PNNL-26809, Rev 0 WTP-RPT-247, Rev 0



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# Standard High Solids Vessel Design De-inventory Simulant Qualification

### September 2017

SK Fiskum CA Burns PA Gauglitz DT Linn RA Peterson MR Smoot



Prepared for the U.S. Department of Energy under Contract DE-AC05-76RL01830

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Test Specification: Work Authorization: Test Plan: Test Exceptions: Focus Area: Test Scoping Statement(s): QA Technology Level: Project Number:

N/A WA# 048 TP-WTPSP-132, Rev 1.0 N/A Pretreatment NA Applied Research 66560

Prepared for the U.S. Department of Energy under Contract DE-AC05-76RL01830

Pacific Northwest National Laboratory Richland, Washington 99352

#### **COMPLETENESS OF TESTING**

This report describes the results of work and testing specified by Test Plan TP-WTPSP-132, Rev 1.0. The work and any associated testing followed the quality assurance requirements outlined in the Test Plan. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Test Plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.

Approved:

t

9/13/17

Reid Peterson, Manager WTP R&T Support Project Date

### **Executive Summary**

The Hanford Tank Waste Treatment and Immobilization Plant (WTP) is working to develop a Standard High Solids Vessel Design (SHSVD) process vessel. To support testing of this new design, WTP engineering staff requested that a Newtonian simulant be developed that would represent the de-inventory (residual high-density tank solids cleanout) process. Its basis and target characteristics are defined in 24590-WTP-ES-ENG-16-021<sup>1</sup> and implemented through PNNL Test Plan TP-WTPSP-132 Rev. 1.0.<sup>2</sup>

This document describes the de-inventory Newtonian carrier fluid (DNCF) simulant composition that will satisfy the basis requirement to mimic the density (1.18 g/mL  $\pm$  0.1 g/mL) and viscosity (2.8 cP  $\pm$  0.5 cP) of 5 M NaOH at 25 °C.<sup>1</sup> The simulant viscosity changes significantly with temperature. Therefore, various solution compositions may be required, dependent on the test stand process temperature range, to meet these requirements. Table ES.1 provides DNCF compositions at selected temperatures that will meet the density and viscosity specifications as well as the temperature range at which the solution will meet the acceptable viscosity tolerance.

Component	Weight Percent					
$Na_2S_2O_3\bullet 5H_2O$	28.49	26.47	25.51	24.45	22.44	
Richland City water	62.27	59.69	58.49	57.11	54.54	
Glycerin	9.24	13.84	16.00	18.43	23.03	
Temp. for 2.8 cP, °C	15	20	22	25	30	
Applicable temp. range, °C $^{(a)}$ 9 - 2314 - 2816 - 3019 - 3324 - 37						
(a) Temperature range where the $2.8 \pm 0.5$ cP viscosity tolerance is met.						

Table ES.1. Component Compositions for DNCF Simulant, 2.8 cP and 1.18 g/mL

The WTP also defined high-density plutonium oxide particles simulant to have suspension and settling properties similar to 100  $\mu$ m plutonium oxide to be mixed with the DNCF. Particles with densities ranging from 8.8 at 150  $\mu$ m to 11.4 g/mLat 100  $\mu$ m would be sufficient to meet this specification (24590-WTP-ES-ENG-16-021<sup>1</sup>). Based on interpolating, as specified in 24590-WTP-ES-ENG-16-021, the target median particle size corresponding to 11.2 g/mL is 104  $\mu$ m. Tungsten carbide particles (particle size range of 90 to 105  $\mu$ mand density of 11.2 g/mL)<sup>3</sup> met this requirement. The tungsten carbide selected is a custom-made product from XL Sci-Tech (Richland, WA). A nickel cladding has been added to the tungsten carbide primary particle to achieve the desired density; hence, the solid particle is a nickel cladded tungsten carbide (NiCladWC). Recovery of the NiCladWC suspended in the DNCF will be accomplished using in-line sock filters within the SHSVD piping system during pumpdown from the test vessel.

<sup>&</sup>lt;sup>1</sup> 24590-WTP-ES-ENG-16-021, Rev. 1. *De-inventory Testing for the Standard High Solids Vessel*. 2017. Bechtel National, Inc., Richland, Washington.

<sup>&</sup>lt;sup>2</sup> TP-WTPSP-132 Rev. 1.0, *Test Plan for PNNL WTPSP-QA Program Support of High Solids Vessel Testing*. 2017 Pacific Northwest National Laboratory, Richland Washington.

<sup>&</sup>lt;sup>3</sup> CCN 285593. April 13, 2017, by MA Trenidad and SM Knight, to RB Daniel and LK Holton, *Meeting Minutes*.

## Acronyms and Abbreviations

AEL	Atkins Engineering Laboratory
ASME	American Society of Mechanical Engineers
BNI	Bechtel National, Inc.
CFR	Code of Federal Regulations
DNCF	de-inventory Newtonian carrier fluid
DOE	U.S. Department of Energy
FIO	for information only
NiCladWC	nickel cladded tungsten carbide
NIST	National Institute of Standards and Technology
NQA	Nuclear Quality Assurance
PNNL	Pacific Northwest National Laboratory
PSD	particle size distribution
QA	quality assurance
R&D	research and development
SDS	safety data sheet (formerly material safety data sheet)
SEM	scanning electron microscopy
SHSVD	Standard High Solids Vessel Design
WTP	Hanford Tank Waste Treatment and Immobilization Plant
WTPSP	Waste Treatment Plant Support Program

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### 1.0 Introduction

The de-inventory simulant is designed to demonstrate the ability of the Standard High Solids Vessel Design (SHSVD) system to remove beyond-design-basis, high density particles from the vessel. Successful removal of 10  $\mu$ m plutonium oxide particles is in the basis of design; beyond-design-basis specifically targets removal of a 100  $\mu$ m plutonium oxide particle. This simulant will be used to test the ability of the (SHSVD) to move large, dense particles (mimicking plutonium oxide) from the vessel in a cleanout or "de-inventory" operation (24590-WTP-ES-ENG-16-021).

This document provides the composition and properties of the proposed simulant for the SHSVD testing for the Hanford Tank Waste Treatment and Immobilization Plant (WTP). It further summarizes testing conducted at the benchtop scale to filter and wash the simulant solids (nickel cladded tungsten carbide [NiCladWC]) to determine solids recovery based on mass.

The simulant is not intended to mimic any particular waste form/feed vector to the WTP, and it is designed to be non-hazardous. Thus, the DNCF is purely a physical/rheological simulant. Similarly, the NiCladWC solids component is a purely physical simulant.

#### 1.1 Target Requirements for the De-inventory Newtonian Carrier Fluid Simulant

The requirements for the DNCF simulant are to achieve the following parameters and are consistent with the properties of 5 M NaOH at 25 °C (24590-WTP-ES-ENG-16-021).

- 1. Density of 1.18 g/mL  $\pm$  0.1 g/mL
- 2. Viscosity of 2.8 cP  $\pm$  0.5 cP

Similarly, the components had to be non-hazardous, commercially available in large quantity, and not cost prohibitive.

### **1.2 Target Requirements for the De-inventory Simulant Solids**

The WTP defined heavy plutonium particles simulant to have suspension and settling properties similar to 100  $\mu$ m plutonium oxide to be mixed with the DNCF. Particles with density ranging from 8.8 to 11.4 g/mL with corresponding particle size ranging from 150 to 100  $\mu$ m meet this specification (24590-WTP-ES-ENG-16-021). The solids need to be non-reactive with the DNCF and non-hazardous. Additional desirable features for these solids include narrow particle size distribution (PSD), non-friable, small aspect ratio, not cost-prohibitive, and commercially available.

### **1.3 Testing Requirements**

Work at PNNL was conducted according to PNNL test plan TP-WTPSP-132, Rev. 1, *Test Plan for PNNL WTPSP-QA Program Support of High Solids Vessel Testing*<sup>1</sup> and two PNNL project plans:

<sup>&</sup>lt;sup>1</sup> Minette, MJ. 2016. Pacific Northwest National Laboratory.

- 1. PP-WTPSP-142, Rev. 0, *Testing Simulants Supporting the Single High Solids Vessel Design* (*SHSVD*),<sup>2</sup> for NiCladWC solids physical property testing (density and particle size per technical procedures defined in Appendix A.
- 2. PP-WTPSP-154, Rev. 2.0, *Testing of De-Inventory Simulant and Added Particles for the Single High Solids Vessel Design (SHSVD)*,<sup>3</sup> for DNCF parametric testing including density and viscosity measurements

The direction of the DNCF development test plan was implemented via a test instruction, TI-WTPSP-160, *Parametric Testing of De-Inventory Newtonian Carrier Fluid Simulating 5 M NaOH*.<sup>4</sup>

Where possible, all testing defined in the PNNL project plans was conducted in compliance with requirements delineated in the Bechtel National, Inc. (BNI) document *Guidelines for Performing Chemical, Physical, and Rheological Properties Measurements*, 24590-WTP-GPG-RTD-001 (hereafter called the BNI Guideline). The BNI Guideline was developed for actual waste testing and as such was somewhat limited. Pacific Northwest National Laboratory (PNNL) instituted several exceptions to the BNI Guideline, as delineated in Table 1.1. Table 1.1 also provides the rationale for the modifications, which mostly result in a more accurate measurement.

Guideline Requirement	Modified Implementation	Rationale
Physical properties Section 4.4 (Note) requires that all masses be recorded to the nearest milligram.	PNNL will measure components on balances that are appropriate to the total measured mass. In cases where small quantities are measured, mass will be recorded to the nearest milligram or tenth of milligram. In cases where the component is >100 g, mass may be measured to the nearest 10 mg (0.01 g). In cases where >1000 g mass is recorded, the mass will be measured to the nearest 100 mg (0.1 g).	The nearest milligram mass measurement makes sense for small mass samples and containers. It is not achievable where the analytical balance capacity would be exceeded and a higher capacity balance (reduced figures past decimal) is required. In all cases, masses recorded that don't meet the nearest milligram requirement will be recorded with at least three significant figures.
Density fluid testing in Section 4.4 is determined from the supernate collected from centrifuged solids. Centrifuged solution is transferred to a tared graduated cylinder; mass is measured and the volume read from the graduation marks.	PNNL testing will measure density on solution that is not contacted with solids, not centrifuged, and using a volumetric flask.	Density measured using the BNI Guideline can only result in at best a two-significant-figure density because volume can only be read to the nearest 0.1 mL in a 10 mL graduated cylinder. Use of larger volumes and volumetric flask will result in a more accurate (four-significant-figure) density measurement. Centrifuging will not be needed because there won't be entrained undissolved solids in the liquid.

**Table 1.1**. Modifications to BNI Guideline Testing<sup>(a)</sup>

<sup>&</sup>lt;sup>2</sup> Fiskum SK. 2016. Pacific Northwest National Laboratory.

<sup>&</sup>lt;sup>3</sup> Fiskum SK. 2017. Pacific Northwest National Laboratory.

<sup>&</sup>lt;sup>4</sup> Fiskum, SK. 2017. Pacific Northwest National Laboratory.

Guideline Requirement	Modified Implementation	Rationale	
Section 5.3 requires the use of National Institute of Standards and Technology (NIST)-traceable viscosity standards.	PNNL will purchase certified viscosity reference standards from Cannon Instrument Company or Poulten Selfe and Lee Ltd. The Cannon Instrument Company was delegated by NIST in 2003 for the responsibility for U.S. national standards for certified liquid viscosity reference material.	Direct NIST-traceable viscosity standards are not commercially available. The production of viscosity reference material is performed by measurement with a certified master viscometer, not by comparison to a certified reference material.	
Per Section 5.6, fitting shear stress versus shear rate data is to be fitted to three non-Newtonian models (Oswald, Bingham Plastic, Herschel-Bulkley). Further, the shear stress versus shear rate is to be measured at 25 and 40 °C. Testing is to be conducted twice on each sample and at least duplicate samples are to be tested.	PNNL will fit the Newtonian carrier fluid to the Newtonian model.	It does not make sense to fit a Newtonian fluid viscosity to models designed for non-Newtonian fluids.	
	PNNL will test at 25 °C for the parametric test samples. Once a formulation is selected, testing will be conducted at 15, 20, 25, and 30 °C.	Testing at 40 °C does not reflect the test conditions at the SHSVD platform. The temperature range of 15 to 30 °C is consistent with the temperature the SHSVD will be exposed to.	
	PNNL will conduct single-sample tests just once during parametric studies. The final selected formulation will be tested in duplicate and each duplicate sample in replicate.	It is not necessary (waste of resources) to obtain multiple data sets on formulations that we won't use.	
(a) SM Barnes, WTP, approved these exceptions via email on July 26, 2016.			

#### **1.4 Quality Requirements**

The PNNL Quality Assurance (QA) Program is based upon the requirements as defined in the United States Department of Energy (DOE) Order 414.1D, Quality Assurance and 10 CFR 830, Energy/Nuclear Safety Management, Subpart A -- Quality Assurance Requirements. PNNL complies with the requirements found in these regulations and implements them in their Waste Treatment Plant Support Program (WTPSP) Quality Assurance (QA) Program by adopting the following consensus standards:

- ASME NQA-1-2000, *Quality Assurance Requirements for Nuclear Facility Applications*, Part I, Requirements for Quality Assurance Programs for Nuclear Facilities
- ASME NQA-1-2000, Part II, Subpart 2.7, Quality Assurance Requirements for Computer Software for Nuclear Facility Applications
- ASME NQA-1-2000, Part IV, Subpart 4.2, Guidance on Graded Application of Quality Assurance (QA) Requirements for Nuclear-Related Research and Development

The WTPSP project is subject to the Price Anderson Amendments Act (PAAA).

This project recognizes that QA applies in varying degrees to a broad spectrum of research and development (R&D) in the technology life cycle. The WTPSP uses a graded approach for the application of the QA controls such that the level of analysis, extent of documentation, and degree of rigor of process control are applied commensurate with their significance, importance to safety, life cycle state of work, or programmatic mission. The technology life cycle is characterized by flexible and informal QA activities in basic research, which becomes more structured and formalized through the applied R&D stages.

The processes and work used as input to this report were conducted at the "Applied Research" level. Applied Research consists of research tasks that acquire data and documentation necessary to assure satisfactory reproducibility of results. The emphasis during this stage of a research task is on achieving adequate documentation and controls necessary to be able to reproduce results.

The analytical work for rheological and density characterizations was conducted under the WTPSP QA Program and was categorized as Applied Research in accordance with the WTPSP QA Program.

Simulant development for small- and full-scale testing was conducted at PNNL under the WTPSP QA Program and categorized as Applied Research in accordance with the WTPSP QA Program.

#### 1.5 Report Organization

This report discusses the characteristics of the DNCF and filtration testing as described in the following sections.

- Section 2.0 describes the parametric testing to determine the best DNCF formulation, selected composition, physical properties (density and viscosity), viscosity as a function of temperature, and temperature stability tests.
- Section 3.0 describes the NiCladWC material and testing.
- Section 4.0 presents the conclusions/summary.
- Section 5.0 provides references.
- Appendix A describes the analysis methodology.
- Appendix B provides the DNCF recipe and formulation in compliance with 24590-WTP-RPT-TE-01-003, Rev. 0 (Townson 2001).
- Appendix C provides the component certificates of analysis provided by the vendor for materials used in testing.
- Appendix D provides the Safety Data Sheets for each component.
- Appendix E discusses the 10 M NaOH simulant derivation.
- Appendix F provides the NiCladWC PSD raw data generated with the Malvern particle size analyzer.

### 2.0 De-inventory Simulant

The targeted physical properties for the DNCF are to achieve a density of  $1.18 \text{ g/mL} \pm 0.1 \text{ g/mL}$  and viscosity of  $2.8 \text{ cP} \pm 0.5 \text{ cP}$ , parameters consistent with the properties of 5 M NaOH at 25 °C (24590-WTP-ES-ENG-16-021, Rev. 1). Sodium thiosulfate pentahydrate and glycerin mixed with Richland City water were selected for use in the DNCF. The salt was selected as a matrix modifier to increase solution density. Glycerin was selected as a matrix modifier to increase solution viscosity. Both materials were selected because they are non-hazardous, and, based on previous testing (Gauglitz et al. 2012), the desired physical properties could be attained with an appropriately formulated mixture. Further, the salt and glycerin are commercially available in large quantities (hundreds of kilograms) at reasonable cost.

A series of test solutions were prepared with a mix of  $Na_2S_2O_3 \bullet 5H_2O$ , Richland City water, and glycerin. Samples of  $Na_2S_2O_3 \bullet 5H_2O$  and glycerin were obtained from Atkins Engineering Laboratory (AEL) under chain of custody AEL-029; these materials were specifically purchased to support SHSVD testing at the AEL test stand. The  $Na_2S_2O_3 \bullet 5H_2O$  was distributed by Sino Chemical Company Ltd. (Zhengzhou, Henan, China), Lot 20161226; see Appendix C for the available product information. This  $Na_2S_2O_3 \bullet 5H_2O$  consisted of large (~1 cm x ~0.5 cm) crystalline solids as shown in Appendix A. The glycerin was produced by Silver Fern Chemical as Glycerin, 99%, USP, Lot 12-1584 (no additional information was made available). This glycerin supply had been stored outside at the Hanford Site in Richland, Washington, in an opaque drum since March 2012. As such, it has been subjected to the outdoor temperature extremes. Some small black specks were noted on the inside bottom of the glycerin sample container (see Appendix A, bubbles at the surface, black specks at bottom of the container). An alternte source of glycerin is being used for the test solution. Richland City water was collected from the municipal water tap.



Figure 2.1. Sample of Sino Chemical Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>•5H<sub>2</sub>O, Lot 20161226 with Centimeter Scale



Figure 2.2. Silver Fern Glycerin with Black Specks

The following sections discuss the development of targeted parametric test compositions, physical properties of the parametric test solutions, and temperature effect on solution viscosities. Further testing was conducted to ensure that the selected DNCF composition would not adversely interact with the selected insoluble solids that mimic  $PuO_2$  physical properties.

#### 2.1 Parametric Testing to Define Target DNCF Composition

The viscosity and density of aqueous solutions of glycerin and sodium thiosulfate can be controlled by varying the proportions of these two additives. The approach used previously to achieve both a density and a viscosity target was to prepare a series of solutions that had a range of glycerin concentration, to vary the viscosity, and to ensure that each had the target density by adjusting the sodium thiosulfate concentration (Gauglitz et al. 2012). To determine the glycerin and sodium thiosulfate concentrations to achieve a specific viscosity and density target, the viscosity of the series of solutions was measured and then a correlation was used to interpolate/achieve a specific density/viscosity target. The target density for the DNCF was selected to match a 5 M NaOH solution at 25 °C, and was determined by interpolation to be 1.182 g/mL based on data reported by Sipos et al. (2000); this is well within the 1.18  $\pm$  0.1 g/mL required in the basis document (24590-WTP-ES-ENG-16-021) and PNNL Project Plan, PP-WTPSP-154, Rev. 2.0.

A simple model for density of glycerin and sodium thiosulfate mixtures can be developed by assuming an ideal mixture (volumes are additive) of pure glycerin with a salt solution whose composition is given by the water and salt content of the mixture. The bulk density of the combination of glycerin and sodium thiosulfate solution is given by the following:

$$\rho = \frac{1}{f_g/\rho_g + (1-f_g)/\rho_{ss}}$$
(2.1)

where

 $\rho$  = bulk solution density of sodium thiosulfate and glycerin solution, g/mL

 $f_g$  = mass fraction of glycerin in the solution mixture

 $1-f_g$  = mass fraction of the solution mixture that is water and dissolved sodium thiosulfate

 $\rho_g$  = density of glycerin, 1.2611 g/mL

 $\rho_{ss}$  = density of the sodium thiosulfate salt solution whose composition is given by the water and salt content of the mixture

The density for the solution of water and sodium thiosulfate can be determined from literature data (CRC 2011), and the following linear equation fits the data for sodium thiosulfate mass fractions between 0.18 and 0.3:

$$\rho_{\rm ss} = 0.9929 + 0.9759 \, f_{\rm salt \, in \, ss} \tag{2.2}$$

where  $f_{salt in ss}$  is the mass fraction of sodium thiosulfate in the salt solution whose composition is given by the water and salt content of the mixture.

The results reported in Gauglitz et al. (2012) were used as guidance for selecting a range of glycerin concentrations for initial solutions and equations 2.1 and 2.2 were used to adjust the mass fraction of sodium thiosulfate to meet the target density of 1.182 g/mL for each solution.

The salt/glycerin/Richland City water test solutions were prepared in a small scale (100 g) from April to June 2017. To prepare the DNCF test solutions, large salt crystals were pulverized such that appropriate quantities could be collected for testing. The crushed salt was then dissolved in Richland City water; the dissolution is an endothermic process. The solution was mixed until dissolution was complete, then allowed to reach ambient temperature. Then glycerin was added to the salt solution to achieve the target glycerin concentration. The composition of the salt and glycerin in solution was calculated based on the measured input component masses.

