL GAS
Time:	14:28	15:42	16:02
H_2	0.000	0.000	0.000
Ar	0.959	0.985	0.984
O_2	15.170	9.838	10.122
N_2	80.214	82.369	82.243
CH_4	0.000	0.000	0.000
CO	0.000	0.000	0.000
CO_2	3.657	6.808	6.651
C_2H_4	0.000	0.000	0.000
C_2H_6	0.000	0.000	0.000
H_2S	0.000	0.000	0.000
COS	0.000	0.000	0.000
C_3H_8	0.000	0.000	0.000
SO_2	0.000	0.000	0.000
CS_2	0.000	0.000	0.000
iC_4H_{10}	0.000	0.000	0.000
nC_4H_{10}	0.000	0.000	0.000
iC_5H_{12}	0.000	0.000	0.000
nC_5H_{12}	0.000	0.000	0.000
$C_6H_{14}+$	0.000	0.000	0.000
	100.000	100.000	100.000

Zero means not detected.

Sampled water- and
sulfur-free.

Global Sulphur Experts Inc.
Tyler, Texas

TABLE A-6

Gas Chromatographic Analyses
(Mole Percent)

Acid Gas Flow Test

Consolidated Natural Gas
Davis, Oklahoma

Feb 24, 1997
File Number: 4309

Sample No:	4	5	6
Site:	ACID GAS	ACID GAS	TAIL GAS
Time:	15:41	16:02	16:35
H_2	0.000	0.000	0.184
Ar	0.000	0.000	0.992
O_2	0.000	0.000	7.812
N_2	0.000	0.020	82.946
CH_4	0.000	0.000	0.000
CO	0.000	0.000	0.045
CO_2	66.500	75.454	5.603
C_2H_4	0.000	0.000	0.000
C_2H_6	0.000	0.000	0.000
H_2S	33.389	24.440	0.414
COS	0.002	0.001	0.011
C_3H_8	0.109	0.085	0.000
SO_2	0.000	0.000	1.991
CS_2	0.000	0.000	0.002
iC_4H_{10}	0.000	0.000	0.000
nC_4H_{10}	0.000	0.000	0.000
iC_5H_{12}	0.000	0.000	0.000
nC_5H_{12}	0.000	0.000	0.000
$C_6H_{14}+$	0.000	0.000	0.000
	100.000	100.000	100.000

Zero means not detected.

Sampled water- and
sulfur-free.

Global Sulphur Experts Inc.
Tyler, Texas

TABLE A-7

Gas Chromatographic Analyses
(Mole Percent)

Utility Gas Analysis

Consolidated Natural Gas
Davis, Oklahoma

Feb 26, 1997
File Number: 4309

Sample No:	17	18
Site:	CO2 TANK	O2 N2 MIX 50-50
Time:	10:16	00:00
H_2	0.000	0.000
Ar	0.016	0.000
O_2	0.366	50.450
N_2	1.303	49.550
CH_4	0.000	0.000
CO	0.000	0.000
CO_2	98.315	0.000
C_2H_4	0.000	0.000
C_2H_6	0.000	0.000
H_2S	0.000	0.000
COS	0.000	0.000
C_3H_8	0.000	0.000
SO_2	0.000	0.000
CS_2	0.000	0.000
iC_4H_{10}	0.000	0.000
nC_4H_{10}	0.000	0.000
iC_5H_{12}	0.000	0.000
nC_5H_{12}	0.000	0.000
$C_6H_{14}+$	0.000	0.000
	100.000	100.000

Zero means not detected.

Sampled water- and sulfur-free.

