



Project Summary

Alkaline and Stretford Scrubbing Tests for H₂S Removal from In-situ Oil Shale Retort Offgas

H. J. Taback, G. C. Quartucy, and R. J. Goldstick

Two mobile pilot-plant scrubbers (one alkaline, the other Stretford) were evaluated for removing reduced sulfur compounds from the offgas of an in-situ retort at Geokinetics. The alkaline scrubber efficiency varied inversely with selectivity: at high solution concentration in the tower, 94 percent removal was achieved at a selectivity of 9; and at low concentration in the venturi, the removal was 50 percent and the selectivity was 79. The Stretford achieved 99+ percent removal with the packed tower and 95 percent with the venturi. A computer model of the alkaline scrubber based on the penetration theory was developed and agrees well with the observed performance. Based on this model, it appears possible to design an alkaline scrubber system including a Claus plant which can achieve 95 percent H₂S removal at a selectivity of 37.

This Project Summary was developed by EPA's Air and Energy Engineering Research Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

Removing hydrogen sulfide (H₂S) and other reduced sulfur compounds (carbonyl sulfide, carbon disulfide, mercaptans, thiophenes, etc.) from shale oil retort offgas with a wet scrubber requires a process that will selectively react with

the sulfur compounds and react as little as possible with the carbon dioxide (CO₂) which is present in much larger amounts than the H₂S. Typically the CO₂ concentration in retort gas is 20 percent, while the H₂S concentration ranges from 0.1 percent (1000 ppm) to 4 percent, depending on the retorting process used. This report covers the tests performed on a direct-fired, in-situ (under the ground) retort for which the lower H₂S concentration applies.

Since both H₂S and CO₂ are acid gases, it is the objective of any scrubbing system to selectively remove as much H₂S and other sulfur compounds as possible while minimizing the reaction with the accompanying CO₂. The reasons for this selectivity are to conserve the scrubbing chemicals and to concentrate the sulfur compounds so that they can be economically converted to a solid recoverable (or safely disposable) form.

Two liquid scrubbing concepts were evaluated, alkaline and Stretford. The tests were conducted on the offgas from a horizontal in-situ oil shale retort at the Geokinetics, Inc. (GKI) site in Eastern Utah during the spring of 1984.

Objectives of the GKI tests were:

1. For the Alkali Scrubber Pilot Plant
 - Shake down the equipment.
 - Investigate the influence of operating parameters (e.g., pH, gas/liquid contact time, and scrubbing agent) on the selectivity and removal efficiency associated

with scrubbing reduced sulfur compounds in the presence of high CO₂ concentration.

2. For the Stretford Pilot Plant
 - Duplicate on retort gas the 99+ percent removal efficiency attained in a previous test of offgas from a coal gasifier.
 - Upon achieving that, explain the low removal efficiency on the 1982 test at GKI by deliberately introducing upsetting changes in the plant chemistry, then returning to the 99+ percent performance.

Site and Process Description

The GKI retort offgas is brought to the surface for processing where it is treated in four steps, shown schematically in Figure 1, before it is discharged to the atmosphere: (1) gas passage through a condenser/demister, upstream of the two blowers; (2) ammonia absorption; (3) sulfur recovery; and (4) incineration. Steps 2, 3, and 4 are performed in series, with the treatment units arranged so that the desired treatment configuration can be obtained by bypassing one or more process steps.

Expected operations during the scrubber test were to bypass the ammonia absorber and treat the gas in the sulfur recovery unit and the incinerator. A maximum of 10 Sm³/s of gas at a maximum temperature of 82°C can be treated in the GKI gas processing operation.

Findings

Alkaline Scrubber

The alkaline scrubber system was operated using both the tray tower and the venturi as the gas/liquid contactor. After relocating the main blower, the equipment performed satisfactorily.

The alkaline scrubber was operated in a simple blowdown process where the alkali solutions were mixed to a specific concentration and fed into either the tray tower or venturi contactors. In a commercial process unit, the scrubber solution would be cycled through a stripper where the absorbed H₂S and CO₂ would be removed. Then the solution would be returned to the original mixing tanks and recycled into the contactor. No significant alkali addition would be required in that case. Since a

stripper was not included as part of the EPA pilot plant, the scrubbing solution was used on a once-through basis, then discharged to the GKI pond.

