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# MARTIN MARIETTA CORPORATION

DENVER DIVISION

ENGINEERING PROPULSION LABORATORY

NITROGEN TETROXIDE VAPOR SCRUBBER USING A RECIRCULATING LIQUID

PREPARED BY:

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Prepared for Presentation at the National Aeronautics and Space Administration/Florida Institute of Technology Conference on Environmental Engineering for Nitrogen Tetroxide  $(N_2^{0} _4)$ .

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#### FOREWORD

The work described herein was conducted by Martin Marietta Corporation, Denver Divison, under NASA Contract NAS10-9095, under the management of the NASA Project Manager, Mr. H. H. Franks, Spacecraft and Storables Section, NASA-Kennedy Center, Florida.

#### 1.0 INTRODUCTION

The Martin Marietta Corporation was placed on contract by the John F. Kennedy Space Center (KSC) in January 1977, to design, manufacture, test and deliver four (4) Hypergolic Oxidizer Vapor Scrubbers. Delivery of the scrubbers was completed in October 1977.

The scrubbers are required to reduce  $N_2O_4$  contamination of nitrogen vent gas streams to a safe level to preclude health hazard to personnel and to preclude adverse environmental effects. The scrubber principle involved is to absorb and neutralize the  $N_2O_4$ component in a closed circuit circulating water/chemical solution in a vertical counter-flow, packed-tower configuration.

The operational and performance test requirements for the scrubbers are defined in KSC Specification No. 79K08492. Basically, this consists of demonstrating that the exit gas contamination level from the scrubbers does not exceed 150 ppm oxidizer under any flow conditions up to 400 scfm with inlet concentrations of up to 100,000 ppm oxidizer.

Early developmental work involving chemical analysis and scrubber liquor selection was performed by the Florida Institute of Technology (FIT). Some of this work consisting of prototype testing was done by Martin Marietta under contract to the FIT.

The basic scrubber concept design was developed by the Planning Research Corporation (PRC) for KSC. This design was identified in KSC Specification No. 79K08492, which specified the functional and physical characteristics of the scrubbers, including: scrubber liquors and concentrations, sump liquor flow rates, scrubber capacity, physical size and shape, construction material, environmental conditions, and quality conformance inspections involving the use of Drager tubes for concentration measurements. This specification provided the design criteria for the detail design produced by Martin Marietta.

During the detail design development, it was evident that several concept changes were required. These were coordinated with KSC and incorporated.

Several problems were encountered during the performance testing that led to a series of investigations and supplementary testing. It was finally necessary to change the scrubber liquors in oxidizer scrubber to successfully achieve performance requirements.

This report provides a description of the scrubbers, thetest configuration, and the various tests performed. Schedule limitations precluded performing the complete test matrix using the final scrubber liquor. Sufficient testing was accomplished, however, to demonstrate that the oxidizer scrubbers fully comply with the performance specification. An Oxidizer Hypergolic Toxic Vapor (HTV) Scrubber has been developed at the Martin Marietta Engineering Propulsion Laboratory in Denver, Colorado. Performance testing was conducted during May through July 1977.

The purpose of the test program was to operate the scrubber with varying inlet propellant concentrations and flow rates and demonstrate that exit gas concentrations were reduced to less than 150 ppm for oxidizer. The oxidizer scrubber inlet concentrations were varied over a range of 440 to 259,000 ppm with flow rates varying from 50 to 400 scfm. In addition, worst case conditions were testing consisting of high and low flow rates with high propellant vapor concentrations.

Several problems were encountered during the performance testing of the scrubber. It was evident from the first series of tests that the performance requirements were not being met. This led to a series of investigations and supplementary testing to determine the cause of below specification performance. This activity included:

- 1. Upgrading the sump liquor flow rates by adjusting the pump clearances.
- 2. Incorporating a viewport in three of the packed towers to observe the uniformity of liquor spray over the packed beds.
- 3. Evaluating the mixing of the  $N_2$  and  $N_2O_4$  inlet gases.
- 4. Inspecting the tower nozzles to determine if any clogging of the nozzles had occurred.
- 5. Evaluating the nozzle spray patterns to determine the spray distribution.
- 6. Conducting smoke tests to evaluate gas flow through the packed towers.
- 7. Conducting tests to determine if the packed towers were flooding.
- 8. Consulting with the packing material manufacturer to determine if application of the packing was proper.
- 9. A thorough evaluation of the gas sampling techniques.

While these investigations improved the knowledge and understanding of the scrubber operation, the scrubber performance was not significantly improved and it was still not per specification.

#### 2.0 <u>SUMMARY</u> (cont'd)

Consultation with the Florida Institute of Technology revealed that a 10-percent solution of sodium sulfite  $(Na_2SO_3)$  may be a better scrubber liquor. The scrubber liquor was changed from sodium hydroxide to sodium sulfite and additional testing was performed. The scrubber performance was within specification under all conditions tested. The use of sodium sulfite, however, presented additional problems:

- 1. The scrubber did not have the capacity to scrub 600 pounds of  $N_2 0_A$  as required; and
- 2. After extended running, a solid precipitate was noted in the bottom of the sump.