Global Sulphur Experts Inc.
Tyler, Texas

APPENDIX B

Appendix B-1

Material Balance Calculations
Test Run Results

```
ULSIM   CASE: CNG01-07                                          98/01/19
NG - Test 1; Sample RF07
                              SULPHUR PLANT PERFORMANCE
                                                                    EFFICIENCY
--------------------------------------------------------------------------------
   Unit:         Thermal       --------------Catalytic Stage--------------
                   Stage
   Efficiency                                 (Percent)
--------------------------------------------------------------------------------
   Conversion:
         Unit        67.55
   Cumulative        67.55

     Recovery:
         Unit        99.77
   Cumulative        67.40

nit Hydrolysis:
          COS          NA
          CS2          NA
--------------------------------------------------------------------------------
verall Recovery Efficiency . . . . . . . . . . . . . . . . . .        67.40

                                                                    PRODUCTION
--------------------------------------------------------------------------------
   Unit:         Thermal       --------------Catalytic Stage--------------
                   Stage
   Production                                    (lb/hr)
--------------------------------------------------------------------------------
   Conversion:
         Unit        11.30
   Cumulative        11.30

     Recovery:
         Unit        11.27
   Cumulative        11.27
--------------------------------------------------------------------------------
otal Inlet Sulphur . . . . . . . . . . . . . . . . . . . . . .        16.72
```

```
SULSIM  CASE: CNG01-07                                          98/01/19
CNG - Test 1; Sample RF07

                                           O/L Known Reaction Furnace (R[illegible])
-------------------------------------------------------------------------------
                                              FD1          AIR            RF
Parameter                                   STREAM       STREAM        STREAM
-------------------------------------------------------------------------------
COMPOSITION (lbmol/hr)
        H2:                                  0.000        0.000         0.026
        Ar:                                  0.000        0.008         0.0[illegible]8
        O2:                                  0.000        0.177         0.003
        N2:                                  0.006        0.660         0.667
        C1:                                  0.000        0.000         0.0[illegible]0
        CO:                                  0.000        0.000         0.0[illegible]2
       CO2:                                  0.053        0.000         0.052
        C2:                                  0.000        0.000         0.000
       H2S:                                  0.521        0.000         0.1[illegible]9
       COS:                                  0.000        0.000         0.001
       SO2:                                  0.000        0.000         0.002
       CS2:                                  0.000        0.000         0.0[illegible]3
       H2O:                                  0.000        0.006         0.3[illegible]9
       NH3:                                  0.000        0.000         0.000
       HCN:                                  0.000        0.000         0.0[illegible]0
        C3:                                  0.002        0.000         0.0[illegible]0
       iC4:                                  0.000        0.000         0.000
       nC4:                                  0.000        0.000         0.000
       iC5:                                  0.000        0.000         0.0[illegible]0
       nC5:                                  0.000        0.000         0.000
        C6:                                  0.000        0.000         0.000
      CH4S:                                  0.000        0.000         0.0[illegible]0
     C2H6S:                                  0.000        0.000         0.0[illegible]0
S vapour as Sx                               0.000        0.000         0.175
S liquid as S1                               0.000        0.000         0.000
                                           ---------    ---------    ---------
        TOTAL                                0.583        0.851         1.448
- - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - -
TEMPERATURE (F)                              60.0         60.0        1888.0
PRESSURE (psig)                              62.00       100.00        14.50
HEAT CAPACITY (BTU/(lbmol.R))                 8.23         6.96         9.53
MOLECULAR WEIGHT                             34.95        28.89        31.06
Sx AVERAGE SPECIES NO. (x)                   0.000        0.000         2.01[illegible]
CUMULATIVE FLOWS (lbmol/hr)
           Feed Stream                       0.583          NA          0.583
           Process Air                       0.000          NA          0.85[illegible]
              Fuel Gas                       0.000          NA          0.000
CUMULATIVE EFFICIENCIES (%)
            Conversion                       0.00           NA         67.55
             Recovery                        0.00           NA          0.00
EXCESS PROCESS AIR (%)                        NA            NA        -30.1
-------------------------------------------------------------------------------
Cumulative Sulphur Conversion as S1 (lbmol/hr) . . . . . . . . .       0.35[illegible]
```

```
ULSIM  CASE: CNG01-08                                           98/01/19
NG - Test 1; Sample TG08
                                SULPHUR PLANT PERFORMANCE
                                                                  EFFICIENCY
----------------------------------------------------------------------------
   Unit:       Thermal      -------------Catalytic Stage-------------
                 Stage
   Efficiency                              (Percent)
----------------------------------------------------------------------------
   Conversion:
         Unit      61.64
   Cumulative      61.64

     Recovery:
         Unit      99.75
   Cumulative      61.48

Unit Hydrolysis:
          COS       NA
          CS2       NA
----------------------------------------------------------------------------
Overall Recovery Efficiency . . . . . . . . . . . . . . . . . . .     61.48


                                                                  PRODUCTION
----------------------------------------------------------------------------
   Unit:       Thermal      -------------Catalytic Stage-------------
                 Stage
   Production                                 (lb/hr)
----------------------------------------------------------------------------
   Conversion:
         Unit      10.31
   Cumulative      10.31

     Recovery:
         Unit      10.28
   Cumulative      10.28
----------------------------------------------------------------------------
Total Inlet Sulphur . . . . . . . . . . . . . . . . . . . . . . .     16.72
```

```
SULSIM  CASE: CNG01-08                                          98/01/19
CNG - Test 1; Sample TG08

                                         Q/L Known Reaction Furnace (R
-----------------------------------------------------------------------
                                           FD1          AIR         RF
Parameter                                STREAM       STREAM      STREA
-----------------------------------------------------------------------
COMPOSITION (lbmol/hr)
         H2:                              0.000        0.000       0.015
         Ar:                              0.000        0.008       0.00
         O2:                              0.000        0.174       0.000
         N2:                              0.006        0.649       0.655
         C1:                              0.000        0.000       0.00
         CO:                              0.000        0.000       0.00
        CO2:                              0.053        0.000       0.054
         C2:                              0.000        0.000       0.00
        H2S:                              0.521        0.000       0.18
        COS:                              0.000        0.000       0.002
        SO2:                              0.000        0.000       0.008
        CS2:                              0.000        0.000       0.00
        H2O:                              0.000        0.006       0.33
        NH3:                              0.000        0.000       0.000
        HCN:                              0.000        0.000       0.0
         C3:                              0.002        0.000       0.0
        iC4:                              0.000        0.000       0.000
        nC4:                              0.000        0.000       0.000
        iC5:                              0.000        0.000       0.00
        nC5:                              0.000        0.000       0.000
         C6:                              0.000        0.000       0.000
       CH4S:                              0.000        0.000       0.00
      C2H6S:                              0.000        0.000       0.00
S vapour as Sx                            0.000        0.000       0.160
S liquid as S1                            0.000        0.000       0.00
                                        ---------    ---------   --------
        TOTAL                             0.583        0.836       1.424
- - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - -
TEMPERATURE (F)                           60.0         60.0       1945.6
PRESSURE (psig)                          62.00       100.00       14.50
HEAT CAPACITY (BTU/(lbmol.R))             8.23         6.96        9.65
MOLECULAR WEIGHT                         34.95        28.89       31.2
Sx AVERAGE SPECIES NO. (x)               0.000        0.000        2.0
CUMULATIVE FLOWS (lbmol/hr)
           Feed Stream                   0.583          NA         0.583
           Process Air                   0.000          NA         0.8
              Fuel Gas                   0.000          NA         0.000
CUMULATIVE EFFICIENCIES (%)
            Conversion                    0.00          NA         61.6
              Recovery                    0.00          NA          0.0
EXCESS PROCESS AIR (%)                     NA           NA        -33.4
-----------------------------------------------------------------------
Cumulative Sulphur Conversion as S1 (lbmol/hr) . . . . . . . . .   0.32
```

```
ULSIM  CASE: CNG01-09                                            98/01/19
NG - Test 2; Sample RF09
                           SULPHUR PLANT PERFORMANCE
                                                                  EFFICIENCY
----------------------------------------------------------------------------
   Unit:        Thermal      -------------Catalytic Stage-------------
                  Stage
   Efficiency                               (Percent)
----------------------------------------------------------------------------
   Conversion:
        Unit       79.47
   Cumulative      79.47

     Recovery:
        Unit       99.76
   Cumulative      79.27

Unit Hydrolysis:
         COS         NA
         CS2         NA
----------------------------------------------------------------------------
verall Recovery Efficiency . . . . . . . . . . . . . . . . . .      79.27

                                                                  PRODUCTION
----------------------------------------------------------------------------
   Unit:        Thermal      -------------Catalytic Stage-------------
                  Stage
   Production                                 (lb/hr)
----------------------------------------------------------------------------
   Conversion:
        Unit       15.82
   Cumulative      15.82

     Recovery:
        Unit       15.79
   Cumulative      15.79
----------------------------------------------------------------------------
otal Inlet Sulphur . . . . . . . . . . . . . . . . . . . . . .      19.91
```

```
SULSIM  CASE: CNG01-09                                          98/01/19
CNG - Test 2; Sample RF09

                                              O/L Known Reaction Furnace (R
--------------------------------------------------------------------------
                                              FD1           AIR          RF
Parameter                                   STREAM        STREAM      STREA
--------------------------------------------------------------------------
COMPOSITION (lbmol/hr)
        H2:                                  0.000         0.000        0.020
        Ar:                                  0.000         0.011        0.01
        O2:                                  0.000         0.252        0.00
        N2:                                  0.001         0.939        0.940
        C1:                                  0.000         0.000        0.00
        CO:                                  0.000         0.000        0.00
       CO2:                                  0.070         0.000        0.067
        C2:                                  0.000         0.000        0.00
       H2S:                                  0.621         0.000        0.11
       COS:                                  0.000         0.000        0.004
       SO2:                                  0.000         0.000        0.000
       CS2:                                  0.000         0.000        0.00
       H2O:                                  0.000         0.008        0.50
       NH3:                                  0.000         0.000        0.000
       HCN:                                  0.000         0.000        0.00
        C3:                                  0.002         0.000        0.00
       iC4:                                  0.000         0.000        0.000
       nC4:                                  0.000         0.000        0.00
       iC5:                                  0.000         0.000        0.00
       nC5:                                  0.000         0.000        0.00
