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Standard High Solids Vessel Design De-inventory Simulant Qualification

September 2017

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



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September 2017

Test Specification: N/A

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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:

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-16-021¹ and implemented through PNNL Test Plan TP-WTPSP-132 Rev. 1.0.²

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. 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.

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

| Component | Weight Percent | | | | |
|--|----------------|---------|---------|---------|---------|
| Na ₂ S ₂ O ₃ •5H ₂ O | 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 – 23 | 14 – 28 | 16 – 30 | 19 – 33 | 24 – 37 |

⁽a) Temperature range where the 2.8 ± 0.5 cP viscosity tolerance is met.

The WTP also defined high-density plutonium oxide particles simulant to have suspension and settling properties similar to 100 µm plutonium oxide to be mixed with the DNCF. Particles with densities ranging from 8.8 at 150 µm to 11.4 g/mLat 100 µm would be sufficient to meet this specification (24590-WTP-ES-ENG-16-021¹). 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 µm. Tungsten carbide particles (particle size range of 90 to 105 µmand density of 11.2 g/mL)³ 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.

¹ 24590-WTP-ES-ENG-16-021, Rev. 1. *De-inventory Testing for the Standard High Solids Vessel*. 2017. Bechtel National, Inc., Richland, Washington.

² 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.

³ 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 correspondling 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-OA Program Support of High Solids Vessel Testing*¹ and two PNNL project plans:

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¹ Minette, MJ. 2016. Pacific Northwest National Laboratory.

- 1. PP-WTPSP-142, Rev. 0, *Testing Simulants Supporting the Single High Solids Vessel Design* (*SHSVD*),² 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)*, ³ 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.*⁴

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.

Table 1.1. Modifications to BNI Guideline Testing^(a)

| 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. |

² Fiskum SK. 2016. Pacific Northwest National Laboratory.

³ Fiskum SK. 2017. Pacific Northwest National Laboratory.

⁴ 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. | 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. |
| Testing is to be conducted twice on each sample and at least duplicate samples are to be tested. | 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. |

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 $25 \,^{\circ}\text{C}$ (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 Na₂S₂O₃•5H₂O, Richland City water, and glycerin. Samples of Na₂S₂O₃•5H₂O 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 Na₂S₂O₃•5H₂O was distributed by Sino Chemical Company Ltd. (Zhengzhou, Henan, China), Lot 20161226; see Appendix C for the available product information. This Na₂S₂O₃•5H₂O 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 Na₂S₂O₃•5H₂O, Lot 20161226 with Centimeter Scale

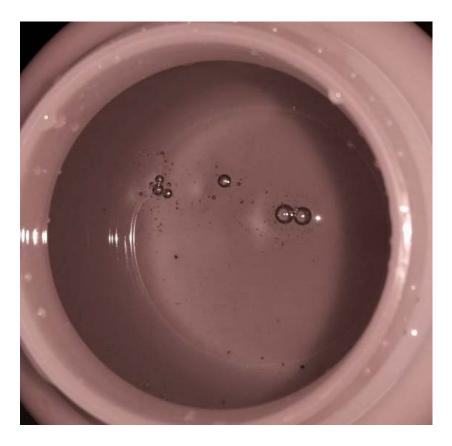


Figure 2.2. Silver Fern Glycerin with Black Specks

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 PuO₂ 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

 ρ = 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

 ρ_g = density of glycerin, 1.2611 g/mL

 ρ_{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:

$$\rho_{ss} = 0.9929 + 0.9759 f_{salt in ss}$$
 (2.2)

where $f_{\text{salt in ss}}$ 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.

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

| Na ₂ S ₂ O ₃ -5H ₂ O, wt% | Glycerin, wt% | Richland City Water, wt% | Density, g/mL | Viscosity at 25 °C, cP |
|--|------------------|-----------------------------|----------------------|---------------------------|
| 28.950 | 8.206 | 62.844 | 1.181 ^(a) | 2.14 |
| 26.709 | 13.307 | 60.042 | 1.180 ^(a) | 2.40 |
| 25.510 | 16.004 | 58.489 | 1.181 ^(a) | 2.60 |
| 23.331 | 21.024 | 55.699 | 1.184 ^(a) | 3.05 |
| 21.346 | 25.533 | 53.139 | 1.181 ^(a) | 3.40 |
| 19.176 | 30.521 | 50.371 | 1.182 ^(a) | 4.00 |
| 22.105 | 25.500 | 52.404 | 1.188 ^(b) | 3.57 |
| 20.596 | 25.507 | 53.917 | 1.176 ^(c) | 3.32 |

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.

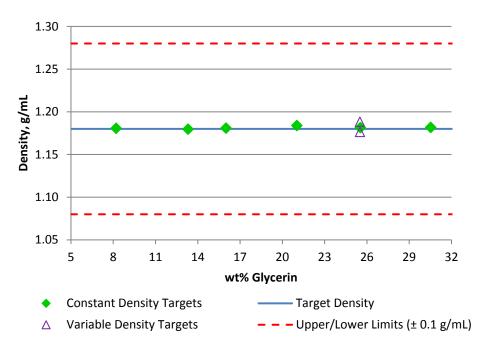


Figure 2.3. Parametric Test Solution Densities at 25 °C as a Function of wt% Glycerin

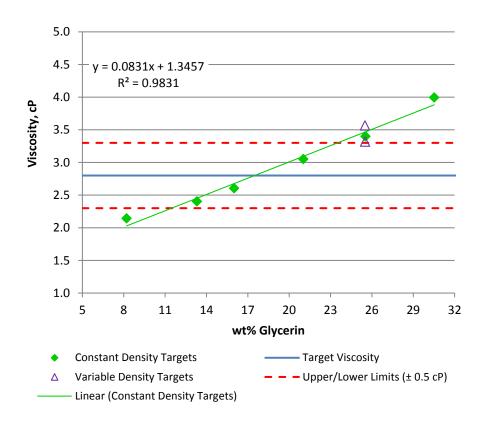


Figure 2.4. Parametric Test Solution Viscosities at 25 °C as a Function of wt% Glycerin

All parametric test solutions easily met the density target of 1.18 ± 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).

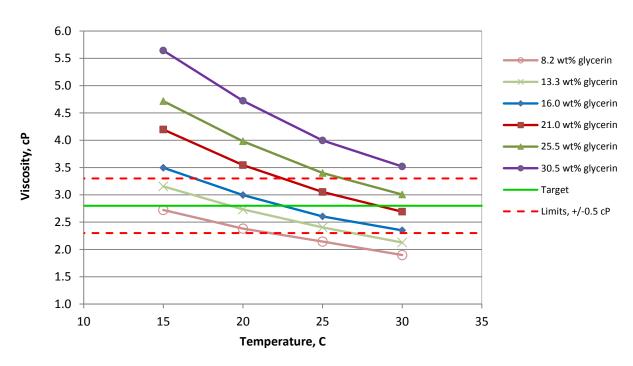


Figure 2.5. Parametric Test Solution Viscosities as a Function of Temperature

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 (± 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% Na₂S₂O₃•5H₂O 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% Na₂S₂O₃•5H₂O 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.