Chemical analysis revealed that both of these problems could be eliminated by using a sump liquor consisting of a mixture of sodium sulfite and sodium hydroxide. Additional testing was performed with successful results, i.e., the capacity was adequate, there was no precipitate formed and the scrubber performance was within specification.

The scrubber performance was determined by analyzing scrubber exit gas samples using detector tubes (Kitagawa and Drager) as required by specification. It was necessary to measure the inlet gas concentrations using a wet chemistry method because of the high concentrations. The wet chemistry method was also used for exit gas analysis as a backup to the detector tube method and to obtain quantitative data. Throughout the test program, good correlation between the two analysis methods was not achieved. The discrepancy is academic, however, because the performance was within specification for both methods of analysis.

#### 3.0 HARDWARE DESCRIPTION

#### 3.1 <u>Scrubber Description</u>

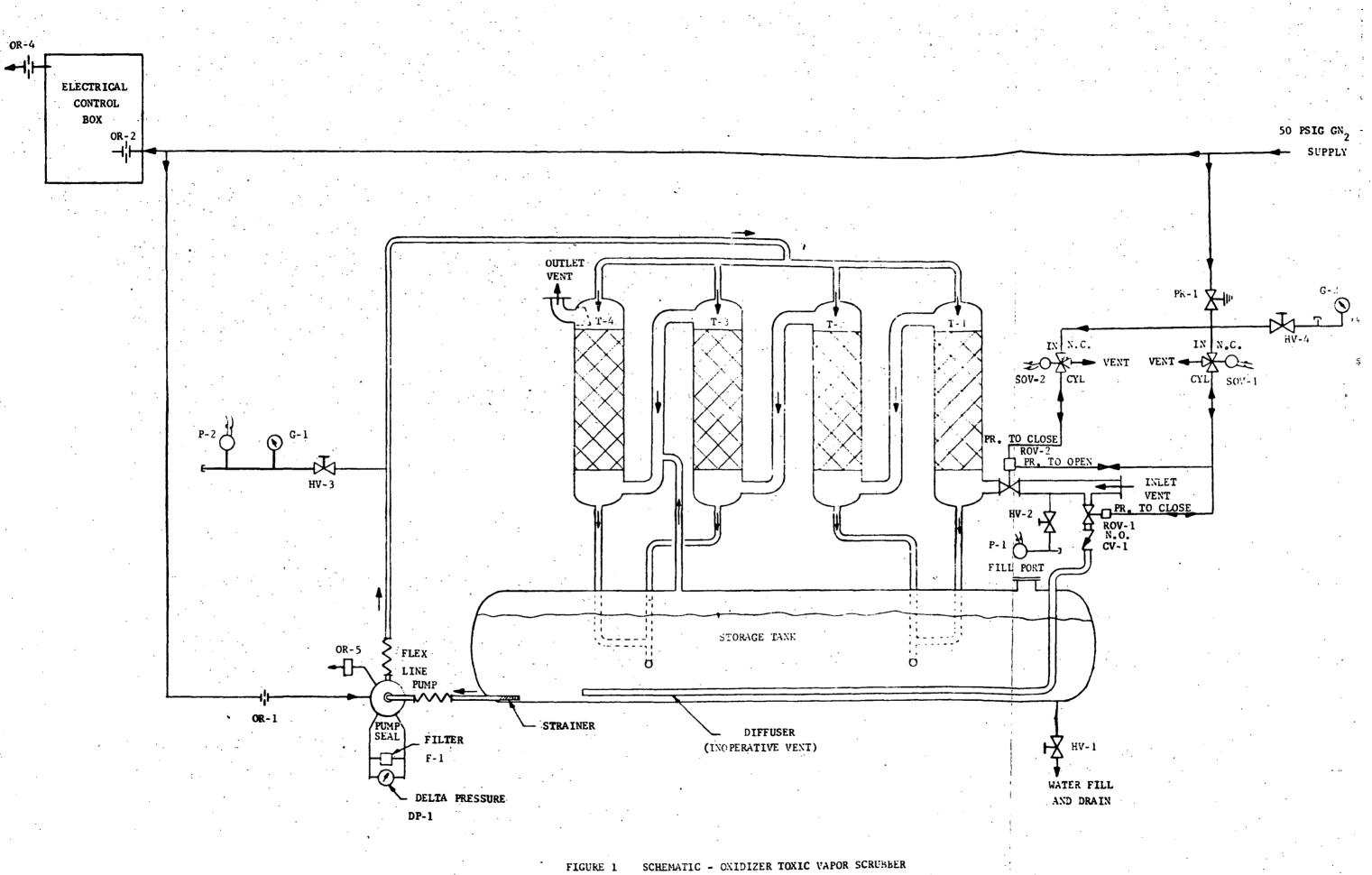
The scrubber contains wet packed bed scrubbing towers, a storage (sump) tank for the chemical solution (liquor) and pneumatic and electrical systems. The scrubber is approximately 8 feet by 8 feet by 11 feet high and weighs approximately 7,000 pounds empty, 10,500 pounds with stoneware loaded, and 18,500 pounds with stoneware and 750 gallons of 18% sodium sulfite and 5% sodium hydroxide solution loaded in the storage tank. The scrubber is shown schematically in Figure 1. It has 4 towers connected in series to minimize the total height and insure good gas/liquid contact. Tower bed packing materials are commerical grade chemical stoneware intalox saddles. The nominal sizes of saddles utilized are 1/2 inch, 3/4 inch and 1 inch. A polypropylene mesh demister is located in the top of twoer #4 to remove liquid droplets from the existing gas stream. The chemical solution is held in the storage tank in the base of the scrubber. When the pump is started, the solution is pumped to a 125 degree teflon spray nozzle at the top of each tower. The chemical solution then counterflows the exhaust gas stream and gravity drains back to the sump.