        C6:                                  0.000         0.000        0.000
      CH4S:                                  0.000         0.000        0.00
     C2H6S:                                  0.000         0.000        0.00
S vapour as Sx                               0.000         0.000        0.245
S liquid as S1                               0.000         0.000        0.00
                                           ---------     ---------    --------
        TOTAL                                0.695         1.210        1.917
- - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - -
TEMPERATURE (F)                             60.0          60.0        2046.8
PRESSURE (psig)                             62.00        100.00         17.00
HEAT CAPACITY (BTU/(lbmol.R))                8.24          6.96          9.51
MOLECULAR WEIGHT                            35.11         28.89         30.96
Sx AVERAGE SPECIES NO. (x)                   0.000         0.000         2.01
CUMULATIVE FLOWS (lbmol/hr)
          Feed Stream                        0.695           NA          0.695
          Process Air                        0.000           NA          1.21
             Fuel Gas                        0.000           NA          0.000
CUMULATIVE EFFICIENCIES (%)
           Conversion                        0.00            NA         79.47
            Recovery                         0.00            NA          0.00
EXCESS PROCESS AIR (%)                         NA            NA        -18.6
--------------------------------------------------------------------------
Cumulative Sulphur Conversion as S1 (lbmol/hr)  . . . . . . . . . .     0.49
```

```
ULSIM  CASE: CNG01-10                                        98/01/19
NG - Test 2; Sample TG10
                                SULPHUR PLANT PERFORMANCE
                                                                   EFFICIENCY
---------------------------------------------------------------------------
    Unit:        Thermal      --------------Catalytic Stage-------------
                   Stage
    Efficiency                                 (Percent)
---------------------------------------------------------------------------
    Conversion:
          Unit      65.17
    Cumulative      65.17

      Recovery:
          Unit      99.70
    Cumulative      64.98

Unit Hydrolysis:
           COS        NA
           CS2        NA
---------------------------------------------------------------------------
Overall Recovery Efficiency . . . . . . . . . . . . . . . . . .      64.98

                                                                   PRODUCTION
---------------------------------------------------------------------------
    Unit:        Thermal      --------------Catalytic Stage-------------
                   Stage
    Production                                    (lb/hr)
---------------------------------------------------------------------------
    Conversion:
          Unit      12.98
    Cumulative      12.98

      Recovery:
          Unit      12.94
    Cumulative      12.94
---------------------------------------------------------------------------
Total Inlet Sulphur . . . . . . . . . . . . . . . . . . . . . .      19.91
```

```
SULSIM  CASE: CNG01-10                                          98/01/19
CNG - Test 2; Sample TG10

                                         O/L Known Reaction Furnace (R[illegible])
--------------------------------------------------------------------------
                                         FD1          AIR            RF
Parameter                              STREAM       STREAM        STREA[illegible]
--------------------------------------------------------------------------
COMPOSITION (lbmol/hr)
        H2:                             0.000        0.000        0.012
        Ar:                             0.000        0.011        0.0[illegible]1
        O2:                             0.000        0.248        0.000
        N2:                             0.001        0.925        0.926
        C1:                             0.000        0.000        0.0[illegible]0
        CO:                             0.000        0.000        0.0[illegible]2
       CO2:                             0.070        0.000        0.071
        C2:                             0.000        0.000        0.0[illegible]0
       H2S:                             0.621        0.000        0.1[illegible]3
       COS:                             0.000        0.000        0.002
       SO2:                             0.000        0.000        0.028
       CS2:                             0.000        0.000        0.0[illegible]1
       H2O:                             0.000        0.008        0.442
       NH3:                             0.000        0.000        0.000
       HCN:                             0.000        0.000        0.0[illegible]0
        C3:                             0.002        0.000        0.0[illegible]0
       iC4:                             0.000        0.000        0.000
       nC4:                             0.000        0.000        0.000
       iC5:                             0.000        0.000        0.0[illegible]0
       nC5:                             0.000        0.000        0.000
        C6:                             0.000        0.000        0.000
      CH4S:                             0.000        0.000        0.0[illegible]0
     C2H6S:                             0.000        0.000        0.0[illegible]0
S vapour as Sx                          0.000        0.000        0.201
S liquid as S1                          0.000        0.000        0.0[illegible]0
                                      ---------    ---------    ---------
        TOTAL                           0.695        1.192        1.881
 - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - -
TEMPERATURE (F)                        60.0         60.0        2127.2[illegible]
PRESSURE (psig)                        62.00       100.00         17.00
HEAT CAPACITY (BTU/(lbmol.R))           8.24         6.96          9.71
MOLECULAR WEIGHT                       35.11        28.89         31.2[illegible]
Sx AVERAGE SPECIES NO. (x)              0.000        0.000         2.0[illegible]
CUMULATIVE FLOWS (lbmol/hr)
           Feed Stream                  0.695          NA          0.695
           Process Air                  0.000          NA          1.1[illegible]2
              Fuel Gas                  0.000          NA          0.000
CUMULATIVE EFFICIENCIES (%)
            Conversion                  0.00           NA         65.1[illegible]
              Recovery                  0.00           NA          0.0[illegible]
EXCESS PROCESS AIR (%)                   NA            NA        -21.3
--------------------------------------------------------------------------
Cumulative Sulphur Conversion as S1 (lbmol/hr) . . . . . . . . .   0.4[illegible]
```

```
ULSIM  CASE: CNG01-11                                           98/01/19
NG - Test 3; Sample RF11
                               SULPHUR PLANT PERFORMANCE
                                                                   EFFICIENCY
-------------------------------------------------------------------------------
    Unit:        Thermal      -------------Catalytic Stage-------------
                   Stage
    Efficiency                                (Percent)
-------------------------------------------------------------------------------
    Conversion:
          Unit       49.70
    Cumulative       49.70

      Recovery:
          Unit       99.86
    Cumulative       49.63

nit Hydrolysis:
           COS         NA
           CS2         NA
-------------------------------------------------------------------------------
verall Recovery Efficiency . . . . . . . . . . . . . . . . . . .      49.63

                                                                   PRODUCTION
-------------------------------------------------------------------------------
    Unit:        Thermal      -------------Catalytic Stage-------------
                   Stage
    Production                                  (lb/hr)
-------------------------------------------------------------------------------
    Conversion:
          Unit        8.54
    Cumulative        8.54

      Recovery:
          Unit        8.53
    Cumulative        8.53
-------------------------------------------------------------------------------
otal Inlet Sulphur . . . . . . . . . . . . . . . . . . . . . . .      17.19
```

```
SULSIM  CASE: CNG01-11                                          98/01/19
CNG - Test 3; Sample RF11

                                                 O/L Known Reaction Furnace (R
---------------------------------------------------------------------------------
                                                   FD1          AIR          RF
Parameter                                         STREAM       STREAM       STREA
---------------------------------------------------------------------------------
COMPOSITION (lbmol/hr)
         H2:                                       0.000        0.000        0.016
         Ar:                                       0.000        0.006        0.0
         O2:                                       0.000        0.137        0.0
         N2:                                       0.001        0.511        0.512
         C1:                                       0.000        0.000        0.0
         CO:                                       0.000        0.000        0.0
        CO2:                                       0.061        0.000        0.059
         C2:                                       0.000        0.000        0.000
        H2S:                                       0.536        0.000        0.25
        COS:                                       0.000        0.000        0.002
        SO2:                                       0.000        0.000        0.002
        CS2:                                       0.000        0.000        0.0
        H2O:                                       0.000        0.004        0.27
        NH3:                                       0.000        0.000        0.000
        HCN:                                       0.000        0.000        0.0
         C3:                                       0.002        0.000        0.0
        iC4:                                       0.000        0.000        0.000
        nC4:                                       0.000        0.000        0.000
        iC5:                                       0.000        0.000        0.0
        nC5:                                       0.000        0.000        0.00
         C6:                                       0.000        0.000        0.000
       CH4S:                                       0.000        0.000        0.0
      C2H6S:                                       0.000        0.000        0.0
S vapour as Sx                                     0.000        0.000        0.131
S liquid as S1                                     0.000        0.000        0.000
                                                ---------    ---------    --------
         TOTAL                                     0.600        0.658        1.26
- - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - -
TEMPERATURE (F)                                   60.0         60.0       1701.9
PRESSURE (psig)                                   62.00       100.00        40.0
HEAT CAPACITY (BTU/(lbmol.R))                      8.24         6.96         9.70
MOLECULAR WEIGHT                                  35.11        28.89        31.67
Sx AVERAGE SPECIES NO. (x)                         0.000        0.000        2.02
CUMULATIVE FLOWS (lbmol/hr)
           Feed Stream                             0.600          NA         0.600
           Process Air                             0.000          NA         0.65
              Fuel Gas                             0.000          NA         0.00
CUMULATIVE EFFICIENCIES (%)
            Conversion                             0.00           NA        49.7
              Recovery                             0.00           NA         0.00
EXCESS PROCESS AIR (%)                              NA            NA       -49.2
---------------------------------------------------------------------------------
Cumulative Sulphur Conversion as S1 (lbmol/hr) . . . . . . . . .            0.26
```

```
ULSIM  CASE: CNG01-12                                      98/01/19
NG - Test 3; Sample TG12
                          SULPHUR PLANT PERFORMANCE
                                                               EFFICIENCY
-----------------------------------------------------------------------------
   Unit:        Thermal     -------------Catalytic Stage-------------
                  Stage
   Efficiency                              (Percent)
-----------------------------------------------------------------------------
   Conversion:
         Unit       43.72
   Cumulative       43.72

     Recovery:
         Unit       99.85
   Cumulative       43.65

nit Hydrolysis:
          COS         NA
          CS2         NA
-----------------------------------------------------------------------------
verall Recovery Efficiency . . . . . . . . . . . . . . . . . .      43.65

                                                               PRODUCTION
-----------------------------------------------------------------------------
   Unit:        Thermal     -------------Catalytic Stage-------------
                  Stage
   Production                                 (lb/hr)
-----------------------------------------------------------------------------
   Conversion:
         Unit        7.51
   Cumulative        7.51

     Recovery:
         Unit        7.50
   Cumulative        7.50
-----------------------------------------------------------------------------
otal Inlet Sulphur . . . . . . . . . . . . . . . . . . . . . .      17.19
```

```
SULSIM   CASE: CNG01-12                                             98/01/19
CNG - Test 3; Sample TG12

                                           O/L Known Reaction Furnace (R[illegible])