The experimental results for the alkaline scrubber are summarized in Table 1 and Figure 2. The runs were conducted using alternately the tower then the venturi at the same solution concentration. Three different solution concentrations were used for each alkali except for the last four runs (No. 31-34) where only the tower was used to make two high concentration runs for both NaOH and KOH.

To analyze these data, a computer model of an alkaline scrubber was developed employing the comprehensive penetration theory. Penetration theory treats the gas/liquid mass transfer to allow contact time to be a significant factor. Other models (e.g., the two-plane theory) have implicit assumptions of equilibrium and cannot account for the contact time difference between a tower and a venturi. The results predicted by the penetration theory agree with the experimental results.

Based on the experimental results and the computer model, an alkaline

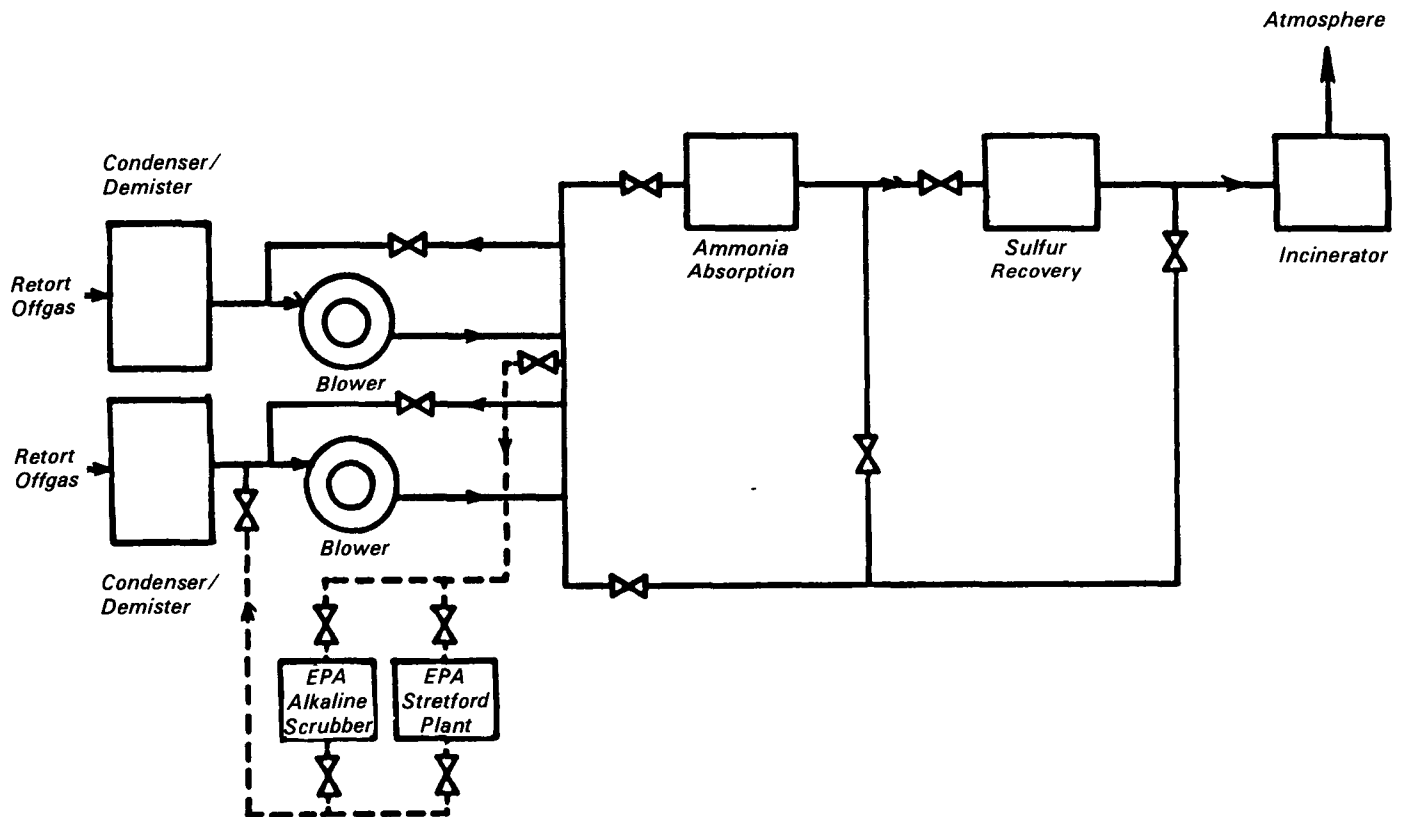


Figure 1. Pilot plant installation in the Geokinetics process.