The density and viscosity of each test solution were evaluated. Density was measured at 21 to 24 °C; the density at 25 °C will not be perceptibly different. The component concentrations (wt%), density, and viscosity at 25 °C results are shown in Table 2.1, Figure 2.3, and Figure 2.4.

$\begin{array}{c} Na_2S_2O_3\text{-}5H_2O,\\ wt\%\end{array}$	Glycerin, wt%	Richland City Water, wt%	Density, g/mL	Viscosity at 25 °C, cP
28.950	8.206	62.844	1.181 <sup>(a)</sup>	2.14
26.709	13.307	60.042	1.180 <sup>(a)</sup>	2.40
25.510	16.004	58.489	1.181 <sup>(a)</sup>	2.60
23.331	21.024	55.699	1.184 <sup>(a)</sup>	3.05
21.346	25.533	53.139	1.181 <sup>(a)</sup>	3.40
19.176	30.521	50.371	1.182 <sup>(a)</sup>	4.00
22.105	25.500	52.404	1.188 <sup>(b)</sup>	3.57
20.596	25.507	53.917	1.176 <sup>(c)</sup>	3.32

Table 2.1. Parametric and Confirmation Test Solution Compositions, Densities, and Viscosities

Densities were measured at 21.0 to 23.6 °C.

(a) Target density is 1.182 g/mL.

(b) Target density is 1.187g/mL.

(c) Target density is 1.177 g/mL.



Figure 2.3. Parametric Test Solution Densities at 25 °C as a Function of wt% Glycerin



Figure 2.4. Parametric Test Solution Viscosities at 25 °C as a Function of wt% Glycerin

All parametric test solutions easily met the density target of  $1.18 \pm 0.1$  g/mL. There was a strong dependency of viscosity with temperature. Therefore, it was not expected that all parametric test solutions would meet the viscosity target at 25 °C.

#### 2.2 Temperature Dependence Testing

The viscosities of the parametric test solutions were also measured at 15, 20, and 30 °C. Figure 2.5 shows the viscosity temperature dependence for the different formulations (inclusive of the 25 °C results). The viscosity is within the 2.3 to 3.3 cP range at a limited temperature range (e.g., 16.4 to 30.1 °C for the 16.0 wt% glycerin solution).



Figure 2.5. Parametric Test Solution Viscosities as a Function of Temperature

Each curve in Figure 2.5 was fit to a polynomial equation to determine the temperature that corresponded to the target viscosity (2.8 cP) and the upper and lower bounds ( $\pm 0.5$  cP) (see Table 2.2). Figure 2.6 shows the process temperature as a function of wt% glycerin to reach the target 2.8 cP viscosity, along with the upper and lower acceptable ranges. For a given wt% glycerin, the operating test temperature window spans about 14 °C while still remaining in the target viscosity range. The 16.0 wt% glycerin / 25.5 wt% Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>•5H<sub>2</sub>O solution was shown to meet the allowed viscosity range of 2.3 cP to 3.3 cP in the temperature range of 16.4 to 30.1 °C. A 15.0 wt% glycerin / 25.96 wt% Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>•5H<sub>2</sub>O solution is interpolated to meet the target temperature range at 15.4 °C (low) to 29.1 °C (high) and still meet the 2.3 to 3.3 cP allowed viscosity range.

		Temperature, °C		
wt% Glycerin	Polynomial Curve Fit	At 2.8 cP	At 2.3 cP	At 3.3 cP
8.2	$T = 5.888 \times cP^{2}-45.571 \times cP+95.366$	13.9	21.7	9.1
13.3	$T = 4.5589 \times cP^2  38.665 \times cP \text{+-} 91.61$	19.1	26.8	13.7
16.0	$T = 5.3871 \times cP^2 - 44.391 \times cP + 104.41$	22.4	30.8	16.6
21.0	$T = 2.8733 \times cP^2  29.687 \times cP \text{+-} 89.006$	28.4	35.9	22.3
25.5	$T = 2.2674 {\times} cP^2 {-} 26.175 {\times} cP {+} 88.065$	32.6	39.9	26.4
30.5	$T = 1.5338 \times cP^2 \text{-} 21.017 \times cP \text{+} 84.818$	38.0	44.6	32.2

Table 2.2. Curve Fits to Reach Target and Upper and Lower Viscosity Targets at Temperature



**Figure 2.6**. Process Temperature as a Function of Glycerin Concentration at 2.8 ± 0.5 cP (where T is Temperature in °C and %G is wt% Glycerin)

#### 2.3 Stability Testing

Aliquots of all parametric test samples were chilled to 10 °C for 2 days and another aliquot set was stored at room temperature (nominally 20 °C) for 7 days. After the storage period at temperature, there were no visible signs of precipitation or other changes in the solution. This was taken to indicate that all compositions are stable at the tested parameters with respect to precipitation.

#### 2.4 Interaction with Solids

The DNCF is to be contacted with high-density solids that mimic the properties/behavior of  $PuO_2$ . Initial work was conducted with bismuth oxide; however, this material reacted with sodium thiosulfate. The bismuth oxide was replaced with NiCladWC.

#### 2.4.1 NiCladWC Reactivity

Tungsten carbide is generally very inert. NiCladWC contact with the DNCF did not show any visual signs of reactivity (Figure 2.7).



**Figure 2.7**. NiCladWC in DNCF (left), Water Blank (center), NiCladWC in Water after 1 Week Fluid Contact (right)

#### 2.4.2 Bismuth Oxide Reactivity

This discussion on bismuth oxide is included for future reference, as other simulant preparations may consider its use. Bismuth oxide was initially selected for use as the heavy plutonium particle simulant due to its general availability and high density (PNNL Project Plan PP-TWPSP-154, Rev. 0, superseded April 2017). However, the bismuth oxide darkened appreciably after contacting DNCF. The reaction was visually obvious after 3 hours contact time. However, contact with glycerin and Richland City water was visually non-reactive (see Figure 2.8). Other salt solutions, NaBr and Na<sub>2</sub>CO<sub>3</sub>, were contacted with bismuth oxide to test any reactivity issues. A 20 wt% Na<sub>2</sub>CO<sub>3</sub> solution resulted in a clumping of the bismuth oxide and creation of a finely dispersed white suspension after ~18 hours contact time. Two reactions were observed: (1) a 20 wt% NaBr solution resulted in very slow darkening of the bismuth oxide (over months) and (2) a 40 wt% solution resulted in creation of a white precipitate (over a couple of days). All tested salt solutions demonstrated that they chemically reacted with the bismuth oxide and thus the bismuth oxide was not an optimal candidate solid for testing.



43.2 wt% Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>-5H<sub>2</sub>O in Richland City water

63.0 wt% glycerin in Richland City water

Richland City water

Figure 2.8. Bismuth Oxide After 21.5-hour Fluid Contact

#### 3.0 NiCladWC Testing

The targeted physical properties for the NiCladWC solid are to achieve a density of  $11.2 \text{ g/mL} \pm 0.1 \text{ g/mL}$ and a particle size of 90 to 105 µm (CCN 285593<sup>1</sup>), although other combinations are acceptable per the simulant basis document (24590-WTP-ES-ENG-16-021). A NiCladWC material custom-made by XL Sci-Tech (Richland, Washington) was selected as the de-inventory solid particle after extensively exploring other options. At the time of this report, 200 g of the NiCladWC material was provided for evaluation and inclusion in this report (lot # XLS7892). This material was sieve cut, 75 to 80 µm and the sieve fractions were evaluated for particle size distribution (PSD) and density. The final mass fraction recovered in the range of interest, 75 to 80 µm, for the NiCladWC powder was approximately 17 wt%. The PSD of the sieve cut, 75 to 80 µm, is given in Figure 3.1; both the general purpose and the single narrow spherical model distributions are plotted. All of the PSD have been included in Appendix F



Figure 3.1. Particle Size Distribution of NiCladWC Powder, Lot# XLS7892, Sieve Cut to 75 to 80 µm

The density of the NiCladWC powder was measured on the bulk material prior to sieving and on the sieve fractions obtained. The results are given in Table 3.1 along with select particle size percentiles. The sieve cuts for the NiCladWC had slightly different density values associated with them. An image of the 75 to 80 µm sieve cut NiCladWc powder is shown in Figure 3.2 below.

<sup>&</sup>lt;sup>1</sup> CCN 285593. Meeting Minutes: "IT Mixing Workshop 2 Mar 2017." March 2, 2016. Bechtel National, Inc., Richland, WA.

Sieve Cut, µm	Density, g/cm <sup>3</sup>	d(10) GP/SN, <sup>(a)</sup> μm	d(50) GP/SN, <sup>(a)</sup> μm	d(90) GP/SN, <sup>(a)</sup> μm
Bulk Material	10.680	NM <sup>(b)</sup>	NM <sup>(b)</sup>	NM <sup>(b)</sup>
> 80	9.860	84.6/87.2	116/116	161/158
75 - 80	10.808	68.1/71.7	93.6/93.5	128/122
< 75	12.044	55.4/57.2	76.3/77.0	105/102

Table 3.1. Density of NiCladWC Sieve Cuts and Select Size Percentiles for Lot# XLS7892

Densities were measured using a gas pycnometer at laboratory temperature, ~ 22 °C.

(a) Model used for data analysis: GP = General Purpose; SN = Single Narrow Spherical.

(b) NM=Not Measured



**Figure 3.2**. Optical Microscopy Images of NiCladWC Powder, Lot# XLS7892, Sieve Cut to 75 to 80 μm (Image is FIO)

The simulant basis document, (24590-WTP-ES-ENG-16-021) provides guidelines for the selection of an undissolved solid simulant (de-inventory solid particle). If the selected particle density falls between the range  $8,800 - 11,460 \text{ kg/m}^3$  (with preference to densities closer to the upper range) the median particle size is to be interpolated from the values plotted in Figure 3.3. The NiCladWC particle evaluated here falls within this density range,  $10,808 \text{ kg/m}^3$ , and hence the target median particle size for this particle density interpolated from Figure 3.3 is approximately  $110 \mu m$ . The median particle size of the selected NiCladWC particle is approximately  $16 \mu m$  smaller than the targeted median value interpolated from the basis of design document specifically states that there is no criterion for an allowable particle size range other than it should be minimized to the extent practical. The selected material crosses the targeted median size as determined by linear interpolation between points.



Figure 3.3. d(10), d(50) (Median) and the d(90) Particle Size distribution of NiCladWC Powder, Lot# XLS7892, Sieve Cut to 75 to 80 μm at a Density of 10.808 g/cm<sup>3</sup> and the Target Median Particle Size.

As discussed above, the NiCladWC particle was also exposed to the DNCF to ensure chemical compatibility.

A limited evaluation of the filter sock, AJR Filtration, part number NMO 75 P2OSS-A, was carried out to evaluate the performance of the sock with the fine NiCladWC material, <75  $\mu$ m sieve cut. The <75  $\mu$ m cut was used for this evaluation because the 75 to 80  $\mu$ m sieve cut was consumed during the particle size analysis and was no longer available for testing. This material was selected for the evaluation because these particles are smaller than those which will be tested; if any of the material was retained then it is likely that the material of interest would also be retained. The mass of <75  $\mu$ m NiCladWC available for the filter sock test was 0.9227 g; 60 % was retained on the sock (see Figure 3.4).



Figure 3.4. NiCladWC <75  $\mu m$  Recovered on the Filter Sock, NMO 75 P2OSS-A

### 4.0 Conclusions

A suite of DNCF simulant compositions were developed for use in testing the SHSVD vessel for the WTP. The components of the DNCF are sodium thiosulfate pentahydrate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>•5H<sub>2</sub>O), glycerin, and Richland City tap water. All components are non-hazardous and commercially available at reasonable cost. A custom made narrow PSD NiCladWC partical was also evaluated for use in the SHSVD testing.

The DNCF compositions were developed for a working test stand temperature range of 15 to 30 °C; the formulation may be selected based on the test stand operating temperature All test solutions met the density requirement of 1.18 g/mL. The DNCF formulations that achieve 2.8 cP viscosity at specific temperatures are given in Table 4.1. All compositions meet the requirements developed in 24590-WTP-ES-ENG-16-021, 2.8 cP  $\pm$  0.5 cP, within the temperature range specified in Table 4.1. The formulation tested that most closely fits the full temperature test range is 16.0 wt% glycerin and the 25.51 wt% Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>•5H<sub>2</sub>O. All simulant compositions are stable with respect to precipitation at 10 °C (at least 2 days) and ~20 °C (at least 7 days).

Component	Weight Percent					
$Na_2S_2O_3\bullet 5H_2O$	28.49	26.47	25.51	24.45	22.44	
Richland City water	62.27	59.69	58.49	57.11	54.54	
Glycerin	9.24	13.84	16.00	18.43	23.03	
Temp. for 2.8 cP, °C	15	20	22.35	25	30	
Applicable temp. range, °C (a) $9.4 - 23.1$ $14.2 - 27.9$ $16.4 - 30.1$ $18.9 - 32.6$ $23.7 - 37.3$						
(a) Temperature range where the 2.8 $\pm$ 0.5 cP viscosity tolerance is met.						

Table 4.1. DNCF Simulant Composition at Selected Temperatures, 2.8 cP and 1.18 g/mL

The NiCladWC particles are qualified for the de-inventory testing as:

- The size range of the particles have been constrained to the extent possible through the use of 75 to 80µm sieves
- The particle density falls within the 8,800 11,460 kg/m3 range
- The d(10), d(50) and d(90) particle size distribution values of 71, 93 and 122 μm (at a density of 10,600 to 10,900 kg/m3) includes the ideal 100μm simulant diameter of 110μm as shown in Figure 3.1.
- This simulant represents the Hanford waste well as the  $100\mu m$  plutonium oxide particle is expected to be the maximum (i.e., the d(100) value) plutonium oxide particle in the waste tanks.

### 5.0 References

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Appendix A

Analysis Methodology
## Appendix A

## **Analysis Method**

This appendix describes the analytical methodology applied for sample analysis.

### A.1 Fluid Density

Fluid densities were determined by measuring the net fluid masses in Class A volumetric flasks. Solution temperatures were recorded when the measurements were taken. This methodology provides a more accurate value with more significant figures than the method provided by Smith and Prindiville (2002), where volume is read from a graduated cylinder.

## A.2 Fluid Viscosity

Characterizations of shear rate versus shear stress (i.e., flow curve) measurements were conducted using the Anton Paar MCR 301 benchtop rheometer. The rheometer uses a concentric cylinder double gap DG26.7 sensor measuring geometry. Each flow-curve measurement consisted of an upward run (0 to 1000 sec-1 of shear rate) and a downward run (1000 to 0 sec-1). Sample temperature control is rheometer dependent. For the MCR 301 system, sample temperature control was accomplished with a combination of a thermal chamber built into the rheometer and a temperature-controlled bath/circulator.

Rheometer performance checks were conducted before initial use and at least once every 30 days of use thereafter with certified Newtonian viscosity standards traceable to the manufacturer's lot number. The rheometer will have demonstrated an accuracy of  $\pm 15\%$  at apparent viscosity measurements less than 10 cP or  $\pm 10\%$  at apparent viscosity measurements greater than 10 cP, as specified in Pacific Northwest National Laboratory (PNNL) technical procedure RPL-COLLOID-02, *Measurement of Physical and Rheological Properties of Solutions, Slurries and Sludges*, Rev. 2.

Rheometers used for this work are generally equipped with thermocouples, thermistors, and/or other devices for measuring the temperature of the sample. These devices are internal to the equipment and cannot be calibrated. A calibrated thermocouple is used to measure the temperature of the circulating water bath and to verify the internal non-calibrated thermocouple. Rheometer performance is evaluated at a set temperature as measured on the calibrated thermocouple, and compared to the certificate of analysis of the viscosity standard. Given that the viscosity standards used to conduct performance checks of the rheometer are highly sensitive to temperature, the performance of the standard at a set temperature was sufficient to confirm proper function of the internal temperature-measuring devices.

## A.3 Solids Sieving

ASTM E-11 sieves were used for sieving the component solids to the desired mesh size. The initial sieve cuts were conducted using a rotap for 10 minutes, 75 to 90 and 90 to 106  $\mu$ m; subsequent sieve cuts were made by hand, notably the 75 to 80  $\mu$ m sieve cut.

### A.4 Particle Size Distribution

Particle size distributions (PSDs) were measured with a Mastersizer 2000 (Malvern Instruments, Inc., Southborough, MA 01772 USA) with a Hydro  $\mu$ P wet dispersion accessory. Malvern lists the Mastersizer particle size measurement range as nominally 0.02 to 2000  $\mu$ m. The actual PSD measurement range depends on the accessory used as well as the properties of the solids being analyzed; when coupled with the Hydro  $\mu$ P dispersion unit, the measurement range is 0.01 to 500  $\mu$ m. The Malvern 2000 uses laser diffraction technology to define PSD. The primary measurement functions of the Malvern analyzer are controlled with the Mastersizer 2000 software, Version 5.6 (Malvern Instruments, Ltd. Copyright<sup>©</sup> 1998-2009).

The Hydro  $\mu$ P wet-dispersion accessory consists of a 20 mL sample flow cell with a continuous variable pump/stirrer and ultrasound. The flow/stirrer rate and sonication can be controlled and altered during measurement. PSD measurements can be made before, during, and after sonication, allowing the influence of sonication on the sample PSD to be determined. Typically, a minimum of three measurements are taken at each condition; the instrument's software generates an average of these measurements.

The sample dispersion is incremental to the dispersion unit (while the pump and stirrer are active) until an obscuration in the range of 5% to 20% is reached. (Note that when fine materials in the <5 micron range are analyzed, the optimal obscuration range is 10%.)

For each condition tested, multiple measurements of PSD were taken, typically a minimum of three. The analyzer software generates an average of these measurements.

Testing was conducted in accordance with PNNL technical procedure OP-WTPSP-003, *Size Analysis Using Malvern MS2000*, Rev. 2. The PSD measurements of the components were conducted in deionized water with a pump speed of 2500 rpm and a stirrer speed of 1000 rpm. Measurements were collected prior to sonication, during sonication (100% power), and post sonication. The results reported herein are the pre-sonication measurements.

## A.5 Optical Microscopy

Solid component morphologies were observed using an optical microscope, Nikon AZ100. The image includes a reference length scale that was calibrated within the microscope software with the aid of a calibrated micro ruler.

### A.6 Solids Density

The nickel cladded tungsten carbide sieve cuts were measured using a Micromeritics AccuPyc II 1340 gas pycnometer with a 10 cc. The density was determined according to PNNL technical procedure OP-WTPSP-008, *Using a Gas Pycnometer*, Rev. 1. System performance was verified using a calibrated sphere. Sample mass ranged from 30 to 50 g with corresponding volumes of 4 to 6 mL. The propagated measurement uncertainty was estimated to be approximately 0.2%.

## A.7 Reference

Smith G and K Prindiville. 2002. *Guidelines for Performing Chemical, Physical and Rheological Properties Measurements*. 24590-WTP-GPG-RTD-001, Bechtel National, Inc., Richland, WA.

# Appendix B

**De-inventory Newtonian Carrier Fluid Simulant** 

## Appendix B

## **De-inventory Newtonian Carrier Fluid Simulant**

This appendix describes the preparation procedure of the de-inventory Newtonian carrier fluid (DNCF) simulant.<sup>1</sup>

### **B.1 Simulant Designation**

The de-inventory simulant is a physical simulant to be used in testing Standard High Solids Vessel Design (SHSVD) with respect to the processing/removal of heavy plutonium particles from the vessel. The de-inventory testing is planned to be performed by conducting three pump-downs from ~11,000 gallons with ~3 kg of NiCladWC and carrier fluid with density and viscosity of 1.18 g/mL and 2.8 cP.

This procedure defines the preparation steps required to produce the DNCF. Specific concerns with this simulant are the carrier fluid density and viscosity.

### B.2 Simulant Waste Stream Composition / Unit Operation Usage / Requirements

#### **B.2.1** Characterization Data Determination

As a physical simulant, the carrier fluid physical properties of the DNCF need to be confirmed. Therefore, the aqueous phase density and viscosity must be measured and meet 1.18 g/mL  $\pm$  0.1 g/mL and 2.8 cP  $\pm$  0.5 cP at the designated processing temperature (15 to 30 °C range).

Nickel clad tungsten carbide will be added to the DNCF at the test stand to test removal processing. Consideration of NiCladWC is not further addressed in this appendix; it is outside of the scope of the liquid component of the simulant.

#### B.2.2 Flowsheet Operations for Which Simulant Was Developed

This simulant is designed to test the efficacy in removing accumulated high-density solids from the SHSVD vessels. This simulant was designed to provide the highest possible viscosity aqueous phase that would be present if the de-inventory process was to be performed. It should be noted that this de-inventory process is a unique process step that is not part of the normal unit operations of the Hanford Tank Waste Treatment and Immobilization Plant (WTP).

