Ammonia Scrubber Testing During IDMS SRAT and SME Processing

by

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Ammonia Scrubber Testing During IDMS SRAT and SME Processing (U)

The transmittal of the attached report fulfills the requirements of the DOE milestone, "Complete TNX testing and Documentation of Ammonia Scrubber Performance". This testing was in support of milestone 22-AA, DWC35.

The ammonia scrubber was successful in removing ammonia from the vapor stream to achieve ammonia concentrations far below the 10 parts per million (ppm) vapor exit design basis during SRAT processing. However, during SME processing, vapor ammonia concentrations as high as 450 ppm were measured exiting the scrubber. Scoping tests are in progress to try to determine the cause of the high ammonia concentrations exiting the scrubber.

The SRTC technical review is complete. Please respond with any comments or acceptance. If you have additional questions, please contact D. A. Crowley (5-6454) or D. P. Lambert (5-6380).

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SUMMARY

This report summarizes the results of the Integrated DWPF Melter System (IDMS) ammonia scrubber testing during the PX-7 run (the seventh IDMS run with a Purex type sludge). Operation of the ammonia scrubber during IDMS Sludge Receipt and Adjustment Tank (SRAT) and Slurry Mix Evaporator (SME) processing has been completed. The ammonia scrubber was successful in removing ammonia from the vapor stream to achieve ammonia concentrations far below the 10 parts per million (ppm) vapor exit design basis during SRAT processing. However, during SME processing, vapor ammonia concentrations as high as 450 ppm were measured exiting the scrubber. The detection limit of the analyzers is 2 ppm.

Four months elapsed from the completion of the SRAT simulation cycle and the start of the SME simulation cycle. This was due to SRAT process foaming that led to the loss of approximately 25% of the aluminum along with smaller quantities of other materials from the SRAT. The original SRAT testing has been reported¹. A summary of the tasks completed prior to resuming PX7 testing have been summarized².

One problem that was discovered during IDMS SRAT processing was vapor bypassing the scrubber. DWPF should ensure that it does not have the same problem. Details of the vapor bypass mechanisms are summarized in Appendix A. Note these two bypass mechanisms were not noted during off-line testing.

A second problem noted during SME testing was the inefficient scrubbing of the ammonia at the end of the SME cycle. The SME scrubber design basis removal efficiency is 99.9% removal of ammonia. The lowest efficiency measured was 50% removal efficiency. It is believed that mass transfer is limiting the absorption of the ammonia by the acidified scrub liquid. Laboratory scale testing is in progress to determine whether the stripped material from the antifoam is interfering with the scrubbing of the ammonia. If the stripped material from the antifoam is the problem, it is possible that it would also affect SRAT scrubber performance.

INTRODUCTION

The IDMS ammonia scrubber was installed to (1) minimize the deposition of ammonium nitrate (a potential explosive³) in the downstream vent piping, and (2) allow testing of a scaled DWPF ammonia scrubber.

The conceptual design for the scrubber was summarized^{4,5} and included with the Defense Waste Processing Facility (DWPF) design documents^{6,7,8} to ensure that the scaled IDMS scrubbers were as prototypic of the DWPF scrubbers as practical. The original design was performed by Ebasco⁹. A cut in Defense Waste Processing Technology (DWPT) funding in FY93 caused the design completion to be delayed approximately nine months¹⁰. The scrubber was fabricated and installed by construction. Construction turned over the scrubber to TNX on May 15, 1994. The results of the off-line testing of the

Integrated DWPF Melter System (IDMS) ammonia scrubbers using ammonia supplied from cylinders was previously summarized¹¹.

The ammonia scrubber installation is part of DWPF Technical Issue 3.7, "Control of Ammonium in DWPF Preparation Processes"¹². DWPF summarized their technical concerns before the scrubber design began¹³. The testing was organized¹⁴ and specific objectives for testing were documented¹⁵. Data collected during testing was recorded in a laboratory notebook¹⁶.

RESULTS

Background

The prototypic SME scrubber (60" of packing in the 5" diameter basket) was used during the SRAT scrubber testing because of the difficulty and expense of replacing the scrubber basket between the SRAT and SME cycles. The SME scrubber basket is appropriately sized for the vapor flows planned during the IDMS PX-7 SRAT and SME processing (see Table 2). Vapor flows less than 100 lb/hr were planned in the SRAT versus the 296 lb/hr design basis (see Table 1).