scrubbing system design concept is suggested which could achieve an H₂S removal efficiency of 95 percent with a selectivity approaching 40. This is a two-stage scrubber with a venturi-contactor first stage and a tray-tower second stage. The first stage removes 50 percent of the H₂S in a highly selective manner. The second stage removes 90 percent of the remaining H₂S at a lower selectivity. Summarized, these performance values are:

TWO-STAGE ALKALINE SCRUBBER - CONCEPT I

Stage No	I	II	Combined
Contactor	Venturi	Tray Tower	
Selectivity Removal	110	40	37
Efficiency, %	50	90	95

Another concept, employing a two-stage tray tower scrubber which results in a higher removal efficiency but a lower selectivity, is summarized as:

Table 1. Summary of Alkali Scrubbing Results

Contactor	Alkali	OH ⁻ Conc. gmole/liter	Removal Efficiency, %	Measured Selectivity ^a	Run No.
Venturi	NaOH	0.012	52	79	21
Venturi	KOH	0.012	53	71	28
Venturi	NaOH	0.023	48	60	24
Venturi	KOH	0.023	48	51	30
Venturi	NaOH	0.045	70	84 ^b	19
Venturi	KOH	0.046	71	21	26
Venturi	NH ₄ OH	0.049	60	71	15
Venturi	NH ₄ OH	0.29	62	56	17
Venturi	NH ₄ OH	2.0	67	11	13
Tower	NaOH	0.012	52	52	20
Tower	KOH	0.012	54	43	27
Tower	NaOH	0.023	54	41	22
Tower	KOH	0.023	59	49	29
Tower	NaOH	0.045	83	36	18
Tower	KOH	0.046	88	41	25
Tower	NH ₄ OH	0.049	64	29	14
Tower	NH ₄ OH	0.29	91	29	16
Tower	NH ₄ OH	2.0	93	9	12
Tower	KOH	0.89	94	N/A ^c	31
Tower	NaOH	1.25	93	N/A	33
Tower	KOH	1.79	92	N/A	32
Tower	NaOH	2.5	94	N/A	34

^aSelectivity is a measure of the preferential removal of H₂S over CO₂, taking into account the relative difference in concentration between the two gases. In this report, selectivity is the ratio of percent removal of H₂S to percent removal of CO₂.

^bData are suspected to be erroneous.

^cSelectivity value was not available because the spent scrubbing solution was not analyzed.

ALKALINE TOWER SCRUBBER - CONCEPT II

Stage No	I	II	Combined
Contactor	Tray Tower	Tray Tower	
Selectivity Removal	40	40	23
Efficiency, %	90	90	99

This two-stage tray tower scrubber can be combined into a single tower of double length.

The alkaline scrubber showed little removal of the organic sulfur compounds. This is similar to previous results reported in the literature.

Stretford Plant

The Stretford operated for over 200 hours. Table 2 gives available measurements of H₂S removal efficiency and pertinent process data taken in conjunction with the removal efficiency. For 140 hours, the plant operated with a venturi contactor, modified from that used in previous tests in that the throat area could be adjusted to handle variable gas flow rates. In this test, the throat was

adjusted to the smallest throat area, 7.1 in. (18 cm), and held constant during most of the testing.

The maximum H₂S removal efficiency measured while using the venturi alone was 95 percent, which was maintained only briefly. Over the period of operation with this contactor alone the removal efficiency averaged 80 percent. A brief attempt was made to experiment with increasing the venturi throat area. When no effect on removal efficiency was observed, the throat area experiment was discontinued.