----------------------------------------------------------------------------
                                              FD1         AIR            RF
Parameter                                    STREAM      STREAM        STREAM
----------------------------------------------------------------------------
COMPOSITION (lbmol/hr)
        H2:                                   0.000       0.000        0.009
        Ar:                                   0.000       0.006        0.0[illegible]6
        O2:                                   0.000       0.124        0.0[illegible]0
        N2:                                   0.001       0.463        0.464
        C1:                                   0.000       0.000        0.000
        CO:                                   0.000       0.000        0.0[illegible]1
       CO2:                                   0.061       0.000        0.061
        C2:                                   0.000       0.000        0.000
       H2S:                                   0.536       0.000        0.2[illegible]2
       COS:                                   0.000       0.000        0.002
       SO2:                                   0.000       0.000        0.002
       CS2:                                   0.000       0.000        0.0[illegible]3
       H2O:                                   0.000       0.004        0.2[illegible]6
       NH3:                                   0.000       0.000        0.000
       HCN:                                   0.000       0.000        0.000
        C3:                                   0.002       0.000        0.0[illegible]0
       iC4:                                   0.000       0.000        0.000
       nC4:                                   0.000       0.000        0.000
       iC5:                                   0.000       0.000        0.0[illegible]0
       nC5:                                   0.000       0.000        0.0[illegible]0
        C6:                                   0.000       0.000        0.000
      CH4S:                                   0.000       0.000        0.000
     C2H6S:                                   0.000       0.000        0.0[illegible]0
S vapour as Sx                                0.000       0.000        0.115
S liquid as S1                                0.000       0.000        0.000
                                            ---------   ---------   ---------
        TOTAL                                 0.600       0.597        1.201
- - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - -
TEMPERATURE (F)                              60.0        60.0       1652.4[illegible]
PRESSURE (psig)                              62.00      100.00        40.0[illegible]
HEAT CAPACITY (BTU/(lbmol.R))                 8.24        6.96         9.77
MOLECULAR WEIGHT                             35.11       28.89        31.90
Sx AVERAGE SPECIES NO. (x)                    0.000       0.000        2.0[illegible]2
CUMULATIVE FLOWS (lbmol/hr)
           Feed Stream                        0.600         NA         0.600
           Process Air                        0.000         NA         0.5[illegible]7
              Fuel Gas                        0.000         NA         0.0[illegible]0
CUMULATIVE EFFICIENCIES (%)
            Conversion                        0.00          NA        43.7[illegible]
              Recovery                        0.00          NA         0.0[illegible]
EXCESS PROCESS AIR (%)                         NA           NA       -55.2
----------------------------------------------------------------------------
Cumulative Sulphur Conversion as S1 (lbmol/hr) . . . . . . . . .    0.2[illegible]
```

```
ULSIM  CASE: CNG01-13                                        98/01/19
NG - Test 4; Sample RF13
                         SULPHUR PLANT PERFORMANCE
                                                                 EFFICIENCY
---------------------------------------------------------------------------
   Unit:        Thermal      --------------Catalytic Stage-------------
                  Stage
   Efficiency                                (Percent)
---------------------------------------------------------------------------
   Conversion:
         Unit        42.16
   Cumulative        42.16

     Recovery:
         Unit        99.84
   Cumulative        42.09

Unit Hydrolysis:
          COS         NA
          CS2         NA
---------------------------------------------------------------------------
Overall Recovery Efficiency . . . . . . . . . . . . . . . . .      42.09

                                                                 PRODUCTION
---------------------------------------------------------------------------
   Unit:        Thermal      --------------Catalytic Stage-------------
                  Stage
   Production                                  (lb/hr)
---------------------------------------------------------------------------
   Conversion:
         Unit         7.25
   Cumulative         7.25

     Recovery:
         Unit         7.23
   Cumulative         7.23
---------------------------------------------------------------------------
Total Inlet Sulphur . . . . . . . . . . . . . . . . . . . . .      17.19
```

```
SULSIM  CASE: CNG01-13                                            98/01/19
CNG - Test 4; Sample RF13

                                       O/L Known Reaction Furnace (R[illegible])
-------------------------------------------------------------------------------
                                       FD1          AIR            RF
Parameter                             STREAM       STREAM        STREAM
-------------------------------------------------------------------------------
COMPOSITION (lbmol/hr)
        H2:                            0.000        0.000         0.017
        Ar:                            0.000        0.006         0.0[illegible]6
        O2:                            0.000        0.124         0.0[illegible]0
        N2:                            0.001        0.463         0.464
        C1:                            0.000        0.000         0.0[illegible]0
        CO:                            0.000        0.000         0.0[illegible]1
       CO2:                            0.061        0.000         0.058
        C2:                            0.000        0.000         0.000
       H2S:                            0.536        0.000         0.2[illegible]1
       COS:                            0.000        0.000         0.0[illegible]2
       SO2:                            0.000        0.000         0.007
       CS2:                            0.000        0.000         0.0[illegible]5
       H2O:                            0.000        0.004         0.2[illegible]9
       NH3:                            0.000        0.000         0.000
       HCN:                            0.000        0.000         0.000
        C3:                            0.002        0.000         0.0[illegible]0
       iC4:                            0.000        0.000         0.0[illegible]
       nC4:                            0.000        0.000         0.000
       iC5:                            0.000        0.000         0.0[illegible]0
       nC5:                            0.000        0.000         0.0[illegible]0
        C6:                            0.000        0.000         0.000
      CH4S:                            0.000        0.000         0.0[illegible]0
     C2H6S:                            0.000        0.000         0.0[illegible]0
S vapour as Sx                         0.000        0.000         0.111
S liquid as S1                         0.000        0.000         0.000
                                     ---------    ---------     ---------
        TOTAL                          0.600        0.597         1.2[illegible]2
- - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - -
TEMPERATURE (F)                       60.0         60.0        1627.2[illegible]
PRESSURE (psig)                       62.00       100.00         40.0[illegible]
HEAT CAPACITY (BTU/(lbmol.R))          8.24         6.96          9.74
MOLECULAR WEIGHT                      35.11        28.89         31.8[illegible]
Sx AVERAGE SPECIES NO. (x)             0.000        0.000         2.0[illegible]
CUMULATIVE FLOWS (lbmol/hr)
          Feed Stream                  0.600          NA          0.600
          Process Air                  0.000          NA          0.5[illegible]7
             Fuel Gas                  0.000          NA          0.0[illegible]0
CUMULATIVE EFFICIENCIES (%)
           Conversion                  0.00           NA         42.1[illegible]
             Recovery                  0.00           NA          0.0[illegible]
EXCESS PROCESS AIR (%)                  NA            NA        -53.7
-------------------------------------------------------------------------------
Cumulative Sulphur Conversion as S1 (lbmol/hr)  . . . . . . . . . .  0.2[illegible]
```

```
ULSIM  CASE: CNG01-14                                          98/01/19
NG - Test 4; Sample TG14
                               SULPHUR PLANT PERFORMANCE
                                                                 EFFICIENCY
---------------------------------------------------------------------------
    Unit:        Thermal      --------------Catalytic Stage-------------
                   Stage
    Efficiency                               (Percent)
---------------------------------------------------------------------------
    Conversion:
          Unit        47.65
    Cumulative        47.65

      Recovery:
          Unit        99.85
    Cumulative        47.58

Unit Hydrolysis:
           COS          NA
           CS2          NA
---------------------------------------------------------------------------
Overall Recovery Efficiency . . . . . . . . . . . . . . . . .      47.58

                                                                 PRODUCTION
---------------------------------------------------------------------------
    Unit:        Thermal      --------------Catalytic Stage-------------
                   Stage
    Production                                  (lb/hr)
---------------------------------------------------------------------------
    Conversion:
          Unit         8.19
    Cumulative         8.19

      Recovery:
          Unit         8.18
    Cumulative         8.18
---------------------------------------------------------------------------
Total Inlet Sulphur . . . . . . . . . . . . . . . . . . . . .      17.19
```

```
SULSIM   CASE: CNG01-14                                        98/01/19
CNG - Test 4; Sample TG14

                                          O/L Known Reaction Furnace (R
-----------------------------------------------------------------------
                                          FD1          AIR          RF
Parameter                               STREAM       STREAM      STREAM
-----------------------------------------------------------------------
COMPOSITION (lbmol/hr)
        H2:                              0.000        0.000       0.009
        Ar:                              0.000        0.006       0.0[illegible]5
        O2:                              0.000        0.141       0.0[illegible]0
        N2:                              0.001        0.527       0.528
        C1:                              0.000        0.000       0.000
        CO:                              0.000        0.000       0.0[illegible]1
       CO2:                              0.061        0.000       0.061
        C2:                              0.000        0.000       0.000
       H2S:                              0.536        0.000       0.2[illegible]3
       COS:                              0.000        0.000       0.0[illegible]2
       SO2:                              0.000        0.000       0.006
       CS2:                              0.000        0.000       0.0[illegible]2
       H2O:                              0.000        0.005       0.2[illegible]2
       NH3:                              0.000        0.000       0.000
       HCN:                              0.000        0.000       0.000
        C3:                              0.002        0.000       0.0[illegible]0
       iC4:                              0.000        0.000       0.000
       nC4:                              0.000        0.000       0.000
       iC5:                              0.000        0.000       0.0[illegible]0
       nC5:                              0.000        0.000       0.0[illegible]0
        C6:                              0.000        0.000       0.000
      CH4S:                              0.000        0.000       0.0[illegible]0
     C2H6S:                              0.000        0.000       0.0[illegible]0
S vapour as Sx                           0.000        0.000       0.126
S liquid as S1                           0.000        0.000       0.000
                                       ---------    ---------   ---------
        TOTAL                            0.600        0.680       1.2[illegible]1
- - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - -
TEMPERATURE (F)                          60.0         60.0       1764.2[illegible]
PRESSURE (psig)                          62.00       100.00        40.0[illegible]
HEAT CAPACITY (BTU/(lbmol.R))            8.24         6.96         9.77
MOLECULAR WEIGHT                         35.11        28.89        31.75
Sx AVERAGE SPECIES NO. (x)               0.000        0.000        2.0[illegible]
CUMULATIVE FLOWS (lbmol/hr)
           Feed Stream                   0.600         NA          0.600
           Process Air                   0.000         NA          0.6[illegible]0
              Fuel Gas                   0.000         NA          0.0[illegible]0
CUMULATIVE EFFICIENCIES (%)
            Conversion                   0.00          NA          47.65
              Recovery                   0.00          NA          0.0[illegible]
EXCESS PROCESS AIR (%)                    NA           NA         -48.9
-----------------------------------------------------------------------
Cumulative Sulphur Conversion as S1 (lbmol/hr) . . . . . . . . .   0.2[illegible]5
```