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

| | | T | 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 | $T = 4.5589 \times cP^2 - 38.665 \times cP + 91.61$ | 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 | $T = 2.8733 \times cP^2 - 29.687 \times cP + 89.006$ | 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 | $T = 1.5338 \times cP^2 - 21.017 \times cP + 84.818$ | 38.0 | 44.6 | 32.2 | |

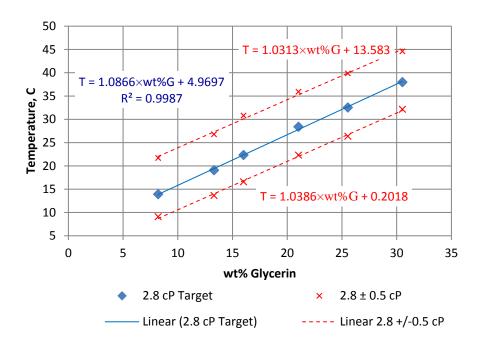


Figure 2.6. Process Temperature as a Function of Glycerin Concentration at 2.8 ± 0.5 cP (where T is Temperature in °C and %G is wt% Glycerin)

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 PuO₂. 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).



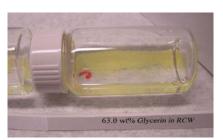
Figure 2.7. NiCladWC in DNCF (left), Water Blank (center), NiCladWC in Water after 1 Week Fluid Contact (right)

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 Na₂CO₃, were contacted with bismuth oxide to test any reactivity issues. A 20 wt% Na₂CO₃ 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% Na₂S₂O₃-5H₂O 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 $105 \mu \text{m}$ (CCN 285593^1), 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 μ m 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 μ m, for the NiCladWC powder was approximately 17 wt%. The PSD of the sieve cut, 75 to 80 μ m, 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

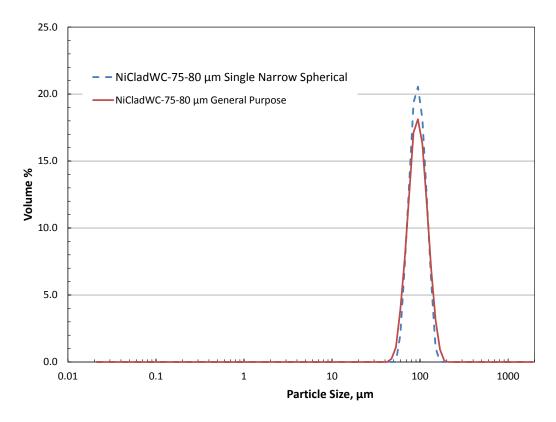


Figure 3.1. Particle Size Distribution of NiCladWC Powder, Lot# XLS7892, Sieve Cut to 75 to 80 μm

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 $80~\mu m$ sieve cut NiCladWc powder is shown in Figure 3.2 below.

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¹ CCN 285593. Meeting Minutes: "IT Mixing Workshop 2 Mar 2017." March 2, 2016. Bechtel National, Inc., Richland, WA.

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

| Sieve Cut, μm | Density, g/cm ³ | d(10) GP/SN, ^(a) μm | d(50) GP/SN, ^(a) μm | d(90) GP/SN, ^(a) μm |
|------------------|----------------------------|--------------------------------------|--------------------------------------|--------------------------------------|
| Bulk Material | 10.680 | NM ^(b) | NM ^(b) | NM ^(b) |
| > 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 |

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

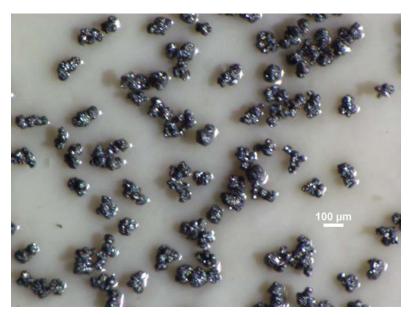


Figure 3.2. Optical Microscopy Images of NiCladWC Powder, Lot# XLS7892, Sieve Cut to 75 to 80 μm (Image is FIO)

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 $8,800-11,460~kg/m^3$ (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, $10,808~kg/m^3$, 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. However, given the relatively tight distribution of the NiCladWC material, and the fact that 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.

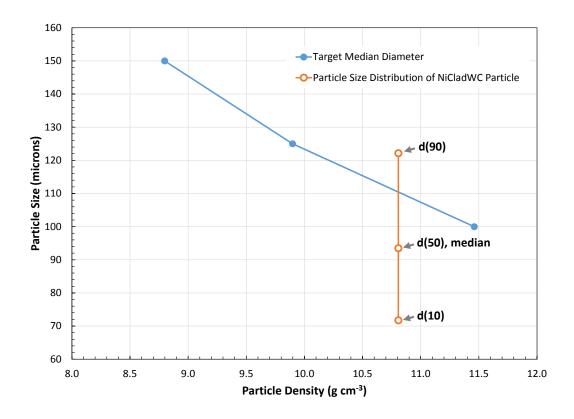


Figure 3.3. d(10), d(50) (Median) and the d(90) Particle Size distribution of NiCladWC Powder, Lot# XLS7892, Sieve Cut to 75 to 80 μm at a Density of 10.808 g/cm³ and the Target Median Particle Size.

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 μ m sieve cut. The <75 μ m cut was used for this evaluation because the 75 to 80 μ 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 μ 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 μm 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 (Na₂S₂O₃•5H₂O), 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% Na₂S₂O₃•5H₂O. All simulant compositions are stable with respect to precipitation at 10 °C (at least 2 days) and ~20 °C (at least 7 days).

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

| Component | Weight Percent | | | | |
|--|----------------|-------------|-------------|-------------|-------------|
| Na ₂ S ₂ O ₃ •5H ₂ O | 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 ± 0.5 cP viscosity tolerance is met.

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 100µm 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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10 CFR 830, Subpart A. 2011. "Quality Assurance Requirements." Code of Federal Regulations, U.S. Department of Energy, Washington, D.C.

10 CFR 830, Subpart B. 2011. "Safety Basis Requirements." Code of Federal Regulations, U.S. Department of Energy, Washington, D.C.

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Gauglitz PA, DN Tran, and WC Buchmiller. 2012. *Simulant Development for Hanford Double-Shell Tank Mixing and Waste Feed Delivery Testing*. PNNL-21791 Rev. 0, Pacific Northwest National Laboratory, Richland, WA.

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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 μ m; subsequent sieve cuts were made by hand, notably the 75 to 80 μ 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 μP wet dispersion accessory. Malvern lists the Mastersizer particle size measurement range as nominally 0.02 to 2000 μm . 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 μP dispersion unit, the measurement range is 0.01 to 500 μm . 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[©] 1998-2009).

The Hydro μP 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.¹

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).

B.1

¹ 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 Na₂S₂O₃•5H₂O is recommended to be American Chemical Society (ACS) reagent grade to ensure that the required physical properties are obtained. ACS reagent grade Na₂S₂O₃•5H₂O is commercially available at reasonable cost. Alternatively, anhydrous Na₂S₂O₃ could be used with appropriate salt and solvent mass adjustments to account for the waters of hydration in Na₂S₂O₃•5H₂O. 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, CH₂OHCHOHCH₂OH) 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 Na₂S₂O₃•5H₂O 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 $Na_2S_2O_3$ •5 H_2O dissolution rate. For small-scale 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% Na₂S₂O₃•5H₂O will accommodate most of the temperature range of interest while still meeting the viscosity and density targets.

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

| | | | Test Temperature | | |
|--|---------------|-------|------------------|-------|-------|
| | 15 °C | 20 °C | 22.4 °C | 25 °C | 30 °C |
| Component | Mass Added, g | | | | |
| Na ₂ S ₂ O ₃ •5H ₂ O | 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 |

Intermediate test temperature compositions may be dialed in based on the function shown in Figure B.1. The Na₂S₂O₃•5H₂O concentration can be determined according to the function in Equation B.1, where %G is wt% glycerin and %S is wt% Na₂S₂O₃•5H₂O (applicable in the range of 8 to 30 wt% glycerin).

