CENTER FOR NUCLEAR WASTE REGULATORY ANALYSES

TECHNICAL OPERATING PROCEDURE

Proc. TOP-010
Revision 0
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Title: TECHNICAL OPERATING PROCEDURE FOR PREPARING SIMULATED J-13 WATER AND ITS MODIFICATIONS

EFFECTIVITY AND APPROVAL

Revision 0 of this procedure became effective on 5-8-90. This procedure consists of the pages and changes listed below.

Page No.	Change	Date Effective
ALL	-	5-8-90

Supersedes Procedure No. NONE

Approvals

Written By	Date	Technical Review	Date
Narasi Sridhar	5/8/90	J. C. Griffin	5/8/90
Quality Assurance	**Date**	**Cognizant Director**	**Date**
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CNWRA Form TOP-1

TOP-010
TECHNICAL OPERATING PROCEDURE FOR PREPARING SIMULATED J-13 WATER AND ITS MODIFICATIONS

1. PURPOSE

The purpose of the document is to describe procedures for preparing aqueous solutions to be used in various corrosion and electrochemical tests on candidate HLW container materials. This procedure establishes controls required by CQAM Section 3, "Scientific Investigations and Analysis Control."

2. SCOPE

This document provides procedures for the preparation of aqueous solutions to be used in conducting corrosion and electrochemical tests on candidate HLW container materials. Specifically, the procedures address the preparation of:

1. An aqueous solution that approximates the reported composition of a water sample obtained from test well J-13 near Yucca Mountain, Nevada. The reported composition of the J-13 well water is shown in Table 1. The solution prepared according to this procedure will be referred to as Simulated J-13 water.

2. Aqueous solutions whose compositions have been modified from the Simulated J-13 water with respect to one or more ionic species. The intents of these modifications may be to identify the quantitative effects of environmental variables on corrosion and conduct corrosion studies under a variety of postulated extreme conditions of the repository environment. The compositions of these modifications may contain fewer ionic species than both the actual J-13 and simulated J-13 waters in order to better identify functional dependencies of corrosion. The solutions prepared under this procedure will be referred to as either Modified Simulated J-13 water or, if the changes are extensive, as Model Solutions.

3. APPLICABLE DOCUMENTS

The following documents are relevant to the procedures described in this document:

1. ASTM E 288 -(Latest Revision), "Standard Specification for Volumetric Flasks"

2. Federal Specification NNN-P-395 (Latest Revision), "Specification for Volumetric Pipets"

4. RESPONSIBILITY

4.1 The cognizant Element Manager shall be responsible for the development and maintenance of this procedure.

4.2 The cognizant Principal Investigator shall be responsible for the implementation of this procedure.

4.3 Personnel performing tests described in this procedure are responsible for complying with its requirements.

5. EQUIPMENT AND CHEMICALS

5.1. Equipment

1. ASTM E288 Class A volumetric flasks calibrated to contain 1000 ml

2. NNN-P-395 Class A volumetric pipets calibrated to deliver 25 ml, 1ml, or other small quantities

3. Analytical balance accurate to at least $\pm$ 0.1 mg and calibrated periodically.

4. pH meter accurate to at least $\pm$ 0.01 pH unit and appropriate glass electrodes, calibrated before use.

5. Selective Ion analyzer accurate to $\pm$ 1% and electrodes, calibrated before use.

5.2 Chemicals

Solutions shall be prepared using high purity water (about 17-18 Mohms.cm resistivity). The chemicals used for preparing the solutions shall meet the purity specified in American Chemical Society Standards (ACS Grade) where possible. In some chemicals ACS Grade may not be available and a certified grade such as Fisher Certified Grade may be used. The lot number of each chemical used in the preparation of a given batch of solution shall be recorded in the Scientific Notebook. The following chemicals are needed for the preparation of Simulated J-13 water:

[1] 0.1 N NaOH

[2] $CaSO_4.2H_2O$

[3] $Ca(NO_3)_2.\ 4H_2O$

[4] $KF.\ 2H_2O$

[5] $MgCl_2.\ 6H_2O$

[6] $NaHCO_3$

[7] KCl

6. IDENTIFICATION AND DESCRIPTION OF SOLUTIONS

The solutions prepared in accordance with the procedure described in this section shall be identified on the label as shown below:

Sample Label:

```
**********************************************************
*                                                        *
* Preparation Date:                  Expiration:         *
*                                                        *
* Identification: SN-CNWRA "notebook #"-"page #"         *
*                                                        *
* Description: J-13 Stock Solution A                     *
*                                                        *
*                                                        *
**********************************************************
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The identification of the solution shall be entered in the appropriate Scientific Notebook. The actual concentrations of the components added to the solution shall be entered in the Scientific Notebook. The units for the components in the solutions shall be milligrams/liter of solution for solids and milliliters/liter of solution for liquids. For the liquid components, the concentration (either as weight percent, molarity or normality) or density of the liquid added shall also be entered.

7. PROCEDURE FOR PREPARATION OF SIMULATED J-13 WATER

7.1 Preparation of Stock Solution A

1. Add the following to 900 ml of high purity water at room temperature in a beaker and stir until dissolved:

   1.377 g $CaSO_4 \cdot 2H_2O$

   Then add the following components to the solution and stir until dissolved fully:

   0.944 g $Ca(NO_3)_2 \cdot 4H_2O$
   0.119 g KCl
   0.569 g $MgCl_2 \cdot 6H_2O$

   Care should be taken to weigh and add $MgCl_2$ quickly since it is hygroscopic and can cause inaccuracies in weight measurement.

2. Pour the solution in a 1000 ml volumetric flask and adjust the volume to 1000 ml with high purity water by washing the beaker with the needed water and pouring the wash water into the flask.

3. Stopper flask and label it as described in section 6. Record preparation steps in the Scientific Notebook as described in section 6.

7.2 Preparation of Stock solution B

1. Add the following chemicals to 900 ml of high purity

water at room temperature in a beaker and stir until dissolved:

6.720 g $NaHCO_3$
0.376 g $KF.2H_2O$

Care should be taken to weigh and add the $KF.2H_2O$ quickly since it is hygroscopic and can cause inaccuracies in weight measurements.

2. Pour solution into a 1000 ml volumetric flask and adjust the total volume to 1000 ml with high purity water by washing the beaker with the needed water and pouring the wash water into the flask.

3. Stopper the flask and label it as described in section 6. Record preparation steps in the Scientific Notebook as described in section 6.

7.3 Preparation of Test Solution

1. Place 925 ml of high purity water in a volumetric flask and pipet 25.0 ml of Stock Solution A and 25.0 ml of Stock Solution B into the flask.

2. Add high purity water to make up the volume to 1000 ml. Stopper the flask and label it as described in section 6. Record preparation steps in the Scientific Notebook as described in section 6.

3. Measure pH and record it in the Scientific Notebook along with the solution batch number and preparation date as described in section 6.

4. Measure the chloride concentration of the test solution with a selective ion electrode (suitably calibrated) and record the value in the Scientific Notebook.

7.4. Storage Of Solutions

The stock solutions shall be used within 15 days of preparation. The test solution shall be used within 5 days of preparation.

8. PREPARATION OF MODIFIED SOLUTIONS BASED ON SIMULATED J-13 WATER

Modifications to the simulated J-13 water may be made to study the effect of environmental variables on corrosion. The types of modifications that may be made are described below. In all these cases, the solutions shall be identified and documented as described in Section 6.

8.1. Increase in the Concentration of Chloride: This increase shall be accomplished by the addition of potassium chloride to Stock Solution A. The total amount of salt added shall be recorded in the Scientific Notebook along with the identification of the solution and preparation date.

8.2. Modification of pH: This shall be accomplished by the use of a suitable reagent (buffer, acid or alkali). Specific recommendations of a reagent solution may depend on the needs of the program and may be a compromise between the need to maintain the pH at the desired value and the need to limit other electrochemical changes to the environment. The reagent solution shall be added to the test solution (Step 6.4 above). If a reagent is used to alter the pH, the type and concentration of reagent used, the pH at the start, and the pH at the finish of the test shall be recorded.

8.3. Other Modifications to Simulated J-13 Solution: These may involve changes in one or more species such as addition of silicic acid or sodium metasilicate. Silicon is one of the major constituents of the natural J-13 well water (Table 1). Silicon has been introduced via silicic acid in some procedures. Since the silicic acid dissolves slowly, changes in silicon concentration with time may be significant [see footnote 1]. Hence, silicic acid has been omitted in this procedure. However, other silicon compounds such as sodium metasilicate may be added to study the effect of silicon. In these cases the silicon concentration shall be measured as a function of storage time to determine stability of the solution.

Footnote 1: R. Pabalan, Memorandum, "Test of Technical Operating Procedure for Preparing Simulated and Modified J-13 Well Water," August 10, 1989.

8.4. Model Solutions: Model solutions may be solutions containing one or more ionic species present in simulated J-13 water at various concentrations. These solutions are generally used to study the effect of environmental variables on corrosion. The model solutions may not involve preparation of stock solutions and may be prepared directly by the addition of necessary amounts of salts to high purity water. The equipment and type of chemicals used shall follow the recommendations given in Section 5. The chemicals used in preparing the model solutions, if they are not listed in section 5.2, shall be identified in the Scientific Notebook regarding their grade, source, and lot number.

9. RECORDS

The files containing the test data and the Scientific Notebooks shall be controlled as records in-process in accordance with CQAM Section 17. These shall be maintained in project files until completion of the project activities at which time they will be compiled into a project data package, and processed as QA records as specified by CQAM Section 17.

Table 1. Chemical Composition of Natural J-13 Well Water (1).

Species	Concentration	
	mg/liter	mmols/liter
Li	0.04 - 0.17	0.006 - 0.024
Na	42 - 50	1.83 - 2.17
K^{+}	3.7 - 6.6	0.10 - 0.17
Mg^{2+}	1.7 - 2.5	0.07 - 0.10
Ca^{2+}	11.5 - 15.0	0.29 - 0.37
Sr^{2+}	0.02 - 0.1	0.0002 - 0.001
Fe^{3+}	<0.01 - 0.16	<0.0002 - 0.003
Al^{3+}	0.008 - 0.11	0.0003 - 0.004
Si (SiO_2)	26.6 - 31.9	0.95 - 1.14
NO_3^{-}	6.8 - 10.1	0.113 - 0.168
F^{-}	1.7 - 2.7	0.029 - 0.135
Cl^{-}	6.3 - 8.4	0.178 - 0.237
HCO_3^{-}	118 - 143	1.93 - 2.34
SO_4^{2-}	17 - 21	0.177 - 0.219
pH	6.8 - 8.3	

[1] - From W. E. Glassley, NWTRB Presentation, January 1990.