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**FINAL WASH PRECIPITATE FEED SIMULANTS FOR DWPF
COLD CHEMICAL RUNS (U)**

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MEMORANDUM

May 15, 1992

To: E. W. Holtzscheiter, 773-A

From: J. C. Marek, 704-T *JCM*

**FINAL WASH PRECIPITATE FEED
SIMULANTS FOR DWPF COLD CHEMICAL RUNS (U)**

SUMMARY

Precipitate feed simulant specifications for Defense Waste Processing Facility (DWPF) Cold Chemical Runs are revised in this report. All previous specifications of precipitate feed simulants for DWPF Cold Chemical Runs (CCRs)¹⁻⁷ are superceded by this document. The revision is needed to 1) eliminate use of hydroxylamine nitrate (HAN) in the precipitate process operation, 2) simulate the projected composition of the precipitate feed to DWPF produced by final washing to reduce the nitrite concentration to $\leq 0.01M$ without washing the simulated feed, 3) delete organic trim chemical additions to the precipitate feed and 4) specify an additive to prevent foaming of the precipitate. Two specifications are provided to operate the Salt Processing Cell (SPC) with mercury (Tests 5&6) and without mercury (Tests 1-4) in the precipitate feed.

INTRODUCTION

The Defense Waste Processing Facility (DWPF) is scheduled to start non-radioactive process operation in September, 1992. The Cold Chemical Runs will demonstrate acceptable process operation and provide data required to satisfy the Waste Compliance Plan⁸. Five basic sludge simulants will be required for these tests⁹. Two basic precipitate simulants will be needed to operate the SPC with and without mercury in the precipitate feed.

Authorized Derivative Classifier

C. T. Randall
C. T. Randall, 704-T
Manager, DWPT

DISCUSSION

Calculational Bases

The calculational bases for the DWPF CCR precipitate feed specifications were derived from WSRC-TR-92-211, Rev. 010. This material balance for the In-Tank Precipitation and DWPF flowsheet incorporates late (or final) washing to reduce the nitrite content of the precipitate feed stored in Tank 49 to $\leq 0.01M$ prior to transfer to the DWPF Salt Process Cell. The precipitate feed, sludge feed, and resulting glass component flows were calculated using the above bases and current tank farm blending schedules, assuming the nitrite concentration in Tank 49 is $0.08M$ and the final washing reduces the nitrite content to $0.01M$. The integrated processing computer model developed using CPES software was used for the calculations.

Variability of Precipitate Feed to DWPF

The precipitate feed to DWPF will have little compositional variability until the existing waste inventory has been processed¹¹. This is because the In-Tank Process (washing the precipitate slurry and filtering to a constant 10 wt% insoluble solids in Tank 48) and the final washing to $\leq 0.01M$ nitrite remove most of the soluble salts, which tend to vary the most in concentration from waste tank to waste tank. The nitrite concentration in Tank 49 during radioactive operation is projected to exceed the $0.08M$ calculational basis assumed in WSRC-TR-92-211. The nitrite concentration will likely be about $0.17M$, but the late washing operation will remove only an additional 6% of the soluble species from the precipitate feed. The greatest variability will likely occur for the minor soluble anions and insoluble sludge components present because sludge is entrained to varying degrees in the salt waste. Glass composition will be kept relatively constant by the appropriate blending of precipitate and sludge feeds.

Simulated Precipitate Slurry Preparation

Preparation of the simulated precipitate feed is outlined in Figure 1. A reliable method to prepare the precipitate simulant is to combine a vendor supplied solution of potassium salts with a vendor supplied sodium tetraphenylborate (NaTPB) solution. Specifications and compositions of the required salt and NaTPB solutions are shown in Tables 1-3. The NaTPB solution is nearly identical in composition to that specified for the In-Tank (Precipitation) Process (ITP), with two minor changes. First, for water balancing between the salt and NaTPB solutions the NaTPB is at a slightly lower concentration of $0.5 M \pm 0.05 M$ rather than $0.55 M \pm 0.05 M$ specified for ITP. Second, the maximum allowable halide content has been reduced to 100 ppm in the NaTPB solution used in preparing the feed simulants.