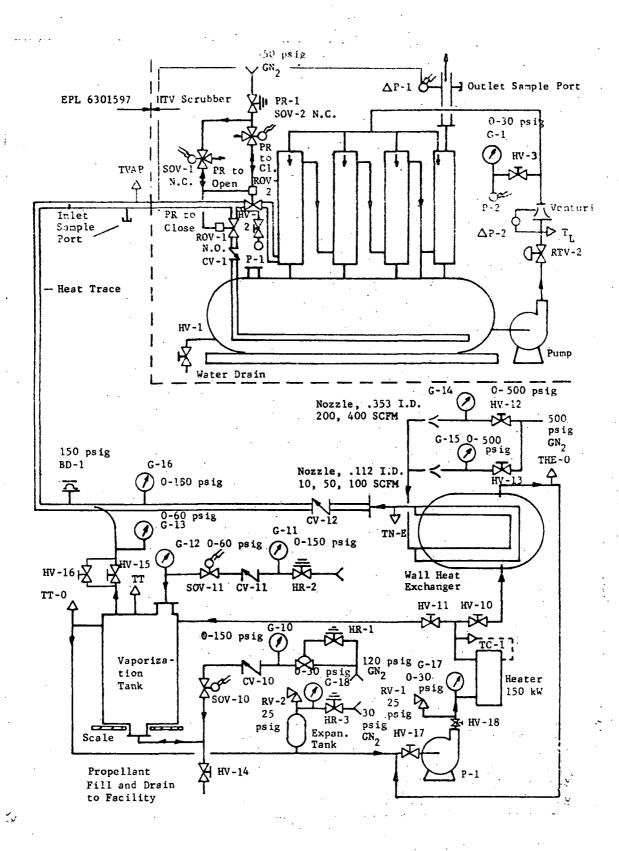
There are two modes of operation for the scrubber. In the first mode, designated the operative mode, the exhaust gas to be scrubbed flows through the scrubber 6-inch inlet line to the towers. In the second mode, designated the inoperative mode, pneumatic valves are positioned to close the 6-inch inlet line and open the 3-inch line. The exhaust gas then enters the storage tank below the chemical solution liquid leve, bubbles through the solution and exits out the last tower.

#### 3.2 Performance Test Setup

The schematic for the oxidizer scrubber test setup is in Figure 2. A photograph of the test setup used for these tests is shown in Figure 3.

Input  $N_2^{0}$  vapors to the scrubber were generated in a water jacketed tank that held liquid  $N_2^{0}$ . The water was heated to approximately 130°F for the  $N_2^{0}$  to get the liquid propellant above its boiling point at the necessary pressure for transfer. The vapors flowed through tubing (which had been warpped with heater tape and insulated) to a mixing pipe where the vapors were mixed with heated nitrogen gas at selected flow rates. A hand value in the propellant flow line was used to throttle the amount of vapor reaching the scrubber.