A schematic of the IDMS ammonia scrubber is shown in Figure 1. The vapor exiting the SRAT/SME condenser is drawn into the bottom of the scrubber through a vapor distributor. The vapor (75 lb/hr air purge plus air inleakage and process generated gases) must travel through a packed column of 1" Intalox saddles before exiting at the top of the scrubber. The scrubber exit vapor is processed downstream in the Process Vessel Vent System's (PVVS) Formic Acid Vent Condenser (FAVC).

Ammonia is removed from the vapor by spraying 1.8 gpm of the SMECT liquid (a nitric acid solution at a pH of 1 to 3) through liquid distributors at the top of the scrubber. Ammonia in the vapor is removed by absorption accompanied by chemical reaction. The acidic liquid prevents ammonia from returning to the vapor by converting virtually all the ammonia to ammonium ($NH_3 + H^+ \rightarrow NH_4^+$). The scrubbing liquid exits the bottom of the scrubber and drains to the Slurry Mix Evaporator Condensate Tank (SMECT). The scrubber liquid flow is provided by the SMECT pump and controlled by throttling a valve in the pump discharge line.

The source of the SMECT liquid is from the SRAT condenser condensate. The SMECT liquid's pH is controlled by adding concentrated nitric acid to the SMECT. The SMECT is unagitated but some mixing is provided by the recirculation of approximately 38 gpm of flow using the SMECT pump.

Ammonia Scrubber Testing During IDMS Processing

SRAT Water Testing (December 1-3, 1994)

Water testing was performed to verify that all equipment and instrumentation were functioning properly prior to the PX-7 SRAT processing. The water testing also served to clean the SRAT, SRAT condenser, SMECT, and the PHA transfer line of material from previous batches. After simulating a transfer of nitric acid to the SRAT, and simulating the feeding of 100 gallons of process water while boiling in the SRAT, the SRAT and SMECT vessels were drained to the sump. The scrubber was backflushed with process water to remove solids that had accumulated during testing that used cylinder supplied ammonia. After backflushing the scrubber, the SMECT was drained again. The SMECT was refilled with process water and nitric acid was added to bring the SMECT pH within the 1-3 operating range. Problems noted during water testing are summarized in Appendix A.

SRAT Processing (December 9-13, 1994)

No ammonia was detected in the vapor at the exit of the scrubber during the SRAT processing. The ammonia scrubber was successful in removing ammonia from the SRAT condenser vapor stream to achieve ammonia vapor concentrations far below the 10 parts per million (ppm) vapor exit design basis. The ammonia concentration in the vapor exit was lower than the detection limit of the Dräger tube analyzers (see Appendix B) in all 52 analyses performed (reported as <2 ppm). The maximum ammonia concentration measured at the scrubber vapor inlet was approximately 300 ppm (the IDMS design basis is 2000 ppm at the IDMS SRAT flow conditions).

SRAT processing lasted approximately 98 hours, beginning December 9, 1994 at 4:00 PM and was completed December 13, 1994 at 6:00 PM. Numerous steam interlocks and a shutdown to install a blind flange (to prevent vapor bypass of the scrubber) caused the SRAT cycle to last longer than planned. The SMECT pH remained in the 1-3 range without any addition of acid throughout SRAT processing because of the acidic condensate that was produced during PHA addition.

The SRAT cycle has three main periods of processing, (1) nitric acid addition, (2) PHA addition, and (3) concentration. The SRAT processing begins with the nitric acid addition at a SRAT liquid temperature of approximately 93°C. The addition of nitric acid drops the SRAT liquid pH to below 7. No ammonia is present at this time and the pH is too low for it to evolve if it were present. Addition of nitric acid lasted approximately three hours.