Surfactant (antifoam) additions to the salt solution before precipitation are recommended to disperse the precipitate solids (which greatly enhances the ability to quantitatively transfer the slurry) and prevent foaming and subsequent segregation of solids from the supernate. The recommended surfactant is 2,4,7,9-tetramethyl-5-decyn-4,7-diol. Tests to determine the minimum effective surfactant

concentration are in progress but for procurement purposes it can be assumed that a maximum of 0.3 wt% of the active ingredient is required in the precipitate slurry. The surfactant is a white, waxy solid material but is available in liquid form from Air Products Co. under the trade name Surfynol® 104E, as a 50/50 weight mixture of the surfactant in ethylene glycol solvent. Surfynol® 104E has been tested in the SRTC laboratories and PHEF at TNX with positive results and must be added at 0.6 wt% to obtain 0.3 wt% active surfactant.

The order of addition of the various reagents to the feed preparation tank is 1) the potassium salt solution, 2) the sodium titanate, 3) the surfactant solution followed by 4) the NaTPB solution to form the white tetraphenylborate precipitate. To generate diphenylmercury in Tests 5&6, the formation of the precipitate is followed by addition of an aqueous mercuric nitrate solution to form diphenylmercury. The tank should be agitated during all additions.

Specifications and compositions of the required trim chemicals are shown in Tables 1-2. Organic trim chemicals that were recommended in previous specifications have been deleted from this specification for the following reasons:

- 1) The latest analytical results for irradiated feeds indicate lower values for organic high-boilers than had been found previously. These data were part of the late washing technical assessment.
- 2) Addition of the organic trim chemicals requires additions of benzene in gross excess of the material balance projections.
- 3) Information to be obtained in Cold Chemical Runs on decanter and stripping performance will not be sacrificed because other organics (e.g. diphenylamine) can be monitored.
- 4) It is not possible to add these materials in the same state they are present in irradiated precipitates - in irradiated precipitates they are present within the solid potassium tetraphenylborate crystal.
- 5) The addition of the organics has at least once caused a severe operating difficulty in PHEF operations¹² that could potentially hinder achieving the objectives and schedule for Cold Chemical runs. It was related to item 4) above and not to any phenomena anticipated for radioactive feeds.

Simulated Precipitate Composition

Compositions of the simulated precipitate feeds for DWPF CCRs are shown in Tables 4-5. The simulants have nitrite concentrations of 0.01M and differ only in mercury content. The alkali content is maintained constant for all the feeds by including the appropriate amount of potassium nitrate where necessary.

All the primary inorganic cations present at greater than 0.1 wt% in the design basis material balance¹⁰ were retained in the washed precipitate feed simulant. Some compounds present at less than 0.1 wt% were retained because they are important for glass product

composition. These include sodium metaborate, silicate, chromate, and molybdate. Other minor compounds were retained because they react in precipitate hydrolysis or are key corrosion species, including sodium carbonate and sulfate. Ammonium ion was deleted from the specification because 1) it generates toxic ammonia fumes when present in alkaline media and 2) ammonia scrubbers will not be installed in the CPC before the start of CCRs. Anions such as aluminate were omitted because including them in the precipitate feed would have little or no effect on glass composition or the Precipitate Process.

Insoluble sodium titanate was included in the precipitate feed simulant because it is important for demonstration of glass quality and process control. Sludge solids were deleted from the precipitate specification because the actual quantity of sludge solids entrained in the salt waste is highly variable, the sludge has no impact on the CCR objectives, and the sludge would contribute excessive nitrite.

There is still considerable activity within the Interim Waste Technology Division regarding sodium titanate requirements for the ITP. The amount of sodium titanate specified here is near the upper level that is currently anticipated for most real feeds to DWPF¹³. The sodium titanate material procured for addition to SRP waste tanks in the ITP is unacceptable for preparing simulants for DWPF Integrated Cold Runs. The sodium titanate specified for the ITP is slurried as 20 wt% titanate in methanol and isopropanol. The alcohols will be substantially removed by washing and filtering before the precipitate is fed to DWPF. The sodium titanate solution desired for the precipitate simulant should have a total alcohol content equivalent to or less than that shown for isopropanol in Tables 1 and 2. The alcohol content of the sodium titanate can be reduced by the vendors by either batch washing and decantation or by distillation. The sodium titanate solution should not contain ethanol or other two-carbon solvents for reasons relating to process safety.

Simulant Compared to Expected Radioactive Feeds

The washed precipitate feed simulant includes potassium at the reference CPES calculated level (molar basis). The precipitate simulant will have considerable excess sodium and nitrate in relation to the CPES material balance with final washing. The excess sodium and nitrate are the result of the simplified precipitation method used to prepare the feed without a subsequent washing operation. The excess sodium in the precipitate simulant has been corrected for by removing the appropriate amount of sodium hydroxide from the sludge simulant specification. This will provide the proper feed specification to the glass melter. An alternative to prepare precipitate simulant with the reference sodium and nitrate levels would be to wash the feed in the pump pits. This alternative was rejected because it would require the addition of washing facilities and the objectives of the CCRs can be met with the specified precipitate simulant.