Appendix B-2

Material Balance Calculations
Thermodynamic Equilibrium Results

```
LSIM  CASE: CNG0107S                                         98/01/19
G - Test 1; Thermodynamic Model
                                SULPHUR PLANT PERFORMANCE
                                                                    EFFICIENCY
-----------------------------------------------------------------------------
   Unit:        Thermal      --------------Catalytic Stage-------------
                 Stage
   Efficiency                                (Percent)
-----------------------------------------------------------------------------
   Conversion:
         Unit       64.39
   Cumulative       64.39

     Recovery:
         Unit       99.76
   Cumulative       64.23

nit Hydrolysis:
          COS        NA
          CS2        NA
-----------------------------------------------------------------------------
verall Recovery Efficiency . . . . . . . . . . . . . . . . . .     64.23

                                                                    PRODUCTION
-----------------------------------------------------------------------------
   Unit:        Thermal      --------------Catalytic Stage-------------
                 Stage
   Production                                   (lb/hr)
-----------------------------------------------------------------------------
   Conversion:
         Unit       10.77
   Cumulative       10.77

     Recovery:
         Unit       10.74
   Cumulative       10.74
-----------------------------------------------------------------------------
otal Inlet Sulphur . . . . . . . . . . . . . . . . . . . . . . .     16.72
```

```
SULSIM  CASE: CNG0107S                                              98/01/19
CNG - Test 1; Thermodynamic Model

                                           Thermodynamic Reaction Furnace (R[redacted]
----------------------------------------------------------------------------------
                                                  FD1         AIR            RF
Parameter                                       STREAM      STREAM        STREA[redacted]
----------------------------------------------------------------------------------
COMPOSITION (lbmol/hr)
        H2:                                      0.000       0.000        0.0[redacted]4
        Ar:                                      0.000       0.008        0.0[redacted]8
        O2:                                      0.000       0.177        0.0[redacted]0
        N2:                                      0.006       0.660        0.667
        C1:                                      0.000       0.000        0.0[redacted]0
        CO:                                      0.000       0.000        0.0[redacted]9
       CO2:                                      0.053       0.000        0.049
        C2:                                      0.000       0.000        0.0[redacted]0
       H2S:                                      0.521       0.000        0.1[redacted]1
       COS:                                      0.000       0.000        0.000
       SO2:                                      0.000       0.000        0.014
       CS2:                                      0.000       0.000        0.0[redacted]0
       H2O:                                      0.000       0.006        0.3[redacted]0
       NH3:                                      0.000       0.000        0.000
       HCN:                                      0.000       0.000        0.0[redacted]0
        C3:                                      0.002       0.000        0.0[redacted]0
       iC4:                                      0.000       0.000        0.000
       nC4:                                      0.000       0.000        0.0[redacted]0
       iC5:                                      0.000       0.000        0.0[redacted]0
       nC5:                                      0.000       0.000        0.0[redacted]0
        C6:                                      0.000       0.000        0.000
      CH4S:                                      0.000       0.000        0.0[redacted]0
     C2H6S:                                      0.000       0.000        0.0[redacted]0
S vapour as Sx                                   0.000       0.000        0.167
S liquid as S1                                   0.000       0.000        0.0[redacted]0
                                               ---------   ---------   --------[redacted]
        TOTAL                                    0.583       0.851        1.449
- - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - -
TEMPERATURE (F)                                 60.0        60.0       1894.7[redacted]
PRESSURE (psig)                                 62.00      100.00        14.5[redacted]
HEAT CAPACITY (BTU/(lbmol.R))                    8.23        6.96         9.54
MOLECULAR WEIGHT                                34.95       28.89        31.[redacted]
Sx AVERAGE SPECIES NO. (x)                       0.000       0.000        2.[redacted]4
CUMULATIVE FLOWS (lbmol/hr)
           Feed Stream                           0.583         NA         0.5[redacted]3
           Process Air                           0.000         NA         0.8[redacted]1
              Fuel Gas                           0.000         NA         0.0[redacted]0
CUMULATIVE EFFICIENCIES (%)
            Conversion                           0.00          NA        64.3[redacted]
              Recovery                           0.00          NA         0.[redacted]
EXCESS PROCESS AIR (%)                           NA            NA       -27.3
----------------------------------------------------------------------------------
Cumulative Sulphur Conversion as S1 (lbmol/hr) . . . . . . . . .          0.3[redacted]6
```

```
ULSIM  CASE: CNG0109S                                      98/01/19
NG - Test 2; Thermodynamic Model
                         SULPHUR PLANT PERFORMANCE
                                                             EFFICIENCY
-------------------------------------------------------------------------
   Unit:       Thermal      -------------Catalytic Stage-------------
                 Stage
   Efficiency                             (Percent)
-------------------------------------------------------------------------
   Conversion:
         Unit    70.24
   Cumulative    70.24

     Recovery:
         Unit    99.72
   Cumulative    70.04

Unit Hydrolysis:
          COS      NA
          CS2      NA
-------------------------------------------------------------------------
Overall Recovery Efficiency . . . . . . . . . . . . . . . . . . .   70.04

                                                             PRODUCTION
-------------------------------------------------------------------------
   Unit:       Thermal      -------------Catalytic Stage-------------
                 Stage
   Production                                (lb/hr)
-------------------------------------------------------------------------
   Conversion:
         Unit    13.99
   Cumulative    13.99

     Recovery:
         Unit    13.95
   Cumulative    13.95
-------------------------------------------------------------------------
Total Inlet Sulphur . . . . . . . . . . . . . . . . . . . . . . .   19.91
```

```
SULSIM  CASE: CNG0109S                                            98/01/19
CNG - Test 2; Thermodynamic Model

                                       Thermodynamic Reaction Furnace (R
-------------------------------------------------------------------------
                                             FD1         AIR          RF
Parameter                                  STREAM      STREAM      STREA
-------------------------------------------------------------------------
COMPOSITION (lbmol/hr)
        H2:                                 0.000       0.000       0.047
        Ar:                                 0.000       0.011       0.01
        O2:                                 0.000       0.252       0.000
        N2:                                 0.001       0.939       0.940
        C1:                                 0.000       0.000       0.00
        CO:                                 0.000       0.000       0.01
       CO2:                                 0.070       0.000       0.063
        C2:                                 0.000       0.000       0.000
       H2S:                                 0.621       0.000       0.14
       COS:                                 0.000       0.000       0.000
       SO2:                                 0.000       0.000       0.036
       CS2:                                 0.000       0.000       0.00
       H2O:                                 0.000       0.008       0.44
       NH3:                                 0.000       0.000       0.000
       HCN:                                 0.000       0.000       0.00
        C3:                                 0.002       0.000       0.00
       iC4:                                 0.000       0.000       0.000
       nC4:                                 0.000       0.000       0.000
       iC5:                                 0.000       0.000       0.00
       nC5:                                 0.000       0.000       0.000
        C6:                                 0.000       0.000       0.000
      CH4S:                                 0.000       0.000       0.00
     C2H6S:                                 0.000       0.000       0.00
S vapour as Sx                              0.000       0.000       0.217
S liquid as S1                              0.000       0.000       0.000
                                          ---------   ---------   --------
        TOTAL                               0.695       1.210       1.918
- - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - -
TEMPERATURE (F)                            60.0        60.0       2042.0
PRESSURE (psig)                            62.00      100.00        17.00
HEAT CAPACITY (BTU/(lbmol.R))               8.24        6.96         9.54
MOLECULAR WEIGHT                           35.11       28.89        30.94
Sx AVERAGE SPECIES NO. (x)                  0.000       0.000        2.01
CUMULATIVE FLOWS (lbmol/hr)
           Feed Stream                      0.695        NA          0.695
           Process Air                      0.000        NA          1.21
              Fuel Gas                      0.000        NA          0.000
CUMULATIVE EFFICIENCIES (%)
            Conversion                      0.00         NA         70.24
              Recovery                      0.00         NA          0.00
EXCESS PROCESS AIR (%)                       NA          NA        -12.5
-------------------------------------------------------------------------
Cumulative Sulphur Conversion as S1 (lbmol/hr) . . . . . . . . .   0.43
```

```
ULSIM  CASE: CNG0111S                                      98/01/19
NG - Test 3; Thermodynamic Model
                          SULPHUR PLANT PERFORMANCE
                                                              EFFICIENCY
-----------------------------------------------------------------------------
   Unit:        Thermal      -------------Catalytic Stage-------------
                 Stage
   Efficiency                                (Percent)
-----------------------------------------------------------------------------
   Conversion:
         Unit       49.08
   Cumulative       49.08

     Recovery:
         Unit       99.86
   Cumulative       49.01

nit Hydrolysis:
          COS         NA
          CS2         NA
-----------------------------------------------------------------------------
verall Recovery Efficiency . . . . . . . . . . . . . . . . . .      49.01

                                                              PRODUCTION
-----------------------------------------------------------------------------
   Unit:        Thermal      -------------Catalytic Stage-------------
                 Stage
   Production                                   (lb/hr)
-----------------------------------------------------------------------------
   Conversion:
         Unit        8.44
   Cumulative        8.44

     Recovery:
         Unit        8.42
   Cumulative        8.42
-----------------------------------------------------------------------------
otal Inlet Sulphur . . . . . . . . . . . . . . . . . . . . . .      17.19
```

```
SULSIM  CASE: CNG0111S                                          98/01/19
CNG - Test 3; Thermodynamic Model

                                       Thermodynamic Reaction Furnace (R
------------------------------------------------------------------------
                                            FD1        AIR          RF
Parameter                                  STREAM     STREAM      STREA
------------------------------------------------------------------------
COMPOSITION (lbmol/hr)
        H2:                                 0.000      0.000       0.019
        Ar:                                 0.000      0.006       0.0[illegible]5
        O2:                                 0.000      0.137       0.0[illegible]0
        N2:                                 0.001      0.511       0.512
        C1:                                 0.000      0.000       0.0[illegible]0
        CO:                                 0.000      0.000       0.0[illegible]5
       CO2:                                 0.061      0.000       0.060
        C2:                                 0.000      0.000       0.000
       H2S:                                 0.536      0.000       0.2[illegible]7
       COS:                                 0.000      0.000       0.001
       SO2:                                 0.000      0.000       0.006
       CS2:                                 0.000      0.000       0.0[illegible]0
       H2O:                                 0.000      0.004       0.2[illegible]
       NH3:                                 0.000      0.000       0.000
       HCN:                                 0.000      0.000       0.[illegible]0
        C3:                                 0.002      0.000       0.0[illegible]0
       iC4:                                 0.000      0.000       0.00[illegible]
       nC4:                                 0.000      0.000       0.000
       iC5:                                 0.000      0.000       0.0[illegible]0
       nC5:                                 0.000      0.000       0.0[illegible]0
        C6:                                 0.000      0.000       0.000
      CH4S:                                 0.000      0.000       0.0[illegible]0
     C2H6S:                                 0.000      0.000       0.0[illegible]0
S vapour as Sx                              0.000      0.000       0.130
S liquid as S1                              0.000      0.000       0.000
                                        ---------  ---------  --------
        TOTAL                               0.600      0.658       1.2[illegible]7
- - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - - -
TEMPERATURE (F)                            60.0       60.0      1694.3
PRESSURE (psig)                            62.00     100.00       40.0
HEAT CAPACITY (BTU/(lbmol.R))              8.24       6.96        9.69
MOLECULAR WEIGHT                           35.11      28.89       31.6[illegible]
Sx AVERAGE SPECIES NO. (x)                 0.000      0.000        2.0[illegible]0
CUMULATIVE FLOWS (lbmol/hr)
          Feed Stream                      0.600        NA         0.600
          Process Air                      0.000        NA         0.6[illegible]
             Fuel Gas                      0.000        NA         0.0[illegible]0
CUMULATIVE EFFICIENCIES (%)
           Conversion                      0.00         NA         49.0
             Recovery                      0.00         NA          0.0
EXCESS PROCESS AIR (%)                       NA         NA        -47.8
------------------------------------------------------------------------
Cumulative Sulphur Conversion as S1 (lbmol/hr) . . . . . . . . .   0.2[illegible]
```

Sulsim™

Performance Summary

01/19/98

CNG1-13S: Train1

CNG - Test 4; Thermodynamic Model

	Thermal Stage	Catalytic Stage
EFFICIENCY (%)		
Conversion:		
Unit	44.41	
Cumulative	44.41	
Recovery:		
Unit		
Cumulative		
Unit Hydrolysis:		
COS	NA	
CS2	NA	

Overall Recovery Efficiency . . . . -

	Thermal Stage	Catalytic Stage
PRODUCTION (LTD)		
Conversion:		
Unit	0.08	
Cumulative	0.08	
Recovery:		
Unit		
Cumulative		

Total Inlet Sulphur 0.18

Unit Output Summary

Thermodynamic Reaction Furnace (RFT)