The second period of processing is PHA addition. After the nitric acid was added, the SRAT is heated to boiling and PHA is added. The PX-7 PHA contained 1100 mg/L of ammonium. Therefore once PHA addition starts, ammonia/ammonium is present in the SRAT, but the pH is too low for

appreciable ammonia to evolve. The condensate from the SRAT condenser drains to the SMECT during PHA addition. Also, the acidic nature of the condensate (there was little pH change in the SMECT during PHA addition so the condensate is very low in pH) allows it to serve as a scrubbing fluid in the condenser. During PHA addition the SMECT pH remained virtually constant (Figure 2). PHA was added at approximately 0.8 gpm with a boilup rate of 325-400 lb/hr. The PHA addition lasted 62 hours including a 6 hour shutdown to replace the PVVS flange (see appendix A).

The third period of processing concentrates the SRAT down to the 1100 gallon level. A 28 hour concentration period was required because the SRAT level was much higher than planned due to a level measurement problem. Ammonia began evolving from the SRAT during the concentration period. Condensate pHs were approximately 10 and the SMECT pH began to rise more rapidly (Figure 2). Note that during nine hours of the concentration period the SRAT was not at boiling because (1) a sample was pulled and the SRAT was rodged to determine the actual concentration and level in the SRAT, and (2) the SMECT was drained to drums since it could not be processed because of the high solids loading of the SMECT liquid. In addition, a five hour reflux period was added at the end of the SRAT cycle to ensure that processing was not stopped before the SRAT hydrogen peak was achieved.

The flow bases for SRAT processing is summarized in Table 2. Problems noted during SRAT processing are summarized in Appendix A.

Modifications Prior to SME Processing (December 14 - April 19, 1995)

An investigation was completed prior to SME processing to determine the cause of the loss of aluminum during SRAT processing. Also, aluminum was added to the SRAT to remediate the SRAT product. Extensive laboratory testing was completed to determine the cause of the loss of aluminum. Laboratory scale testing was completed to develop an antifoam strategy for the PX-7 SME cycle since a major objective of the SME cycle was to prevent foaming. Because of the high concentration of solids in the SMECT, the SMECT was emptied and cleaned. Four major processing changes were made between the SRAT and SME cycle:

- (1) The SMECT was emptied and cleaned. Process water (710 gallons) and nitric acid were added to prepare the scrub liquid for SME processing.
- (2) Aluminum in the form of Gibbsite ($\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$) was added to the SRAT product to remediate the SRAT product prior to SME processing.
- (3) Ammonium nitrate was added to the SME prior to processing to increase the ammonium concentration to 1000 mg/L. This was to make up for the ammonia that evolved during the four month idle period between the SRAT and SME processing.
- (4) A new antifoam strategy was recommended for the PX-7 SME processing based on laboratory scale testing. 200 g

Dow Corning 544 antifoam were added per 1000 kg SME total mass before processing and each 12 hours during processing. This was approximately 10 times the antifoam added during the SRAT processing.

SME Processing (April 19 15:25 - April 20, 1995 12:20)

SME processing begins with the initiation of boiling to concentrate the SME after the addition of the frit slurry (1st SME concentration). The SME volume was 1280 gallons after the first addition of the frit slurry. The SME was concentrated down to a volume approximately 1150 gallons before the second addition of frit slurry. The SME volume was 1280 gallons after the second addition of the frit slurry (2nd SME concentration). The SME was concentrated down to a volume of 1150 gallons after the second addition of frit slurry. The condensate collects in the SMECT during the concentration portion of SME processing but can be returned to the SME (reflux) as needed.

The ammonia scrubber was successful in removing ammonia from the SME condenser vapor stream to achieve ammonia vapor concentrations below 2 ppm until 90 minutes prior to the completion of SME processing. During the last 90 minutes of SME processing the ammonia vapor concentration exiting the scrubber continually increased and was measured at >70 ppm (limit of detection of Dräger tube used). The maximum ammonia concentration measured at the scrubber vapor inlet was approximately 3000 ppm (the IDMS design basis is 10,000 ppm inlet and 10 ppm outlet at the IDMS SME flow conditions). Table 4 summarizes the ammonia data collected during SME processing.

SME processing lasted approximately 21 hours, beginning April 19, 1995 at 15:25 PM and was completed April 20, 1995 at 12:20 PM. The SMECT pH remained in the 1-3 range except during a 90 minute period (07:30 - 09:00 on April 20, 1995). The pH increased to 4.3 when nitric acid could not be added to the SMECT (Figure 4). The SME condensate was refluxed back to the SRAT during this 90 minute period until nitric acid had been added. pH control was accomplished with the addition of 90 wt % nitric. Three antifoam additions totaling approximately 9.2 lbs were added during the SME processing.