Quantities of Materials

Table 6 shows the quantities of feed and product simulants that will be required for each DWPF Integrated Cold Run. These quantities were

calculated using blending rates for the PHA and glass¹⁴ and the appropriate melter turnover volumes¹⁵. Over 1.1 million pounds of precipitate slurry will be needed for the six tests.

Allowable Variance in Component Concentrations

The task team responsible for developing specifications for all feed materials has recommended maximum allowable variances for the simulant specifications⁹. The allowable variances are summarized in Table 7. The specification for nitrite in the precipitate slurry simulant is $\pm 10\%$ to control the nitrite concentration at the late washing target of 0.01M. The specifications for the total halide (chloride plus fluoride) content of the salt solution (20 ppm) and the NaTPB solution (100 ppm) as well as the allowable variance on sulfate ($\pm 10\%$) are important for corrosion control purposes. Other components present at levels of greater than 1.5 wt% (dry basis) can vary $\pm 10\%$. Components present in the precipitate simulant at less than 1.5 wt% but greater than 0.01 wt% (dry basis) can vary $\pm 25\%$, and components specified in the precipitate simulant at less than 0.01 wt% (dry basis) can vary $\pm 75\%$. The latter specification is based on analytical sensitivity limitations. Iron and aluminum impurities should be less than 200 ppm (dry basis). Because the precipitate simulant is prepared by the combination of two solutions, the total quantity of salt solution and NaTPB solution combined to produce the feed should be controlled to within $\pm 2.5\%$ of the amounts shown in Tables 1&2 to obtain an acceptable variance in the component concentrations.

QUALITY ASSURANCE

A computer model was developed by the author to calculate the compositions and quantities of chemicals required to prepare the precipitate slurry simulant and the precipitate hydrolysis aqueous product simulant. The model is contained on a Microsoft® Excel spreadsheet that runs on an Apple® Macintosh™ personal computer. The computer model contains internal checks for errors by mass balance calculations. The same model has been used successfully to calculate precipitate feed simulants for research and development work in the Precipitate Hydrolysis Experimental Facility at TNX.

Calculations were verified by the author and by the leader of the SRTC Cold Feed Task Team. Verification included comparison of hand calculations to output of the computer model and a check of each calculational cell formula. A copy of the hand calculations and spreadsheet formulas have been added to the task team leader's file of pertinent DWPF Cold Feed Run documents. These documents will be placed in a permanent QA file upon completion of all task team feed specification work. A copy of each set of calculations has been placed on floppy disks that will be stored permanently in the DWPF Cold Feed Run QA file.

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FIGURE 1

PREPARATION OF PRECIPITATE SIMULANT

TABLE 1. REAGENTS FOR PRECIPITATE WITH NO MERCURYSALT SOLUTION

<u>COMPCUND</u>	<u>LB/1000 LB SLURRY</u>	<u>WT% WET BASIS</u>	<u>WT% DRY BASIS</u>
H2O	437.32	94.89	0
KNO3	19.53	4.24	83.0
K2CO3	1.73	0.375	7.34
KNO2	0.76	0.165	3.24
CsNO3	0.52	0.11	2.23
C6H5OH	0.45	0.098	1.92
K2C2O4	0.29	0.063	1.24
B(OH)3	0.20	0.043	0.83
K2SO4	0.043	0.009	0.18
K3PO4	0.0033	0.0007	0.14 0.014
K2CrO4	0.001	0.0002	0.004 <i>gem</i>
K2SiO3	0.0009	0.0002	0.004
K2MoO4	0.00016	0.000035	0.0007
TOTAL	460.85	100	100

SODIUM TITANATE SLURRY

<u>COMPOUND</u>	<u>LB/1000 LB SLURRY</u>	<u>WT% WET BASIS</u>	<u>WT% DRY BASIS</u>
NaTi2O5H	1.85	19.9992	99.996
(CH3)2CHOH	0.0001	0.0008	0.004
H2O	7.4	80.0	0.0
TOTAL	9.25	100	100

SURFACTANT: 2,4,7,9-tetramethyl-5-decyn-4,7-diol, 50 wt% in ethylene glycol. Quantity to be specified.