Because of the failure of the plant to achieve the 99+ percent removal efficiency objective observed in the previous coal gasifier test, a field-fabricated, packed-column contactor was added in series with and downstream of the venturi. This device increased the removal efficiency to the 99+ percent range during its period of operation. Because of the makeshift nature of this field modification, there was no instrumentation to measure the flow rate of the scrubber liquid through the tower. Thus, it was not possible to optimize liquid distribution between the venturi and the tower.

Conclusions

Based on the findings reported here, the following conclusions were reached:

1. For shale oil retort offgas similar in composition to that from the GKI process, the alkaline scrubber, in combination with a stripper and a Claus plant, could be a viable means of H₂S removal. This overall conclusion is based on the following conclusions.
2. For GKI-type process offgas and based on these tests, the performance of an alkaline scrubber with a tray tower contactor similar to that in the EPA pilot plant can achieve an H₂S removal efficiency of at least 90 percent with a selectivity of approximately 30. Under the same conditions a single venturi contactor in place of the tray tower would remove only 50 to 60 percent H₂S, but with a selectivity of 70 to 80.
3. Based on the computer model developed to analyze these test results, the removal efficiencies and selectivity above are applicable to offgas with lower H₂S concentrations than found at GKI. This suggests a concept of multiple scrubbing actions to increase the H₂S

removal. Because this increased removal efficiency is accompanied by a reduced selectivity which could present a problem for the Claus plant, the cost effectiveness of this concept requires a design study.

4. Based on a three gas component (H_2S , NH_3 , and CO_2) analysis by the computer program, the principal reactant for the H_2S in the retort offgas is the NH_3 in that same offgas. In that NH_3 is present in the GKI offgas in similar molar quantities to that of the H_2S , the scrubber performance observed on these tests may not be applicable to retort offgas with little or no NH_3 . This suggests that water with just the NH_3 in the gas stream would be an effective scrubbing agent. Scrubbing in this manner would certainly improve the selectivity; but, the effect on removal achievable is uncertain.
5. The alkaline scrubber removal efficiency and selectivity seemed to have little dependency on the alkali used. This is consistent with the above concept that it is the NH_3 in the offgas itself that is reacting the H_2S . Since the NH_3 and H_2S concentrations are variable, it is likely that some of the H_2S is reacted by the alkali. Therefore, it is likely some alkali will always be needed. However, the choice of scrubbing alkali may be made on such factors as cost, maintenance, safety, availability, and crew comfort (rather than performance).
6. The absorption of H_2S and CO_2 in the alkaline solution appears to be fully reversible by distillation. The sulfur in the scrubber solution is primarily in the form of sulfide. The sulfate or sulfite level determined in the scrubbing solution was equal to that in the water supply. The sulfide will distill off as H_2S (along with CO_2) while the sulfate will not.
7. With an adequate contactor the Stretford process can obtain H_2S removal efficiencies of 99 percent. These tests suggest that, if adequate H_2S removal cannot be achieved with a venturi, a packed tower is a workable option for improving performance.
8. To ensure continued satisfactory performance of a Stretford plant in processing retort offgas, it is im-