<sup>&</sup>lt;sup>1</sup> This appendix was prepared to align with requirements defined in 24590-WTP-GPG-RTD-004 and implemented according to PNNL Project Plan PP-WTPSP-154, Rev. 2.

#### **B.2.3** Simulant Design Requirements and Acceptance Criteria

As indicated, PNNL was not directed to address the solid phase of this simulant. Therefore, the only requirements associated with this simulant are whether the physical properties of the simulant meet those specified, in particular the density and viscosity of the simulant.

### **B.3 Simulant Preparation Procedure**

#### B.3.1 Chemicals to Use

The  $Na_2S_2O_3 \cdot 5H_2O$  is recommended to be American Chemical Society (ACS) reagent grade to ensure that the required physical properties are obtained. ACS reagent grade  $Na_2S_2O_3 \cdot 5H_2O$  is commercially available at reasonable cost. Alternatively, anhydrous  $Na_2S_2O_3$  could be used with appropriate salt and solvent mass adjustments to account for the waters of hydration in  $Na_2S_2O_3 \cdot 5H_2O$ . The parametric testing described in this report to meet the 2.8 cP viscosity and 1.18 g/mL density targets was prepared from a product purchased from Sino Chem ("Photo Grade, 99%" as subsampled and provided by Atkins Engineering Laboratory [AEL]).

Glycerin (1, 2,3-Propantriol, CH<sub>2</sub>OHCHOHCH<sub>2</sub>OH) can be obtained in bulk quantity at a reasonable cost from a variety of suppliers. Glycerin of 99% or better purity is recommended. The parametric testing described in this report to meet the 2.8 cP viscosity and 1.18 g/mL density targets was prepared from Silver Fern glycerin that had been stored outside at the Hanford Site for 5 years (since March of 2012; no other information was provided by AEL or Bechtel National, Inc.).

Municipal water is used for salt dissolution. All testing has been conducted with Richland City water collected from the tap in June of 2017.

#### B.3.2 Chemical Addition Order

The  $Na_2S_2O_3 \cdot 5H_2O$  salt must be dissolved in water before addition of glycerin. The addition order of salt to water is recommended with mixing to prevent the salt from clumping. Once the salt is completely dissolved, the glycerin can be added.

The high salt content and endothermic reaction retards the  $Na_2S_2O_3 \cdot 5H_2O$  dissolution rate. For smallscale tests (100 g), approximately an hour with gentle mixing and warming was needed to fully dissolve the salt. Large-scale production will likely require extended thermal equilibrium time for salt dissolution. Once the salt is dissolved, the glycerin is simply added to the solution and mixed.

The formulations for a 10.0 L (11.8 kg) batch of de-inventory simulant carrier fluid is shown in Table B.1. Note that 16% glycerin and 25.51%  $Na_2S_2O_3\bullet 5H_2O$  will accommodate most of the temperature range of interest while still meeting the viscosity and density targets.

	Test Temperature				
	15 °C	20 °C	22.4 °C	25 °C	30 °C
Component	Mass Added, g				
$Na_2S_2O_3\bullet 5H_2O$	3,362	3,124	3,010	2,885	2,647
Richland City water	7,348	7,044	6,901	6,740	6,435
Glycerin, 99% or better	1,090	1,633	1,889	2,175	2,717

Table B.1. Component Mass Additions Needed for a 10 L DNCF Simulant

Intermediate test temperature compositions may be dialed in based on the function shown in Figure B.1. The  $Na_2S_2O_3 \bullet 5H_2O$  concentration can be determined according to the function in Equation B.1, where %G is wt% glycerin and %S is wt%  $Na_2S_2O_3 \bullet 5H_2O$  (applicable in the range of 8 to 30 wt% glycerin).



Figure B.1. 2.8 cP Viscosity Temperature Dependence as a Function of wt% Glycerin

$$-0.4388 \times \%G + 32.538 = \%S \tag{B.1}$$

#### **B.3.3** Precautions

Appropriate safety apparel should be worn when working with the salt and glycerin. This includes a lab coat or lab apron with long-sleeve shirt, safety goggles, and gloves. The Safety Data Sheets recommend using a suitable respirator around high concentrations of  $Na_2S_2O_3 \cdot 5H_2O$  and glycerin. However,  $Na_2S_2O_3 \cdot 5H_2O$  is not particularly dusty and glycerin has a low volatility. Chemical handling should be conducted in well-ventilated work spaces. The salt may create dust that should not contact eyes or the respiratory system. The Safety Data Sheets should be consulted for response to contact with material.

Dissolution of the sodium thiosulfate salt is an endothermic process; that is, the solution will become very cold. Salt dissolution (129 g of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>•5H<sub>2</sub>O into 89.5 g Richland City water for preparing 300 g salt

solution) reduced the solution temperature to 3.3 °C. Solution warming to room temperature or above is needed for complete dissolution of the salt.

#### **B.3.4 Other Considerations**

The  $Na_2S_2O_3 \cdot 5H_2O$  is considered hygroscopic; it must be protected from high humidity and should be well sealed to mitigate ambient interaction with water vapor.

Glycerin is hygroscopic and will result in partial decomposition at 54 °C.<sup>1</sup> Therefore, it should be stored in a cool, dry place and protected from heat.

Once the de-inventory carrier fluid is prepared, it is expected to be stable with respect to physical property changes. The salt solution is not expected to support microbial life; this assertion is based on observations from similar preparations from previous tests.

### **B.4 Key Characteristics and Limitations of Simulant**

#### **B.4.1 Key Characteristics**

The key characteristics of the DNCF are density (1.18 g/mL) and viscosity (2.8 cP at the test temperature).

#### **B.4.2** Limitations

This simulant is purely physical—it must not be construed as a chemical simulant.

The NiCladWC was not selected to represent any particular tank waste; it was selected to mimic heavy plutonium particle settling characteristics (beyond the 10 micron WTP design basis). The basis for this simulant is predicated almost entirely on the design basis for the vessels, and as such the simulant is not intended to represent any expected feed to the WTP.

The DNCF viscosity is temperature dependent. A given formulation will meet the specification within a defined temperature range.

### **B.5** Verification and Validation of the Simulant

The only recommended verification activity is to measure the component input masses and measure the mixed fluid density and viscosity to ensure they are within the specification of  $1.18 \pm 0.1$  g/mL and  $2.8 \pm 0.5$  cP at the test temperature.

<sup>1</sup> Product Safety Assessment: Glycerin, September 2014. Dow Chemical Company <u>http://msdssearch.dow.com/PublishedLiteratureDOWCOM/dh\_091a/0901b8038091a41a.pdf?filepath=productsafety</u> /pdfs/noreg/233-00490.pdf&fromPage=GetDoc

### **B.6 Simulant Properties Comparison to Actual Waste Properties**

No comparisons are possible. The DNCF does not emulate an actual waste, but is intended to emulate a possible WTP vessel flush solution.

### **B.7 Simulant Development Organization**

The DNCF formulation was developed at Battelle, Pacific Northwest National Laboratory under the River Protection Project – Waste Treatment Plant R&T project. The following PNNL staff contributed to the formulation of the DNCF: Reid Peterson, Phil Gauglitz, Sandra Fiskum, Diana Linn, and Carolyn Burns. Staff may be reached at the following address:

PO Box 999 Battelle, PNNL Richland WA 99352

Appendix C

Material Certificates of Analysis

## Appendix C

## **Material Certificates of Analysis**

This appendix provides the available certificates of analysis or conformance from each of the products used for the de-inventory Newtonian carrier fluid simulant and the simulant described in Appendix E. Note that the Silver Fern glycerin certificate of analysis was not made available. The certificates of analysis include the following:

- Sodium thiosulfate pentahydrate, Sino Chemical, Batch 20161226
- Sodium thiosulfate pentahydrate, Noah Technologies, Lot 0275037/1.1
- Glycerin, Noah Technologies, Lot 28717/0.0

#### **CERTIFICATE OF ANALYSIS**

NAME	SODIUM THIOSULFATE PENTAHYDRATE (PHOTO GRADE)			
DATE OF MANUFACTURE	DEC. 26, 2016 BATCH NO.			20161226
DATE OF ANALYSIS	DEC. 26, 2016 DATE OF EXPIRY		DEC.25,2018	
	ANALYSIS R	ESULT	Г	
ITEM	STANDARD		TEST RESULT	
PURITY%	99.9MIN	99.91		
WATER INSOLUBLE%	0.01MAX		0.006	
NA2S%	0.001MAX		0.008	
FE%	0.002MAX		0.001	
PH	6.5-9.5		8	
CONCLUSION: COMPLY	HG/T2328-2015			
NSPECTOR: WANG ZH	AOYI	REINSPE	CTOR: XI	J YA

ISSUED BY

ZHENGZHOU SINO CHEMICAL CO., LTD.





### CERTIFICATE OF ANALYSIS

Code 90425

SODIUM THIOSULFATE, PENTAHYDRATE, ACS Reagent, crystal, Na2S2O3.5H2O

Lot 0275037/1.1		
TEST	REQUIREMENTS	FOUND
Assay	99.5 - 101.0%	100.0%
Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> .5H <sub>2</sub> O		
pH of a 5% solution at a 25 C	6.0 - 8.4	7.1
	MAXIMUM ALLOWABLE	
Insoluble matter	0.005%	< 0.005%
Nitrogen compounds	0.002%	< 0.002%
Sulfate and Sulfite (as SO $_4$ )	0.1%	0.1%
Sulfide (S)	Passes Test	Passes Test
	(limit about 1 ppm)	

According to ACS, Reagent Chemicals, Tenth Edition, 2006

All values are maximum and may represent detection limits.

NOAH CHEMICAL DIV. NOAH TECHNOLOGIES CORPORATION 1 Noah Park San Antonio, TX 78249-3419 Telephone 210-691-2000 Fax 210-691-2600

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### **CERTIFICATE OF ANALYSIS**

Code 90785	i		
GLYCEROL, 2,3-Propantri	99.5% pure, ACS Reagent, liquid, iol), CH <sub>2</sub> OHCHOHCH <sub>2</sub> OH	, (Glycerin, 1,	
Lot 28717/0	.0		
TEST		REQUIREMENTS	FOUND
Assay, C3H5(OH	)3	> 99.5%	99.8%
by volume			
		MAXIMUM ALLOWA	BLE
Color (APHA)		10	5
Residue after ign	ition	0.005%	0.001%
Neutrality		Passes test	Passes test
Chlorinated comp	pounds (as Cl)	0.003%	0.002%
Sulfate (SO <sub>4</sub> )		0.001%	< 0.001%
Acrolein and glue	cose	Passes test	Passes test
Fatty acid esters		0.05%	< 0.05%
(as butyric aci	d)		
Substances darke	ened by	Passes test	Passes test
sulfuric acid			
Heavy metals (as	Pb)	2 ppm	<1 ppm
Water (H <sub>2</sub> O)		0.5%	< 0.5%

Conforms to ACS, Reagent Chemicals Tenth Edition, 2006

All values are maximum and may represent detection limits.

NOAH CHEMICAL DIV. NOAH TECHNOLOGIES CORPORATION 1 Noah Park San Antonio, TX 78249-3419 Telephone 210-691-2000 Fax 210-691-2600

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Appendix D

Safety Data Sheets

## Appendix D

## **Safety Data Sheets**

This appendix provides prototypic safety data sheets (SDSs) associated with each of the products used for the de-inventory Newtonian carrier fluid simulant. SDSs prepared by Sino Chem and Silver Fern were not provided. The SDSs included herein are as follows:

- Glycerin, Noah Technologies
- Sodium thiosulfate pentahydrate, Noah Technologies



# Safety Data Sheet According to 29 CFR 1910.1200

SDS No. 850	Review date June 1, 2015
	1 Identification of substance and company
Product details	
Product name:	<u>Glycerol</u>
Product code:	90785
Manufacturer/Supplier:	Noah Technologies Corporation
	1 Noah Park
	San Antonio, Texas 78249-3419
	Phone: (210) 691-2000
	Web site: www.noahtech.com
Emergency information:	CHEMTREC
	703-527-3887
	2 Hazards identification
Hazard designation:	This product is not classified hazardous according to the Regulation (EC) No. 1272/2008 and the Council
	Directives 67/548/EEC and 99/45/EEC and the OSHA Hazard Communication Standard (29 CFR 1910.1200)
GHS label elements:	None
Signal word:	None
Hazard/precautionary statements:	Avoid consumption
Information pertaining to particular dangers	
for man and environment:	None
HMIS ratings (scale 0-4):	Health: 1
	Flammability: O
	Physical hazard: 0
	3 Composition Information on ingradiante
Chamical name:	3 Composition/information on ingretitents
Designation: (CAS#):	69,916
Energiation. (CAG#).	
Fulfitura:	HUCH2CCH(UH)CH2OH
Synonyms:	Giycenn, innydroxypropane, 1, 2, 3-propanei.noi
Idenuncation number(s):	200 200 S
EC Number.	200-269-0
	4 First aid measures
After inhalation:	Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen.
	Get medical attention
After skin contact:	Instantly wash with water and soap and rinse thoroughly
	For symptoms of exposure consult a physician
After eye contact:	Rinse opened eye for at least 15 minutes under running water. Assure adequate flushing by separating
	the eyelids with fingers. Consult a physician.
After swallowing:	If conscious, give large quantities of water and seek medical attention.
	Never give anything by mouth to an unconscious individual.
Information for doctor:	Prolonged or repeated exposure may cause nausea, headache, vomiting
Cuidable autin mulabing anguta	5 Fire-fighting measures
Suitable excinguishing agents:	Ose extinguishing media most appropriate to surrounding fire conditions
Special nazards caused by the material, its	
products of compustion or resulting gases:	in case of fire, the following can be released:
	Carbon monoxide
	Carbon dioxide
Protective equipment:	Wear self-contained breathing apparatus
	vvear fully protective suit.
	6 Accidental release measures
Person-related safety precautions:	Wear personal protective equipment. Keep unprotected persons away.
	Ensure adequate ventilation
Measures for environmental protection:	Do not allow material to be released to the environment without proper governmental permits

Page 1 of 4

Measures for cleaning/collecting:	Dispose of contaminated material as waste according to item 13		
Additional information:	See Section 7 for information on safe handling		
	See Section 8 for information on personal protective equipment		
	see section 15 ion miorination on disposal		
	7 Handling and storage		
Information for safe handling:	Keep containers tightly sealed		
	Store in cool, dry place in tightly closed containers		
Information about protection against	Ensure good ventilation/exhaustion at the workplace		
explosions and fires:	Mixtures with hydrogen peroxide are highly explosive. Ignites on contact with potassium permanganate.		
	calcium hypochlorite. Mixture with nitric acid & sulfuric acid forms the explosive glyceryl nitrate. Mixture		
	with perchloric acid & lead oxide forms explosive perchlorate esters.		
Storage requirements to be met by storerooms			
and containers:	Keep container tightly closed in a dry and well-ventilated place. Hygroscopic.		
information about storage in one common	Do not stars together with avidiming against		
Further information about storage conditions:	Keep container tightly sealed		
	Store in cool, dry conditions in well sealed containers		
	8 Exposure controls/personal protection		
Additional information about design of technical systems:	Pronetly onersting chemical time bood designed for hazardoue chamicate and having an average free		
technical systems.	velocity of at least 100 feet per minute		
Components with critical values that require			
monitoring at the workplace:	Glycerol		
	mg/m3		
	ACGIH TLV 10; (nuisance particulate)		
Additional information	USA PEL 5 (respirable fraction); 15 (total dust)		
Personal protective equipment:	No udia		
General protective and hygienic measures:	The usual precautionary measures should be adhered to in handling the chemicals		
	Keep away from foodstuffs, beverages and food		
	Instantly remove any solled and impregnated garments		
	Wash hands during breaks and at the end of the work		
Respiratory protection:	Use suitable respirator when high concentrations are present Use only NIOSHIMESA or CEN encryved duet mark type NIS or TYPE P1 (EN 143)		
Hand protection:	Impervious gloves		
Eye protection:	Safety glasses		
Skin protection:	Protective work clothing		
Additional protective equipment:	Sufficient to prevent contact		
	Emergency eyewash and safety shower		
	9 Physical and chemical properties		
General Information:			
Physical state:	Viscous liquid		
Color:	Clear to pale yellow		
Smell: Melecular Weight (Calculated):	Odorless		
Melting/Freezing point/range:	20 C		
Boiling point/range:	182 C		
Sublimation temperature/start:	Not determined		
Flash point:	160 C (closed cup)		
Autoignition temperature:	370 C		
Decomposition temperature:	290 C		
Lower:	Not determined		
Upper:	Not determined		
Vapor pressure (mm Hg):	0.0025 mm Hg @ 50 C		
Vapor density:	3.18		
Specific gravity at 20 °C	1.25 Isochible		
Solubility invelocibility with water at 0 °C	Insoluble		
	10 Stability and reactivity		
Conditions to be avoided:	No decomposition if used and stored according to specifications		
	See section 7 for information on proper handling and storage		
Materials to be avoided:	Oxidizing agents		
Dangerous reactions:	See section /		

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Hazardous decomposition products:

Carbon monoxide Carbon dioxide

A suite tourisiteur	11 Toxicological information
Acute toxicity:	
closeification:	Oral art I DE0: 12 E00 mailes
classification.	Skin-rahbit LD50: 12,000 mg/kg
Primary irritant effect	Shireaddic 2000. 2 To,000 Higing
on the skin:	Initiant for skin and mucous membranes
on the eve:	Irritant offact
Sensitization	No sensitizing effect known
Additional toxicological information:	no avising you concerned a many and choose to vicity of this substance is not fully known.
Carcinogenicity:	To the best of our knowledge the acute and chronic toxicity of this substance is not fully known.
calentogenery.	To are best of our knowledge are acate and onlene toxicity of the substance is not kny known
	12 Ecological information
General notes:	Generally not nazardous for water.
	Do not allow material to be released to the environment without proper governmental permits.
	13 Disposal considerations
Recommendation:	Consult state, local or national regulation for proper disposal
	Allow professional disposal company to handle waste
	Must be specially treated under adherence to official regulations
Unclean packagings	
Recommendation:	Disposal must be made according to official regulations
	14 Transport information
Land transport DOT	
Proper shipping name:	Chemicals Non-Hazardous
Technical name:	Glycerol
DOT Hazard Class:	STYLE STYLE
Subsidiary risk:	
UN Identification number:	
I shelle):	
Packing amun:	
Packing group:	
Maging label(a):	10
Warning label(s).	10
North American Emergency Response	
Notes:	
Air transport ICAO-TI and IATA-DGR:	
Proper shipping name:	Chemicale Non-Hazardous
Technical name:	Glucerol
DOT Hazard Class:	
Subsidiary risk:	
UN Identification number:	
Label(s):	
Packing group:	
Reportable quantity (RQ):	
Warning label(s):	10
North American Emergency Response	
Guidebook No.:	
Notes:	
UPS Ground / FedEx Ground	
Proper shipping name:	Chemicals Non-Hazardous
Technical name:	Giveerol
DOT Hazard Class:	
Subsidiary risk:	
UN Identification number:	
Label/s):	
Packing group:	
Reportable quantity (BQ):	
repercente destinct (ros).	

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Warning label(s): 10 North American Emergency Response Guidebook No.: Notes: UPS Air Proper shipping name: Chemicals Non-Hazardous Technical name: Glycerol DOT Hazard Class: Subsidiary risk: UN Identification number: Label(s): Packing group: Reportable quantity (RQ): Warning label(s): 10 North American Emergency Response Guidebook No.: Notes: 15 Regulatory information Designation according to EC guidelines: Observe normal safety regulations when handling chemicals. This product is not subject to identification regulations under EC Directives and the Ordinance on Hazardous Materials National regulations: Information about limitation of use: For use only by technically gualified individuals. Water hazard class: Generally not hazardous for water. Other regulations, limitations and Although this chemical may sometimes be used as a food or drug or cosmetic, our products are not prohibitive regulations approved or suitable for such use or for human consumption

16 Other information

The above information is accurate to the best of our knowledge. However, since data, safety standards and government regulation are subject to change and the

The above information is accurate to the best of our knowledge. However, since data, safety standards and government regulation are subject to change and the conditions of handling and use, or misuse are beyond our control. NOAH MAKES NO WARRANTY, EITHER EXPRESSED OR IMPLIED, WITH RESPECT TO THE COMPLETENESS OR CONTINUING ACCURACY OF THE INFORMATION CONTAINED HEREIN AND DISCLAIMS ALL LIABILITY FOR RELIANCE THEREON. User should satisfy himself that he has all current data relevant to his particular use.