During the period of poor scrubber performance, all scrubber parameters were normal. The scrubber liquid flow was steady at 1.85 gpm (design basis 1.79 gpm), the vapor inlet flow was steady at 76 lb/hr (design basis 72 lb/hr), the pH was 2.0 (design basis <3), and the scrubber differential pressure was constant at 0.5 inwc (Figure 5). It should also be noted that the vapor exit piping from the scrubber was dry and all vapor instrumentation was operating as designed indicating there were no signs of excessive liquid entrainment as observed during previous scrubber testing. The scrubber inlet and outlet samples were submitted to the lab to determine whether the samples were saturated with ammonia. The ammonia concentration was only 650 mg/L, much less than saturation. The pH of the inlet sample was

2.0 and the pH of the exit sample was 2.3 indicating that very little ammonia scrubbing was taking place.

In past testing at these conditions with cylinder supplied ammonia at vapor inlet concentrations of 17,000 ppm, no ammonia was detected exiting the scrubber¹⁷. Table 3 summarizes the difference between testing with cylinder supplied ammonia and SME processing. Note there is no obvious sign of hydraulic problems in the scrubber that would explain the poor scrubber performance.

Post SME Testing (April 20, 1995 12:20 - April 21, 1995 02:00)

SME testing was continued to determine the cause of the poor scrubber performance noted during the last 90 minutes of SME processing. The SME condensate was refluxed back to the SME to allow further testing of the scrubber although the SME cycle was complete. This testing continued for 30 minutes until a low SME level interlock interrupted processing. 100 gallons of process water was added. A fourth antifoam addition prior to boiling was made to ensure foam did not form during the extended processing (antifoam concentration now at 800 ppm). Boiling resumed at 17:45 on April 20, 1995. The SME cycle condensate was again refluxed back to the SME until 23:00. During this period the ammonia concentration exiting the scrubber was relatively constant at approximately 250 ppm (Figure 6). There was no change in the pH of the scrubbing liquid indicating little ammonia was being scrubbed since this would have caused the pH of the SMECT contents (scrub solution) to increase.

At 23:00, the SME was placed in the concentrate mode by diverting the condensate to the SMECT. During this period the scrubber performance seemed to degrade more with the ammonia concentration exiting the scrubber increasing to approximately 450 ppm (Figure 6). The pH of the SMECT increased rapidly during this period but it was probably caused by the high condensate pH that was being returned to the SMECT and not due to scrubbing of the ammonia.

Recommended Path Forward

The cause of the poor scrubber performance must be determined. There are at least three possible explanations for the poor scrubber performance:

- (1) Channeling or some other hydraulic problem that allows poor liquid/vapor contact and poor scrubbing of ammonia.
- (2) Liquid resistance to mass transfer.
- (3) Vapor resistance to mass transfer.

Scoping tests will be completed to determine whether there is a problem related to liquid resistance to the absorption of ammonia. The major processing change between the SRAT and SME cycles was the increase in the antifoam additive. The Dow-Corning 544 solution contains approximately 5% strippable components (per the vendor). These strippable components would

increase in concentration as more antifoam is added and as more concentrating (stripping) occurs. If they were to coat the water droplets in the condensate (scrub solution) entering the scrubber, they may increase the liquid resistance to mass transfer. Another possible factor is the presence of sludge solids in the condensate during the SRAT cycle which may have mitigated the loss of scrubber efficiency.

Scoping tests are being performed in the laboratory by bubbling a 1000 ppm ammonia/nitrogen vapor through the following liquids:

- (1) PX-7 SMECT condensate from the SME cycle.
- (2) PX-7 SMECT condensate from the SRAT cycle.
- (3) Distilled water/nitric acid at pH of sample 1.
- (4) Condensate produced from SRAT slurry with antifoam added (this condensate will contain strippable components from antifoam) at pH of sample 1.