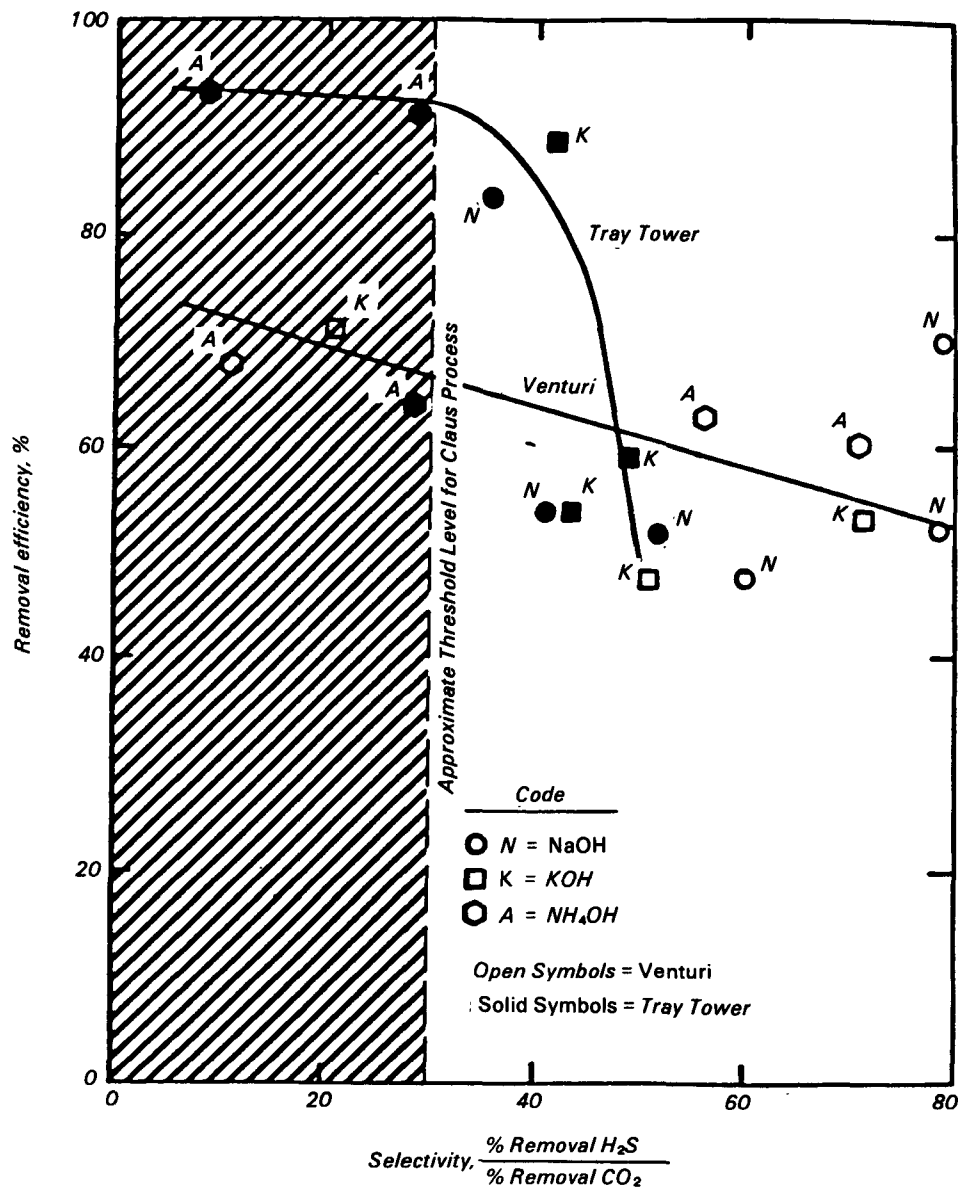


Figure 2. Removal efficiency vs. selectivity for alkaline scrubber.

portant to effectively remove the hydrocarbon mist and other particulate matter from the gas before it enters the plant.

Table 2. Stretford Operating Conditions and Removal Efficiency During GKI Test Program