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# Safety Data Sheet According to 29 CFR 1910.1200 (OSHA HCS)

SDS No. 2340	Review date October 5, 2015
	1 Identification of substance and company
Product details	
Product name:	Sodium thiosulfate, pentahydrate
Product code:	90425, C2843
Manufacturer/Supplier:	Noah Technologies Corporation
	1 Noah Park
	San Antonio, Texas 78249-3419
	Phone: 210-691-2000
	Fax: 210-691-2600
	Web site: www.noahtech.com
Emergency information:	CHEMTREC
	800-424-9300
	2 Havarde identification
Hazard designation:	None
Information pertaining to particular dangers	1010
for man and environment:	Not applicable
Hazards not otherwise classified	Reaction with acids produces toxic sulfur diovide ras
HMIS ratings (scale 0.4):	Health: 1
	Flammability: 0
	Physical hazard: 0
	3 Composition/Information on ingredients
Chemical name:	Sodium thiosulfate, pentahydrate
Designation: (CAS#):	10102-17-7
E C Number:	231-867-5
Formula:	Na2S2O3.5H2O
Synonyms:	Ametox, Antichlor, Hypo, sodium hyposulfite
Ingredients of known acute toxicity:	Not applicable
	4 First aid measures
After inhalation:	If breathed in, move person into fresh air. If not breathing, give artificial respiration.
After skin contact:	Instantly wash with water and soap and rinse thoroughly
	If irritation persists, consult a physician
After eye contact:	Rinse opened eye for at least 15 minutes under running water. Assure adequate flushing by separating
	the eyelids with fingers. If irritation persists, seek medical attention.
After ingestion:	Never give anything by mouth to an unconscious person. Rinse mouth with water
Information for doctor:	Show this safety data sheet to the doctor in attendance
Immediate medical attention and special	
treatment needed:	Ingestion causes cyanosis in humans. Large oral doses have a cathartic effect.
	5 Eiro fichting mozeuroe
Suitable extinguishing agents:	Use extinguishing media most suitable to surrounding fire conditions
Succial hazards caused by the material its	ose oxingetoring mean most senable to senouncing me contentions
products of combustion or resulting gases:	Ovides of sodium and sulfur (SOV), sodium diavide
Special fire fighting procedures:	Wear self-contained breathing annaratus
Special file lighting procedures.	Wear fully protective fire fighting equipment/clothing in fire situations
Unusual fire and explosion hazard:	Not applicable
	6 Accidental release measures
Person-related safety precautions:	Avoid dust formation. Avoid breathing vapours, mist or gas. Ensure adequate ventilation.
Measures for environmental protection:	Do not allow material to be released to the environment without proper governmental permits
Measures for cleaning/collecting:	Sweep up and shove. Keep in suitable, closed containers for proper disposal.
Additional information:	See Section 7 for information on safe handling
	See Section 8 for information on personal protective equipment
	See Section 13 for information on disposal

7 Handling and storage

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Information for ante handling:	Veen eenteinen tieblijveeded
information for safe handling.	Store in cool, dry place in tightly closed containers
	Ensure good ventilation/exhaustion at the workplace
Information about protection against	
explosions and fires:	Explosion hazard with sodium nitrite and metal nitrites
Storage requirements to be met by storerooms	
and containers:	Keep container tightly closed in a dry and well-ventilated place. Do not store near acids. Air and moisture
Incompatibility (avaid contrast with)	sensitive. Streng ovidinam or golds. Contact with ovidinars courses evolthermic reactions. Contact with opids releases
incompatibility (avoid contact with).	strong oxidizers or acros. Contact with oxidizers causes exothernic reactions, Contact with acros releases toxic sulfur dioxide cas. Sodium nitrate, sodium nitrite, lead, sodium, silver and mercury salts and iodides
Further information about storage conditions:	None
	8 Exposure controls/personal protection
Ventilation requirements:	Properly operating chemical fume hood designed for hazardous chemicals and having an average face
Components with critical values that require	velocity of at least 100 feet per minute
components with chucal values that require	None
Additional information:	None
Personal protective equipment:	
General protective and hygienic measures:	The usual precautionary measures should be adhered to in handling the chemicals
	Keep away from foodstuffs, beverages and food
	Instantly remove any soiled and impregnated garments
	Wash hands during breaks and at the end of the work
Personal and the sector sector	Avoid contact with the eyes and skin
Personal protective equipment:	Use suitable consister when high concentrations are recent
(Use only NIOSH or CEN approved Equipment)	Use only NIOSH/MESA or CEN approved dust mask type N95 or TYPE P1 (EN 143)
Hand protection:	Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique
· · · · · · · · · · · · · · · · · · ·	to avoid skin contact.
Eye protection:	Safety glasses
Skin protection:	Protective work clothing
Additional protective equipment:	Sufficient to prevent contact
	Emergency eyewash and safety shower
Precautionary labeling:	Wash thoroughly after handling
	Do not get in eyes, on skin or on dotning Do not breathe dust vanor, mist, cas
	Store in tightly closed containers
	Store in a cool, dry place
	0 Division and showled was added
General Information:	9 Physical and chemical properties
Physical state:	Crystals or granules
Color:	Clear to white
Odor:	Odorless
Odor threshold:	Not determined
Molecular Weight (Calculated):	248.18
pH (5% solution)	Not determined
Melting point/reezing point/range: Relling point/range:	48 C Not determined
Sublimation temperature/start:	Not determined
Decomposition temperature:	100 C (-H-O)
Flammability (solid, gas):	
Flash point:	Non-flammable
Autoignition temperature:	Not determined
Danger of explosion:	Not determined
Flammable limits:	
Lower:	Not determined
Upper:	Not determined
Vapor pressure (mm Hg):	Not determined
Vapor density:	Not determined
Specific gravity:	1.69
Bulk density:	Not determined
Solubility in/Miscibility with water:	700 g/L @ 20 C
Partition coefficient n-octanol/water.	Not determined
Viscosity:	Not determined
Other information:	No additional information
	10 Stability and reactivity

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Penetiulty	Makdataminad
Chemical stability:	Stable under recommended storage conditions
Possibility of barardous reactions:	Stable under recommended storage conditions. Explosion her and with early mitrite and matel nitrites
Conditions to be avoided:	Explosion nazara wan social mate and motor maters
	See section 7 for information on proper handling and storage
Materials to be avoided:	Strong oxidizers or acids. Contact with oxidizers causes exothermic reactions. Contact with acids releases
	toxic sulfur dioxide gas. Sodium nitrate, sodium nitrite, lead, sodium, silver and mercury salts and iodides.
Dangerous reactions:	Contact with acids releases toxic sulfur dioxide gas
Hazardous decomposition products:	Oxides of sodium and sulfur (SOx)
(thermal and other)	
	11 Toxicological information
Acute toxicity:	
LD/LC50 values that are relevant for	
classification:	intravenous-rat LD <sub>50</sub> : > 2,500 mg/kg
Primary irritant or corrosive effect:	
on the skin:	None
on the eye:	Millo Resigned or received surgering may as use allocate specifications in contain constitute individuals.
Sense and sumptoms of exposition	Froiongeo or repeated exposure may cause anel doese have a softwate site of at
orgina and symptoms of exposule;	To the best of our knowledge, the chemical, physical and toxicological organizes have not been
	thoroughly investigated.
Carcinogenicity:	No classification data on carcinogenic properties of this material is available from the EPA_LARC_NTP
	OSHA or ACGIH
Additional information:	RTECS: Not available
	12 Ecological information
Toxicity:	
Toxicity to fish:	Not determined
Toxicity to daphnia and other aquatic	
invertebrates:	Not determined
Toxicity to algae:	Not determined
Persistence and degradability:	Multiple sector of
Biodegradability:	Not determined
Bioaccumulative potential:	Not determined
Bioaccumulation:	Not determined
Other adverse effects:	Not determined
other adverse effects.	Not determined
	13 Disposal considerations
Recommendation:	Consult state, local or national regulation for proper disposal
	Allow professional disposal company to handle waste
	Must be specially treated under adherence to official regulations
Unclean packaging recommendation:	Disposal must be made according to official regulations
Land tensors at DOT	14 Transport information
Land transport DOT	
Proper shipping name:	Chemicals Non-Hazardous
Technical name:	Sodium thiosulfate, pentahydrate
DOT Hazard Class:	
Subsidiary risk:	
UN Identification number:	
Label(s):	
Packing group:	
Reportable quantity (RQ):	
Warning label(s):	10
North American Emergency Response	
Guidebook No.:	
Notes:	
Air transport ICAO-TI and IATA-DGR:	
Proper shipping name:	Chemicals Non-Hazardous
Technical name:	Sodium thiosulfate, pentahydrate
DOT Hazard Class:	
Subsidiary risk:	
UN Identification number:	
Label(s):	
Packing group:	

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Reportable quantity (RQ):	
Warning label(s):	10
North American Emergency Response	
Guidebook No.:	
UPS Ground / FedEx Ground	
Proper shipping name:	Chemicals Non-Hazardous
Technical name:	Sodium thiosulfate, pentahydrate
DOT Hazard Class:	
Subsidiary risk:	
UN Identification number:	
Label(s):	
Packing group:	
Reportable quantity (RQ):	
Warning label(s):	10
North American Emergency Response	
Guidebook No.:	
Notes:	
UPS Air	
Proper shipping name:	Chemicals Non-Hazardous
Technical name:	Sodium thiosulfate, pentahydrate
DOT Hazard Class:	
Subsidiary risk:	
UN Identification number:	
Label(s):	
Packing group:	
Reportable quantity (RQ):	
Warning label(s):	10
North American Emergency Response	
Guidebook No.:	
Notes:	
	15 Regulatory information
SARA Section 302 Extremely Hazardous	
components and corresponding TPQs:	No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.
SARA Section 311 / 312 hazards:	No SARA hazards
SARA Section 313 components:	This material does not contain any chemical components with known CAS numbers that exceed the
	threshold (De Minimis) reporting levels established by SARA Title III, Section 313
California Proposition 65 components:	This product does not contain any chemicals known to the State of California to cause cancer, birth
	defects or any other reproductive harm.
TSCA:	This product is listed in the TSCA inventory
	16 Other information

18 Other information
The above information is accurate to the best of our knowledge. However, since data, safety standards and government regulation are subject to change and the
conditions of handling and use, or misuse are beyond our control. NOAH MAKES NO WARRANTY, ETHER EXPRESSED OR IMPLIED, WITH RESPECT TO THE
COMPLETENESS OR CONTINUING ACCURACY OF THE INFORMATION CONTAINED HEREIN AND DISCLAIMS ALL LIABILITY FOR RELIANCE THEREON.
User should satisfy himself that he has all current data relevant to his particular use.

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Appendix E 10 M NaOH Simulant

## Appendix E

## **10 M NaOH Simulant**

The Hanford Tank Waste Treatment and Immobilization Plant (WTP) originally requested a simulant solution for 10 M NaOH solution with a density of 1.32 g/mL and viscosity of 10.5 cP at 25 °C. A large amount of work went into this simulant development, and the data are reported herein so that they won't be lost, as they could prove useful elsewhere. The same three components tested for the de-inventory Newtonian carrier fluid (DNCF) simulant, Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>•5H<sub>2</sub>O, Richland City water, and glycerin, were used for the 10 M NaOH simulant.

The DNCF development test plan was implemented via two test instructions:

- TI-WTPSP-155, Preparations and Physical Property Testing of De-Inventory Newtonian Carrier Fluid and Slurry for the SHSVD
- TI-WTPSP-158, Dilution Testing of De-Inventory Newtonian Carrier Fluid for the SHSVD

A series of test solutions were prepared with a mix of  $Na_2S_2O_3 \bullet 5H_2O$ , Richland City water, and glycerin. The  $Na_2S_2O_3 \bullet 5H_2O$  was obtained from Noah Technologies Corporation (San Antonio, TX) as American Chemical Society (ACS) reagent grade, crystal  $Na_2S_2O_3 \bullet 5H_2O$  salt, Catalog Number 90425, Lot 0275037/1.1, which was assayed by the vendor to be 100%  $Na_2S_2O_3 \bullet 5H_2O$ . The glycerin was obtained from Noah Technologies, Product Code 90785, ACS reagent grade, Lot 28717/0.0. It was assayed to be 99.5% pure. Both reagents were stored at room temperature for less than 1 year, and protected from the environment. Richland City water was collected from the municipal water tap.

### E.1 Parametric Testing to Define Target DNCF Composition

The salt/glycerin/Richland City water tests solutions were prepared in a small scale (100 g). To prepare the simulant test solutions, the salt was first dissolved in Richland City water and warmed and mixed until dissolution was complete. Then glycerin was added to the salt solution. Composition of the salt/glycerin solution was calculated based on the measured input component masses.

The density and viscosity of each test solution were evaluated. The viscosity data were collected for information only so that the optimal salt/glycerin concentrations targets could be determined. Density and viscosity results are shown in Table E.1 and Figure E.1. The recommended upper and lower limits in Figure E.1 are  $\pm 0.1$  g/mL for density and  $\pm 1$  cP for viscosity.

$\begin{array}{c} Na_2S_2O_3\text{-}5H_2O,\\ wt\%\end{array}$	Glycerin, wt%	Richland City Water, wt%	Density, g/mL	Viscosity, cP
Parametric Tests				
44.284	24.517	31.226	1.327	9.75
43.605	26.007	30.389	1.326	9.98
43.387	26.513	30.105	1.325	10.29
43.196 <sup>(a)</sup>	26.953 <sup>(a)</sup>	29.850 <sup>(a)</sup>	1.328	10.50
42.492	28.496	29.013	1.325	11.25
44.653	25.405	29.942	1.331	10.61
42.124	27.541	30.335	1.323	10.43
Confirmation Tests				
43.163	27.005	29.845	1.327 <sup>(b)</sup>	11.07
43.161	27.004	29.841	1.327 <sup>(b)</sup>	10.98
		• •		

Table E.1. Parametric and Confirmation Test Solution Compositions, Densities, and Viscosities at 25 °C

(a) Selected target composition for the simulant.(b) Density was measured at 21 °C for the confirmation test samples.





The 43.2 wt% hydrated salt concentration (equivalent to 27.5 wt% anhydrous salt basis) mixed with 27.0 wt% glycerin met the target physical properties at 1.33 g/mL density and 10.5 cP viscosity. The selected composition was re-prepared in two confirmation tests, which resulted in reproducible density values, but the viscosity values were offset high (see Table E.1 and Figure E.2).

All parametric test samples were chilled to 10 °C for 2 days and stored at room temperature (nominally 20 °C) for 7 days. There were no visible signs of precipitation or other changes in the solution, indicating good temperature stability.

#### E.2 Temperature Dependence Testing

The viscosity as a function of temperature was tested between 15 and 30  $^{\circ}$ C (see Figure E.2). The viscosity is within the 9.5 to 11.5 cP range at a very narrow temperature range of 24 to 29  $^{\circ}$ C.



Figure E.2. Simulant Viscosity as a Function of Temperature

### E.3 Dilution Testing

It was apparent that one solution would not suffice to be effective at the conceived test stand temperatures. Therefore, another approach was selected—dilute a stock high-concentration glycerin-salt solution with additional salt solution to lower the viscosity needed for a given test temperature.

Table E.2 shows the compositions of the two stock solutions. The glycerin/salt solution composition was formulated to provide an upper bound in viscosity at high temperature (30 °C). The salt-only solution composition was formulated to match the target density of 1.32 g/mL such that dilution of the glycerin/salt solution with the salt-only solution would not alter the diluted solution density, only the solution viscosity would be altered. Table E.3 provides the percent by weight dilution, diluted test sample compositions, and densities.

Identification	Solution	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> - 5H <sub>2</sub> O, wt%	Glycerin, wt%	Richland City Water, wt%	Density, g/mL	Viscosity, cP at 25 °C
158-GS	Glycerin Salt	40.461	31.921	27.625	1.321	13.4
158-SS	Salt Solution	59.433	0.000	40.570	1.335	NA

Table E.2. Stock Solutions
Identification	% Dilution	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> - 5H <sub>2</sub> O, wt%	Glycerin, wt%	Richland City Water, wt%	Density, g/mL
158-GS	0	40.461	31.921	27.625	1.321
158-1	10.7	42.497	28.494	29.014	1.326
158-2	19.4	44.134	25.742	30.131	1.328
158-3	26.5	45.487	23.465	31.054	1.332
158-4	37.5	47.580	19.949	32.482	1.333
158-5	51.9	50.311	15.347	34.345	1.341

Table E.3. Diluted Solutions

The viscosities were measured for each of the solutions at 15, 20, 25, and 30  $^{\circ}$ C. Table E.4 and Figure E.3 show the measured viscosities.

	No.S.O.	Clusorin		Visc	cosity, cP at	Temperature	e, °C
Identification	$Ma_2S_2O_3-5H_2O, wt\%$	wt%	Dilution %	15	20	25	30
158-GS	40.461	31.9	0	21.0	16.4	13.4	11.0
158-1	42.497	28.5	10.7	18.8	14.9	11.7	9.69
158-2	44.134	25.7	19.4	16.8	13.4	10.6	8.82
158-3	45.487	23.5	26.5	15.3	12.3	9.63	8.18
158-4	47.580	19.9	37.5	13.2	10.7	8.49	7.24
158-5	50.311	15.3	51.9	10.8	8.82	7.20	6.26

Table E.4. Measured Viscosities at Four Temperatures



Figure E.3. Viscosity as a Function of Temperature at Various Glycerin Compositions

Each curve in Figure E.3 was fit to a polynomial equation to determine the temperature that corresponded to the target viscosity (10.5 cP) and the upper and lower bounds ( $\pm 1$  cP) (see Table E.5). Figure E.4 shows the process temperature as a function of wt% glycerin to reach the target 10.5 cP viscosity, along with the upper and lower acceptable range. For a given wt% glycerin, the operating test temperature range is about 5 °C while still remaining in the target viscosity range

Table E.5.	Curve Fits to	Reach Target	and Upper and	l Lower Visco	sity Targets at	Temperature
------------	---------------	--------------	---------------	---------------	-----------------	-------------

		Ter	mperature, °C	
Identification	Polynomial Curve Fit	10.5 cP	9.5 cP	11.5 cP
158-GS	$T = 0.076 cP^2 - 3.9358 cP + 64.118$	31.2	33.6	28.9
158-1	$T = 0.0787 cP^2 - 3.8615 cP + 59.837$	28.0	30.3	25.8
158-2	$T = 0.1044 cP^2 - 4.5181 cP + 61.527$	25.6	28.0	23.4
158-3	$T = 0.1327 cP^2 - 5.1641 cP + 63.049$	23.5	26.0	21.2
158-4	$T = 0.19 cP^2 - 6.3358 cP + 65.65$	20.1	22.6	17.9
158-5	$T = 0.36 cP^2 - 9.3767 cP + 74.535$	15.8	17.9	14.3



Figure E.4. Process Temperature as a Function of Glycerin Concentration at  $10.5 \pm 1$  cP

# Appendix F

### Malvern Particle Size Analyzer Output Records for the NiCladWC Solid Material

## Appendix F Malvern Particle Size Analyzer Output Records for the NiCladWC Solid Material

The particle size distribution (PSD) of the samples listed in Table F.1 were measured in accordance with WTPSP operating procedure OP-WTPSP-003, Rev. 3, using a Malvern Mastersizer 2000 with a Hydro G measuring accessory. The solids listed in Table F.1 include sieved fractions of a XLSciTech nickel clad tungsten carbide powder (lot # XLS7892). Data were collected by the Malvern Mastersizer 2000 software (Version 5.6) in Malvern data file "SHSVD WC samples Feb 2017.mea". A subsample of the sample solids is suspended in water by direct addition to the Hydro G dispersion unit. A dispersion unit flow and stir speed of 2500 and 1000 RPM are used, respectively. The PSD measurement protocol is outlined in Table F.2, and results in a set of pre-sonication, during-sonication, and post-sonication averages for each sample. The sample naming convention is "[ID]-[N].[M]", where ID is the test ID from Table F.1, N is the replicate number (1 for the primary measurement, 2 for the first replicate, 3 for the second replicate, and so on), and M is denotes the measurement type (1 – pre-sonciation, 2 – during-sonication, and 3 – post-sonication).

Table F.1: De-inventory solid samples evaluated for PSD and reported in this appendix.