These liquids will have the same volumetric flow of ammonia at the same concentration and will be analyzed for pH and ammonium to determine their ability to scrub ammonia. If it is determined that the antifoam has caused the scrubber performance problem, a new antifoam agent will likely be needed prior to DWPF's WP-17 testing. Further testing may be required to determine if cause (1) or (3) is hindering scrubber performance if liquid resistance is not the cause.

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Appendix A - Problems Noted During Testing

SRAT Water Testing

A problem was noted during water testing that allowed vapor to bypass the scrubber. The vapor was drawn from the SRAT through the Mercury Water Wash Tank (MWWT) to the PVVS subheader, allowing approximately 50% of the vapor flow to bypass the scrubber. Closing a valve isolating the MWWT directly from the PVVS subheader eliminated this leak path. No other scrubber problems were noted during water testing.

Scrubber Testing During SRAT Processing

A second problem was encountered during PHA addition with vapor bypassing the scrubber similar to the above problem with the MWWT. There is a three-way valve that allows the SRAT condenser condensate to drain to either the SRAT or the SMECT. When the three way valve was open to drain condensate to the SMECT, a vapor path from the SRAT through the condensate drain leg, to the SMECT tank, and finally to the PVVS subheader caused approximately 50% of the vapor flow to bypass the scrubber. A blind flange was installed on the SMECT isolating the SMECT directly from the PVVS subheader. This modification eliminated the vapor leak path.

The scrubber exit vapor stream was noted to be very wet during PHA addition. At times fog was observed in the scrubber vapor exit during the PHA addition to the SRAT. These problems cleared up during the later part of the PHA addition.

Solids buildup in the scrubber is suspected because of the differential pressure increase from 0.8 - 2.5 inwc (Figure 3). This is similar to increase in differential pressure noted during ammonia scrubber testing with cylinder supplied ammonia. The solids buildup was caused by the carryover of solids from the SRAT to the SMECT during SRAT processing.

Appendix B - Ammonia Analysis of Vapor Streams

Ammonia Scrubber Testing During SRAT Processing

Dräger tubes specific for ammonia were used to determine the ammonia vapor concentration during SRAT processing. The ammonia Dräger tube uses a bromphenol blue indicator specific for ammonia. The Ammonia 2/a tube has a detection range of 2-30 ppm ammonia. Also, to ensure the Dräger tube analysis was within the 10-15% accuracy quoted in the technical literature¹⁸, the vapor sample must have an absolute humidity between 3 and 12 mg/L water vapor.

The water vapor 0.1 Dräger tube was used prior to the ammonia measurement to verify the humidity was within the 3-12 mg/L range. Twenty-four measurements were made with the humidity tube. The absolute humidity ranged from 1 mg/L to 22 mg/L with five samples above 12 mg/L. These high humidity readings (13, 22, 13, 14, 13) occurred during the later part of PHA addition. At this time the SRAT level was much higher than planned. A fog was noted in the vapor exiting the scrubber at this point.

The sample point for the scrubber exit vapor sample is a sample tap directly at the outlet of the scrubber. This modification was made because of concerns that the sample pumped to the analyzer house may not be representative of the process. The sample for the scrubber entrance vapor sample is in the analyzer house.

An ADS developed analyzer was installed prior to operations, but because of materials coating the lenses of the analyzer and line pluggage (with mercury compounds), it was not reliable during SRAT processing.

Ammonia Scrubber Testing During SME Processing

Various Dräger tubes specific for ammonia were used during the SME cycle and during subsequent testing. The humidity was constant at approximately 35 mg/L during the cycle, higher than the 3-12 mg/L required to ensure the sample is within the 10-15% accuracy promised.

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- ¹⁸ Dräger tube CH 31901, *Ammonia, Instructions for Use*, 10th Edition, June 1991.