CNG - Test 4; Thermodynamic Model

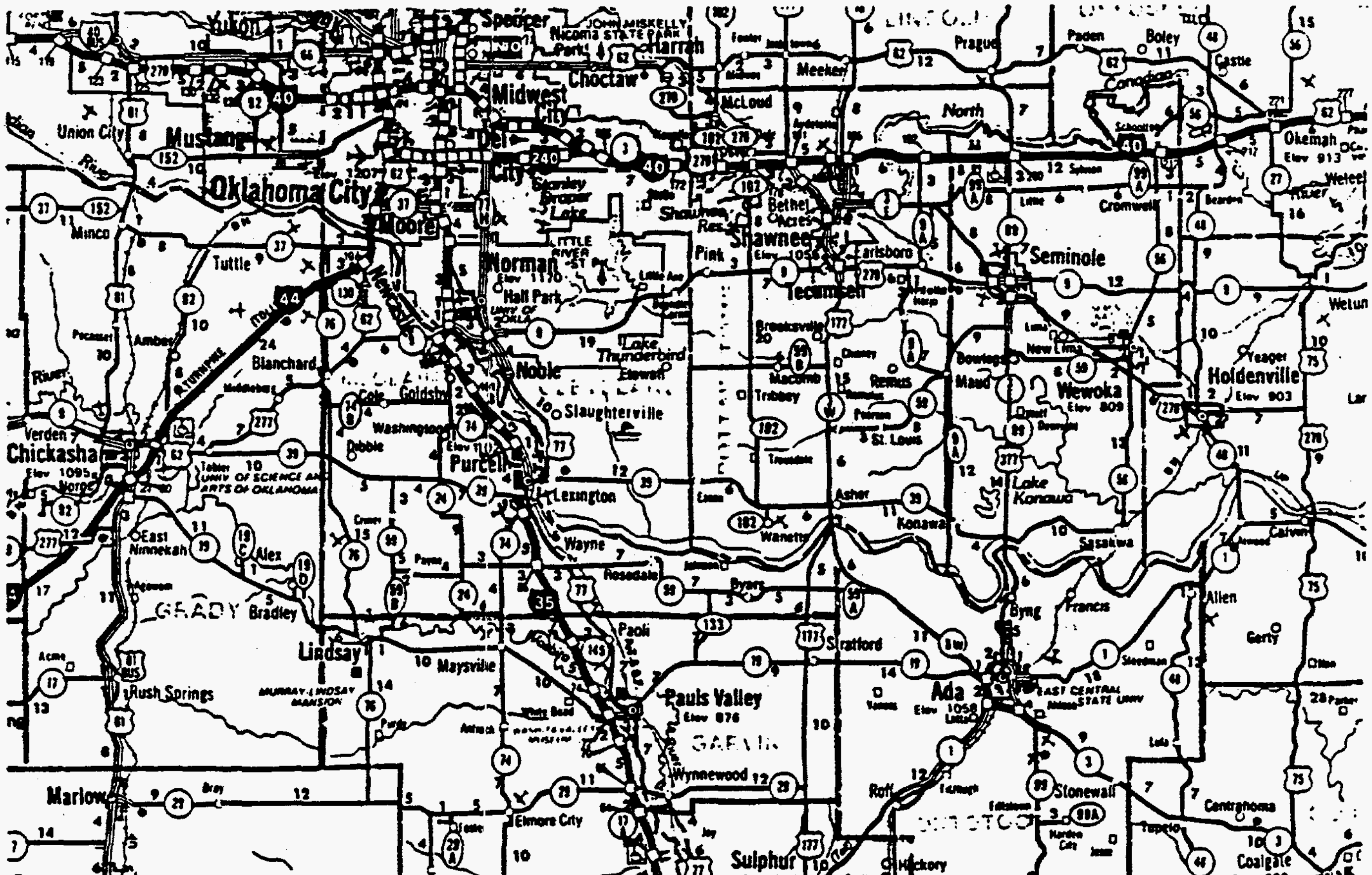
Wet Composition	AG16 (Outlet)	(Air)	RFT (Outlet)
H2	-	-	0.015
Ar	-	0.006	0.006
O2	-	0.124	-
N2	0.001	0.463	0.464
C1	-	-	-
CO	-	-	0.005
CO2	0.061	-	0.061
C2	-	-	-
H2S	0.536	-	0.293
COS	-	-	-
SO2	-	-	0.004
CS2	-	-	-
H2O	-	0.004	0.239
NH3	-	-	-
HCN	-	-	-
C3	0.002	-	-
iC4	-	-	-
nC4	-	-	-
iC5	-	-	-
nC5	-	-	-
C6+	-	-	-
CH4S	-	-	-
C2H6S	-	-	-
S Vapour as Sx	-	-	0.117
S Liquid as S1	-	-	-
Total (lbmol/hr)	0.600	0.597	1.205
Stream Data			
Temperature (F)	60.0	60.0	1623.7
Pressure (psig)	62.000	100.000	40.000
Enthalpy (MMBTU/hr)	-0.02	-	-0.02
Molecular Weight	35.11	28.89	31.80
Sx Avg. Spec. No.	-	-	2.04
Unit Specific Outlet Data			
Air Demand (%)			53.20
S Conv. as S1 (lbmol/hr)			0.24
S Conv. Efficiency (%)			44.41

APPENDIX C

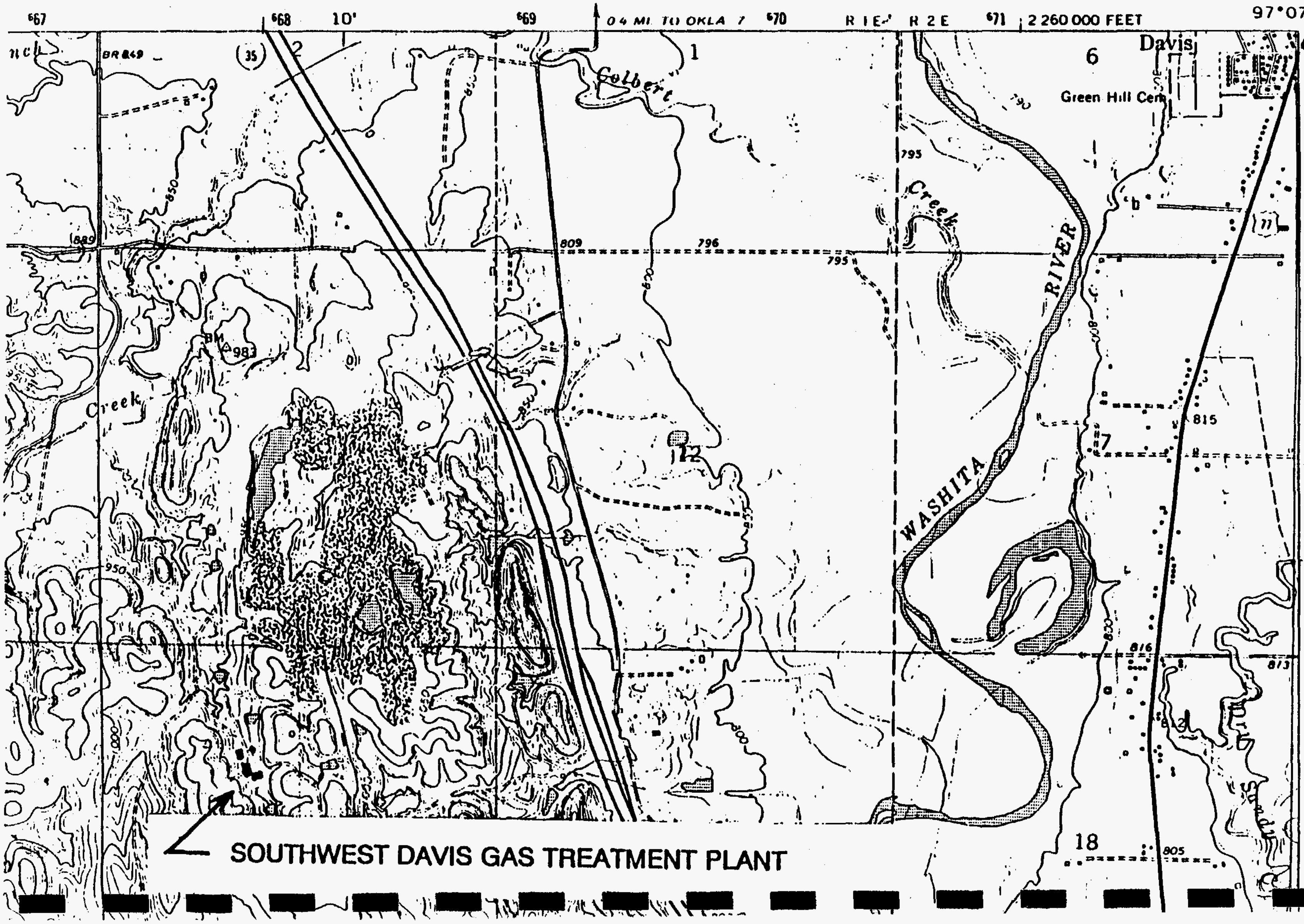
Appendix C

Site Maps and Equipment Photographs

South Central Oklahoma Test Site
Southwest Davis Gas Treatment Plant



Turner Falls Quadrangle (Oklahoma)
Detail of Southwest Davis Gas Treatment Plant Location