#### Figure 2 Scrubber Performance Test Fixture Setup

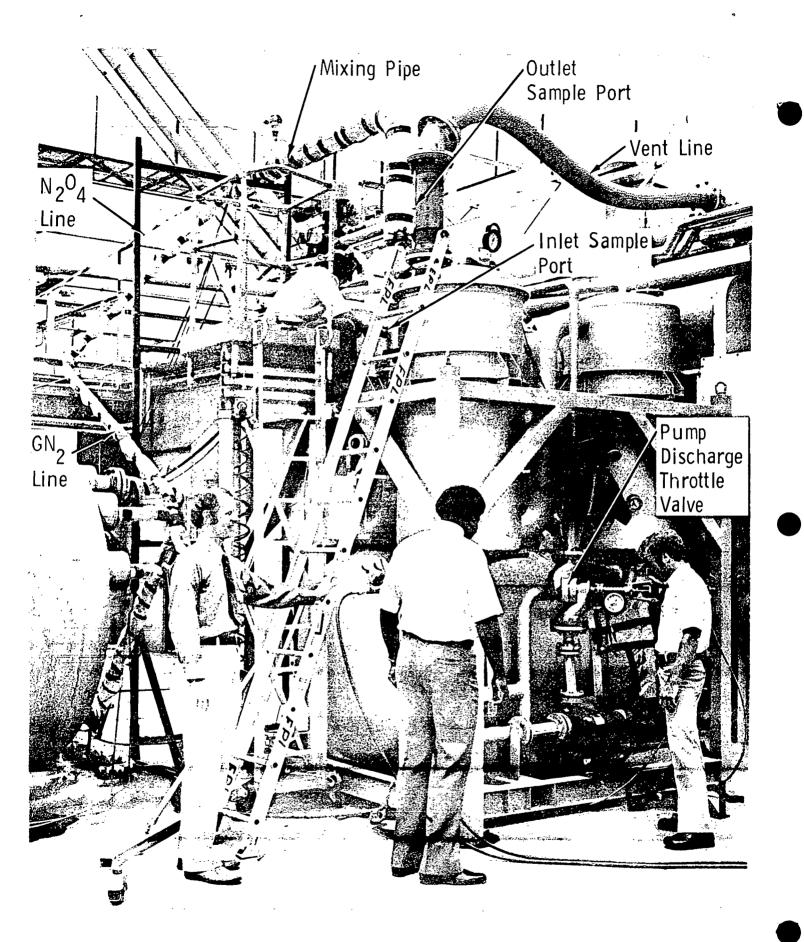


FIGURE 3  $N_2O_4$  Scrubber Test Setup

#### 3.0 HARDWARE DESCRIPTION (cont'd)

#### 3.2 <u>Performance Test Setup</u> (cont'd)

Input gas samples for the wet chemistry analysis were taken with a gas sample syringe inserted through a rubber septum into the input gas flow approximately 1-12/ feet upstream of the scrubber inlet port. Output gas samples were taken with a separate syringe inserted through a rubber septum into the vent gas flow approximately 2 feet downstream of the scrubber outlet port. The sampling approach was to take three samples of the inlet and outlet gas concentrations for one steady state condition. The samples were taken in the same manner by the same person for each data point with a time of two to five minutes between each of the three points. The gas concentrations were then analyzed by the wet chemistry methods described in Reference 1.

Gas detector tube measurements were made in the outlet gas flow stream through a 3/8-inch AN port at the same elevation as the wet chemistry sample port.

The sump liquor flow was varied by adjusting the remotely controlled throttle value on the scrubber pump discharge line. This value was installed for test purposes only.

The performance test matrix for the  $N_2^0 q_4$  scrubber is defined in Table 1.

N <sub>2</sub> 0 <sub>4</sub> Input Vapor	Nitrogen Gas Flow Rate, scfm							
Concentrations, ppm	<b>50</b> .	100	200	400				
500	X	X	х	x				
	x	X	x	x				
	x	х	<b>x</b> .					
	x	· x ·	•					
	х	х						
	x	x `						
	x	· · ·						
100,000	X			,				

Table 1. Test Matrix - N<sub>2</sub>0<sub>4</sub> Performance Tests

X = 100, 150 and 200 gpm liquid flow rates.

Worst Case Condition No. 1

Nitrogen Flow Rate: 10 scfm

N204 Input Vapor Concentration: 900,000 ppm

Liquid Flow Rate: Maximum

Worst Case Condition No. 2

Nitrogen Flow Rate: 400 scfm

N204 Input Vapor Concentration: 100,000 ppm

Liquid Flow Rate: Maximum

#### 4.0 <u>TEST DESCRIPTION FOR N.O. SCRUBBER</u>

The testing of the  $N_2O_4$  scrubbers consisted of three separate phases. These phases were test runs made to evaluate the scrubber using three different sump liquors: sodium hydroxide (NaOH), sodium sulfite (Na<sub>2</sub>SO<sub>3</sub>) and a mixture of NaOH and Na<sub>2</sub>SO<sub>2</sub>.

#### 4.1 <u>Sodium Hydroxide (NaOH)</u>

Forty-seven runs were made during the period from 04/27/77 through 06/16/77. During the forty-seven runs, the sump liquor flow rate was varied from 85 gpm to 181 gpm. The strength of the sump liquor varied from 7.95% NaOH to 2.16% NaOH. The scrubber was run in the operative and in the inoperative modes. Summaries of the test results for the NaOH sump liquor operative and inoperative mode test runs are found in Tables 2 and 3.

4.1.1 Post Test Inspection - After run 47 was completed, the NaOH sump liquor was drained. The towers were inspected and it was noted that the stoneware had settled 1 to 1-1/2 inches in all four towers. There was no caking of precipitates on the stoneware or the nozzles. Liquid was detected in the test setup vent line and identified as nitric acid. The scrubber pump was disassembled. Stoneware chips had been circulated by the pump and scoring of the impeller, impeller housing and pump shaft was noted.

#### 4.1.2 <u>Conclusions: NaOH Sump Liquor Test Series</u>

- 1. The desired outlet gas concentration of 150 ppm NO<sub>2</sub> or less could not be obtained with the NaOH solution.
- 2. Higher pump flow rate gives a lower outlet gas concentration.
- 3. Higher inlet gas concentration gives a higher outlet gas concentration.
- 4. Higher nitrogen flow rate gives a higher outlet gas concentration.
- 5. Proper circulation is occurring in the sump as designed.
- 6. Inoperative mode scrubs less efficiently than operative mode.
- 7. When scrubber pump is shut off, scrubber bed will contain enough sump liquor to scrub for a minimum of four minutes at selected flow rates of nitrogen and inlet gas concentrations.
- 8. Best scrubbing performance is with low sump strength (2 to 3% NaOH) rather than high sump strength (6 to 8% NaOH).
- 9. Majority of scrubbing is performed in the first tower.