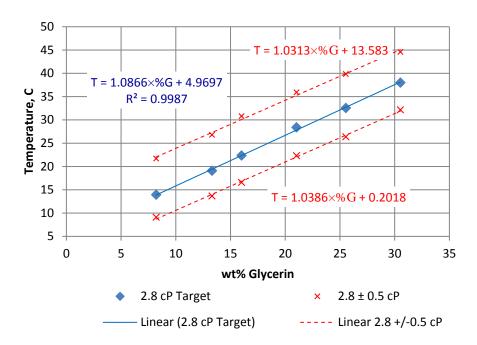


Figure B.1. 2.8 cP Viscosity Temperature Dependence as a Function of wt% Glycerin

$$-0.4388 \times \%G + 32.538 = \%S$$
 (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 Na₂S₂O₃•5H₂O and glycerin. However, Na₂S₂O₃•5H₂O 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 Na₂S₂O₃•5H₂O 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 Na₂S₂O₃•5H₂O 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. ¹ 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 ± 0.1 g/mL and 2.8 ± 0.5 cP at the test temperature.

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¹ Product Safety Assessment: Glycerin, September 2014. Dow Chemical Company http://msdssearch.dow.com/PublishedLiteratureDOWCOM/dh_091a/0901b8038091a41a.pdf?filepath=productsafety/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

ZHENGZHOU SINO CHEMICAL CO.,LTD.
NO.8 QINGONG ROAD,ZHENGZHOU, CHINA
TEL:0086 371 63368978 FAX: 0086 371 63369578

CERTIFICATE OF ANALYSIS

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

ISSUED BY

ZHENGZHOU SINO CHEMICAL CO.,LTD.



CERTIFICATE OF ANALYSIS

Code 90425

SODIUM THIOSULFATE, PENTAHYDRATE, ACS Reagent, crystal, $Na_2S_2O_3.5H_2O$

Lot 0275037/1.1

TEST REQUIREMENTS FOUND

Assay 99.5 - 101.0% 100.0%

Na₂S₂O₃.5H₂O

Sulfide (S)

pH of a 5% solution at a 25 C 6.0 - 8.4 7.1

MAXIMUM ALLOWABLE

Passes Test

 $\begin{tabular}{ll} Insoluble matter & 0.005\% & <0.005\% \\ Nitrogen compounds & 0.002\% & <0.002\% \\ Sulfate and Sulfite (as SO_4) & 0.1\% & 0.1\% \\ \end{tabular}$

(limit about 1 ppm)

Passes Test

According to ACS, Reagent Chemicals, Tenth Edition, 2006

All values are maximum and may represent detection limits.

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CERTIFICATE OF ANALYSIS

Code 90785

GLYCEROL, 99.5% pure, ACS Reagent, liquid, (Glycerin, 1, 2,3-Propantriol), CH $_2$ OHCHOHCH $_2$ OH

Lot 28717/0.0

| TEST | REQUIREMENTS | FOUND |
|--|-------------------|-------------|
| Assay, C ₃ H ₅ (OH) ₃ by volume | > 99.5% | 99.8% |
| | MAXIMUM ALLOWABLE | |
| Color (APHA) | 10 | 5 |
| Residue after ignition | 0.005% | 0.001% |
| Neutrality | Passes test | Passes test |
| Chlorinated compounds (as CI) | 0.003% | 0.002% |
| Sulfate (SO ₄) | 0.001% | < 0.001% |
| Acrolein and glucose | Passes test | Passes test |
| Fatty acid esters | 0.05% | < 0.05% |
| (as butyric acid) | | |
| Substances darkened by | Passes test | Passes test |
| sulfuric acid | | |
| Heavy metals (as Pb) | 2 ppm | <1 ppm |
| Water (H ₂ O) | 0.5% | < 0.5% |

Conforms to ACS, Reagent Chemicals Tenth Edition, 2006

All values are maximum and may represent detection limits.

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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



Emergency information:

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 **Glycerol** Product name:

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 CHEMTREC 703-527-3887

2 Hazards identification

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) Hazard designation:

GHS label elements: None

Signal word:

Hazard/precautionary statements: Avoid consumption

Information pertaining to particular dangers

for man and environment: None HMIS ratings (scale 0.4): Health: 1 Flammability: 0

Physical hazard: 0

Chemical name: Glycerol 56-81-5 Designation: (CAS#):

Formula: HOCH₂CCH(OH)CH₂OH

Synonyms: Glycerin, trihydroxypropane, 1, 2, 3-propanetriol

Identification number(s): 200-289-5 EC Number:

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

5 Fire-fighting measures

Suitable extinguishing agents:

Use extinguishing media most appropriate to surrounding fire conditions Special hazards caused by the material, its

products of combustion or resulting gases: In case of fire, the following can be released: Carbon monoxide

Carbon dioxide

Protective equipment: Wear self-contained breathing apparatus

Wear 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

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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 13 for information on disposal

7 Handling and storage

Information for safe handling: Keep containers tightly sealed

Store in cool, dry place in tightly closed containers

Ensure good ventilation/exhaustion at the workplace Information about protection against

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 storage facility:

Do not store together with oxidizing agents

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: Properly operating chemical furne hood designed for hazardous chemicals and having an average face

velocity of at least 100 feet per minute

Components with critical values that require

monitoring at the workplace:

mg/m3

ACGIH TLV 10; (nuisance particulate)

USA PEL 5 (respirable fraction); 15 (total dust)

Additional information: No data

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

Respiratory protection: Use suitable respirator when high concentrations are present

Glycerol

Use only NIOSH/MESA or CEN approved dust mask type N95 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 sevewash and safety shower

9 Physical and chemical properties

General Information:

Viscous liquid Physical state: Clear to pale yellow Color: Smell: Odorless Molecular Weight (Calculated): 92.09 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: Danger of explosion: See section 7 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

 Solubility in/Miscibility 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 7

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Hazardous decomposition products: Carbon monoxide

Carbon dioxide

11 Toxicological information

Acute toxicity:

LD/LC50 values that are relevant for

classification: Oral-rat LD50: 12,600 mg/kg

Skin-rabbit LD50: > 10,000 mg/kg Primary irritant effect:

on the skin: Irritant for skin and mucous membranes

on the eye: Irritant effect

Sensitization No sensitizing effect known

To the best of our knowledge the acute and chronic toxicity of this substance is not fully known. Additional toxicological information: To the best of our knowledge the acute and chronic toxicity of the substance is not fully known Carcinogenicity:

General notes: Generally not hazardous 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

Disposal must be made according to official regulations Recommendation:

14 Transport information

Land transport DOT

Chemicals Non-Hazardous Proper shipping name:

Technical name: 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: Glycerol

DOT Hazard Class: Subsidiary risk: UN Identification number:

Label(s):

Packing group: Reportable quantity (RQ):

Warning label(s):

North American Emergency Response

Guidebook No.:

Notes:

10

Chemicals Non-Hazardous Proper shipping name: Technical name: Glycerol

DOT Hazard Class: Subsidiary risk: UN Identification number:

UPS Ground / FedEx Ground

Label(s): Packing group:

Reportable quantity (RQ):

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Warning label(s):

10

North American Emergency Response

Guidebook No.:

Notes:

UPS Air

Proper shipping name:

Chemicals Non-Hazardous Glycerol

Technical name: DOT Hazard Class: Subsidiary risk:

UN Identification number:

Label(s):

Packing group: Reportable quantity (RQ):

Waming label(s):

10

North American Emergency Response

Guidebook No.: Notes:

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 qualified 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 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.