SODIUM TETRAPHENYLBORATE (NaTPB) SOLUTION

<u>COMPOUND</u>	<u>LB/1000 LB SLURRY</u>	<u>WT% WET BASIS</u>	<u>WT% DRY BASIS</u>
H2O	437.29	82.52	0
NaTPB	90.67	17.11	97.9
NaOH	1.93	0.37	2.1
TOTAL	529.89	100	100

TABLE 2. REAGENTS FOR PRECIPITATE WITH MERCURYSALT SOLUTION

<u>COMPOUND</u>	<u>LB/1000 LB SLURRY</u>	<u>WT% WET BASIS</u>	<u>WT% DRY BASIS</u>
H2O	417.33	94.69	0
KNO3	19.53	4.43	83.5
K2CO3	1.73	0.39	7.39
KNO2	0.76	0.17	3.26
CsNO3	0.52	0.12	2.24
C6H5OH	0.45	0.10	1.94
K2C2O4	0.29	0.066	1.25
B(OH)3	0.05	0.011	0.21
K2SO4	0.043	0.010	0.18
K3PO4	0.0033	0.00075	0.014
K2CrO4	0.001	0.0002	0.004
K2SiO3	0.0009	0.0002	0.004
K2MoO4	0.00016	0.000036	0.0007
TOTAL	440.72	100	100

SODIUM TITANATE SLURRY

<u>COMPOUND</u>	<u>LB/1000 LB SLURRY</u>	<u>WT% WET BASIS</u>	<u>WT% DRY BASIS</u>
NaTi2O5H	1.85	19.9992	99.996
(CH3)2CHOH	0.0001	0.0008	0.004
H2O	7.4	80.0	0.0
TOTAL	9.25	100	100

SURFACTANT: 2,4,7,9-tetramethyl-5-decyn-4,7-diol, 50 wt% in ethylene glycol. Quantity to be specified.

SODIUM TETRAPHENYLBORATE (NaTPB) SOLUTION

<u>COMPOUND</u>	<u>LB/1000 LB SLURRY</u>	<u>WT% WET BASIS</u>	<u>WT% DRY BASIS</u>
H2O	441.2	82.52	0
NaTPB	91.48	17.11	97.9
NaOH	1.94	0.37	2.1
TOTAL	534.6	100	100

MERCURIC NITRATE SOLUTION

<u>COMPOUND</u>	<u>LB/1000 LB SLURRY</u>	<u>WT% WET BASIS</u>	<u>WT% DRY BASIS</u>
Hg(NO3)2	1.54	10.0	100
H2O	13.86	90.0	0
TOTAL	15.4	100	100

**TABLE 3: Anion, Cation, Organic and Elemental
Content of Salt and Sodium Tetraphenylborate Solutions**

<u>Salt Solution</u>	<u>Wt% DRY BASIS</u>	
<u>Specie</u>	<u>Tests 1-4</u>	<u>Tests 5&6</u>
K ⁺	38.4	38.65
NO ₃ ⁻	51.6	51.9
CO ₃ ²⁻	3.2	3.2
C ₆ H ₅ OH	1.92	1.94
NO ₂ ⁻	1.75	1.76
Cs ⁺	1.52	1.53
B(OH) ₃	0.83	0.21
C ₂ O ₄ ²⁻	0.66	0.66
B	0.15	0.037
SO ₄ ²⁻	0.10	0.10
PO ₄ ³⁻	0.0062	0.0063
CrO ₄ ²⁻	0.0025	0.0026
SiO ₃ ²⁻	0.0020	0.0019
Cr	0.0011	0.0012
Si	0.00074	0.00070
MoO ₄ ²⁻	0.00045	0.00046
Mo	0.00027	0.00028
<u>NaTPB Solution</u>	<u>Wt% DRY BASIS (all tests)</u>	
<u>Specie</u>	<u>All Tests</u>	
Na ⁺	7.77	
OH ⁻	0.885	
TPB ⁻	91.35	

TABLE 4. COMPOSITION OF PRECIPITATE SIMULANT FOR TESTS 1-4

<u>COMPOUND</u>	<u>WT% WET BASIS</u>	<u>WT% DRY BASIS</u>
H ₂ O	88.2	0
KTPB	8.28	70.1
NaNO ₃	1.66	14.1
NaTPB	0.97	8.21
CsTPB	0.243	2.06
NaOH	0.186	1.58
NaTi ₂ O ₅ H	0.185	1.57
Na ₂ CO ₃	0.13	1.12
NaNO ₂	0.062	0.52
C ₆ H ₅ OH	0.045	0.38
Na ₂ C ₂ O ₄	0.024	0.20
Na ₂ B ₄ O ₇	0.016	0.135
Na ₂ SO ₄	0.0035	0.03
Na ₃ PO ₄	0.00025	0.0022
Na ₂ CrO ₄	0.00003	0.0007
Na ₂ SiO ₃	0.00007	0.0006
Na ₂ MoO ₄	0.00001	0.0001