Sample ID	Sample Description
NiCladWC-75-80 um	Sieved XlSciTech XLS7892, 75-80 µm fraction
NiCladWC- $<75$ um	Sieved XlSciTech XLS7892, <75 µm fraction
NiCladWC->80 um	Sieved XlSciTech XLS7892, $>80\mu\mathrm{m}$ fraction

Step	Operation	Sonicator	Duration [s]
1	Recirculate dispersion	Off	60
2	Measure pre-sonicated PSD	Off	120
3	Recirculate dispersion	On $(100\% \text{ power})$	60
4	Measure during-sonication PSD	On $(100\% \text{ power})$	120
5	Recirculate dispersion	Off	60
6	Measure post-sonication PSD	Off	120

Table F.2: Measuring protocol applied for determination of sample PSD

The active measurement of PSD is conducted at 1000 Hz, with a 20 second period of red light measurement followed immediately by a 20 second period of blue light measurement. Three separate periods of blue and red light measurement (taking a total of 120 seconds) make up each average PSD measurement reported herein. All average measurements were exported to PDF (file: "NiCladWC xls7892 psd SN-GP.pdf") using the Malvern Mastersizer 2000 Software (Version 5.6) and are included on the pages that follow. The measured PSD results are interpreted using two pre-programmed light scattering models available in the Malvern Mastersizer 2000 Software: 1) a "general purpose" model (for irregular powders with broad size range) and 2) a "single narrow mode (spherical)" model (typically intended for mono-disperse spheres but applied here to evaluate any changes to the distribution caused by the assumption of mono-dispersity).

#### F.1 Malvern Export: "NiCladWC xls7892 psd SN-GP.pdf"

On the following pages, the direct Malvern software PDF export for file "NiCladWC xls7892 psd SN-GP.pdf" is reproduced.





	Name:		20.00	SOP Name:				Measured:	10t 21 2017	1-22-14 DM		
Sample S	Source & type	ce & type: Measured by: Anal							131 21, 2017 -	+.52.111 W		
D3M966 Sample bulk lot ref: Result Source:							Monday, Augu	ıst 21, 2017 4	:32:12 PM			
eampie :				Averaged								
Particle I	Name:	Accessory Name:						Analysis mo	Sensitivity:			
Tungsten Particle I	powder RI-			Hydro 2000G (/	A)			Single narrow	mode (spher	ical)	Enhanced	n•
2.240	NI.			0.1				0.020	to 2000.000	) um	9.44 %	
Dispersa Water	int Name:			Dispersant RI 1.330	:			Weighted Re 0.624	sidual: %		Result Emu Off	lation:
<b>Concent</b> 0.1277	ration: %Vol			<b>Span :</b> 0.539				Uniformity: 0.17			Result units	5:
Specific 0.0655	Surface Area m²/g	:		Surface Weigł 91.615 u	n <b>ted Mean</b> I m	D[3,2]:		Vol. Weighted 95.455	<b>d Mean D[4,3</b> um	]:		
d(0.1):	71.721	um			d(0.5):	93.458	um			d(0.9):	122.079	um
Γ	30				Particl	e Size Dist	ributior					
								Δ				
	25	D D										
	୍ <u>ଚ</u> 20	) — — — — — — — — — — — — — — — — — — —										
	8											
	Ĕ <u></u> 15	5									-	
	īo 10										_	
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	(											
	t	0.01	0.1		1		0	100		1000 3	000	
ŀ	N// 01 - 11	0.75.00		•	Part	ticle Size	(µm)	7 4 66 44 -				_
	-NICladW	U-15-80 U	m-SIN-1.1	- Average,	IVIONday,	, August 2	21, 201	/ 4:32:11 P	VI Size (up)	Volume In %	Size (um)	Volume In %
	0.020	0.00	0.142	0.00	1.002	0.00	7.0	96 0.00	50.238	0.00	355.656	0.00
	0.022	0.00	0.159 0.178	0.00	1.125 1.262	0.00	7.9 8.9	62 0.00 34 0.00	56.368 63.246	2.01	399.052 447.744	0.00
	0.028	0.00	0.200	0.00	1.416	0.00	10.0	24 0.00	70.963	7.20 13.30	502.377	0.00
	0.032	0.00	0.224	0.00	1.589	0.00	11.2	47 0.00	79.621	19.39	563.677	0.00
	0.036	0.00	0.252	0.00	2.000	0.00	14.1	59 0.00	100.237	20.57	709.627	0.00
	0.045	0.00	0.317	0.00	2.244	0.00	15.8	87 0.00	112.468	18.15 12.24	796.214	0.00
	0.050	0.00	0.356	0.00	2.518	0.00	17.8	25 0.00	126.191	6.07	893.367	0.00
	0.063	0.00	0.448	0.00	3.170	0.00	20.0	40 0.00	158.866	1.07	1124.683	0.00
	0.071	0.00	0.502	0.00	3.557	0.00	25.1	79 0.00	178.250	0.00	1261.915	0.00
	0.080	0.00	0.564	0.00	3.991	0.00	28.2	51 0.00	200.000	0.00	1415.892 1588.656	0.00
	0.009	0.00	0.032	0.00	5.024	0.00	35.5	0.00	251.785	0.00	1782.502	0.00
	0.112	0.00	0.796	0.00	5.637	0.00	39.9	0.00	282.508	0.00	2000.000	0.00
	0.126	0.00	0.893	0.00	6.325 7.096	0.00	44.7 50.2	74 38 0.00	316.979 355.656	0.00		

Operator notes: XLS7892 sieved, 75-80 um fraction

Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545



![](_page_80_Picture_1.jpeg)

Sample NiCladW	<b>Name:</b> 'C-75-80 u	m-Sl	N-1.2 - Aver	ag	je	SOP Name:							Me Mo	easured: onday, August	21, 2017	4:34:06 PM			
Sample	Source & t	ype:				Measured by:							Ar M	nalysed:	21 2017	1-34-07 DM			
Sample	bulk lot re	ef:				Result Sourc Averaged	e:						IVIC	onday, August i	21, 2017 4	4.34.07 PW			
Particle Tungster	Name: powder					Accessory Na Hydro 2000G	an (A	ne: )					Ar Sir	nalysis model ngle narrow mo	: ode (spher	ical)	Se Er	ensitivity: hanced	
Particle	RI:					Absorption:							Si	ze range:	2000.000		0	bscuratio	n:
Dispersa Water	ant Name:					Dispersant R 1.330	:						<b>W</b> 0.0	eighted Resid	2000.000	J um	Re Of	esult Emu	lation:
<b>Concent</b> 0.1363	t <b>ration:</b> %Vol					<b>Span :</b> 0.538							Ur 0.1	niformity: 17			Re Vo	esult units	5:
<b>Specific</b> 0.0655	Surface A m²/g	rea:				Surface Weig 91.561 u	<b>jht</b> um	ed Mean	D[3,	,2]:			<b>Vo</b> 95	<b>5.394</b> um	ean D[4,3	3]:			
d(0.1):	71.69	93	um					d(0.5):	9	3.394		um				d(0.9):		121.978	um
		30		_			_	Partic	:le S	ize Di:	stril	oution					7		
														Δ					
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	(%	20		+			_												
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								Par	rticle	e Size	e (h	ım)							
	-NiClao	JWC	-75-80 u	m	-SN-1.2	2 - Average	, I	Monday	<i>,</i> Αι	ugust	21	, 2017	74	:34:06 PM					
	Size (µ	m) V 20	olume In %		Size (µm) 0.142	Volume In %		Size (µm) 1.002	Volu	me In %	0	Size (µn 7.09	n) 16	Volume In %	Size (µm) 50.238	Volume In %		Size (µm) 355.656	Volume In %
	0.0	22	0.00		0.159	0.00		1.125		0.00		7.96	2	0.00	56.368	2.02		399.052	0.00
	0.0	25 28	0.00		0.178	0.00		1.262		0.00		10.02	24	0.00	70.963	7.22		447.744 502.377	0.00
	0.0	32	0.00		0.224	0.00		1.589		0.00		11.24	7	0.00	79.621	13.34 19.43		563.677	0.00
	0.0	36 40	0.00		0.252	0.00		1.783		0.00	•	12.61 14.15	9	0.00	89.337 100.237	20.59		632.456 709.627	0.00
	0.0	45	0.00		0.317	0.00		2.244		0.00		15.88	17	0.00	112.468	18.12		796.214	0.00
	0.0	50	0.00		0.356	0.00		2.518		0.00		17.82	5	0.00	126.191	6.03		893.367	0.00
	0.0	63	0.00		0.399	0.00		2.825		0.00	)	20.00	10	0.00	141.589	1.06		1124.683	0.00
	0.0	71	0.00		0.502	0.00		3.557		0.00		25.17	9	0.00	178.250	0.00		1261.915	0.00
	0.0	80	0.00		0.564	0.00		3.991		0.00		28.25	1	0.00	200.000	0.00		1415.892	0.00
	0.0	00	0.00		0.632	0.00		4.477		0.00	2	31.69	6	0.00	224.404 251.785	0.00		1782.502	0.00
	0.1	12	0.00		0.796	0.00		5.637		0.00		39.90	95	0.00	282.508	0.00		2000.000	0.00
	0.1: 0.1	26 42	0.00		0.893 1.002	0.00		6.325 7.096		0.00		44.77 50.23	4 8	0.00	316.979 355.656	0.00			

Operator notes: XLS7892 sieved, 75-80 um fraction

Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545 File name: SHSVD WC samples Feb 2017.mea Record Number: 190 9/5/2017 3:40:27 PM

![](_page_81_Picture_0.jpeg)

![](_page_81_Picture_1.jpeg)

Sample Nar NiCladWC-	<b>me:</b> 75-80 um-S	SN-1.3 - Aver	age	SOP Name:				<b>Measured:</b> Monday, Augusi	t 21, 2017 4	4:36:01 PM				
Sample Sou	urce & type	<b>):</b>		Measured by:				Analysed: Monday August 21, 2017 4:36:02 PM						
Sample bu	Ik lot ref:			Result Source Averaged			Monday, August 21, 2017 4.00.02 F M							
Particle Na Tungsten po Particle RI: 2.240 Dispersant Water	ame: owder : t Name:	Accessory Name: Hydro 2000G (A) Absorption: 0.1 Dispersant RI: 1.330						Analysis mode Single narrow m Size range: 0.020 to Weighted Resi 0.650 %	l: ode (spher 2000.000 dual:	ical) ) um	Sensitivity: Enhanced Obscuration: 10.12 % Result Emulation: Off			
Concentrat 0.1374	tion: %Vol			<b>Span :</b> 0.540				Uniformity: 0.17			Result unit Volume	s:		
Specific Su 0.0655	u <b>rface Area</b> m²/g	:		<b>Surface Weigh</b> 91.633 ui	n <b>ted Mean</b> m	D[3,2]:		<b>Vol. Weighted I</b> 95.484 un	<b>Mean D[4,3</b> า	3]:				
d(0.1):	71.718	um			d(0.5):	93.481	um			d(0.9):	122.165	um		
					Partic	le Size Distr	ibution							
Volume (92)	25 (% 20 15 10 10 5 (%)									1000 2				
	(	J.01	0.1		I		) 、	100		1000 3	000			
_		C 7E 00 ···	m CN 1 7	A. 10 70 71	Monder		µIII) 1 2017	1.24.01 DM				-		
	-NiCladW Size (µm) 0.020 0.022 0.025 0.028 0.032 0.036 0.040 0.045 0.056 0.056 0.063 0.071 0.080 0.089 0.100 0.112 0.126 0.142	C-75-80 u Volume In % 0.00	m-SN-1.3 Size (µm) 0.142 0.159 0.178 0.200 0.224 0.252 0.283 0.317 0.356 0.399 0.448 0.502 0.564 0.632 0.564 0.632 0.710 0.796 0.893 1.002	<ul> <li>Average,</li> <li>Volume In %</li> <li>0.00</li> </ul>	Monday Size (µm) 1.002 1.125 1.262 1.416 1.589 1.783 2.000 2.244 2.518 2.825 3.170 3.557 3.991 4.477 5.024 4.537 6.325 7.096	Y, August 2:           Volume In %           0.00	1, 2017 Size (µn 7.09 7.96 8.93 10.02 11.24 12.61 14.15 15.88 17.82 20.000 22.44 25.17 28.25 31.69 35.56 39.90 44.77 50.23	4:36:01 PM	Size (µm) 50.238 56.368 63.246 70.963 79.621 89.337 100.237 112.468 126.191 141.589 158.866 178.250 200.000 224.404 251.785 282.508 316.979 355.656	Volume In % 0.00 2.02 7.19 13.28 19.36 20.55 18.13 12.25 6.11 1.09 0.00 0.00 0.00 0.00 0.00 0.00 0	Size (µm) 355.656 399.052 447.744 502.377 632.456 709.627 796.214 893.367 1002.374 1124.683 1261.915 1415.892 1588.656 1782.502 2000.000	Volume In % 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.		

Operator notes: XLS7892 sieved, 75-80 um fraction

Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545 File name: SHSVD WC samples Feb 2017.mea Record Number: 194 9/5/2017 3:40:27 PM

![](_page_82_Picture_0.jpeg)

![](_page_82_Picture_1.jpeg)

Sample I NiCladW	Name: SOP Name: /C-<75 um-SN-1.1 - Average								Measured: Monday, August 21, 2017 4:49:01 PM						
Sample S	Sourc	e & type:		1	Measured by: D3M966				<b>Analysed:</b> Monday, August 21, 2017 4:49:02 PM						
Sample I	bulk	ot ref:		1	Result Source Averaged	:									
Particle Tungsten Particle 2.240	icle Name:     Accessory Name:       gsten powder     Hydro 2000G (A)       icle RI:     Absorption:       0     0.1       persant Name:     Dispersant RI:							Analysis mod Single narrow ( Size range: 0.020 to Weighted Page	n:						
Water		anie.			1.330	•			0.616 9	%		Off	nation.		
<b>Concent</b> 0.0862	ratio	n: 6Vol		:	<b>Span :</b> 0.586				Uniformity: 0.177	s:					
Specific 0.08	Surfa n	n <b>ce Area:</b> n²/g		-	<b>Surface Weigh</b> 74.996 u	n <b>ted Mean</b> m	D[3,2]:		<b>Vol. Weighted</b> 78.769 u	Mean D[4,3	]:				
d(0.1):		57.348	um			d(0.5):	77.134	um			d(0.9):	102.555	um		
[						Partic	le Size Dist	ributior	n			-			
		25							Λ						
	(%)	20										-			
	lume (	15													
	٨٥	10													
		5													
		8	.01	0.1		1	1	0	100		1000 3	000			
						Par	ticle Size	(um)							
	—Ni	CladW0	C-<75 um-	-SN-1.1 -	Average. N	londay.	August 21	. 2017	4:49:01 PM						
L	S	ize (µm) V	/olume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µ	m) Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %		
		0.020	0.00	0.142	0.00	1.002	0.00	7.0	96 62 0.00	50.238 56.368	6.23	355.656	0.00		
		0.025	0.00	0.178	0.00	1.262	0.00	8.9	0.00 34 0.00	63.246	11.35 16.32	447.744	0.00		
		0.028	0.00	0.200	0.00	1.416	0.00	10.0	24 0.00	70.963	18.99	502.377	0.00		
		0.032	0.00	0.224	0.00	1.589	0.00	11.2	47 0.00 19	89.337	18.22	632.456	0.00		
		0.040	0.00	0.283	0.00	2.000	0.00	14.1	59 0.00 0.00	100.237	14.28 8.68	709.627	0.00		
		0.045	0.00	0.317	0.00	2.244	0.00	15.8	87 0.00	112.468	3.28	796.214	0.00		
		0.050	0.00	0.356	0.00	2.518	0.00	20.0	0.00	126.191	0.23	1002.374	0.00		
		0.063	0.00	0.448	0.00	3.170	0.00	22.4	40 0.00	158.866	0.00	1124.683	0.00		
		0.071 0.080	0.00	0.502	0.00	3.557 3.991	0.00	25.1 28.2	79 0.00 51	178.250 200.000	0.00	1261.915 1415.892	0.00		

Operator notes: XLS7892 sieved, < 75 um fraction

0.00

0.00

0.00

0.00

0.00

0.632

0.710

0.796

0.893

0.00

0.00

0.00

0.00

0.00

4.477

5.024

5.637

6.325 7.096

0.089

0.100

0.112

0.126 0.142

Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545

0.00

0.00

0.00

0.00

0.00

31.698

35.566

39.905

44.774 50.238

0.00

0.00

0.00

0.20

2.22

224.404

251.785

282.508

316.979 355.656

File name: SHSVD WC samples Feb 2017.mea Record Number: 198 9/5/2017 3:40:27 PM

1588.656

1782.502

2000.000

0.00

0.00

0.00

0.00

0.00

0.00

0.00

0.00

![](_page_83_Picture_0.jpeg)

![](_page_83_Picture_1.jpeg)

Sample I NiCladW	<b>Name:</b> /C-<75 ւ	um-SN-	1.2 - Average	e	SOP Nam	e:					Measured: Monday, August 21, 2017 4:51:22 PM					
Sample	Source	& type	:		Measured	l by:					Analysed: Monday, Aug	aust 21. 2017 4:	51:23 PM			
Sample	bulk lot	t ref:			Result So Averaged	ource:										
Particle Tungsten	Name:	r			Accessory Name: Hydro 2000G (A)						Analysis m Single narro	odel: w mode (spheric	al)	Sensitivity: Enhanced		
Particle 2,240	RI:				Absorption:					Size range: 0.020	to 2000.000	um	Obscura 8.24	ition: %		
Dispersa Water	ant Nan	ne:			Dispersant RI: 1.330					Weighted R 0.614	esidual:	um	Result E	mulation:		
<b>Concent</b> 0.0900	tration: %V	′ol			<b>Span :</b> 0.587						Uniformity: 0.178			<b>Result u</b> Volume	nits:	
<b>Specific</b> 0.0802	Surface m²/	<b>e Area:</b> ′g			Surface V 74.831	Veighte um	ed Mean	D[3,2]:			Vol. Weight 78.612	ed Mean D[4,3] um	:			
d(0.1):	57	.202	um				d(0.5):	76.95	8	um			d(0.9):	102.41	2 um	
							Partic	le Size I	Distri	bution				_		
		25									Λ			_		
	(%	20														
	) er	15														
	olun	10														
	>	10														
		5												_		
		0														
		8	.01	0.1			1		10	)	100	)	1000 3	000		
							Par	ticle Si	ze (µ	ım)						
	-NiC	ladW(	C-<75 um	<u>-SN-1.2</u>	- Averag	e, Mo	onday, i	August	21,	2017	4:51:22 PI	<u>И</u>		1		
	Size	e (µm) \ 0.020	0.00	Size (µm) 0.142	Volume In	% : 00	Size (µm) 1.002	Volume In	00	Size (µ 7.09	m) Volume In 9	6 Size (μm) 50.238	6.34	Size (µ 355.6	m) Volume In %	
		0.022 0.025	0.00	0.159	0.0	00	1.125 1.262	0.	00	7.9	62 0.0 34 0.0	0 56.368 63.246	11.47	399.0 447.7	52 0.00	
		0.028	0.00	0.200	0.0	00	1.416	0. 0.	00	10.0	24 0.0	70.963	16.39 18.97	502.3	77 0.00 77	
		0.032	0.00	0.224	0.0	00	1.589	0.	00	11.24	0.0	0 79.621	18.12	563.6	0.00	

0.00 0.00 0.00 0.00 18.12 0.036 0.252 12.619 89.337 632.456 1.783 0.00 0.00 0.00 0.00 14.15 0.040 0.283 2.000 14.159 100.237 709.627 0.00 0.00 0.00 8.58 0.00 0.045 0.317 2.244 15.887 112.468 796.214 0.00 3.24 0.00 0.00 0.00 0.050 0.356 2.518 17.825 126.191 893.367 0.00 0.00 0.00 0.00 0.22 0.056 141.589 1002.374 0.399 2.825 20.000 0.00 0.00 0.00 0.00 0.00 3.170 158.866 1124.683 0.063 0.448 22,440 0.00 0.00 0.00 0.00 0.00 1261.915 0.071 0.502 3.557 25.179 178.250 0.00 0.00 0.00 0.00 0.00 3.991 1415.892 0.080 0.564 28.251 200.000 0.00 0.00 0.00 0.00 0.00 0.089 0.632 4.477 31.698 224.404 1588.656 0.00 0.00 0.00 0.00 0.00 0.100 0.710 5.024 35.566 251.785 1782.502 0.00 0.00 0.00 0.00 0.00 0.112 0.796 5.637 39.905 282.508 2000.000 0.00 0.00 0.00 0.21 0.00 0.126 0.142 0.893 6.325 7.096 44.774 316.979 355.656 0.00 0.00 0.00 2.29 0.00 50.238

**Operator notes:** XLS7892 sieved, < 75 um fraction

Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545 File name: SHSVD WC samples Feb 2017.mea Record Number: 202 9/5/2017 3:40:28 PM

0.00

0.00

0.00

0.00

0.00

0.00

0.00

0.00

0.00

0.00

![](_page_84_Picture_0.jpeg)

![](_page_84_Picture_1.jpeg)