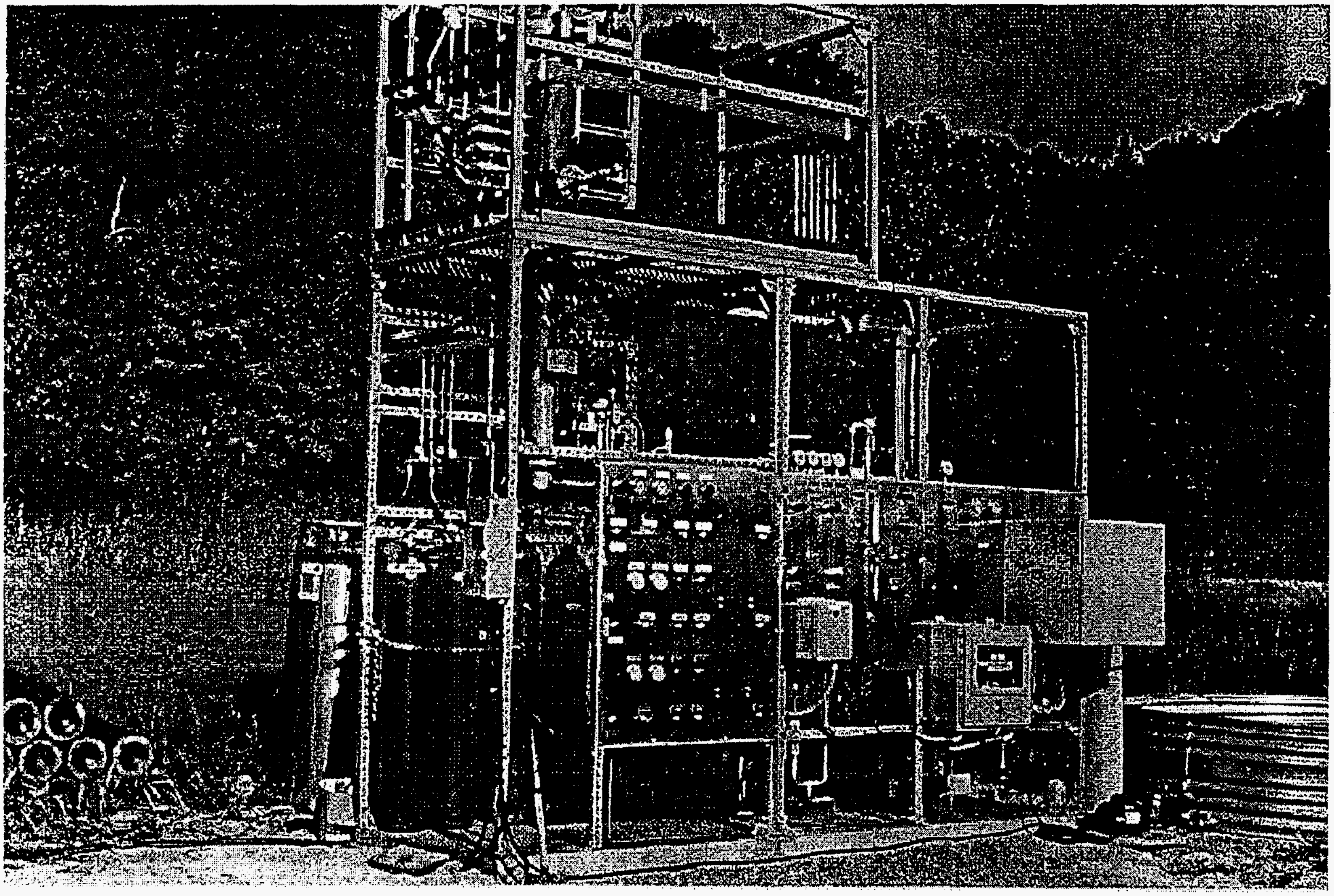


SOUTHWEST DAVIS GAS TREATMENT PLANT

NEPA Information / DE-AC21-92MC29470

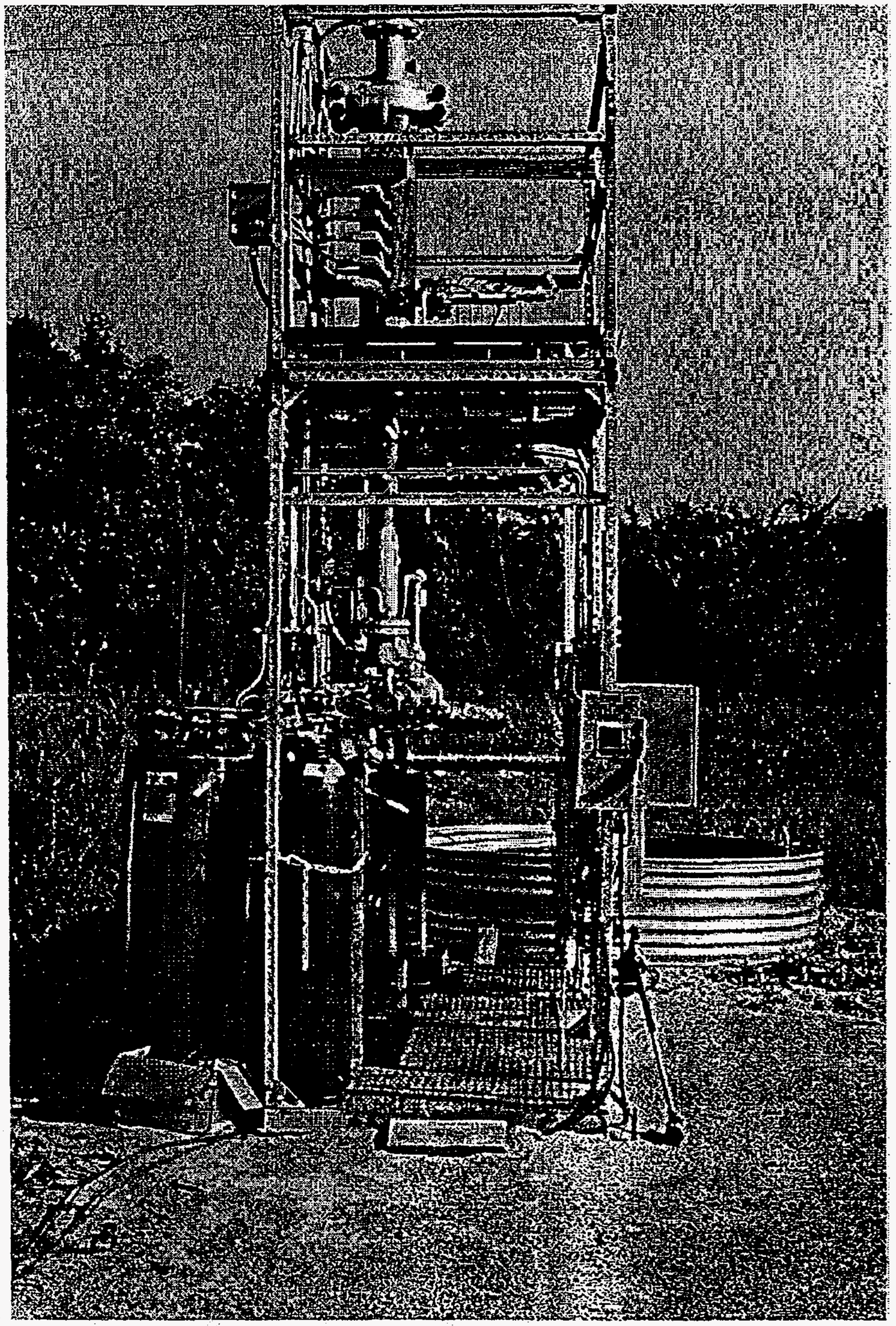
CNG Claus Pilot Plant

Pilot Plant



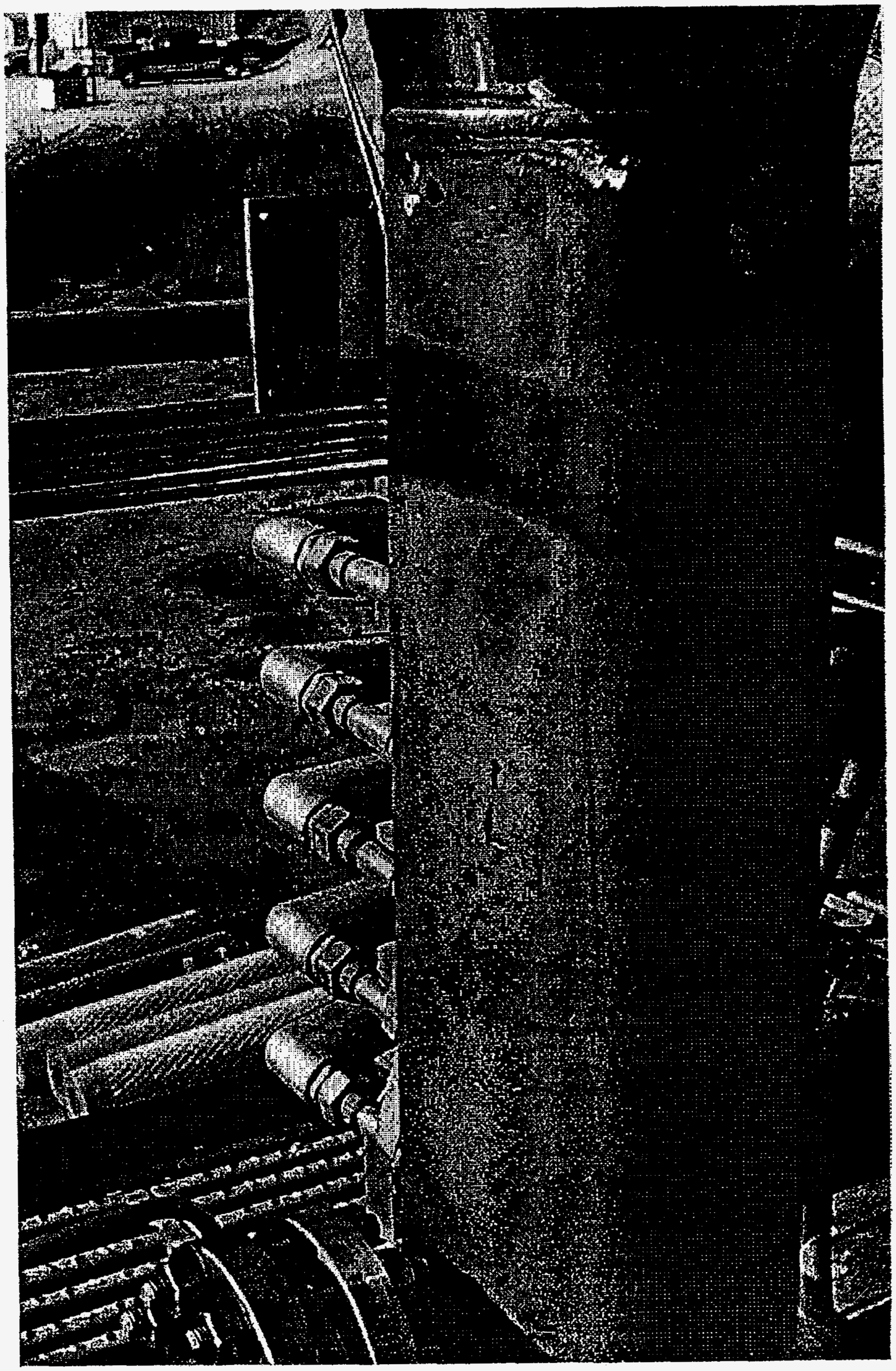
CNG Claus Pilot Plant

Pilot Plant

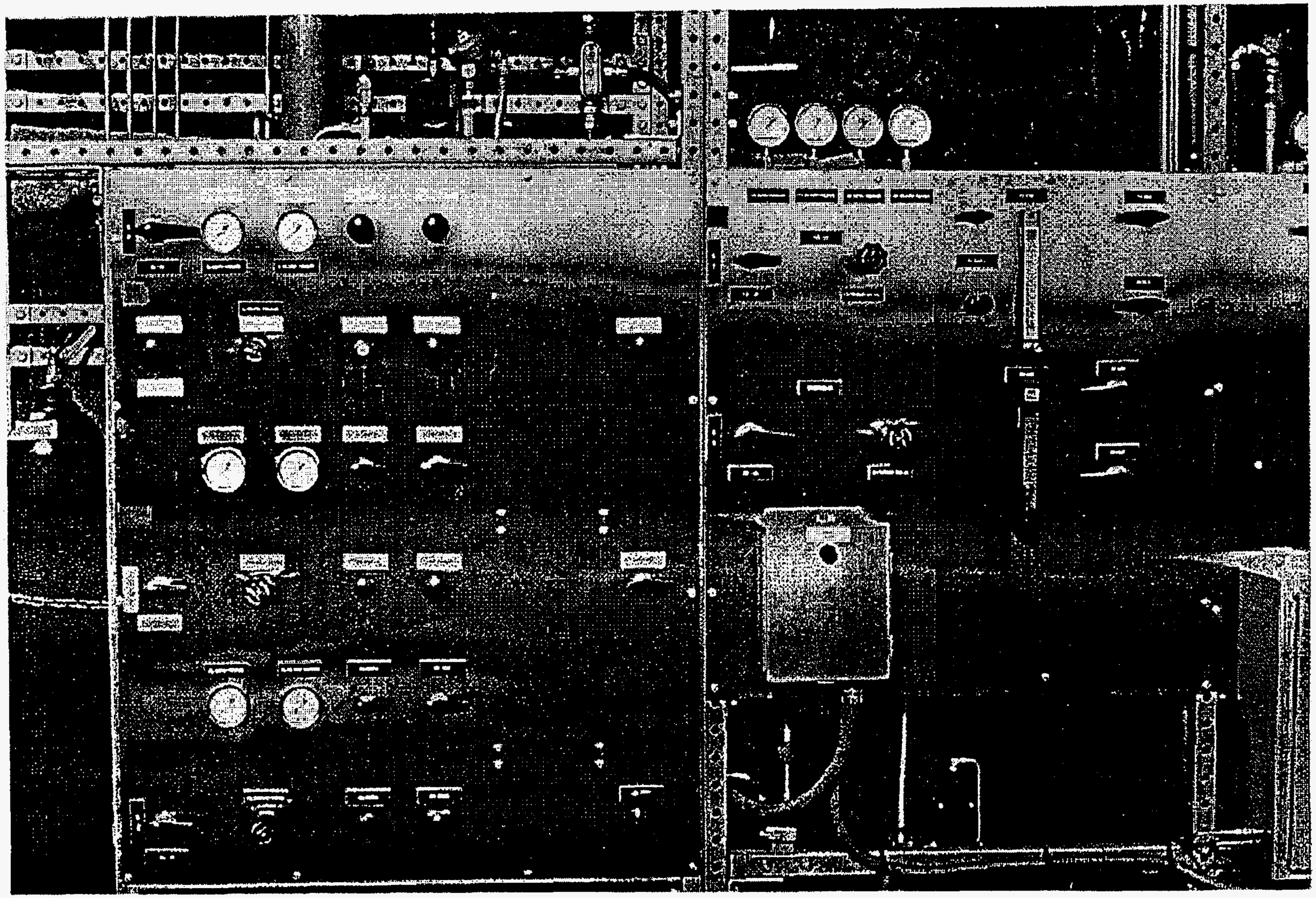