TABLE 5. COMPOSITION OF PRECIPITATE SIMULANT FOR TESTS 5&6

<u>COMPOUND</u>	<u>WT% WET BASIS</u>	<u>WT% DRY BASIS</u>
H ₂ O	88.0	0
KTPB	8.28	68.8
NaNO ₃	1.75	14.5
NaTPB	0.97	8.06
CsTPB	0.243	2.02
NaTi ₂ O ₅ H	0.185	1.54
Hg(C ₆ H ₅) ₂	0.168	1.4
NaOH	0.16	1.33
Na ₂ CO ₃	0.132	1.10
NaNO ₂	0.062	0.51
C ₆ H ₅ OH	0.045	0.38
Na ₂ C ₂ O ₄	0.024	0.20
Na ₂ B ₄ O ₇	0.016	0.133
Na ₂ SO ₄	0.0035	0.03
Na ₃ PO ₄	0.00025	0.002
Na ₂ CrO ₄	0.00008	0.0007
Na ₂ SiO ₃	0.00007	0.0006
Na ₂ MoO ₄	0.00001	0.0001

TABLE 6. QUANTITIES OF COLD FEED SIMULANTS FOR SPC

<u>CAMPAIGN</u>	<u>LBS PRECIPITATE SIMULANT</u>	
	<u>Minimum*</u>	<u>10% Excess</u>
Test 1 Blended Sludge	281,050	309,160
Test 2 Blend w/Nd Tracer	234,310	257,740
Test 3 Low Viscosity, Purex	126,930	139,620
Test 4 High Viscosity, HM	133,940	147,340
Test 5 Batch 1 with Hg	208,280	229,110
Test 6 <u>Batch 1/Spike</u>	<u>208,280</u>	<u>229,110</u>
Total	1,192,790	1,312,080

* Represents value required for the Cold Chemical Runs with no allowance for excess quantities, rounded up to nearest 10 lbs.

TABLE 7. ALLOWABLE VARIANCES IN PRECIPITATE FEED SIMULANTSGENERAL VARIANCES

<u>COMPONENT</u>	<u>ALLOWABLE VARIANCE</u>
NO ₂ -, SO ₄ =	± 10%
Other Components > 1.5 wt% (dry basis)	± 10%
Other Components < 1.5 wt% but > 0.01 wt% (dry basis)	± 25%
Other Components < 0.01 wt% (dry basis)	± 75%
Fe, Al (dry basis)	200 ppm max.
Maximum variation in batch size quantities of potassium salt solution and NaTPB solution	± 2.5%
Halides (Chloride, Fluoride)	
- Salt Solution	20 ppm max.
- NaTPB Solution	100 ppm max.

NaTPB SOLUTION

<u>COMPONENT</u>	<u>ALLOWABLE VARIANCE</u>
Sodium Tetraphenylboron, NaTPB	± 0.05 M
Sodium Hydroxide, NaOH	± 0.025 M
Benzene	< 650 ppm
Halides (Cl, F)	< 100 ppm
Copper	< 5 ppm
pH	12.5 ± 1.5

TABLE 7, cont'd. ALLOWABLE VARIANCES
IN PRECIPITATE FEED SIMULANTSPOTASSIUM SALT SOLUTION

<u>COMPONENT</u>	<u>ALLOWABLE VARIANCE</u>
NO ₂ -, SO ₄ =	± 10%
Other Components > 1.5 wt% (dry basis)	± 10%
Other Components < 1.5 wt% but > 0.01 wt% (dry basis)	± 25%
B, Cr, Mo, Si	± 75%
Benzene	< 650 ppm
Halides (Cl, F)	< 20 ppm
Copper	< 10 ppm
Fe, Al	< 200 ppm
Total Insoluble Solids	< 2.0 wt% wet basis
pH	8 - 12

SODIUM TITANATE SLURRY

<u>COMPONENT</u>	<u>ALLOWABLE VARIANCE</u>
Monosodium Titanate, NaTi ₂ O ₅ H	200 ± 20 g/l
Total Alcohol Content	≤ 0.0008 wt%
NaOH	0.10 ± 0.02 M
Total Soluble Inorganic Salts	< 20 g/l
Total Halide (Cl, F)	< 300 ppm
Total Carbon	< 100 ppm
Particle Size distribution	
Percent smaller than 1 micron	< 1%
Percent larger than 35.5 microns	< 1%

END

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