Sample Name:SOP Name:Measured:NiCladWC-<75 um-SN-1.3 - AverageMonday, August 21, 2017 4	Measured: Monday, August 21, 2017 4:53:20 PM						
Sample Source & type:         Measured by:         Analysed:           D3M966         Monday, August 21, 2017 4:	53:22 PM						
Sample bulk lot ref: Result Source: Averaged							
Particle Name:         Accessory Name:         Analysis model:           Tungsten powder         Hydro 2000G (A)         Single narrow mode (spheric	Sensitivity: cal) Enhanced						
Particle RI:         Absorption:         Size range:           2.240         0.1         0.020         to         2000.000	Obscuration: um 8.35 %						
Dispersant Name:Dispersant RI:Weighted Residual:Water1.3300.613%	Result Emulation: Off						
Concentration:         Span :         Uniformity:           0.0913         %Vol         0.588         0.178	<b>Result units:</b> Volume						
Specific Surface Area:         Surface Weighted Mean D[3,2]:         Vol. Weighted Mean D[4,3]           0.0802         m²/g         74.849         um         78.633         um	:						
d(0.1): 57.216 um d(0.5): 76.950 um	d(0.9): 102.490 um						
Particle Size Distribution							
25							
$\odot$ 20							
3 10							
5							
8.01 0.1 1 10 100	1000 3000						
Particle Size (µm)							
NiCladWC-<75 um-SN-1.3 - Average, Monday, August 21, 2017 4:53:20 PM							
Size (um)   Volume In 9/   Size (um)							
0.020         0.00         0.142         0.00         1.002         0.00         7.096         0.00         50.238	Volume In %         Size (µm)         Volume In %           6.36         355.656         0.00						
Size (µn)         Volume n %	Volume In %         Size (µm)         Volume In %           6.36         355.656         0.00           11.50         447.744         0.00						
Size (µII)         Volume III %         Size (µII)         Volume III %         Size (µIII)         Volume III %         Size µIII         Volume III %         Si	Volume In %         Size (µm)         Volume In %           6.36         355.656         0.00           11.50         399.052         0.00           16.41         502.377         0.00           18.96         502.377         0.00						

0.036 0.040 0.045 0.050 0.056 0.063 0.071 0.080 0.089 0.100	0.00       0.00       0.00       0.00       0.00       0.00       0.00       0.00       0.00       0.00       0.00       0.00       0.00	0.252 0.283 0.317 0.356 0.399 0.448 0.502 0.564 0.632 0.710	0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0	1.783 2.000 2.244 2.518 2.825 3.170 3.557 3.991 4.477 5.024	0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0	12.618 14.159 15.887 17.825 20.000 22.440 25.179 28.251 31.698 35.566	0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0	89.3 100.2 112.4 126.1 141.5 158.8 178.2 200.0 224.4 251.7	7         14.12           7         8.58           8         3.29           9         0.23           6         0.00           0         0.00           0         0.00           4         0.00           5         0.00	632.456 709.627 796.214 893.367 1002.374 1124.683 1261.915 1415.892 1588.656 1782.502	0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0
0.080 0.089 0.100 0.112 0.126 0.142	0.00 0.00 0.00 0.00	0.584 0.632 0.710 0.796 0.893 1.002	0.00 0.00 0.00 0.00 0.00	4.477 5.024 5.637 6.325 7.096	0.00 0.00 0.00 0.00 0.00	28.25 31.698 35.566 39.905 44.774 50.238	0.00 0.00 0.19 2.28	200.00 224.40 251.78 282.50 316.97 355.68	0         0.00           4         0.00           5         0.00           8         0.00           9         0.00           6         0.00	1415.892 1588.656 1782.502 2000.000	0.00 0.00 0.00

**Operator notes:** XLS7892 sieved, < 75 um fraction

Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545

![](_page_85_Picture_0.jpeg)

![](_page_85_Picture_1.jpeg)

Sample I NiCladW	Name: C->80 um-S	SN-1.1 - Ave	rage		SOF	P Name:						Me Mo	easured: onday, August	21, 2017 :	5:05:40 PM			
Sample \$	Source & ty	pe:			Mea D3M	sured by	<b>y</b> :					An Mo	nalysed: onday, August	21, 2017 5	5:05:41 PM			
Sample	bulk lot ref	1			Res Avei	ult Sour raged	ce:											
Particle Tungsten Particle 2.240 Dispersa Water	Name: powder RI: ant Name:				Acc Hydr Abs 0.1 Disp 1.33	essory N ro 2000G corption: persant I	Nan 6 (A) RI:	ne: )				Ar Sir Si: 0.0 We 0.5	nalysis mode ngle narrow m ze range: 020 to eighted Resid 553 %	l: ode (spher 2000.000 dual:	ical) ) um	Sensitiv Enhance Obscura 8.56 Result E Off	ity: d ation: % Emula	ition:
<b>Concent</b> 0.1437	ration: %Vol				<b>Spa</b> 0.61	<b>n :</b> 5						<b>Ur</b> 0.1	<b>hiformity:</b> 194			<b>Result u</b> Volume	nits:	
Specific 0.0528	Surface Are m²/g	ea:			<b>Sur</b> 113.	<b>face Wei</b> 610	ght um	ed Mean	D[3,2]	:		<b>Vo</b> 11	9.676 um	/lean D[4,3	3]:			
d(0.1):	87.253	um						d(0.5):	115	.760	um				d(0.9):	158.45	8	um
]								Partic	e Size	Distr	ibutior	ı						]
		25																
		20											Λ					
	(% )	20														-		
	me (9	15														_		
	Volu	10																
		5																
		8.01		0	.1			1		1(	)		100		1000 3	000		
								Part	icle S	Size (	um)							
	-NiClad	NC- \ 80 I	ım_9	SN_1 1	- Δ	orado	N/u	onday	\uau	ct 21	2017	5.0						1
L	Size (um	Volume In 9	%	Size (un	n)   Volu	ume In %		Size (um)	Volume	st 21,	Size (u		Volume In %	Size (um)	Volume In %	Size (L	um) V	J olume In %
	0.020	0.0	0	0.14	2	0.00		1.002		0.00	7.0	96	0.00	50.238	0.00	355.6	56	0.00
	0.022	0.0	0	0.15	9 8	0.00		1.125		0.00	7.9	62 34	0.00	56.368 63.246	0.00	399.0	52 44	0.00
	0.028	0.0	0	0.20	0	0.00		1.416		0.00	10.0	24	0.00	70.963	0.15	502.3	77	0.00
	0.032	0.0	0	0.22	4	0.00		1.589		0.00	11.2	47	0.00	79.621	8.04	563.6	77	0.00
	0.036	0.0	0	0.25	2	0.00		1.783		0.00	12.6	19 59	0.00	89.337	14.90	632.4	56 27	0.00
	0.045	0.0	0	0.31	7	0.00		2.244		0.00	15.8	87	0.00	112.468	18.09	796.2	14	0.00
	0.050	0.0	0	0.35	6	0.00		2.518		0.00	17.8	25	0.00	126.191	16.11	893.3	67	0.00
	0.056	0.0	0	0.39	9 8	0.00		2.825		0.00	20.0	40	0.00	141.589 158.866	10.24	1002.3	74 83	0.00
	0.071	0.0	0	0.50	2	0.00		3.557		0.00	25.1	79	0.00	178.250	6.51	1261.9	15	0.00
	0.080	0.0	0	0.56	4	0.00		3.991		0.00	28.2	51	0.00	200.000	0.62	1415.8	92	0.00
	0.089	0.0	0	0.63	0	0.00		4.477 5.024		0.00	31.6	98 66	0.00	224.404	0.05	1588.6	56 02	0.00
	0.112	0.0	0	0.79	6	0.00		5.637		0.00	39.9	05	0.00	282.508	0.00	2000.0	00	0.00
	0.126	0.0	0	0.89	3 2	0.00		6.325 7.096		0.00	44.7	74 38	0.00	316.979 355.656	0.00			

XLS7892 sieved, >80 um fraction Operator notes:

Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545 File name: SHSVD WC samples Feb 2017.mea Record Number: 210 9/5/2017 3:40:28 PM

![](_page_86_Picture_0.jpeg)

![](_page_86_Picture_1.jpeg)

Sample I NiCladW	Name: C->80 um-SN-	-1.2 - Average	sc	OP Name:				Measured: Monday, August	t 21, 2017 5	5:07:42 PM		
Sample S	Source & type	:	Me	easured by:				Analysed:	21 2017 5	·07·43 PM		
Sample I	bulk lot ref:		Re Av	esult Source eraged	:			wonday, August	21, 2017 3	.07.431 1		
Particle Tungsten Particle 2.240 Dispersa Water	Name: powder RI: unt Name:		Ac Hy 0.1 Dis 1.3	cessory Na dro 2000G (/ psorption: spersant RI:	<b>me</b> : \)			Analysis mode Single narrow m Size range: 0.020 to Weighted Resi 0.554 %	l: ode (spheri 2000.000 dual:	cal) um	Sensitivity: Enhanced Obscuratio 8.93 % Result Emu Off	n: Ilation:
<b>Concent</b> 0.1502	ration: %Vol		<b>Sp</b> 0.6	<b>an :</b> 620				Uniformity: 0.198			Result units	S:
<b>Specific</b> 0.0528	Surface Area: m²/g		<b>Su</b> 11:	r <b>face Weigh</b> 3.661 ur	n <b>ted Mean</b> m	D[3,2]:		Vol. Weighted I 120.100 un	<b>Mean D[4,3</b> ] า	]:		
d(0.1):	87.153	um			d(0.5):	115.688	um			d(0.9):	158.845	um
	25 20 (%) = 15 10 10 5										-	
	8	01	0.1		1	1	0	100		1000 3	000	
	Ū		011		Par	ticle Size (	μm)					
	-NiCladW	C->80 um-	SN-1.2 - A	verage, N	londay, <i>i</i>	August 21	, 2017	5:07:42 PM				
	Size (µm)	Volume In %	Size (µm) Vo	olume In %	Size (µm)	Volume In %	Size (µ	m) Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
	0.022 0.022 0.025 0.032 0.036 0.040 0.045 0.050 0.056 0.056 0.055 0.071 0.080 0.089 0.100	0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0	0.159 0.178 0.200 0.224 0.252 0.283 0.317 0.356 0.399 0.448 0.502 0.564 0.632 0.710 0.776	0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0	1.125 1.262 1.416 1.589 1.783 2.000 2.244 2.518 2.825 3.170 3.557 3.991 4.477 5.024 5.637	0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0	7.05 8.93 10.02 11.24 12.67 14.15 17.82 20.00 22.44 25.17 28.25 31.66 35.56 35.57	0.00           42         0.00           44         0.00           99         0.00           99         0.00           99         0.00           90         0.00           90         0.00           90         0.00           90         0.00           90         0.00           90         0.00           90         0.00           90         0.00           90         0.00           90         0.00           90         0.00           90         0.00           90         0.00           90         0.00           90         0.00           91         0.00           92         0.00           93         0.00           94         0.00           95         0.00	56.368 63.246 70.963 79.621 89.337 100.237 112.468 126.191 141.589 158.866 178.250 200.000 224.404 251.785 282.508	0.00 0.00 0.17 4.16 8.08 14.92 18.07 18.47 16.01 10.13 6.41 2.59 0.61 0.08 0.03	399.052 447.744 502.377 632.456 709.627 796.214 893.367 1002.374 1124.683 1261.915 1415.892 1588.656 1782.502 2000.000	0.04 0.00 0.00 0.00 0.00 0.00 0.00 0.00
	0.126	0.00	0.893	0.00	6.325	0.00	44.77	4 0.00	316.979	0.13		

XLS7892 sieved, >80 um fraction Operator notes:

1.002

0.142

Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545

50.238

355.656

7.096

![](_page_87_Picture_0.jpeg)

![](_page_87_Picture_1.jpeg)

Sample I NiCladW	Name: C->80 um-SI	N-1.3 - Avera	ge	SOP Name:				<b>Measured:</b> Monday, Augu	st 21, 2017 5	i:09:37 PM		
Sample \$	Source & typ	e:		Measured by: D3M966				Analysed: Monday, Augus	st 21. 2017 5	:09:38 PM		
Sample	e Name: WC->80 um-SN-1.3 - Average e Source & type: e bulk lot ref: e Name: e npowder e RI: sant Name: ntration: %Vol ic Surface Area: m <sup>2</sup> /g 1): 87.175 um 25 20 25 20 15 15 20 15 15 8.01 NiCladWC->80 um-S Size (um) Volume In % 0.022 0.002 0.002 0.0000 0.0000 0.0000 0.0000000000000000000000000000000			Result Source Averaged					. ,			
Particle Tungsten Particle 2.240 Dispersa Water	He Name: dWC->80 um-SN-1.3 - Average He Source & type: He bulk lot ref: the powder the powder the RI: entration: 2 %Vol fic Surface Area: 9 m <sup>2</sup> /g 1): 87.175 um $25 \frac{25}{20} \frac{15}{10} \frac{15}{10$			Accessory Na Hydro 2000G (. Absorption: 0.1 Dispersant RI 1.330	<b>me</b> : 4) :			Analysis modSingle narrow inSize range:0.020trWeighted Res0.5639	el: mode (spheri o 2000.000 sidual: 6	cal) um	Sensitivity: Enhanced Obscuration 8.88 % Result Emu Off	n: lation:
<b>Concent</b> 0.1492	ration: %Vol			<b>Span :</b> 0.614				Uniformity: 0.194			Result units Volume	<b>S:</b>
<b>Specific</b> 0.0529	Surface Area m²/g	a:		Surface Weigl 113.497 u	n <b>ted Mean</b> m	D[3,2]:		<b>Vol. Weighted</b> 119.547 u	<b>Mean D[4,3</b> m	]:		
d(0.1):	87.175	um			d(0.5):	115.674	um			d(0.9):	158.194	um
[					Partic	le Size Dist	ribution					
	2	5										
	2	.5						Λ				
	2	0										
	5) 1 me	5									_	
	nlov 1	0									-	
		5										
		8.01	0.1		1	1	0	100		1000 3	000	
					Par	ticle Size	(µm)					
	-NiCladW	/C->80 um	n-SN-1.3	- Average, N	londay, <i>l</i>	August 21	, 2017	5:09:37 PM				
	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µ	m) Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
	0.022	0.00	0.142	0.00	1.125	0.00	7.9	0.00	56.368	0.00	399.052	0.00
	0.025	0.00	0.178	0.00	1.262	0.00	8.93	0.00	63.246	0.00	447.744	0.00
	0.028	0.00	0.200	0.00	1.416 1.589	0.00	10.0	0.00	70.963	4.15	502.377	0.00
	0.036	0.00	0.252	0.00	1.783	0.00	12.6	0.00	89.337	8.05 14.91	632.456	0.00
	0.040	0.00	0.283	0.00	2.000	0.00	14.1	0.00	100.237	18.11	709.627	0.00
	0.045	0.00	0.317	0.00	2.244	0.00	15.8	0.00	12.468	18.55	893.367	0.00
	0.056	0.00	0.399	0.00	2.825	0.00	20.00	0.00	141.589	16.12 10.21	1002.374	0.00
	0.063	0.00	0.448	0.00	3.170	0.00	22.4	0.00	158.866	6.47	1124.683	0.00
	0.071	0.00	0.502	0.00	3.557	0.00	25.1	0.00	200.000	2.60	1415.892	0.00
	0.089	0.00	0.632	0.00	4.477	0.00	31.69	0.00	224.404	0.59	1588.656	0.00
	0.100	0.00	0.710	0.00	5.024	0.00	35.5	0.00	251.785	0.00	1782.502	0.00
	0.112	0.00	0.796	0.00	6.325	0.00	44.7	0.00	316.979	0.00	2000.000	
	0.142	0.00	1.002	0.00	7.096	0.00	50.23	0.00	355.656	0.00		

XLS7892 sieved, >80 um fraction Operator notes:

Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545

![](_page_88_Picture_0.jpeg)

![](_page_88_Picture_1.jpeg)

Sample N NiCladW	<b>lame:</b> C-75-80 um	-1.1-Average		SOP Name:				Measured: Monday, Augus	it 21, 2017 4	4:32:11 PM		
Sample S	Source & ty	pe:		Measured by: D3M966				Analysed: Thursday, Augu	st 31, 2017	4:48:41 PM		
Sample I	oulk lot ref			Result Source Averaged	:							
Particle I Tungsten Particle I	Name: powder RI:			Accessory Na Hydro 2000G (/ Absorption:	<b>me:</b> A)			Analysis mode General purpos Size range:	e		Sensitivity: Normal Obscuratio	n:
Dispersa Water	int Name:			Dispersant RI 1.330				Weighted Resi           0.672         %	idual:	) un	Result Emu Off	lation:
<b>Concent</b> 0.1267	ration: %Vol			<b>Span :</b> 0.638				Uniformity: 0.201			Result units	s:
Specific 0.066	Surface Are m²/g	ea:		<b>Surface Weigh</b> 90.891 ui	n <b>ted Mean</b>	D[3,2]:		Vol. Weighted 96.360 ur	<b>Mean D[4,3</b> n	]:		
d(0.1):	68.116	um			d(0.5):	93.583	um			d(0.9):	127.868	um
Γ					Partic	le Size Distr	ibution					
		25						Λ			_	
		20						/			_	
	(%											
	) el	15									-	
	olum	10										
	×											
		5										
		8.01	0.1		1	1(	C	100		1000 3	000	
					Par	ticle Size (	µm)					_
Ľ	-NiClad	NC-75-80 ι	um-1.1-Av	erage, Mon	day, Auc	gust 21, 20	17 4:32	2:11 PM	Cine (ver)			Values la 9
	0.020	0.00	0.142	0.00	1.002	0.00	7.09	6 0.00	50.238	1.11	355.656	0.00
	0.022	0.00	0.159	0.00	1.125 1.262	0.00	7.96	2 0.00	56.368 63.246	3.93	399.052 447.744	0.00
	0.028	0.00	0.200	0.00	1.416	0.00	10.02	4 0.00	70.963	7.75 12.50	502.377	0.00
	0.032	0.00	0.224	0.00	1.589	0.00	11.24	0.00	79.621	17.13	563.677 632.456	0.00
	0.040	0.00	0.283	0.00	2.000	0.00	14.15	9 0.00	100.237	18.13	709.627	0.00
	0.045	0.00	0.317	0.00	2.244	0.00	15.88	7 0.00	112.468	11.84	796.214	0.00
	0.050	0.00	0.356	0.00	2.518	0.00	20.00	0.00	126.191	6.95	893.367 1002.374	0.00
	0.063	0.00	0.448	0.00	3.170	0.00	22.44	0.00	158.866	3.12 0.92	1124.683	0.00
	0.071	0.00	0.502	0.00	3.557	0.00	25.17	9 0.00	178.250	0.07	1261.915	0.00
	0.089	0.00	0.632	0.00	4.477	0.00	31.69	0.00	224.404	0.00	1588.656	0.00
	0.100	0.00	0.710	0.00	5.024	0.00	35.56	6 0.00	251.785	0.00	1782.502	0.00
	0.112	0.00	0.796	0.00	5.637 6.325	0.00	39.90 44.77	0.00	282.508 316.979	0.00	2000.000	
	0.142	0.00	1.002	0.00	7.096	0.00	50.23	8 0.24	355.656	0.00		

Operator notes: Average of 3 measurements from SHSVD WC samples Feb 2017.mea

Malvern Instruments Ltd. Malvern, UK Tel := +[44] (0) 1684-892456 Fax +[44] (0) 1684-892789 Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545 File name: SHSVD WC samples Feb 2017.mea Record Number: 238 9/5/2017 3:40:28 PM

![](_page_89_Picture_0.jpeg)

![](_page_89_Picture_1.jpeg)