Date 1984	Time	Gas Contct Device	Removal Eff %	Gas Flow Rate Sm ³ /s	Gas Out Temp °C	Gas Inlt Pres mm Hg	Inlt H ₂ S Conc ppmV	Out H ₂ S Conc ppmV	Sol Flow Rate S1/s	Sol Htr T in °C	Sol Htr T out °C	Oxidizer		ADA Conc kg/m ³	Van. Conc kg/m ³	Carb. Conc kg/m ³	Thio- sul. Conc kg/m ³	pH	Oxidation Potential mV
												Air Flow Sm ³ /s	Motor Curr amps						
5/5	18:05	V	75.2	0.190	40	27.8	1617	401	1.93	43	40	0.036	7.5	7.5	2.3	16.8	0.2	10.8	+38
	18:55	V	74.7	0.255	41	30.4	1579	400	1.91	43	43	0.035	7.3	7.5	2.2	16.4	0.2	10.5	+19
	20:30	V	73.7	0.233	43	30.4	1633	429	1.79	44	44	0.029	7.4	7.5	2.2	15.8	0.2	10.2	0
	21:45	V	62.1	0.256	40	30.4	1571	595	1.77	43	43	0.029	7.4	8.7	2.5	15.3	0.2	9.9	-21
	22:35	V	51.7	0.269	43	30.4	1322	639	1.86	46	44	0.026	7.3	8.7	2.4	14.9	0.2	9.9	-15
	23:30	V	61.3	0.269	44	32.9	1618	626	1.54	47	47	0.026	7.3	8.7	2.4	14.5	0.2	9.9	-8
5/6	00:30	V	72.7	0.272	44	29.3	1623	443	1.60	47	47	0.026	7.3	8.7	2.4	14.1	0.2	9.8	-2
	01:30	V	79.5	0.192	38	29.9	2169	444	1.48	47	41	0.026	7.4	8.6	2.4	13.7	0.2	9.8	+5
	02:30	V	90.3	0.327	44	24.3	2018	195	1.48	46	46	0.025	7.4	8.6	2.3	13.3	0.2	9.1	+4
	03:30	V	93.4	0.254	45	30.4	1886	125	1.51	46	46	0.024	7.3	8.6	2.3	12.9	0.2	8.4	+2
	04:25	V	94.3	0.189	46	30.4	1741	100	1.48	47	47	0.024	7.3	8.5	2.3	12.5	0.3	7.7	+1
	05:30	V	93.7	0.189	47	30.9	1590	101	1.50	48	47	0.026	7.3	8.5	2.2	12.0	0.3	7.0	0
5/7	21:21	V	63.0	0.276	46	60.1	763	282	1.67	46	46	0.039	7.5	10.0	3.3	16.0	0.5	10.0	-50
	22:02	V	70.4	0.206	43	65.8	1157	343	1.64	47	46	0.038	7.4	9.9	3.3	15.9	0.5	9.9	-37
5/8	18:23	V	84.5	0.280	42	43.0	1369	212	1.80	43	41	0.034	7.5	8.2	3.3	30.0	0.6	7.4	-39
	20:00	V	94.5	--	--	--	1372	75	--	--	--	--	--	8.0	3.3	30.2	0.6	7.4	-33
	22:05	V	76.8	--	--	--	1378	320	--	--	--	--	--	7.7	3.3	30.4	0.6	7.4	-27
	22:17	V	81.1	0.483	37	53.1	1392	263	1.74	39	36	0.030	7.4	7.6	3.3	30.4	0.6	7.4	-21
	23:12	V	80.2	0.489	37	70.8	1395	276	1.71	39	34	0.034	7.4	7.5	3.3	30.5	0.6	7.8	-21
	23:45	V	80.8	0.443	37	60.7	1398	269	1.73	39	34	0.034	7.4	7.4	3.3	30.6	0.6	8.2	-21
5/9	00:45	V	81.4	0.443	36	65.8	1399	260	1.72	39	35	0.034	7.3	7.3	3.3	30.7	0.6	8.9	-21
	16:10	V	85.6	0.208	42	140.2	1689	243	1.81	43	43	0.039	7.3	4.6	3.2	30.1	0.7	9.1	-69
	16:30	V	82.8	0.218	42	146.7	1700	293	1.82	43	43	0.038	7.3	4.6	3.2	30.1	0.7	8.9	-78
	18:02	V	86.1	0.215	42	126.5	1818	253	1.82	43	43	0.038	7.4	4.6	3.2	29.9	0.7	8.6	-87