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

Sodium thiosulfate, pentahydrate Product name:

Product code:

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 CHEMTREC Emergency information: 800-424-9300

None

Hazard designation:

Information pertaining to particular dangers

for man and environment: Not applicable

Reaction with acids produces toxic sulfur dioxide gas Hazards not otherwise classified HMIS ratings (scale 0.4):

Flammability: 0 Physical hazard: 0

3 Composition/Information on ingredients

Chemical name: Sodium thiosulfate, pentahydrate Designation: (CAS#): 10102-17-7

EC Number: 231-867-5 Formula: Na₂S₂O₃.5H₂O

Synonyms Ametox, Antichlor, Hypo, sodium hyposulfite Not applicable

Ingredients of known acute toxicity:

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

Rinse opened eye for at least 15 minutes under running water. Assure adequate flushing by separating After eye contact:

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 Fire-fighting measures
Use extinguishing media most suitable to surrounding fire conditions Suitable extinguishing agents:

Special hazards caused by the material, its

Oxides of sodium and sulfur (SOx), sodium dioxide Wear self-contained breathing apparatus products of combustion or resulting gases: Special fire fighting procedures:

Wear fully protective fire fighting equipment/clothing in fire situations

Unusual fire and explosion hazard:

6 Accidental release measures

Avoid dust formation. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Person-related safety precautions: Measures for environmental protection: Do not allow material to be released to the environment without proper governmental permits

Measures for cleaning/collecting: Additional information: Sweep up and shove. Keep in suitable, closed containers for proper disposal. 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

Information for safe handling: Keep containers tightly sealed

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

Keep container tightly closed in a dry and well-ventilated place. Do not store near acids. Air and moisture

Incompatibility (avoid contact with): 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.

Further information about storage conditions:

 Exposure controls/personal protection
 Properly operating chemical fume hood designed for hazardous chemicals and having an average face. Ventilation requirements:

velocity of at least 100 feet per minute

Components with critical values that require

monitoring at the workplace: 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

Avoid contact with the eyes and skin

Personal protective equipment:

Respiratory protection:

Precautionary labeling:

Use suitable respirator when high concentrations are present

(Use only NIOSH or CEN approved Equipment) Hand protection:

Use only NIOSH/MESA or CEN approved dust mask type N95 or TYPE P1 (EN 143) 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

Wash thoroughly after handling

Do not get in eyes, on skin or on clothing Do not breathe dust, vapor, mist, gas Store in tightly closed containers Store in a cool, dry place

9 Physical and chemical properties

General Information: 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/freezing point/range: 48 C Boiling point/range: Not determined Sublimation temperature/start: Not determined Decomposition temperature: 100 C (-H₂O)

Flammability (solid, gas): Flash point: Non-flammable Autoignition temperature: Not determined Danger of explosion: Not determined

Flammable limits: Not determined Lower: Upper: Evaporation rate: Not determined Vapor pressure (mm Hg): Not determined Vapor density: Not determined Specific gravity:

Bulk density: Not determined Solubility in/Miscibility with water: 700 g/L @ 20 C Partition coefficient n-octanol/water: Not determined Not determined Viscosity: Other information: No additional information

10 Stability and reactivity

Page 2 of 4

Reactivity: Not determined

Chemical stability: Stable under recommended storage conditions. Possibility of hazardous reactions: Explosion hazard with sodium nitrite and metal nitrites Conditions to be avoided: Exposure to air and moisture may affect product quality See section 7 for information on proper handling and storage

Strong oxidizers or acids. Contact with oxidizers causes exothermic reactions. Contact with acids releases Materials to be avoided:

Oxides of sodium and sulfur (SOx)

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: (thermal and other)

11 Toxicological information

Acute toxicity:

LD/LC50 values that are relevant for

classification: intravenous-rat LD_{so}: > 2,500 mg/kg Primary irritant or corrosive effect:

on the skin: None Mild on the eye:

Sensitization: Prolonged or repeated exposure may cause allergic reactions in certain sensitive individuals

Signs and symptoms of exposure: Ingestion causes cyanosis in humans. Large oral doses have a cathartic effect.

To the best of our knowledge, the chemical, physical and toxicological properties have not been

thoroughly investigated.

Carcinogenicity: No classification data on carcinogenic properties of this material is available from the EPA, IARC, NTP,

OSHA or ACGIH RTECS: Not available

Additional information:

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: Biodegradability: Bioaccumulative potential: Not determined Bioaccumulation: Not determined Mobility in soil: 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:

Not determined

Disposal must be made according to official regulations

10

Land transport DOT

Other adverse effects:

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):

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):

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):

North American Emergency Response

Guldebook No.: Notes:

UPS Air

Proper shipping name: Chemicals Non-Hazardous Sodium thiosulfate, pentahydrate Technical name:

DOT Hazard Class: Subsidiary risk: UN Identification number:

Label(s): Packing group:

Reportable quantity (RQ):

Warning label(s): North American Emergency Response

SARA Section 311 / 312 hazards:

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.

No SARA hazards

10

10

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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

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

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, Na₂S₂O₃•5H₂O, 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 Na₂S₂O₃•5H₂O, Richland City water, and glycerin. The Na₂S₂O₃•5H₂O was obtained from Noah Technologies Corporation (San Antonio, TX) as American Chemical Society (ACS) reagent grade, crystal Na₂S₂O₃•5H₂O salt, Catalog Number 90425, Lot 0275037/1.1, which was assayed by the vendor to be 100% Na₂S₂O₃•5H₂O. 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 ± 0.1 g/mL for density and ± 1 cP for viscosity.

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

| Na ₂ S ₂ O ₃ -5H ₂ O, wt% | 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 ^(a) | 26.953 ^(a) | $29.850^{(a)}$ | 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 ^(b) | 11.07 |
| 43.161 | 27.004 | 29.841 | 1.327 ^(b) | 10.98 |

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

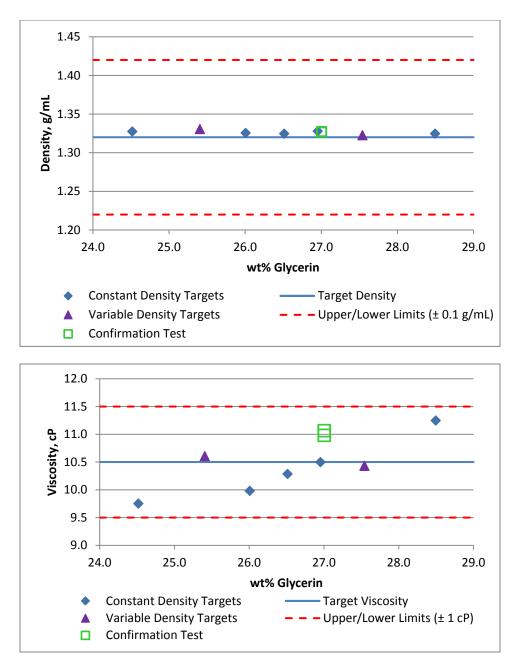


Figure E.1. Parametric and Confirmation Test Solutions Density and Viscosity Results

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 $10\,^{\circ}\text{C}$ for 2 days and stored at room temperature (nominally $20\,^{\circ}\text{C}$) 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 °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 °C.