Sample N NiCladW	<b>lame:</b> C-75-80 um	-1.2-Average		SOP Name:				Measured: Monday, Augus	t 21, 2017 4	4:34:06 PM		
Sample S	Source & ty	pe:		Measured by: D3M966				Analysed: Thursday, Augu	st 31. 2017	4:49:59 PM		
Sample I	oulk lot ref	:		Result Source Averaged	:				, .			
Particle I Tungsten Particle I 2.240 Dispersa Water	Name: powder RI: unt Name:			Accessory Na Hydro 2000G ( <i>A</i> Absorption: 0.1 Dispersant RI 1.330	<b>me</b> : A)			Analysis mode General purpos Size range: 0.020 to Weighted Resi 0.675 %	el: e 2000.000 idual:	) um	Sensitivity: Normal Obscuratio 10.06 % Result Emu Off	n: , ilation:
<b>Concent</b> 0.1352	ration: %Vol			<b>Span :</b> 0.639				Uniformity: 0.201			Result units	s:
Specific 0.066	Surface Ar m²/g	ea:		Surface Weigh 90.847 u	n <b>ted Mean</b> m	D[3,2]:		Vol. Weighted 96.313 ur	<b>Mean D[4,3</b> n	i]:		
d(0.1):	68.084	um			d(0.5):	93.536	um			d(0.9):	127.808	um
Γ					Partic	le Size Distr	ibution					
		25						Δ			_	
		20									_	
	(%)											
	ne (	15									_	
	Volui	10									_	
	-	F										
		5										
		8.01	0.1	1	1	1(	0	100		1000 3	000	
					Par	ticle Size (	µm)					
Ŀ	-NiClad	NC-75-80	um-1.2-A	verage, Mon	day, Au	gust 21, 20	17 4:34	1:06 PM				
	Size (µm 0.020	) Volume In %	Size (µm) 0.142	Volume In %	Size (µm) 1.002	Volume In %	Size (µr 7.09	h) Volume In %	Size (µm) 50.238	Volume In %	Size (µm) 355.656	Volume In %
	0.022	0.00	0.159	0.00	1.125	0.00	7.96	2 0.00	56.368	3.94	399.052	0.00
	0.028	0.00	0.200	0.00	1.416	0.00	10.02	4 0.00	70.963	7.77	502.377	0.00
	0.032	0.00	0.224	0.00	1.589	0.00	11.24	7 0.00	79.621	12.55	563.677	0.00
	0.030	0.00	0.252	0.00	2.000	0.00	12.61	9 0.00	89.337	18.13	632.456 709.627	0.00
	0.045	0.00	0.317	0.00	2.244	0.00	15.88	7 0.00	112.468	16.29 11.82	796.214	0.00
	0.050	0.00	0.356	0.00	2.518	0.00	17.82	5 0.00	126.191 141.589	6.93	893.367 1002.374	0.00
	0.063	0.00	0.399	0.00	3.170	0.00	22.44	0.00	158.866	3.11	1124.683	0.00
	0.071	0.00	0.502	0.00	3.557	0.00	25.17	9 0.00	178.250	0.92	1261.915	0.00
	0.080	0.00	0.564	0.00	3.991 4.477	0.00	28.25	1 0.00	200.000	0.00	1415.892 1588.656	0.00
	0.100	0.00	0.710	0.00	5.024	0.00	35.56	6 0.00	251.785	0.00	1782.502	0.00
	0.112	0.00	0.796	0.00	5.637	0.00	39.90	5 0.00	282.508	0.00	2000.000	0.00
	0.120	0.00	1.002	0.00	6.325 7.096	0.00	50.23	4 0.24	315.979	0.00		

Malvern Instruments Ltd. Malvern, UK Tel := +[44] (0) 1684-892456 Fax +[44] (0) 1684-892789 Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545 File name: SHSVD WC samples Feb 2017.mea Record Number: 242 9/5/2017 3:40:28 PM

![](_page_90_Picture_0.jpeg)

![](_page_90_Picture_1.jpeg)

Sample Nar NiCladWC-	<b>me:</b> 75-80 um-′	1.3-Average		SOP Name:				<b>Measured:</b> Monday, Augus	it 21, 2017 4	4:36:01 PM		
Sample Sou	urce & typ	e:		Measured by: D3M966				Analysed: Thursday, Augu	st 31, 2017	4:50:58 PM		
Sample bu	lk lot ref:			Result Source Averaged	:				, -			
Particle Na Tungsten po Particle RI: 2.240 Dispersant Water	owder			Accessory Na Hydro 2000G (/ Absorption: 0.1 Dispersant RI: 1.330	<b>me</b> : A)			Analysis mode General purpos Size range: 0.020 to Weighted Res 0.669 %	el: e 2000.000 idual:	) um	Sensitivity: Normal Obscuration 10.12 % Result Emu Off	n: Ilation:
Concentrat 0.1362	tion: %Vol			<b>Span :</b> 0.639				Uniformity: 0.201			Result units	S:
Specific Su 0.066	n <b>rface Area</b> m²/g	1:		Surface Weigh 90.877 ur	n <b>ted Mean</b>	D[3,2]:		Vol. Weighted 96.351 ur	<b>Mean D[4,3</b> m	]:		
d(0.1):	68.099	um			d(0.5):	93.571	um			d(0.9):	127.873	um
					Partic	le Size Distr	ibution				7	
(%) (%)	2 2 (%) 2	5									-	
											_	
		0										
		8.01	0.1		1	1(	C	100		1000 3	000	
					Par	ticle Size (	µm)					
	NiCladW	/C-75-80 u	m-1.3-Av	erage, Mon	day, Au	gust 21, 20	17 4:36	:01 PM				
	0.020 0.022 0.025 0.028 0.032 0.036 0.040 0.045 0.050 0.050 0.050 0.050 0.050 0.063 0.063 0.071 0.080 0.089 0.100 0.112	0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0	5126 (JH7) 0.142 0.159 0.178 0.200 0.224 0.252 0.283 0.317 0.356 0.399 0.448 0.502 0.564 0.632 0.710 0.796	0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0	1.002 1.125 1.262 1.416 1.589 1.783 2.000 2.244 2.518 2.825 3.170 3.557 3.991 4.477 5.024 5.637	0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0	7.96 8.93 10.02 11.24 12.61 14.15 15.88 17.82 20.00 22.44 25.17 28.25 31.69 35.56 39.90	Volume III %           2         0.00           2         0.00           4         0.00           7         0.00           9         0.00           9         0.00           7         0.00           9         0.00           9         0.00           0         0.00           0         0.00           9         0.00           9         0.00           9         0.00           9         0.00           9         0.00           9         0.00           9         0.00           9         0.00           9         0.00           9         0.00           9         0.00           9         0.00           9         0.00           1         0.00           5         0.00	5.24 (JH) 50.23 56.368 63.246 70.963 79.621 89.337 100.237 112.468 126.191 141.589 158.866 178.250 200.000 224.404 251.785 282.508	1.11 3.93 7.76 12.51 17.12 18.12 16.30 11.83 6.95 3.13 0.93 0.07 0.00 0.00 0.00 0.00	355.656 399.052 447.744 502.377 632.456 709.627 796.214 893.367 1002.374 1124.683 1261.915 1415.892 1588.656 1782.502 2000.000	0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0
	0.142	0.00	1.002	0.00	7.096	0.00	50.23	B 0.24	355.656	0.00		

Operator notes: Average of 3 measurements from SHSVD WC samples Feb 2017.mea

Malvern Instruments Ltd. Malvern, UK Tel := +[44] (0) 1684-892456 Fax +[44] (0) 1684-892789 Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545 File name: SHSVD WC samples Feb 2017.mea Record Number: 246 9/5/2017 3:40:28 PM

![](_page_91_Picture_0.jpeg)

![](_page_91_Picture_1.jpeg)

Sample N NiCladW	<b>lame:</b> C-<75 um-1.1	-Average	so	P Name:				Measured: Monday, Augus	st 21, 2017 4	4:49:01 PM		
Sample S	Source & type	:	Me D3	asured by: M966				Analysed: Thursday, Augu	ıst 31. 2017	4:52:16 PM		
Sample I	oulk lot ref:		Re Ave	sult Source eraged	:							
Particle I Tungsten	Name: powder		Ac Hyd	cessory Na dro 2000G (A	<b>me:</b> A)			Analysis mode General purpos	el: e		Sensitivity: Normal	n.
2.240	NI.		0.1	sorption.				0.020 to	2000.000	) um	7.89 %	
Dispersa Water	int Name:		<b>Dis</b> 1.3	spersant RI: 330	:			Weighted Res 0.528 %	<b>idual:</b>		Result Emu Off	lation:
<b>Concent</b> 0.0851	ration: %Vol		<b>Sp</b> 0.6	<b>an :</b> 648				Uniformity: 0.2			Result units	5:
<b>Specific</b> 0.0811	Surface Area m²/g	:	<b>Su</b> 74.	rface Weigh .005 ur	n <b>ted Mean I</b> m	D[3,2]:		Vol. Weighted 78.664 ui	<b>Mean D[4,3</b> m	]:		
d(0.1):	55.432	um			d(0.5):	76.373	um			d(0.9):	104.950	um
[					Particl	e Size Distr	ibution				7	
	25	5						^				
	20	)									_	
	8	-										
	e la	)										
	10 10	)										
	5	5						_/			_	
	{	3.01	0.1		1	1	0	100		1000 3	000	
					Part	icle Size (	µm)					
-	-NiCladW	C-<75 um	-1.1-Avera	ge, Monda	ay, Augus	st 21, 201 <sup>.</sup>	7 4:49:	D1 PM				
	Size (µm)	Volume In %	Size (µm) Vo	olume In %	Size (µm)	Volume In %	Size (µr	1) Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
	0.022	0.00	0.142	0.00	1.125	0.00	7.96	0.00 2 0.00	56.368	6.90 11.54	399.052	0.00
	0.025	0.00	0.178	0.00	1.262	0.00	8.93	4 0.00	63.246	15.82	447.744	0.00
	0.032	0.00	0.220	0.00	1.589	0.00	11.24	7 0.00	79.621	17.85	563.677	0.00
	0.036	0.00	0.252	0.00	1.783	0.00	12.61	9 0.00	89.337	10.71	632.456	0.00
	0.040	0.00	0.283	0.00	2.000	0.00	14.15	9 0.00	100.237	8.16	709.627	0.00
	0.050	0.00	0.356	0.00	2.518	0.00	17.82	5 0.00	126.191	3.98	893.367	0.00
	0.056	0.00	0.399	0.00	2.825	0.00	20.00	0.00	141.589	0.23	1002.374	0.00
	0.063	0.00	0.448	0.00	3.170	0.00	22.44	0.00	158.866	0.00	1124.683	0.00
	0.080	0.00	0.564	0.00	3.991	0.00	28.25	0.00	200.000	0.00	1415.892	0.00
	0.089	0.00	0.632	0.00	4.477	0.00	31.69	8 0.02	224.404	0.00	1588.656	0.00
	0.100	0.00	0.710	0.00	5.024	0.00	35.56	6 0.16	251.785 282.508	0.00	1782.502	0.00
	0.126	0.00	0.893	0.00	6.325	0.00	44.77	4 1.06	316.979	0.00	200000	
	0.142	0.00	1.002	0.00	7.096	0.00	50.23	8	355.656	0.00		

Average of 3 measurements from SHSVD WC samples Feb 2017.mea Operator notes:

Malvern Instruments Ltd. Malvern, UK Tel := +[44] (0) 1684-892456 Fax +[44] (0) 1684-892789

Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545 File name: SHSVD WC samples Feb 2017.mea Record Number: 250 9/5/2017 3:40:28 PM

![](_page_92_Picture_0.jpeg)

![](_page_92_Picture_1.jpeg)

Sample N NiCladW0	<b>lame:</b> C-<75 um-1.2	2-Average	s	OP Name:				Measured: Monday, Augus	st 21, 2017 4	4:51:22 PM		
Sample S	Source & typ	e:	N	leasured by: ISM966				Analysed: Thursday Aug	ıst 31 2017	4:53:02 PM		
Sample b	oulk lot ref:		R	esult Source	:			maroady, rage		1.00.02 1 1		
Particle I	Name:		A	ccessory Na	me:			Analysis mod	el: se		Sensitivity:	
Particle F	RI:		A	bsorption:	·)			Size range:			Obscuratio	n:
2.240 Dispersa	nt Name:		0	.1 Dispersant RI:				0.020 to Weighted Res	o 2000.000 idual:	) um	8.24 %	lation:
Water			1	.330				0.527 %	, D		Off	
<b>Concent</b> 0.0889	ration: %Vol		<b>S</b> 0	<b>pan :</b> .649				Uniformity: 0.2			Result units	S:
<b>Specific</b> 0.0812	Surface Area m²/g	1:	<b>S</b> 7	<b>urface Weigh</b> 3.883 ur	<b>ted Mean</b> ท	D[3,2]:		<b>Vol. Weighted</b> 78.538 u	<b>Mean D[4,3</b> m	i]:		
d(0.1):	55.334	um			d(0.5):	76.245	um			d(0.9):	104.785	um
[	2				Partic	le Size Dist:	ribution				7	
	2	5						Δ				
	2	0									_	
	(%											
	) 1 mue (	5										
	N 1	0										
		5										
		8.01	0.1		1	1	0	100		1000 3	000	
					Par	ticle Size	(µm)					_
F	-NiCladW	/C-<75 um	-1.2-Avera	age, Monda	iy, Augu	ist 21, 201	7 4:51:	22 PM				
	Size (µm) 0.020	Volume In %	Size (µm) 1 0.142	olume In %	Size (µm) 1.002	Volume In %	Size (µ	n) Volume In %	Size (µm) 50.238	Volume In %	Size (µm) 355.656	Volume In %
	0.022	0.00	0.159	0.00	1.125	0.00	7.96	2 0.00	56.368 63.246	11.61	399.052 447.744	0.00
	0.028	0.00	0.200	0.00	1.416	0.00	10.02	4 0.00	70.963	15.87 17.85	502.377	0.00
	0.032	0.00	0.224	0.00	1.589	0.00	11.24	0.00	79.621 89.337	16.67	563.677 632.456	0.00
	0.040	0.00	0.283	0.00	2.000	0.00	14.15	0.00 9 0.00	100.237	12.89 8.10	709.627	0.00
	0.045	0.00	0.317	0.00	2.244	0.00	15.88	0.00	112.468	3.93	796.214	0.00
	0.056	0.00	0.399	0.00	2.825	0.00	20.00	0.00	141.589	1.43	1002.374	0.00
	0.063	0.00	0.448	0.00	3.170	0.00	22.44	0.00	158.866	0.22	1124.683	0.00
	0.071	0.00	0.502	0.00	3.557 3.991	0.00	25.17	9 0.00	178.250	0.00	1261.915 1415.892	0.00
	0.089	0.00	0.632	0.00	4.477	0.00	31.69	0.00	224.404	0.00	1588.656	0.00
	0.100	0.00	0.710	0.00	5.024	0.00	35.56	6 0.02	251.785	0.00	1782.502	0.00
	NiCladWC-<75 um-1           Size (µm)         Volume in %           0.022         0.00           0.022         0.00           0.022         0.00           0.022         0.00           0.025         0.00           0.036         0.00           0.046         0.00           0.056         0.00           0.056         0.00           0.056         0.00           0.056         0.00           0.056         0.00           0.056         0.00           0.071         0.00           0.089         0.00           0.112         0.00           0.112         0.00           0.142         0.00			0.00	5.637 6.325	0.00	39.90 44.77	4 1.08	282.508 316.979	0.00	2000.000	
	0.142	0.00	1.002	0.00	7.096	0.00	50.23	8 3.21	355.656	0.00		

Operator notes: Average of 3 measurements from SHSVD WC samples Feb 2017.mea

Malvern Instruments Ltd. Malvern, UK Tel := +[44] (0) 1684-892456 Fax +[44] (0) 1684-892789 Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545 File name: SHSVD WC samples Feb 2017.mea Record Number: 254 9/5/2017 3:40:28 PM

![](_page_93_Picture_0.jpeg)

![](_page_93_Picture_1.jpeg)

Sample I NiCladW	<b>\ame:</b> C-<75 um-1.3∙	-Average	so	P Name:				Measured: Monday, Augus	it 21, 2017 4	:53:20 PM		
Sample S	Source & type	:	Me D3	asured by:				Analysed: Thursday Augu	st 31 2017	4·54·04 PM		
Sample I	bulk lot ref:		Re Ave	sult Source eraged	:			muisuay, Augu	31 31, 2017	4.04.04 T M		
Particle Tungsten Particle 2.240 Dispersa Water	Name: powder RI: ant Name:		Aca Hya 0.1 Dis 1.3	cessory Na dro 2000G (/ sorption: spersant RI: 30	<b>me:</b> \)			Analysis mode General purpos Size range: 0.020 to Weighted Resi 0.524 %	el: e 2000.000 idual:	um	Sensitivity: Normal Obscuration 8.35 % Result Emu Off	n: lation:
<b>Concent</b> 0.0901	ration: %Vol		<b>Sp</b> 0.6	<b>an :</b> 49				Uniformity: 0.2			Result units	:
<b>Specific</b> 0.0812	Surface Area: m²/g	:	<b>Su</b> 73.	<b>rface Weigh</b> 911 ur	n <b>ted Mean I</b> m	D[3,2]:		Vol. Weighted 78.569 ur	<b>Mean D[4,3</b> ) n	<b>]:</b>		
d(0.1):	55.356	um			d(0.5):	76.271	um			d(0.9):	104.835	um
[					Particle	e Size Distr	ibution					7
	25	5									-	
	20	)									-	
	<b>%</b>											
	<u>)</u> 15	5									-	
	ň											
	ō 10	)									-	
	5	5										
	8	8.01	0.1		1	1	0	100		1000 3	000	
					Part	icle Size (	um)					
	-NiCladW	С-<75 um	-1.3-Avera	ne Monda		t 21 201	7 4.53	20 PM				
L	Size (µm)	Volume In %	Size (µm) Vo	lume In %	Size (µm)	Volume In %	Size (µ	m) Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
	0.020	0.00	0.142	0.00	1.002	0.00	7.09	0.00	50.238 56.368	6.95	355.656	0.00
	0.025	0.00	0.178	0.00	1.262	0.00	8.93	0.00	63.246	11.60 15.86	447.744	0.00
	0.028	0.00	0.200	0.00	1.416	0.00	10.02	0.00	70.963	17.85	502.377	0.00
	0.032	0.00	0.252	0.00	1.783	0.00	12.6	0.00	89.337	16.67	632.456	0.00
	0.040	0.00	0.283	0.00	2.000	0.00	14.15	0.00	100.237	12.90	709.627	0.00
	0.045	0.00	0.317	0.00	2.244	0.00	15.88	0.00	112.468	3.95	796.214	0.00
	0.056	0.00	0.399	0.00	2.825	0.00	20.00	0.00	141.589	1.44	1002.374	0.00
	0.063	0.00	0.448	0.00	3.170	0.00	22.44	0.00	158.866	0.22	1124.683	0.00
	0.071	0.00	0.502	0.00	3.557	0.00	25.17	0.00	178.250	0.00	1261.915 1415.892	0.00
	0.089	0.00	0.632	0.00	4.477	0.00	31.69	0.00	224.404	0.00	1588.656	0.00
	0.100	0.00	0.710	0.00	5.024	0.00	35.56	0.02	251.785	0.00	1782.502	0.00
	0.112	0.00	0.796	0.00	5.637 6.325	0.00	39.90	1.07	282.508	0.00	2000.000	
	0.142	0.00	1.002	0.00	7.096	0.00	50.23	3.20	355.656	0.00		

Operator notes: Average of 3 measurements from SHSVD WC samples Feb 2017.mea

Malvern Instruments Ltd. Malvern, UK Tel := +[44] (0) 1684-892456 Fax +[44] (0) 1684-892789 Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545 File name: SHSVD WC samples Feb 2017.mea Record Number: 258 9/5/2017 3:40:28 PM

![](_page_94_Picture_0.jpeg)

![](_page_94_Picture_1.jpeg)

Sample I NiCladW	Name: C->80 um-1	.1-Average	SOP	Name:				Measured: Monday, Augus	st 21, 2017 5	:05:40 PM		
Sample	Source & ty	pe:	Meas D3M9	ured by:				Analysed: Thursday Augu	ist 31 2017 4	4·54·58 PM		
Sample	bulk lot ref	:	<b>Resu</b> Avera	It Source	:							
Particle Tungsten Particle 2.240 Dispersa Water	Name: powder RI: ant Name:		Acce Hydro 0.1 Dispo 1.330	ssory Nar 2000G (A rption: ersant RI:	me: \)			Analysis mode General purpos Size range: 0.020 to Weighted Res 0.540 %	el: e 2000.000 idual:	um	Sensitivity: Normal Obscuratior 8.56 % Result Emu Off	n: lation:
<b>Concent</b> 0.1428	ration: %Vol		<b>Span</b> 0.660	:				Uniformity: 0.207			Result units Volume	::
<b>Specific</b> 0.0531	Surface Are m²/g	ea:	<b>Surfa</b> 112.9	i <b>ce Weigh</b> 80 ur	ted Mean I n	D[3,2]:		Vol. Weighted 120.188 ur	<b>Mean D[4,3]</b> m	:		
d(0.1):	84.689	) um			d(0.5):	116.543	um			d(0.9):	161.639	um
		0.5			Particle	e Size Disti	ibution				<b></b>	
		20						$\wedge$			_	
	lume (%	15										
	Vo											
		5										
		8.01	0.1		1	1	0	100		1000 30	000	
					Part	icle Size (	um)					
	-NiClad	NC->80 un	n-1.1-Average	, Monda	iv, Augus	st 21, 201	7 5:05:	40 PM				
	Size (µm	) Volume In %	Size (µm) Volur	ne In %	Size (µm)	/olume In %	Size (µ	m) Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
	0.020	0.00	0.142 0.159	0.00	1.002 1.125	0.00	7.09	0.00	50.238 56.368	0.03	355.656 399.052	0.00
	0.025	0.00	0.178	0.00	1.262	0.00	8.93	0.00	63.246	1.59	447.744	0.00
	0.028	0.00	0.220	0.00	1.416	0.00	10.02	0.00	70.963	4.63	563.677	0.00
	0.036	0.00	0.252	0.00	1.783	0.00	12.61	0.00	89.337	7.82 13.49	632.456	0.00
	0.040	0.00	0.283	0.00	2.000	0.00	14.15	0.00	100.237	16.68	709.627	0.00
	0.050	0.00	0.356	0.00	2.518	0.00	17.82	0.00	126.191	17.61	893.367	0.00
	0.056	0.00	0.399	0.00	2.825	0.00	20.00	0.00	141.589	10.69	1002.374	0.00
	0.063	0.00	0.448	0.00	3.170	0.00	22.44	0.00	158.866	7.07	1124.683	0.00
	0.080	0.00	0.564	0.00	3.991	0.00	28.25	0.00	200.000	3.11 0.94	1415.892	0.00
	0.089	0.00	0.632	0.00	4.477	0.00	31.69	0.00	224.404	0.13	1588.656	0.00
	0.100	0.00	0.796	0.00	5.637	0.00	39.90	0.00	282.508	0.00	2000.000	0.00
	0.126	0.00	0.893	0.00	6.325	0.00	44.77	4 0.00	316.979	0.00		