	20:20	V	82.7	0.205	42	126.5	1768	306	1.90	43	43	0.038	7.4	4.6	3.2	29.6	0.7	9.9	-75
5/10	00:30	V	82.9	--	39	32.9	1761	301	1.93	43	39	0.030	7.4	5.5	3.5	31.5	0.7	9.7	-42
	17:16	V	80.7	0.189	38	15.2	1362	263	1.36	41	39	0.025	7.7	9.9	3.4	30.1	0.8	9.4	-54
	17:48	V	80.8	0.247	38	15.2	1330	255	1.36	41	39	0.024	7.7	9.9	3.4	29.9	0.8	9.4	-48
	18:27	V	80.9	0.190	37	15.2	1305	249	1.36	40	39	0.024	7.7	9.8	3.4	29.8	0.8	9.5	-42
	19:30	V	81.1	0.267	40	15.2	1283	243	1.35	41	42	0.024	7.7	9.7	3.4	29.5	0.8	9.5	-46
	20:15	V	81.1	0.298	40	15.2	1270	240	1.36	41	42	0.020	7.7	9.7	3.3	29.3	0.8	9.4	-51
	21:28	V	80.9	0.189	40	15.2	1259	240	1.36	42	42	0.019	7.7	9.6	3.3	28.9	0.8	9.4	-55
	22:15	V	81.3	0.220	41	17.7	1255	235	1.36	43	43	0.019	7.8	9.5	3.3	28.7	0.8	9.5	-51
	23:21	V	81.0	0.189	42	17.7	1250	237	1.38	44	44	0.019	7.7	9.5	3.3	28.4	0.8	9.7	-48
5/11	00:30	V	81.2	N/A	42	12.7	1248	235	1.36	43	44	0.024	7.7	9.4	3.3	28.1	0.8	9.8	-44
	01:30	V	79.7	N/A	39	12.7	1134	230	1.36	43	41	0.019	7.7	9.3	3.3	27.8	0.8	10.0	-41
	02:30	V	80.7	N/A	39	12.7	1250	241	1.36	43	41	0.027	7.7	9.2	3.3	27.5	0.8	10.1	-37
	03:30	V	80.6	N/A	39	12.7	1232	239	1.38	43	41	0.017	7.7	9.1	3.3	27.3	0.8	9.9	-23
	04:35	V	81.8	N/A	40	12.7	1242	226	1.67	42	42	0.017	7.7	9.1	3.2	27.0	0.8	9.6	-10
	05:30	V	81.3	N/A	40	10.1	1240	232	1.53	42	41	0.021	7.7	9.0	3.2	26.7	0.8	9.4	+4
	06:35	V	84.3	N/A	39	10.1	1236	194	2.12	43	41	0.021	7.7	8.9	3.2	26.7	0.8	9.4	0
5/12	13:30	V&T	99.1	N/A	38	19.5	1024	9	1.63	42	42	0.026	7.8	7.5	3.0	30.9	1.0	9.3	+2
	14:30	V&T	99.2	N/A	41	19.5	1036	8	1.64	43	45	0.027	7.8	7.5	3.0	30.7	1.0	9.3	-15
	15:30	V&T	99.1	N/A	38	18.2	985	9	1.60	42	42	0.029	7.7	7.4	2.9	30.4	1.0	10.1	-15
	16:22	V&T	98.8	N/A	38	25.3	1001	12	1.59	42	43	0.029	7.5	7.3	2.9	30.2	1.0	10.8	-16
	17:19	V&T	98.7	N/A	38	22.8	1138	15	1.61	42	42	0.029	7.7	7.3	2.9	30.0	1.0	11.6	-16
	18:25	V&T	98.8	N/A	39	22.8	1199	14	1.62	42	43	0.027	7.7	7.2	2.8	29.7	1.0	12.3	-17
	19:19	V&T	99.1	N/A	39	25.3	1171	11	1.69	42	43	0.031	7.7	7.1	2.8	29.5	1.0	11.5	-22
	20:18	V&T	99.0	N/A	38	25.3	1218	12	1.70	42	42	0.031	7.7	7.1	2.7	29.3	1.0	10.7	-27
	21:25	V&T	99.3	N/A	39	25.3	1237	9	1.86	42	42	0.033	7.6	7.0	2.7	29.0	1.0	9.9	-32
	22:24	V&T	99.4	N/A	100	27.8	1232	8	1.96	43	41	0.035	7.5	6.9	2.6	28.7	1.0	9.8	-23
	23:21	V&T	99.4	N/A	39	25.3	1228	7	2.05	42	41	0.035	7.6	6.8	2.6	28.5	1.0	9.6	-13