CNG Claus Pilot Plant

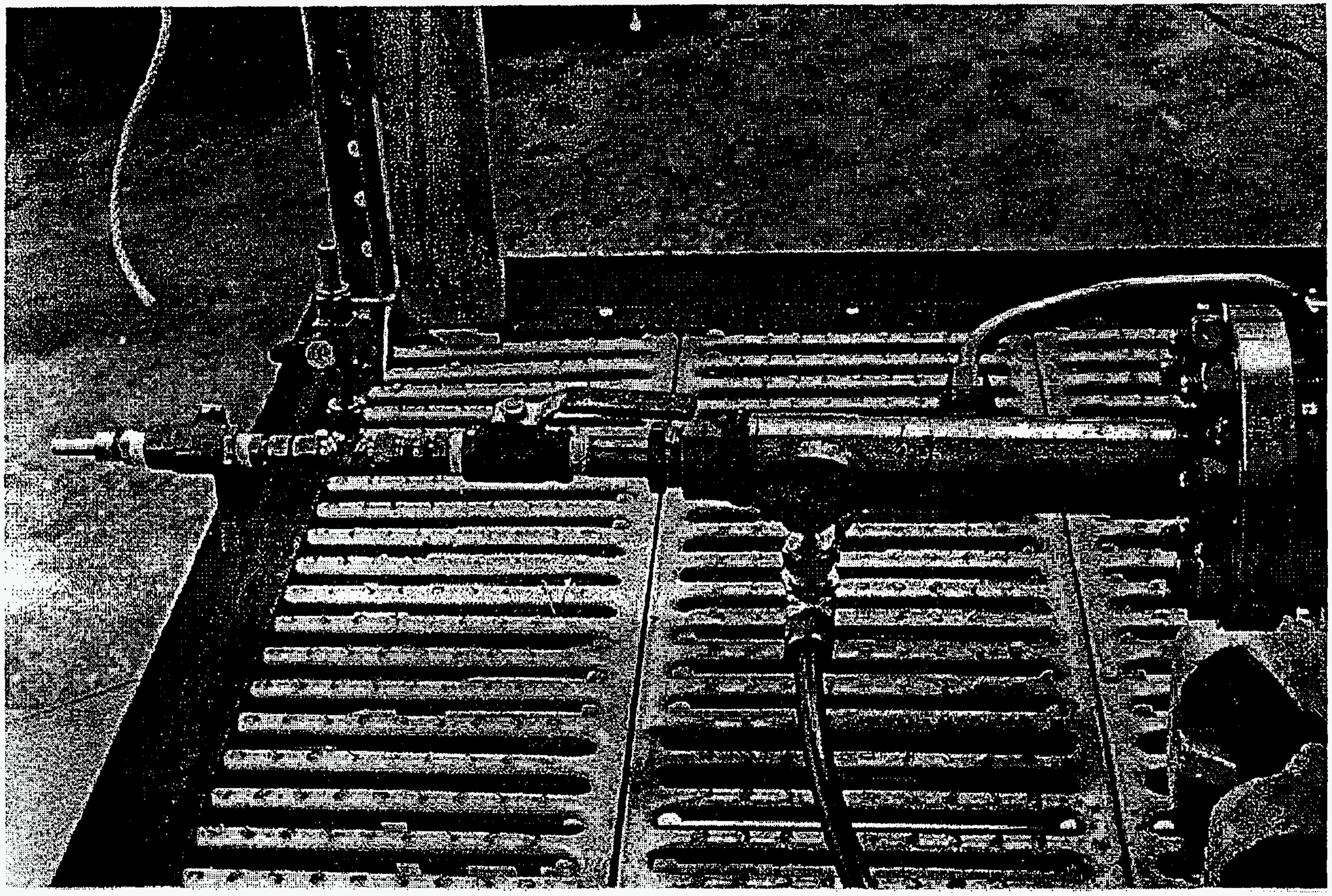
Reaction Furnace



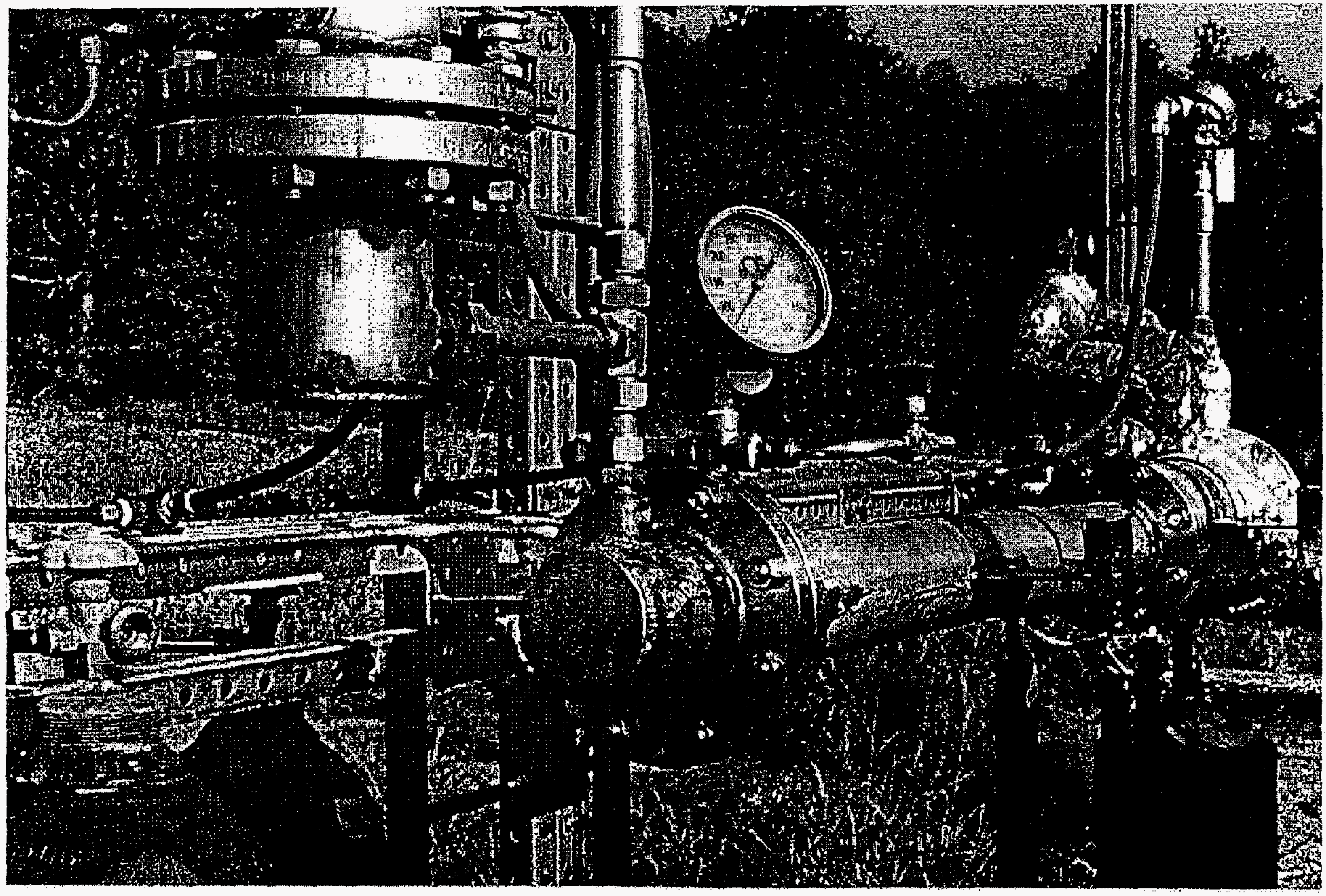
CNG Claus Pilot Plant
Control Panel



CNG Claus Pilot Plant
Water Cooled Sample Probe



CNG Claus Pilot Plant
Sulfur Condenser



CNG Claus Pilot Plant

Sulfur Collector