# Table 2 Test Data Summary, N<sub>2</sub>O<sub>4</sub> Scrubber with NaOH Sump Liquor, Operative Mode

			Wet Chemist		Average	Outlet Ga Detector		Líq	uor
SN Flowrate,		•	Average Inlet Gas,	Average Outlet Gas.	Liquor Flowrate,	ppm	Strength		
scfm	Date	Run	ppm	ppm	gpm	Drager	Kitagawa	pli	% Conc
10	5-27-77	3	91,200	280	133	1 .	750		
_	5-27-77	2	95,500	443	171		500		
	5-27-77	4	98,100	107	95	· .	850		3.96
	5-27-77	1.	217,000	3,330	172		500		7.85
	6-6-77	· 31	484,000	636	160	· ·			· .
50 ·	6-1-77	13	1,960	398	128		·	·	
50	6-1-77	12	2,350	299	144		1		
	6-1-77	7	5,310	275	135				
	6-1-77	6	6,680	342	149		1		
	6-1-77	. 5	7,990	301	181				6.64
	6-1-77	11	34,200	276	181				0.04
	6-7-77	33		258	157	· · ·	· · ·		6.48
• •			34,900	179	165	1 · · · · ·			2.88
	6-16-77	46	47,200	1 ·		1			7.92
	6-6-77	26	54,700	304	154	1			
	6-8-77	37	58,500	199	172	500			5.04
	6-3-77	23	229,000	107	178	500			· • •
	6-3-77	25	249,000	65	115	1,000			2.76
	6-3-77	24	259,000	32	145	1,000		· ·	
100	6-1-77	10	6.440	454	172	50			
	6-1-77	9	7,080	504	148	100-150	1		· ·
	6-1-77	8	7,600	501	110	100 250			
	6-16-77	47	18,800	504	167				2.16
	6-7-77.	34	22,200	634	157				6.1
	·6-1-77	16	23,800	951	129	500	·		. 0.1
	6-1-77	14	24,400	693	181	500		·	
	6-1-77	14		957	154	500			
			26,800			500			1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 -
Í	6-6-77	27	30,900	653	157				· .
	6-8-77	38	31,100	538	176				•
150	6-6-77	29	22,600	1,100	160				
200	6-7-77	35	12,800	1,040	157				
200	6-8-77	39	17,000	951	181				
ļ	6-6-77	28	17,700	1.070	159	· ·			
1	6-3-77	28 17		928	158				
			20,100	1,130	131				•
	6-3-77	18	20,700						
	6-3-77	19	22,500	1,040	102				
400	6-3-77	20	4,910	1,680	72	·			
	6-3-77	21	5,530	1,390	128	1,000+			
	6-3-77	22	6.470	1,570	167				
	6-7-77	36	7,100	1,140	159	Į.			
l	6-6-77	30	12,000	903	160	Į [		·	6.30
	6-6-77	32	80,000	2,400	160				

Table 3	Test Data	Summary	$^{NO}_{24}$ Scrubl	ber with	
•	NaOH Sump	Liquor,	Inoperative	Mode	

			Wet Chemis	try	Outlet				
GN <sub>2</sub> Flowrate,			Average Inlet Gas,	Average Outlet Gas,	Detecto ppm	r Tube,	Liquor Strength		
scfm	Date	Run	ppm	ppm	Drager	Kitagawa	pН	% Conc.	
10	6-9-77	44	922,000	3,720				. ·	
50	6-9-77	40	42,500	538				4.56	
100	6 <b>-9</b> -77	45	22,100	400		· .		• •	
	6-9-77	41	24,700	1,760				3.72	
200	6-9-77	42	12,300	2,810	·			•	
400	6-9-77	43	74,400	9,100					

#### 4.0 <u>TEST DESCRIPTION FOR N<sub>2</sub>O<sub>4</sub> SCRUBBER</u> (cont'd)

#### 4.2 Scrubber Tower Operation Tests

The desired output of 150 ppm NO<sub>2</sub> or less was not obtained with the NaOH sump liquor. Several possibilities were investigated to evaluate if the scrubber towers were flooding at certain test conditions or if the gas was channeling in the towers so that maximum scrubbing would not be achieved. It was hoped that these tests could help explain the high outlet gas concentrations.

A dry bed smoke test was performed to observe the gas flow in a dry tower. With the pump off and the values in the operative mode position, nitrogen was flowed at 10 to 400 scfm through the scrubber. A smoke candle was moved across the diameter of the tower along the surface of the packed bed and the gas flow observed. At 10 scfm, a smoke candle indicated that the flow at the edges of the tower was more pronounced than in the center. At 400 scfm, the flow dispersion in the tower was nearly uniform.