Table 2. (Continued)

Date 1984	Time	Gas Contct Device	Removal Eff %	Gas Flow Rate Sm ³ /s	Gas Out Temp °C	Gas Inlt Pres mm Hg	Inlt H ₂ S Conc ppmV	Out H ₂ S Conc ppmV	Sol Flow Rate S1/s	Sol Htr T in °C	Sol Htr T out °C	Oxidizer		ADA Conc kg/m ³	Van. Conc kg/m ³	Carb. Conc kg/m ³	Thio- sul. Conc kg/m ³	pH	Oxidation Potential mV
												Air Flow Sm ³ /s	Motor Curr amps						
5/13	15:30	V*	86.9	N/A	38	20.0	990	130	1.88	39	41	0.042	7.6	7.6	2.1	--	1.1	9.9	-9
	16:12	V*	84.3	N/A	39	25.3	797	125	1.86	41	41	0.043	7.6	9.7	2.8	--	1.1	10.1	-19
	17:17	V*	83.3	N/A	39	25.3	749	125	1.79	42	42	0.044	7.6	9.7	2.7	--	1.1	10.4	-28
	18:57	V*	85.4	0.250	39	25.3	940	137	1.82	40	41	0.043	7.6	9.7	2.6	--	1.1	10.6	-23
	19:32	V*	86.2	0.251	38	25.3	1001	138	1.86	40	42	0.039	7.7	11.0	3.0	--	1.1	10.7	-18
	20:16	V*	86.9	0.251	38	25.3	1063	139	1.83	42	42	0.038	7.6		3.0	--	1.1	10.9	-13
	21:13	V*	87.3	0.251	38	25.3	1094	139	1.83	43	43	0.037	7.6		3.0	--	1.1	--	--
	22:18	V*	87.4	0.250	39	26.3	1113	140	1.87	43	43	0.036	7.6		2.9	--	1.1	--	--
	23:25	V*	87.6	0.250	39	22.8	1119	139	1.82	44	43	0.036	7.6		2.9	--	1.1	--	--
5/14	00:00	V*	87.7	0.250	38	22.8	1125	138	1.83	44	43	0.036	7.6	11.0	2.8	--	1.1	--	--
	01:00	V*	87.9	0.211	36	22.8	1133	137	1.80	40	38	0.033	7.6	11.0	2.8	--	1.1	--	--
	02:00	V*	87.9	0.210	36	22.8	1133	137	1.77	40	41	0.031	7.6	11.0	2.7	--	1.1	--	--
	03:00	V*	88.3	0.210	36	22.8	1128	132	2.02	41	42	0.031	7.6	11.0	2.7	--	1.1	--	--
	04:00	V&T	95.1	0.210	37	22.8	1116	55	2.08	41	42	0.028	7.6	11.0	3.1	--	1.1	--	--
	05:00	V&T	95.0	0.210	36	22.8	1127	56	2.05	38	37	0.031	7.6	11.0	3.0	--	1.1	--	--
	06:00	V&T	94.5	0.210	37	22.8	1115	61	2.05	40	39	0.033	--	11.0	3.0	--	1.1	--	--
	07:00	V&T	94.4	0.192	37	22.0	1106	62	2.27	39	39	0.033	7.6	11.0	2.9	--	1.1	--	--
	07:30	V&T	97.3	0.182	37	17.5	1101	30	2.26	39	39	0.034	7.7	--	--	--	1.1	--	--
	08:15	V&T	98.7	0.182	--	17.5	1091	14	2.26	39	39	0.034	7.7	--	--	--	1.1	--	--

V = venturi.

V&T = venturi plus packed tower.

*On these tests, the packed tower was in place but no solution was flowing to it.

H. Taback and G. Quartucy are with KVB, Inc., Irvine, CA 92714; and R. Goldstick is with Energy Design Service, Ojai, CA 93023.

Edward R. Bates is the EPA Project Officer (see below).

The complete report, entitled "Alkaline and Stretford Scrubbing Tests for H₂S Removal from In-Situ Oil Shale Retort Offgas," (Order No. PB 85-246 965/AS; Cost: \$22.95, subject to change) will be available only from:

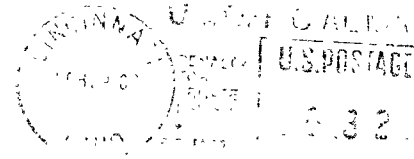
National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
Telephone: 703-487-4650

The EPA Project Officer can be contacted at:

Air and Energy Engineering Research Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

United States
Environmental Protection
Agency

Center for Environmental Research
Information
Cincinnati OH 45268



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