Table 1
Design basis for DWPF and IDMS scrubbers

Design Parameter	DWPF		IDMS	
	SRAT	SME	SRAT	SME
Height, inches	111	60	111	60
Diameter, inches	18.812	11.938	7.981	5.047
Liquid Flow, gpm	20	10	3.60	1.79
Vapor Flow, lb/hr	1645	404	296	72
Ammonia, lb/hr	1.2	2.5	0.22	0.45
DWPF Scale			1:5.56	1:5.59

Note: The SME scrubber basket is installed in IDMS

Table 2
PX-7 Ammonia Scrubber Testing Conditions

	IDMS Scaled Design Basis	IDMS SRAT Testing	IDMS SME Testing
Vapor Flow , lb/hr	72	95-100	68-76
Liquid Flow, gpm	1.79	1.85	1.85
Measured pH	<3	1-2.5	1.8-4.5

Table 3
Ammonia Scrubber Comparison

Test	Liquid Flow, gpm	Vapor Flow, lb/hr	Differential Pressure, inwc	Ammonia Inlet, ppm	Ammonia Outlet, ppm
SME-9,11 ¹	1.7-1.8	77-78	0.9-1.5	17,100	<2
SME-10,12 ¹	1.7-1.8	76-77	0.9-1.1	12,600 ¹	<2
PX-7 SME	1.85	78	0.5	1,000-3,000	>70

1. Off-line testing using cylinder supplied ammonia.

Table 4
PX-7 SME Ammonia Scrubber Data

Date	Time	Scrubber Inlet Ammonia	Scrubber Outlet Ammonia	Comment
4/19/95	5:30		<2	4/13/95 Added Frit, water and ammonium nitrate 02:00 Added 1400 g Antifoam
4/19/95	15:12		<2	15:25 Started SRAT Concentration
4/19/95	16:35		<2	15:45 1400 g Added Antifoam
4/19/95	18:45		<2	19:00 Cooldown Initiated
4/20/95	1:33		<2	Added Frit and Water and 1400 g antifoam
4/20/95	2:33		<2	2:40 Started Steam to SRAT
4/20/95	3:33		<2	
4/20/95	4:33		<2	
4/20/95	5:33		<2	
4/20/95	6:33		<2	
4/20/95	7:33		<2	7:30 Started SRAT Refluxing
4/20/95	8:33	400	<2	8:50 Added acid to SMECT
4/20/95	9:45	670	<2	9:00 Started SRAT Concentration
4/20/95	10:45	1000	2	
4/20/95	11:15	3000	20	
4/20/95	11:30		>70	
4/20/95	11:40		>70	
4/20/95	12:00		>70	
4/20/95	12:25		5	12:20 Started SRAT Refluxing
4/20/95	12:30	2000	5	
4/20/95	12:40		23	
4/20/95	12:50		17	12:50 Cooldown Initiated
4/20/95	13:00	500	6	
4/20/95	13:10	700	2	
4/20/95	13:20	530	1.3	
4/20/95	13:30	520	1.1	
4/20/95	13:40	430	0.9	
4/20/95	13:50	350	1	
4/20/95	14:00	300	0.6	
4/20/95	14:10	230	0.9	
4/20/95	14:30	130	0.2	

Table 4 Continued
PX-7 SME Ammonia Scrubber Data

Date	Time	Scrubber Inlet Ammonia	Scrubber Outlet Ammonia	Comment
4/20/95	15:05	75	0.6	
4/20/95	17:40	42	5	17:00 Started Steam to SRAT
4/20/95	18:10	200	5	17:20 Added 1400 g antifoam
4/20/95	18:55	350	4	18:45 Started SRAT Refluxing
4/20/95	19:10	600	18	
4/20/95	19:30	1000	50	
4/20/95	20:00	1500	225	
4/20/95	20:25	1000	250	
4/20/95	20:55	1000	150	
4/20/95	21:25	1000	275	
4/20/95	21:55	1000	350	
4/20/95	22:25	1000	200	
4/20/95	22:55	900	350	23:00 Started SRAT Concentration
4/20/95	23:25	900	400	
4/20/95	23:55	900	450	
4/21/95	0:25	900	400	
4/21/95	0:55	800	450	12:50 Cooldown Initiated
4/21/95	1:25	800	350	
4/21/95	1:55	1000	350	
4/21/95	2:25	500	150	
4/21/95	2:55	500	100	
4/21/95	3:25	200	80	
4/21/95	3:55	160	50

Figure 2
SMECT pH during SRAT Processing

Figure 3
Scrubber Differential Pressure during SRAT Processing

Figure 4
SMECT pH during SME Processing

Figure 5
Scrubber Differential Pressure during SME Processing

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