A wet bed smoke test was also performed to determine the gas flow characteristics in an operational or wet bed tower. With the lid and demister of tower #4 removed, a nozzle was installed to spray at approximately the same height as the nozzle in tower #4 when the lid is installed. The pump was turned on and tower #4 was thoroughly soaked, the pump shut off and  $GN_2$  flow measurements were made with a smoke candle for 1 to 2 min utes as the water gravity drained through the tower. This technique was repeated for  $GN_2$  flows of 10, 50, 100, 200 and 400 scfm. At 10 scfm, the  $GN_2$  flow was uniform over the bed surface as measured by a smoke candle. At 50 and 100 scfm, the flow around the edges of the tower was slightly less than the flow in the center. At 200 and 400 scfm, the flow pattern seemed uniform as measured by the smoke candle.

Another test was performed to determine if flooding had occurred in tower #1. In order to increase the liquid flow rate to Tower 1, the nozzles in towers #3 and #4 were removed and the supply line capped. The scrubber pump was turned on and 60 gpm was flowed through tower #1 spray nozzle. Nitrogen flow was varied from 10 scfm to 400 scfm. No flooding of the tower was observed under these conditions.

A test to evaluate the spray pattern of the scrubber teflon nozzles was conducted to determine the distribution of liquor to the packed bed surface during scrubber operation. The 2-piece nozzle was tested with the inner diffuser vane pinned and with it free to rotate. A series of plastic cups were arranged along a line representing the diameter of the tower. A teflon spray nozzle was positioned at a height above the cups that approximated the nozzle height above the scrubber bed. The nozzle was connected to a water line with a flow capacity of 40 gallons per minute. The water was turned on for a brief period to partially fill the cups. From the amount of liquid in the cups, a distribution was determined. The results obtained indicated that the center portion of the tower packed bed surface received about 1/3 more water than the outer portions of the surface with inner diffuser vane pinned and with the diffuser vane free to rotate.

#### 4.0 <u>TEST DESCRIPTION FOR N.O. SCRUBBER</u> (cont'd)

#### 4.2' <u>Scrubber Tower Operation Tests</u> (cont'd).

It was concluded from these tests that the gas and liquid flow distribution in the towers was good for the conditions tested and no changes to the tower configuration needed to be made. The spray nozzles in the towers are satisfactory and should continue to be used in the scrubbers.

#### 4.3 <u>Sodium Sulfite (Na<sub>2</sub>SO<sub>2</sub>) Sump Liquor</u>

The initial tests of the oxidizer scrubber were performed utilizing a solution of sodium hydroxide as the sump liquor. This test series indicated that the desired output of 150 ppm NO<sub>2</sub> or less could not be obtained with the NaOH solution. References<sup>2</sup> in the report, "Hypergolic Propellant Vapor Disposal", Florida Institute of Technology, Contract NAS10-8399, indicated good results could be obtained by use of sodium sulfite solution as the scrubbing liquor.

Thirty-eight (38) test runs using sodium sulfite as the sump liquor were made during the period from 06/23/77 through 07/06/77. Tests were made with sump liquor concentrations at 10% and 25% Na<sub>2</sub>SO<sub>3</sub>. The actual concentration of the Na<sub>2</sub>SO<sub>3</sub> could not be easily measured so pH readings were used as an indication of sump liquor strength. Na<sub>2</sub>SO<sub>3</sub> was also added to partially depleted sump solution to determine if liquor strength could be increased during usage without a complete drain and refill operation. Summaries of the test data for the Na<sub>2</sub>SO<sub>3</sub> sump liquor operative and inoperative mode test runs are presented in Tables 4 and 5.

#### Conclusions: Na SO, Sump Liquor Test Series

As a result of the tests conducted, the following conclusions were reached:

- 1. Sodium sulfite gave much better overall performance than sodium hydroxide as a sump liquor and met the scrubber specification requirement that the output gas concentration be less than 150 ppm of NO<sub>2</sub>.
- 2. Sodium sulfite formed a one-inch layer of precipitate in the bottom of the sump tank during the capacity test.
- 3. Higher pump flow rate gives a lower outlet gas concentration.
- 4. Higher inlet gas concentration gives a higher outlet gas concentration.