Operator notes: Average of 3 measurements from SHSVD WC samples Feb 2017.mea

Malvern Instruments Ltd. Malvern, UK Tel := +[44] (0) 1684-892456 Fax +[44] (0) 1684-892789 Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545 File name: SHSVD WC samples Feb 2017.mea Record Number: 262 9/5/2017 3:40:28 PM

![](_page_95_Picture_0.jpeg)

![](_page_95_Picture_1.jpeg)

Sample Nar NiCladWC-	<b>me:</b> >80 um-1.2	2-Average		SOP Name:					Measured: Monday, Augu	ıst 21, 2017	5:07:42 PM		
Sample So	urce & typ	e:		Measured by D3M966	<b>'</b> :				Analysed: Thursday, Aud	ust 31. 2017	4:56:05 PM		
Sample bu	lk lot ref:			Result Source Averaged	ce:					, , .			
Particle Na Tungsten po	i <b>me:</b> owder			Accessory N Hydro 2000G	lame: (A)				Analysis moo General purpo	<b>lel:</b> se		Sensitivity: Normal	
Particle RI:	:			Absorption:					Size range:	~ 2000 00		Obscuratio	n:
Dispersant	Name:			Dispersant F	RI:				Weighted Re	sidual:	J un	Result Emu	, Ilation:
Water				1.330					0.557	%		Off	
Concentrat 0.1491	tion: %Vol			<b>Span :</b> 0.661					Uniformity: 0.207			Result unit	s:
Specific Su 0.0532	nface Area m²/g	1:		Surface Weig 112.845	<b>ghted Me</b> um	an D[3,2]:			<b>Vol. Weighted</b> 120.051 ເ	<b>i Mean D[4,</b> 3 um	3]:		
d(0.1):	84.564	um			d(0.5	): 116	.406	um			d(0.9):	161.469	um
					Par	ticle Size	Distr	ibution				_	
	2	5											
									$\square$				
	2	0											
		_											
	~ 1 <u>ຍ</u>	5											
	5 1	0											
		5											
										$\mathbf{X}$			
		0.01	0.1		1		1(	C	100		1000 3	000	
					Р	article S	Size (	µm)					
	NiCladW	/C->80 um	-1.2-Ave	rage, Mono	day, Au	gust 21	, 2017	7 5:07:	42 PM				
	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µr	n) Volume	In %	Size (µr	n) Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
	0.020	0.00	0.142	0.00	1.12	:5	0.00	7.96	0.00 2 0.00	56.368	0.04	399.052	0.00
	0.025	0.00	0.178	0.00	1.26	2	0.00	8.93	4 0.00	63.246 70.963	1.61	447.744	0.00
	0.020	0.00	0.220	0.00	1.58	9	0.00	11.24	7 0.00	79.621	4.67	563.677	0.00
	0.036	0.00	0.252	0.00	1.78	3	0.00	12.61	9 0.00	89.337	7.86	632.456	0.00
	0.040	0.00	0.283	0.00	2.00	4	0.00	14.15	9 7 0.00	100.237	16.70	709.627	0.00
	0.050	0.00	0.356	0.00	2.51	8	0.00	17.82	5 0.00	126.191	17.60	893.367	0.00
	0.056	0.00	0.399	0.00	2.82	5	0.00	20.00	0 0.00	141.589	10.64	1002.374	0.00
	0.063	0.00	0.448	0.00	3.17	7	0.00	22.44	9 0.00	178.250	7.03	124.683	0.00
	0.080	0.00	0.564	0.00	3.99	11	0.00	28.25	1 0.00	200.000	3.09	1415.892	0.00
	0.089	0.00	0.632	0.00	4.47	7	0.00	31.69	8 0.00	224.404	0.13	1588.656	0.00
	0.112	0.00	0.796	0.00	5.63	7	0.00	39.90	5 0.00	282.508	0.00	2000.000	0.00
	0.126	0.00	0.893	0.00	6.32	5	0.00	44.77	4 0.00	316.979	0.00		
	0.142		1.002		7.09	6		50.23	8	355.656			

Operator notes: Average of 3 measurements from SHSVD WC samples Feb 2017.mea

Malvern Instruments Ltd. Malvern, UK Tel := +[44] (0) 1684-892456 Fax +[44] (0) 1684-892789 Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545 File name: SHSVD WC samples Feb 2017.mea Record Number: 266 9/5/2017 3:40:28 PM

![](_page_96_Picture_0.jpeg)

![](_page_96_Picture_1.jpeg)

Sample I NiCladW	<b>Vame:</b> C->80 um-1	.3-Average		SOP Name:				Measured: Monday, August 21, 2017 5:09:37 PM					
Sample Source & type:				Measured by: D3M966	1			Analysed: Thursday, August 31, 2017 4:56:50 PM					
Sample	bulk lot ref	1		Result Source Averaged	e:								
Particle Tungsten Particle 2.240 Dispersa Water	Name: powder RI: ant Name:			Accessory N Hydro 2000G Absorption: 0.1 Dispersant R 1.330	ame: (A) I:			Analysis mod General purpo Size range: 0.020 t Weighted Res 0.541 S	<b>iei:</b> se o 2000.000 <b>siduai:</b> %	) um	Sensitivity: Normal Obscuration: 8.88 % Result Emulation: Off		
<b>Concent</b> 0.1483	ration: %Vol			<b>Span :</b> 0.660				Uniformity: 0.207			Result units: Volume		
Specific Surface Area:0.0532m²/g				Surface Weig 112.862	<b>hted Mear</b> um	n D[3,2]:		Vol. Weighted Mean D[4,3]: 120.059 um					
d(0.1):	84.590	) um			d(0.5):	116.425	um			d(0.9):	161.450	um	
]					Parti	cle Size Dist	ribution				_		
	(%	20						$\land$			_		
	ume (9	15											
	Vol	10											
		5						J					
		8.01	0.	1	1		10	100		1000 3	000		
					Pa	rticle Size	(um)						
			n 1 2 Av	orado Mond		1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1		27 DM				-	
l	Size (µm	) Volume In %	Size (µm	) Volume In %	Size (µm)	Volume In %	Size (µ	m) Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	
	0.020	0.00	0.14	2 0.00	1.002	0.00	7.0	0.00	50.238	0.04	355.656	0.00	
	0.022	0.00	0.15	0.00	1.125	0.00	7.9	0.00	63.246	0.31	447.744	0.00	
	0.028	0.00	0.20	0.00	1.416	0.00	10.0	24 0.00	70.963	1.61 4.66	502.377	0.00	
	0.032	0.00	0.22	4 0.00	1.589	0.00	11.2	47 0.00	79.621	7.85	563.677	0.00	
	0.030	0.00	0.25	0.00	2.000	0.00	14.1	0.00	100.237	13.53	709.627	0.00	
	0.045	0.00	0.31	7 0.00	2.244	0.00	15.8	37 0.00	112.468	16.70 17.61	796.214	0.00	
	0.050	0.00	0.35	6 0.00	2.518	0.00	17.8	25 0.00	126.191	15.89	893.367	0.00	
	0.063	0.00	0.39	0.00	3.170	0.00	20.0	0.00	158.866	10.65	1124.683	0.00	
	0.071	0.00	0.50	2 0.00	3.557	0.00	25.1	79 0.00	178.250	7.03	1261.915	0.00	
	0.080	0.00	0.56	4 0.00	3.991	0.00	28.2	0.00	200.000	0.93	1415.892	0.00	
	0.100	0.00	0.03	0.00	5.024	0.00	35.5	0.00	251.785	0.12	1782.502	0.00	
	0.112	0.00	0.79	6 0.00	5.637	0.00	39.9	0.00	282.508	0.00	2000.000	0.00	
	0.126	0.00	0.89	0.00	6.325 7.096	0.00	44.7 50.2	0.00	316.979 355.656	0.00			

Operator notes: Average of 3 measurements from SHSVD WC samples Feb 2017.mea

Malvern Instruments Ltd. Malvern, UK Tel := +[44] (0) 1684-892456 Fax +[44] (0) 1684-892789 Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545 File name: SHSVD WC samples Feb 2017.mea Record Number: 270 9/5/2017 3:40:28 PM

### F.2 Malvern Performance Check Results

As per WTPSP operating procedure OP-WTPSP-003, Rev. 3, the performance of the Malvern Mastersizer 2000 is checked using NIST Standard Reference Material 1003c. Opening, continuation, and closing performance checks for the Malvern Mastersizer 2000 were collected under Malvern data file "LSL-2015.mea" (Records 52, 56, and 84, respectively). These performance checks were exported to PDF (file: "PC Checks.pdf") using the Malvern Mastersizer 2000 Software (Version 5.6) and are included on the pages that follow.

![](_page_98_Picture_0.jpeg)

![](_page_98_Picture_1.jpeg)

Sample Name: 10003C-1.1 - Average				SOP Name:				Measured: Wednesday, June 14, 2017 6:17:16 PM					
Sample Source & type: NIST				Measured by: D3M966				Analysed: Wednesday, June 14, 2017 6:17:17 PM					
Sample	bulk lot ref:			Result Source Averaged	e:								
Particle Glass be	Name: ads (typical)			Accessory Na Hydro 2000G (	ame: A)			Analysis mod Single narrow r	el: node (spher	ical)	Sensitivity: Enhanced		
Particle	RI:			Absorption:				Size range:	2000.000	) um	Obscuration:		
Dispersa Water	ant Name:			Dispersant RI	l:			Weighted Res 0.305 %	idual:		Result Emulation: Off		
<b>Concent</b> 0.0448	t <b>ration:</b> %Vol			<b>Span :</b> 0.661				Uniformity: 0.209			Result units: Volume		
<b>Specific</b> 0.0773	Surface Are m²/g	a:		Surface Weig 31.699 u	<b>hted Mean</b> Im	D[3,2]:		Vol. Weighted 33.758 u	<b>Mean D[4,</b> 3 m	3]:			
d(0.1):	23.411	um			d(0.5):	32.985	um			d(0.9):	45.209	um	
					Partic	cle Size Dis	ribution						
								Λ					
	2	20									-		
	(%	-											
	ue (	5											
	unio,	0									_		
	~	_											
		5											
		8.01	0.1		1		10	100		1000 3	] 000		
					Par	rticle Size	(µm)						
	-10003C	-1.1 - Ave	rage, Weo	dnesday, Ju	ne 14, 2	017 6:17:	16 PM						
	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µ	m) Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	
	0.020	0.00	0.142	0.00	1.002	0.00	7.09	0.00	50.238 56.368	2.95	355.656	0.00	
	0.025	0.00	0.178	0.00	1.262	0.00	8.93	0.00	63.246	0.35	447.744	0.00	
	0.028	0.00	0.200	0.00	1.416	0.00	10.0	0.00	70.963	0.00	502.377	0.00	
	0.032	0.00	0.224	0.00	1.783	0.00	12.6	0.00	89.337	0.00	632.456	0.00	
	0.040	0.00	0.283	0.00	2.000	0.00	14.1	0.00	100.237	0.00	709.627	0.00	
	0.045	0.00	0.317	0.00	2.244	0.00	15.8	0.45	112.468	0.00	796.214	0.00	
	0.050	0.00	0.356	0.00	2.518	0.00	20.00	2.06	141.589	0.00	1002.374	0.00	
	0.063	0.00	0.448	0.00	3.170	0.00	22.4	4.80	158.866	0.00	1124.683	0.00	
	0.071	0.00	0.502	0.00	3.557	0.00	25.1	12.53	178.250	0.00	1261.915	0.00	
	0.080	0.00	0.564	0.00	3.991 4.477	0.00	31.6	15.75	200.000	0.00	1588.656	0.00	
	0.100	0.00	0.710	0.00	5.024	0.00	35.5	6 17.03 15.79	251.785	0.00	1782.502	0.00	
	0.112	0.00	0.796	0.00	5.637	0.00	39.90	12.21	282.508	0.00	2000.000	0.00	
	0.126	0.00	1.002	0.00	6.325 7.096	0.00	44.7 50.2	7.52	316.979	0.00			

Operator notes:

Malvern Instruments Ltd. Malvern, UK Tel := +[44] (0) 1684-892456 Fax +[44] (0) 1684-892789 Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545 File name: Standards LSL2-2015.mea Record Number: 52 9/7/2017 1:33:45 PM

![](_page_99_Picture_0.jpeg)

![](_page_99_Picture_1.jpeg)

Sample Name: SOP Name: 1003c-1.1 - Average								N T	Measured: Tuesday, August 22, 2017 6:32:07 PM					
Sample Source & type: Measured by: D3M966						A T	Analysed: Tuesday, August 22, 2017 6:32:08 DM							
Sample bulk lot ref: Result Source: Averaged							ucousy, Augus	. 22, 2017	5.02.00 T W					
Particle Name: Glass beads (typical) Particle RI: 1.520 Dispersant Name: Water					Accessory Name: Hydro 2000G (A) Absorption: 0 Dispersant RI: 1.330					Analysis model: Single narrow mode (spherical) Size range: 0.020 to 2000.000 um Weighted Residual: 0.339 %			Sensitivity: Enhanced Obscuration: 15.50 % Result Emulation: Off	
<b>Concent</b> 0.0723	t <b>ration:</b> %Vol			:	<b>Span :</b> 0.667				L C	<b>Jniformity:</b> 0.209			Result units	S:
<b>Specific</b> 0.0787	Surface m²/g	Area:			Surface Weigh 31.128 ur	n <b>ted Mean</b>	D[3,2]:		۱ ع	<b>/ol. Weighted M</b> 33.176 um	<b>/lean D[4,3</b>	]:		
d(0.1):	22.9	969	um			d(0.5):	32.369		um			d(0.9):	44.544	um
						Partic	le Size Dis	ribu	ution					
		20							$\int$				-	
	ne (%)	15											_	
	Volun	10							+					
		5											_	
		8.	01	0.1		1		10		100		1000 30	000	
						Pai	ticle Size	(µn	n)					
	-1003	c-1.1	- Averad	e, Tuesc	lav, August	22, 201	7 6:32:07	PM						1
I	Size (	µm) V	olume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	S	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
	0.0	020	0.00	0.142	0.00	1.002	0.00		7.096	0.00	50.238 56.368	2.54	355.656 399.052	0.00
	0.0	025	0.00	0.178	0.00	1.262	0.00		8.934	0.00	63.246	0.27	447.744	0.00
	0.0	028	0.00	0.200	0.00	1.416	0.00		10.024	0.00	70.963	0.00	502.377	0.00
	0.0	032	0.00	0.224	0.00	1.783	0.00		12.619	0.00	89.337	0.00	632.456	0.00
	0.0	040	0.00	0.283	0.00	2.000	0.00		14.159	0.00	100.237	0.00	709.627	0.00
	0.0	045	0.00	0.317	0.00	2.244	0.00		15.887	0.63	112.468	0.00	796.214	0.00
	0.0	056	0.00	0.399	0.00	2.825	0.00		20.000	2.44	141.589	0.00	1002.374	0.00
	0.0	063	0.00	0.448	0.00	3.170	0.00		22.440	9.25	158.866	0.00	1124.683	0.00
	0.0	071 080	0.00	0.502	0.00	3.557	0.00		25.179	13.15	178.250	0.00	1261.915	0.00
	0.0	089	0.00	0.632	0.00	4.477	0.00		31.698	16.06	224.404	0.00	1588.656	0.00
	0.	100	0.00	0.710	0.00	5.024	0.00		35.566	15.21	251.785	0.00	1782.502	0.00
	0.	112 126	0.00	0.796	0.00	5.637 6.325	0.00		39.905 44.774	11.40	282.508	0.00	2000.000	
	0.1	142	0.00	1.002	0.00	7.096	0.00		50.238	6.79	355.656	0.00		

Operator notes:

Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545 File name: Standards LSL2-2015.mea Record Number: 56 9/7/2017 1:33:45 PM

![](_page_100_Picture_0.jpeg)

![](_page_100_Picture_1.jpeg)

Sample Name: 1003C-3.2 - Average					\$	SOP Name:					Measured: Wednesday, September 06, 2017 4:27:55 PM							
Sample Source & type:				<b>I</b> [	Measured by: D3M966						Analysed: Wednesday, September 06, 2017 4:27:56 PM							
Sample	bulk lot	ref:			1	Result Source: Averaged												
Particle Glass be	Name: ads (typic	cal)			ł	Accessory Na Hydro 2000G (	ame: (A)					Analysis model: Single narrow mode (spherical)				Sensitivity: Enhanced		
Particle 1.520	RI:				(	Absorption:						<b>Siz</b> 0.0	<b>e range:</b> 20 to	2000.000	) um	Obscuration: 9.78 %		
Dispersa Water	ant Nam	e:			ļ	Dispersant R	l:					<b>We</b> 0.3	eighted Resid	lual:		Result Emulation: Off		
<b>Concent</b> 0.0441	t <b>ration:</b> %Vo	ol			<b>:</b> (	<b>Span :</b> ).614						<b>Un</b> 0.1	<b>iformity:</b> 94			Result unit	s:	
<b>Specific</b> 0.0788	Surface m²/g	Area:			5	<b>Surface Weig</b> 31.071 ເ	hted Me um	an I	D[3,2]:			<b>Vol</b> 32.	I. Weighted M 848 um	lean D[4,3	i]:			
d(0.1):	23.	410	um				d(0.	i):	32.11	4	um				d(0.9):	43.123	um	
							Pa	ticl	e Size [	Distri	bution							
		25	; <u> </u>									Λ				-		
		20	)															
	(%																	
	ne (	15																
	'olur	10																
	>																	
		5																
		C																
		C	0.01		0.1		1	) o rt	iala Ci	10 	)		100		1000 3	000		
	1002	00.2	2 Avora	ao M	lodn	ocday Son	tomb			ze († 7 4.4	лп) 27.55						_	
I	Size	(µm)	Z - AVELA Volume In %	Size	(µm)	Volume In %	Size (µ	m) \	Volume In	/ 4	Z7.33 Size (µr	r IVI n)   \	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	
	0.	.020	0.00	0	142	0.00	1.0	02	0.	00	7.09	96	0.00	50.238	2.08	355.656	0.00	
	0.	.022	0.00	0	159	0.00	1.1	25 62	0.	00	8.93	34	0.00	63.246	0.00	447.744	0.00	
	0.	.028	0.00	0	200	0.00	1.4	16	0.	00	10.02	24	0.00	70.963	0.00	502.377	0.00	
	0.	.032	0.00	0	224	0.00	1.5	89 83	0.	00	11.24	17	0.00	79.621	0.00	563.677 632.456	0.00	
	0.	.040	0.00	0	283	0.00	2.0	00	0.	00	14.15	59	0.00	100.237	0.00	709.627	0.00	
	0.	.045	0.00	0	317	0.00	2.2	44	0.	00	15.88	37	0.33	112.468	0.00	796.214	0.00	
	0.	.050	0.00	0	356 399	0.00	2.5	25	0.	00	20.00	25 00	1.88	126.191	0.00	893.367	0.00	
	0.	.063	0.00	0	448	0.00	3.1	70	0.	00	22.44	10	4.91 9.31	158.866	0.00	1124.683	0.00	
	0.	.071	0.00	0	502	0.00	3.5	57	0.	00	25.17	79	13.99	178.250	0.00	1261.915	0.00	
	0.	.089	0.00	0	632	0.00	3.9 4.4	77	0.	00	31.69	98	17.50	224.404	0.00	1588.656	0.00	
	0.	100	0.00	0	710	0.00	5.0	24	0. 0.	00	35.56	66	15.86	251.785	0.00	1782.502	0.00	
	0.	.112	0.00	0	796 893	0.00	5.6	37 25	0.	00	39.90	05 74	10.89	282.508	0.00	2000.000		
	0.	.142	0.00	1	.002	0.00	7.0	96	0.	00	50.23	88	5.19	355.656	0.00			

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![](_page_104_Picture_0.jpeg)

![](_page_104_Picture_1.jpeg)

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