5. Higher nitrogen flow rate gives a higher outlet gas concentration.

6. Inoperative mode scrubs less efficiently than operative mode.

# Table 4 Test Data Summary $N_2^0{}_4$ Scrubber with $Na_2SO_3$ Sump Liquor, Operative Mode

<b>C</b> 1:			Wet Chemist		Average	Outlet G Detector		Liquor	
GN <sub>2</sub> Flowrate,	· ·	1	Average Inlet Gas.	Average Outlet Gas,	Liquor Flowrate,	ppm		Streng	th _
scfm.	Date	Run	ppm	ppm	gpm	Drager	Kitagawa	рH	5. Conc
10	6-25-77	15	606,000	0	150		0	·7 ·	
50	6-25-77	11	634	0	148		0.	9 ·	
	6-24-77	5	1,490	0	151		0	8-9	
	6-29-77	17	9,570	0	150			10	
	6-24-77	9	11,300	0	151		C	<u>8-9</u>	
	6-23-77	3	16,800	2.0	147	<5	. 0 .	. 8	
	6-23-77	1	21,200	3.1	. 150			9-10	
	7-1-77	28	22,000	413*	127				
	6-23-77	4	22,400	6.1	154	0.	0	<u>8-9</u>	
• • •	7-1-77	30	28,900	453*	146 .			7 ·	
	7-6-77	31	29,300	0	147			10	
	6-29-77	18	25,500	0	153 <sup></sup>			8-9	
	7-1-77	27	30,200	377*	154				
	7-1-77	25	30,700	187	154			•	
·	6-23-77	2	30,700	6.1	154	<5	<10	. 8	
·	7-1-77	26	31,300	377*	154	· · ·			
	7-1-77	29	31,700	893*	103		• .	7-8	
	7-6-77	33	147,000	417	148		( ) (		
100	6-24-77	ó	442	5.3	154	· ·	0	<sup>.</sup> 8	
	6-25-77	12	6,950	0	154		0	•	
	6-24-77	10	7,050	0	146		0	8-9	
	6-29-77	19	12,400 24,600	46 0	98 153				
	7-6-77	36	24,800	183	113		10.	9-10	
	7-6-77	37	29,500	193	146			8-9	
200	6-24-77	7	683	0	151	•	. 0	8	
	6-25-77	13	4,770	0	154 🔗		0	8	
	6-29-77	20	5,230 5,720 5,550	30 44 48	148 127 106			8-9	
400	6-24-77	8	561	1.2	154		с	8-9	
	6-25-77	14	3,110	0	151		<sup>-</sup> 0	, S. j	
	6-30-77	21	4,360 4,000 3,880	0	149 128 98	-		9-10	
	6-25-77	16	41,600	0.7	149		ę.	7-8	

.

Table 5	Test Data Summary, $N_{24}^{0}$ Scrubber with
	Na, SO3 Sump Liquor, Inoperative Mode

			Wet Chemis	try	Outlet (				
GN <sub>2</sub> Flowrate,			Average Inlet Gas,	Average Outlet Gas,	Detector ppm	r Tube,	Liquor Strength		
scfm	Date	Run	ppm	bbw	Drager	Kitagawa	PH	% Conc.	
10 ·	7-6-77	35	565,000	680		· ·			
50	6-30-77	22	14,900	o		· ·	9		
100	6-30-77	23	18,400	15					
200	6-30-77	24	10,200	54					
400	7-6-77	34	51,100	53					

## 4.0 <u>TEST DESCRIPTION FOR N\_0</u> SCRUBBER (cont'd)

#### 4.3 <u>Sodium Sulfite (Na, SO, ) Sump Liquor</u>

- 7. A sharp breaking point of reduced scrubber efficiency did not occur as the 600 lb  $N_2 O_4$  goal was reached in the capacity run.
- 8. When the scrubber pump is shut off, the scrubber bed will contain enough sump liquor to scrub for a minimum of five minutes at selected flow rates of nitrogen and inlet gas concentrations.

#### 4.4 <u>Sodium Hydroxide (NaOH)/Sodium Sulfite (Na<sub>2</sub>SO<sub>2</sub>) Sump Liquor</u>

A mixture of Na  $_{2}SO_{3}$  and NaOH was made in the sump to determine if capacity could be increased and the precipitate from the sodium sulfite kept in solution. Twenty runs were made during the period from 07/07/77 through 07/09/77. One sump solution mixture was used for the entire series. The sump pH remained at 10 for the twenty runs. The scrubber was run in the operative and inoperative mode. Summaries of the test results for the NaOH/Na  $_{2}SO_{3}$  sump liquor operative and inoperative mode test runs are found in Tables 6 and 7.

<u>Post Test Inspection</u> - The sump liquor was drained and no salts were noted. The drop in sump liquor flow rate from 172 gpm to 139 gpm between run 8 and previous runs could be explained by partially plugged tower spray nozzles. An examination of the tower spray nozzles were made and whole 1/2-inch saddles from the packed bed were found in the following quantities: Tower 1, 4 saddles; tower 2, 16 saddles; tower 3, 3 saddles; tower 4, 1 saddle. Apparently, 1/2inch saddles had migrated downward through the 1-inch saddles and holding screen into the sump storage tank. During the inoperative mode, runs 5, 6 and 7, the saddles were suspended in the sump solution by the agitation in the sump from the inlet nitrogen bubbling through the inlet diffuser in the sump storage tank bottom. When run 8 was performed and the scrubber pump turned on, the saddles were passed through the pump and into the tower spray nozzles. Runs 8 through 20 were performed at reduced sump liquor flow rate.

#### 5.0 <u>CONCLUSIONS: NaOH/Na, SO, SUMP LIQUOR</u>

As a result of the tests conducted, the following conclusions were reached:

- 1. The sump liquor was as efficient as  $Na_2SO_3$  for scrubbing in the inoperative mode.
- 2. NaOH/Na<sub>2</sub>SO<sub>2</sub> did not precipitate a solid.
- 3. Half inch saddles appeared in the sump tank during the test and were ingested through the pump and lodged in the nozzles. This indicated the need to prevent the pump from ingesting the saddles, either by filtering the pump suction line or by insuring that the half inch saddles would not migrate through the one inch saddles and support plates in the towers.
- 4. Six hundred sixteen pounds of  $N_2 O_4$  can be absorbed by the sump liquor although the efficiency drops after 560 pounds of  $N_2 O_4$  is absorbed.
- 5. The operative mode test results for the NaOH/Na $_2$ SO $_3$  mixture were generally not as good as for the Na $_2$ SO $_3$  by itself.
- Early in the test (after run 7), the nozzles became partially 6. clogged with whole stoneware saddles which had been ingested through the pump. This reduced the sump liquor flow rate and probably distorted the spray patterns in the towers. It this problem had not existed, results probably would have been better. Test results show that after 560 pounds of oxidizer had been reacted in the unit, the measured concentration of the outlet gas was 124 ppm for an input concentration of 137,000 ppm. Not until 600 pounds had been reacted, did the outlet concentration indicate that the specification of less than 150 ppm had been exceeded. Visual observations of the vent stack during all of these tests was made by the entire test crew and no brown vapors were observed at any time. On the basis of these results, it is believed that with full liquor flow and unclogged tower nozzles, that the mixture of sodium sulfite and sodium hydroxide would scrub as well as the sodium sulfite by itself and that the capacity to react 600 pounds of oxidizer could be achieved.
- 7. A mixture of 5% NaOH and 18%  $Na_2SO_3$  in water is recommended as the sump liquor to be used in the oxidizer scrubber.

Wet Chemistry Nutlet Cas Average Detector Tube, Liquor 63.<sup>2</sup> Average Average Liquor Strengel. ppm Outlet Gas. Flowrace, Inlet Gas, Flowrate, yH / Cone Date scrm kun ppm ppm gpm Drager Kitagawa 10 7-9-77 12 489,000 258 133 7-9-77 50 16 14 1,070 133 7-7-77 5 1 4,230 172 :0 7-9-77 20 117,000 752 133 7-9-77 19 132,000 825 135 7-9-77 18 137,000 124 133 7-9-77 176,000 17 949 133

39

36

153

0

23

38

546

# Table 6 Test Data Summary, $N_2O_4$ Scrubber with NaOH/Na $_2SO_3$ Sump Liquor, Operative Mode

Table 7

7-9-77

7-7-77

7-9-77

7-7-77

7-9-77

7-9-77

7-9-77

106

200

-00

15

9

10

8

14

13

11

6,170

12,900

22,400

3,270

4,770

3,900

27,500

Test Data Summary, N<sub>2</sub>0<sub>4</sub> Scrubber with NaOH/Na<sub>2</sub>SO<sub>3</sub> Sump Liquor, Inoperative Mode

133

133

139

139

133

1/32

139

		1	Wet Chemis	try	Outlet Gas			
GN2 Flowrate,			Average Inlet Gas,	Average Outlet Gas,	Detector ppm	r Tube	Liquo	
scfm	Date	Run	ppm	bbm	Drager	Kitagawa	рН	% Cont.
10	7-7-77	3.	737,000	61		·.		
50	7-8-77	5	11,600	0			10	1
	7-7-77	4	49,900	-		l	10	
100	7-8-77	6 ·	10,800	0				
200	7-8-77	7	5,830	0		· ·		
400	7-7-77	2 · ·	49,900	60				

#### **REFERENCES:**

1. Test Report for Performance Test of Hypergolic Propellant Vapor Scrubbers, T. A. Murdock, October 1977.

## APPENDIX A - ABBREVIATIONS

	· ·			
cc	Cubic centimeter		· .	
DR	Drager Gas Detector Tube	• 4 • •		· · ·
E PL	Engineering Propulsion Laboratory Martin Marietta Corporation Denver, Colorado			
FIT	Florida Institute of Technology Melbourne, Florida		· · · · · · · · · · · · · · · · · · ·	N
gal.	Gallon			
GN <sub>2</sub>	Gaseous nitrogen	· · · . ·		
gpm	Gallons per minute		· ` .	
HTV	Hypergolic Toxic Vapor		•	· ·
KIT	Kitagawa Gas Detector Tube	·	· · ·	
KSC	Kennedy Space Center	:	• •	· ·
ln <sub>2</sub>	A Liquid Nitrogen			
m1	Milliliter		•	
MMC	Martin Marietta Corporation	•	. •	
N204	Nitrogen tetroxide	•		•
NaOH	Sodium Hydroxide			
Na2SO3	Sodium sulfite	•		
NO <sub>2</sub>	Nitrogen dioxide		<i>.</i>	
рН	Measure of acidity and alkalinity		•	
psid	Pounds per square inch, different	ial		
psig	Pounds per square inch, gage			
P/N	Part number			
ppm	Parts per million			
scfm	Standard cubic feet per minute			
	Microns		•	

APPENDIX B - SCHEMATIC SYMBOL LIST