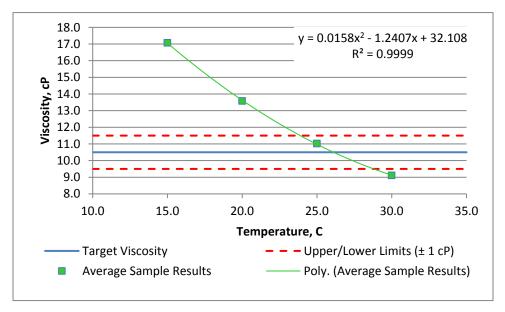


Figure E.2. Simulant Viscosity as a Function of Temperature

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 ₂ S ₂ O ₃ - 5H ₂ 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

Table E.3. Diluted Solutions

| Identification | % Dilution | Na ₂ S ₂ O ₃ - 5H ₂ 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 |

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.

Table E.4. Measured Viscosities at Four Temperatures

| Na ₂ S ₂ O ₃ | | Glycerin | | Viscosity, cP at Temperature, °C | | | | | |
|---|------------------------|----------|------------|----------------------------------|------|------|------|--|--|
| Identification | 5H ₂ O, 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 | | |

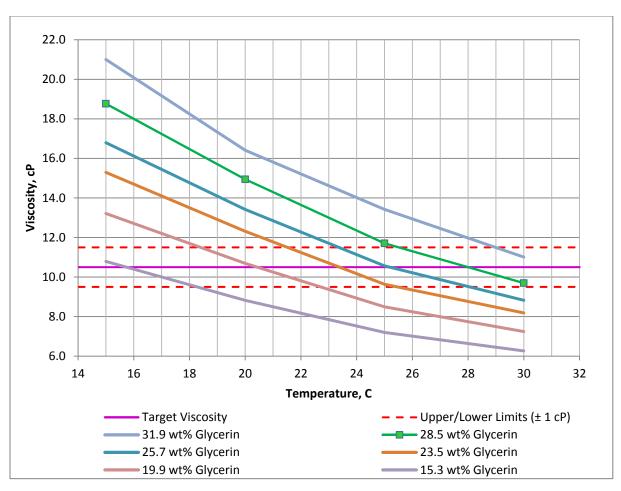


Figure E.3. Viscosity as a Function of Temperature at Various Glycerin Compositions

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 (± 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 Lower Viscosity Targets at Temperature

| | | Ter | | |
|----------------|--|---------|--------|---------|
| Identification | Polynomial Curve Fit | 10.5 cP | 9.5 cP | 11.5 cP |
| 158-GS | $T = 0.076 \text{ cP}^2 - 3.9358 \text{ cP} + 64.118$ | 31.2 | 33.6 | 28.9 |
| 158-1 | $T = 0.0787 \text{ cP}^2 - 3.8615 \text{ cP} + 59.837$ | 28.0 | 30.3 | 25.8 |
| 158-2 | $T = 0.1044 \text{ cP}^2 - 4.5181 \text{ cP} + 61.527$ | 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 | $T = 0.19 \text{ cP}^2 -6.3358 \text{ cP} + 65.65$ | 20.1 | 22.6 | 17.9 |
| 158-5 | T= 0.36*cP^2 -9.3767*cP+74.535 | 15.8 | 17.9 | 14.3 |

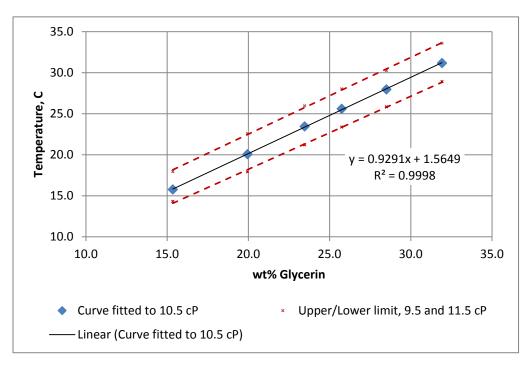


Figure E.4. Process Temperature as a Function of Glycerin Concentration at 10.5 ± 1 cP

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 μm fraction |

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

| 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 |

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.





Sample Name:

NiCladWC-75-80 um-SN-1.1 - Average

Sample Source & type:

Sample bulk lot ref:

SOP Name:

Measured by:

D3M966

Monday, August 21, 2017 4:32:11 PM

Analysed:

Monday, August 21, 2017 4:32:12 PM

to 2000.000

Result Source: Averaged

Particle Name: Tungsten powder

Particle RI: 2.240

Concentration:

Dispersant Name:

Water

0.1277

d(0.1):

Accessory Name: Hydro 2000G (A) Absorption: 0.1

Dispersant RI:

1.330 Span:

0.539

Analysis model:

Single narrow mode (spherical)

Size range:

0.020

95.455

Weighted Residual:

0.624

Sensitivity:

Obscuration:

Result Emulation:

Enhanced

9.44

Uniformity: Result units:

Volume

Specific Surface Area: 0.0655

m²/g

%Vol

71.721

um

Surface Weighted Mean D[3,2]: 91.615 um

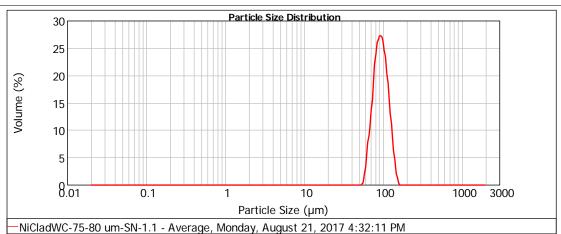
d(0.5):

93.458

Vol. Weighted Mean D[4,3]:

d(0.9): 122.079

um



| Size (µm) V 0.020 0.022 0.025 0.028 0.032 0.036 0.040 0.045 0.056 0.056 0.063 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | 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 | Volume in % 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0 | 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 | Volume In % 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0 | Size (µm) 7.096 7.962 8.934 10.024 11.247 12.619 14.159 15.887 17.825 20.000 22.440 25.179 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | 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 | Volume In % 0.00 2.01 7.20 13.30 19.39 20.57 18.15 12.24 6.07 1.07 0.00 | Size (µm) 355.656 399.052 447.744 502.377 632.456 709.627 796.214 893.367 1002.374 1124.683 1261.915 | Volume In % 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0 |
|--|--|---|---|---|---|--|---|---|--|--|---|
| 0.045 | 0.00 | 0.317 | 0.00 | 2.244 | 0.00 | 15.887 | 0.00 | 112.468 | 18.15 | 796.214 | 0.00 |
| 0.050 | 0.00 | 0.356 | 0.00 | 2.518 | 0.00 | 17.825 | 0.00 | 126.191 | 12.24 | 893.367 | 0.00 |
| 0.056 | 0.00 | 0.399 | 0.00 | 2.825 | 0.00 | 20.000 | 0.00 | 141.589 | 6.07 | 1002.374 | 0.00 |
| 0.063 | 0.00 | 0.448 | 0.00 | 3.170 | 0.00 | 22.440 | 0.00 | 158.866 | 1.07 | 1124.683 | 0.00 |

Operator notes:

XLS7892 sieved, 75-80 um fraction

Malvern Instruments Ltd. 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: 186 9/5/2017 3:40:27 PM





Sample Name:

NiCladWC-75-80 um-SN-1.2 - Average

Sample Source & type:

Sample bulk lot ref:

SOP Name:

Measured by: D3M966

Result Source:

Averaged

Monday, August 21, 2017 4:34:06 PM

Analysed:

Monday, August 21, 2017 4:34:07 PM

Particle Name: Tungsten powder Particle RI:

2.240 **Dispersant Name:**

Water

Concentration: 0.1363 %Vol

d(0.1):

Specific Surface Area: 0.0655 m²/g

71.693

um

Accessory Name: Hydro 2000G (A) Absorption:

0.1 Dispersant RI:

1.330 Span:

0.538

Surface Weighted Mean D[3,2]: 91.561 um

d(0.5):

Analysis model:

Single narrow mode (spherical)

Size range:

to 2000.000 0.020 Weighted Residual:

0.647

Uniformity:

Vol. Weighted Mean D[4,3]:

95.394 um

d(0.9): 121.978

Volume

Sensitivity:

Obscuration:

Result units:

Result Emulation:

um

Enhanced

10.06

Particle Size Distribution 30 25 20 Volume (%) 15 10 5 8.01 0.1 100 1000 3000 Particle Size (µm) -NiCladWC-75-80 um-SN-1.2 - Average, Monday, August 21, 2017 4:34:06 PM

93.394

| _ | W Claur | 70 70 00 0 | 41.1 | . 0.4 1.2 | rttorago | iviorida | ,, riagast |
|---|-----------|-------------|------|-----------|-------------|-----------|-------------|
| | Size (µm) | Volume In % | | Size (µm) | Volume In % | Size (µm) | Volume In % |
| | 0.020 | 0.00 | | 0.142 | 0.00 | 1.002 | 0.00 |
| | 0.022 | 0.00 | | 0.159 | 0.00 | 1.125 | 0.00 |
| | 0.025 | 0.00 | | 0.178 | 0.00 | 1.262 | 0.00 |
| | 0.028 | | | 0.200 | | 1.416 | |
| | 0.032 | 0.00 | | 0.224 | 0.00 | 1.589 | 0.00 |
| | 0.036 | 0.00 | | 0.252 | 0.00 | 1.783 | 0.00 |
| | 0.040 | 0.00 | | 0.283 | 0.00 | 2.000 | 0.00 |
| | 0.045 | 0.00 | | 0.317 | 0.00 | 2,244 | 0.00 |
| | 0.050 | 0.00 | | 0.356 | 0.00 | 2,518 | 0.00 |
| | 0.056 | 0.00 | | 0.399 | 0.00 | 2.825 | 0.00 |
| | 0.063 | 0.00 | | 0.448 | 0.00 | 3,170 | 0.00 |
| | 0.071 | 0.00 | | 0.502 | 0.00 | 3.557 | 0.00 |
| | 0.071 | 0.00 | | 0.564 | 0.00 | 3.991 | 0.00 |
| | 0.089 | 0.00 | | 0.632 | 0.00 | 4.477 | 0.00 |
| | 0.009 | 0.00 | | 0.632 | 0.00 | 5.024 | 0.00 |
| | | 0.00 | | 0.710 | 0.00 | | 0.00 |
| | 0.112 | 0.00 | | | 0.00 | 5.637 | 0.00 |
| | 0.126 | 0.00 | | 0.893 | 0.00 | 6.325 | 0.00 |
| | 0.142 | | | 1.002 | | 7.096 | |

| 11, 2017 4:34:00 PW | | | | | | | | | | |
|---------------------|-----------|-------------|---|-----------|-------------|--|--|--|--|--|
| | Size (µm) | Volume In % | 1 | Size (µm) | Volume In % | | | | | |
| | 7.096 | 0.00 | | 50.238 | 0.00 | | | | | |
| | 7.962 | 0.00 | | 56.368 | 2.02 | | | | | |
| | 8.934 | 0.00 | | 63.246 | | | | | | |
| | 10.024 | | | 70.963 | 7.22 | | | | | |
| | 11.247 | 0.00 | | 79.621 | 13.34 | | | | | |
| | 12,619 | 0.00 | | 89.337 | 19.43 | | | | | |
| | 14.159 | 0.00 | | 100.237 | 20.59 | | | | | |
| | 15.887 | 0.00 | | 112,468 | 18.12 | | | | | |
| | 17.825 | 0.00 | | 126,191 | 12.19 | | | | | |
| | 20.000 | 0.00 | | 141.589 | 6.03 | | | | | |
| | 22,440 | 0.00 | | 158.866 | 1.06 | | | | | |
| | 25.179 | 0.00 | | 178.250 | 0.00 | | | | | |
| | | 0.00 | | 200.000 | 0.00 | | | | | |
| | 28.251 | 0.00 | | | 0.00 | | | | | |
| | 31.698 | 0.00 | | 224.404 | 0.00 | | | | | |

0.00

0.00

282.508

316.979 355.656

39.905

44.774

50.238

| e In % | Size (µm) | Volume In % |
|--------|-----------|-------------|
| 0.00 | 355.656 | 0.00 |
| 2.02 | 399.052 | 0.00 |
| 7.22 | 447.744 | 0.00 |
| 13.34 | 502.377 | 0.00 |
| 19.43 | 563.677 | 0.00 |
| 20.59 | 632.456 | 0.00 |
| 18.12 | 709.627 | 0.00 |
| 12.19 | 796.214 | 0.00 |
| 6.03 | 893.367 | 0.00 |
| 1.06 | 1002.374 | 0.00 |
| 0.00 | 1124.683 | 0.00 |
| 0.00 | 1261.915 | 0.00 |
| 0.00 | 1415.892 | 0.00 |
| 0.00 | 1588.656 | 0.00 |
| 0.00 | 1782.502 | 0.00 |
| 0.00 | 2000.000 | 0.00 |
| 0.00 | | |
| 0.00 | | |

Operator notes:

XLS7892 sieved, 75-80 um fraction

Malvern Instruments Ltd. 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: 190 9/5/2017 3:40:27 PM





Sample Name:

NiCladWC-75-80 um-SN-1.3 - Average

Sample Source & type:

Sample bulk lot ref:

SOP Name:

D3M966

Monday, August 21, 2017 4:36:01 PM

Analysed:

Monday, August 21, 2017 4:36:02 PM

Result Source: Averaged

Measured by:

Particle Name: Tungsten powder Particle RI:

2.240

Dispersant Name:

Water Concentration:

0.1374

d(0.1):

Accessory Name: Hydro 2000G (A) Absorption: 0.1

Dispersant RI:

1.330

Span:

0.540

Specific Surface Area: 0.0655 m²/g

71.718

um

%Vol

Surface Weighted Mean D[3,2]: 91.633 um

d(0.5):

Analysis model:

Single narrow mode (spherical)

Size range:

to 2000.000 0.020

Weighted Residual: 0.650

Uniformity:

Vol. Weighted Mean D[4,3]:

95.484

d(0.9): 122.165 um

112.468 126.191 141 589 158.866

178.250

200.000

224.404

251.785

282.508

316.979 355.656

Sensitivity:

Obscuration:

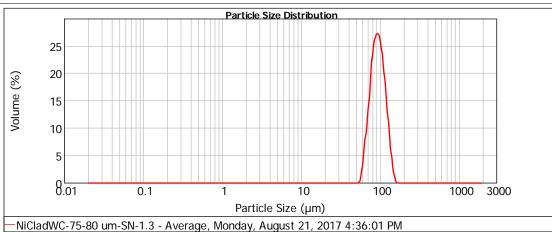
Result units:

Result Emulation:

Enhanced

10.12

Volume



93.481

| Size (µm) | Volume In % | I | Size (µm) | Volume In % | Size (µm) | Volume In % | Ī | Size (µm) | Volume In % | 1 | Size (µm) | Volume |
|-----------|-------------|---|-----------|-------------|-----------|-------------|---|-----------|-------------|---|-----------|--------|
| 0.020 | 0.00 | | 0.142 | 0.00 | 1.002 | 0.00 | I | 7.096 | 0.00 | | 50.238 | |
| 0.022 | 0.00 | | 0.159 | 0.00 | 1.125 | 0.00 | | 7.962 | 0.00 | | 56.368 | |
| 0.025 | 0.00 | | 0.178 | 0.00 | 1.262 | 0.00 | | 8.934 | 0.00 | | 63.246 | |
| 0.028 | | | 0.200 | | 1.416 | | | 10.024 | | | 70.963 | |
| 0.032 | 0.00 | | 0.224 | 0.00 | 1.589 | 0.00 | | 11.247 | 0.00 | | 79.621 | 1. |
| 0.036 | 0.00 | | 0.252 | 0.00 | 1.783 | 0.00 | | 12.619 | 0.00 | | 89.337 | 1: |
| 0.040 | 0.00 | | 0.283 | 0.00 | 2.000 | 0.00 | | 14.159 | 0.00 | | 100.237 | 2 |
| 5.0.10 | 0.00 | | 2.200 | 0.00 | 2.000 | 0.00 | | | 0.00 | | | 1: |

| 0.032 | 0.00 | 0.224 | 0.00 |
|-------|------|-------|------|
| | 0.00 | | 0.00 |
| 0.036 | 0.00 | 0.252 | 0.00 |
| 0.040 | 0.00 | 0.283 | 0.00 |
| 0.045 | | 0.317 | |
| 0.050 | 0.00 | 0.356 | 0.00 |
| 0.056 | 0.00 | 0.399 | 0.00 |
| | 0.00 | | 0.00 |
| 0.063 | 0.00 | 0.448 | 0.00 |
| 0.071 | 0.00 | 0.502 | 0.00 |
| 0.080 | | 0.564 | |
| 0.089 | 0.00 | 0.632 | 0.00 |
| 0.100 | 0.00 | 0.710 | 0.00 |
| | 0.00 | | 0.00 |
| 0.112 | 0.00 | 0.796 | 0.00 |
| 0.126 | 0.00 | 0.893 | 0.00 |
| | | | |

| 1.262 | 0.00 | |
|-------|------|--|
| | 0.00 | |
| 1.416 | 0.00 | |
| 1.589 | | |
| 1.783 | 0.00 | |
| | 0.00 | |
| 2.000 | 0.00 | |
| 2.244 | 0.00 | |
| 2.518 | | |
| 2.825 | 0.00 | |
| 3,170 | 0.00 | |
| | 0.00 | |
| 3.557 | 0.00 | |
| 3.991 | 0.00 | |
| 4.477 | | |
| 5.024 | 0.00 | |
| | 0.00 | |
| 5.637 | 0.00 | |

6.325

7.096

| 1 | , 2017 4 | 1:36:01 PN | / |
|---|-----------|-------------|---|
| | Size (µm) | Volume In % | 1 |
| | 7.096 | 0.00 | |
| | 7.962 | 0.00 | |
| | 8.934 | 0.00 | |
| | 10.024 | 0.00 | |
| | 11.247 | 0.00 | |
| | 12.619 | 0.00 | |
| | 14.159 | 0.00 | |
| | 15.887 | 0.00 | |
| | 17.825 | 0.00 | |
| | 20.000 | 0.00 | |
| | | | |

0.00

0.00

0.00

0.00

0.00

25.179

28.251

31.698

35.566

39.905

44.774

50.238

| e In % | | Size (µm) | Volume In % |
|--------|---|-----------|-------------|
| 0.00 | | 355.656 | 0.00 |
| 2.02 | | 399.052 | 0.00 |
| 7.19 | | 447.744 | 0.00 |
| 13.28 | | 502.377 | 0.00 |
| - | | 563.677 | |
| 19.36 | | 632,456 | 0.00 |
| 20.55 | | 709.627 | 0.00 |
| 18.13 | | 796.214 | 0.00 |
| 12.25 | | | 0.00 |
| 6.11 | | 893.367 | 0.00 |
| 1.09 | | 1002.374 | 0.00 |
| 0.00 | | 1124.683 | 0.00 |
| | | 1261.915 | |
| 0.00 | | 1415.892 | 0.00 |
| 0.00 | | 1588.656 | 0.00 |
| 0.00 | | 1782.502 | 0.00 |
| 0.00 | | | 0.00 |
| 0.00 | | 2000.000 | |
| | ı | | |

Operator notes:

XLS7892 sieved, 75-80 um fraction

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

Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545

0.00

File name: SHSVD WC samples Feb 2017.mea Record Number: 194 9/5/2017 3:40:27 PM





Sample Name:

NiCladWC-<75 um-SN-1.1 - Average

Sample Source & type:

Sample bulk lot ref:

SOP Name:

Measured by: D3M966

Result Source: Averaged

Monday, August 21, 2017 4:49:01 PM

Analysed:

Monday, August 21, 2017 4:49:02 PM

Particle Name: Tungsten powder Particle RI: 2.240 Dispersant Name:

57.348

um

Water

Concentration: 0.0862 %Vol

Specific Surface Area: 0.08 m²/g

d(0.1):

Accessory Name: Hydro 2000G (A) Absorption:

0.1 Dispersant RI:

1.330 Span:

0.586 Surface Weighted Mean D[3,2]:

74.996 um

d(0.5):

Analysis model:

Single narrow mode (spherical)

Size range:

to 2000.000 0.020

Weighted Residual: 0.616

Uniformity: 0.177

Vol. Weighted Mean D[4,3]:

78.769 um

Sensitivity:

Obscuration:

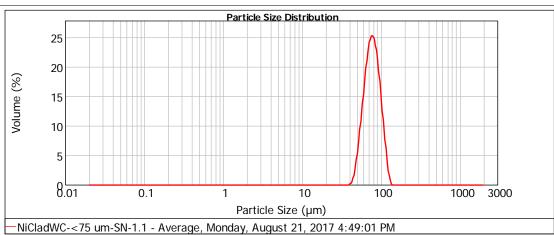
Result Emulation:

Enhanced

7.89

Result units: Volume

d(0.9): 102.555 um



77.134

Operator notes:

XLS7892 sieved, < 75 um fraction





Sample Name:

NiCladWC-<75 um-SN-1.2 - Average

Sample Source & type:

Sample bulk lot ref:

SOP Name:

Measured by: D3M966

Result Source:

Averaged

Monday, August 21, 2017 4:51:22 PM

Analysed:

Monday, August 21, 2017 4:51:23 PM

Particle Name: Tungsten powder Particle RI: 2.240

Dispersant Name: Water

Concentration: 0.0900 %Vol

Specific Surface Area: 0.0802 m²/g

Accessory Name: Hydro 2000G (A) Absorption:

Dispersant RI:

0.1

1.330 Span:

0.587 Surface Weighted Mean D[3,2]:

74.831 um Analysis model:

Single narrow mode (spherical)

Size range:

to 2000.000 0.020

Weighted Residual: 0.614

Uniformity: 0.178

Vol. Weighted Mean D[4,3]:

78.612 um

> d(0.9): 102.412 um

Sensitivity:

Obscuration:

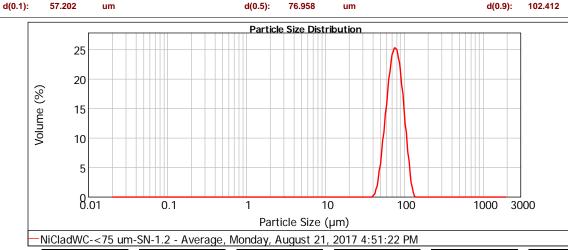
Result units:

Volume

Result Emulation:

Enhanced

8.24



| Size (µm) | | Size (µm) | Volume In % | Size (µm) | Volume In % | Size (µm) | Volume In % |] | Size (µm) | Volume In % | Size (µm) | Volume In % |
|-----------|------|-----------|-------------|-----------|-------------|-----------|-------------|---|-----------|-------------|-----------|-------------|
| 0.020 | 0.00 | 0.142 | 0.00 | 1.002 | 0.00 | 7.096 | 0.00 | | 50.238 | 6.34 | 355.656 | 0.00 |
| 0.022 | 0.00 | 0.159 | 0.00 | 1.125 | 0.00 | 7.962 | 0.00 | | 56.368 | 11.47 | 399.052 | 0.00 |
| 0.025 | 0.00 | 0.178 | 0.00 | 1.262 | 0.00 | 8.934 | 0.00 | | 63.246 | 16.39 | 447.744 | 0.00 |
| 0.028 | 0.00 | 0.200 | 0.00 | 1.416 | 0.00 | 10.024 | 0.00 | | 70.963 | 18.97 | 502.377 | 0.00 |
| 0.032 | 0.00 | 0.224 | 0.00 | 1.589 | 0.00 | 11.247 | 0.00 | | 79.621 | 18.12 | 563.677 | 0.00 |
| 0.036 | 0.00 | 0.252 | 0.00 | 1.783 | 0.00 | 12.619 | 0.00 | | 89.337 | 14.15 | 632.456 | 0.00 |
| 0.040 | 0.00 | 0.283 | 0.00 | 2.000 | 0.00 | 14.159 | 0.00 | | 100.237 | 8.58 | 709.627 | 0.00 |
| 0.045 | 0.00 | 0.317 | 0.00 | 2.244 | 0.00 | 15.887 | 0.00 | | 112.468 | 3.24 | 796.214 | 0.00 |
| 0.050 | 0.00 | 0.356 | 0.00 | 2.518 | 0.00 | 17.825 | 0.00 | | 126.191 | 0.22 | 893.367 | 0.00 |
| 0.056 | 0.00 | 0.399 | 0.00 | 2.825 | 0.00 | 20.000 | 0.00 | | 141.589 | 0.22 | 1002.374 | 0.00 |
| 0.063 | 0.00 | 0.448 | 0.00 | 3.170 | 0.00 | 22.440 | 0.00 | | 158.866 | 0.00 | 1124.683 | 0.00 |
| 0.071 | | 0.502 | | 3.557 | | 25.179 | | | 178.250 | 0.00 | 1261.915 | |
| 0.080 | 0.00 | 0.564 | 0.00 | 3.991 | 0.00 | 28.251 | 0.00 | | 200.000 | | 1415.892 | 0.00 |
| 0.089 | 0.00 | 0.632 | 0.00 | 4.477 | 0.00 | 31.698 | 0.00 | | 224.404 | 0.00 | 1588.656 | 0.00 |
| 0.100 | 0.00 | 0.710 | 0.00 | 5.024 | 0.00 | 35.566 | 0.00 | | 251.785 | 0.00 | 1782.502 | 0.00 |
| 0.112 | 0.00 | 0.796 | 0.00 | 5.637 | 0.00 | 39.905 | 0.00 | | 282.508 | 0.00 | 2000.000 | 0.00 |
| 0.126 | 0.00 | 0.893 | 0.00 | 6.325 | 0.00 | 44.774 | 0.21 | | 316.979 | 0.00 | | |
| 0.142 | 0.00 | 1.002 | 0.00 | 7.096 | 0.00 | 50.238 | 2.29 | | 355.656 | 0.00 | | |

Operator notes:

XLS7892 sieved, < 75 um fraction





Sample Name:

NiCladWC-<75 um-SN-1.3 - Average

Sample Source & type:

Sample bulk lot ref:

SOP Name:

Measured by: D3M966

Result Source:

Averaged

Monday, August 21, 2017 4:53:20 PM

Analysed:

Monday, August 21, 2017 4:53:22 PM

Sensitivity:

Obscuration:

Result units:

102.490

um

Volume

d(0.9):

Result Emulation:

Enhanced

8.35

Particle Name: Tungsten powder Particle RI: 2.240

Dispersant Name:

Water

Concentration: 0.0913 %Vol

Specific Surface Area: 0.0802 m²/g

Accessory Name: Hydro 2000G (A)

Absorption: 0.1

Dispersant RI: 1.330

Span: 0.588

Surface Weighted Mean D[3,2]: 74.849 um

Analysis model:

Single narrow mode (spherical)

Size range:

to 2000.000 0.020 Weighted Residual:

0.613

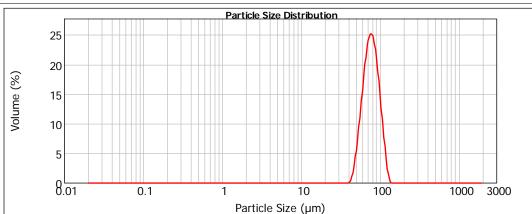
Uniformity:

0.178

Vol. Weighted Mean D[4,3]:

78.633 um

d(0.1): 57.216 d(0.5): 76.950



| Size (µm) | volume in % | Size (µm) | volume in % | Size (µIII) | VO |
|-----------|-------------|-----------|-------------|-------------|----|
| 0.020 | 0.00 | 0.142 | 0.00 | 1.002 | |
| 0.022 | 0.00 | 0.159 | 0.00 | 1.125 | |
| 0.025 | | 0.178 | 0.00 | 1.262 | |
| 0.028 | 0.00 | 0.200 | | 1.416 | |
| 0.032 | 0.00 | 0.224 | 0.00 | 1.589 | |
| 0.036 | 0.00 | 0.252 | 0.00 | 1.783 | |
| 0.040 | 0.00 | 0.283 | 0.00 | 2.000 | |
| 0.045 | 0.00 | 0.200 | 0.00 | 2.244 | |
| | 0.00 | | 0.00 | | |
| 0.050 | 0.00 | 0.356 | 0.00 | 2.518 | |
| 0.056 | 0.00 | 0.399 | 0.00 | 2.825 | |
| 0.063 | 0.00 | 0.448 | 0.00 | 3.170 | |
| 0.071 | 0.00 | 0.502 | 0.00 | 3.557 | |
| 0.080 | 0.00 | 0.564 | 0.00 | 3.991 | |
| 0.089 | 0.00 | 0.632 | 0.00 | 4.477 | |
| 0.100 | 0.00 | 0.710 | 0.00 | 5.024 | |
| | | | | | |

NiCladWC-<75 um-SN-1.3 - Average, Monday, August 21,

| Size (µm) | Volume In % |
|-----------|-------------|
| 1.002 | 0.00 |
| 1.125 | 0.00 |
| 1.262 | 0.00 |
| 1.416 | 0.00 |
| 1.589 | 0.00 |
| 1.783 | 0.00 |
| 2.000 | |
| 2.244 | 0.00 |
| 2.518 | 0.00 |
| 2.825 | 0.00 |
| 3,170 | 0.00 |
| 3.557 | 0.00 |
| 3.991 | 0.00 |
| 4.477 | 0.00 |
| 7.477 | 0.00 |

5.637

6.325 7.096

| 2017 4: | 53:20 PM | |
|-------------|-------------|--|
| Size (µm) | Volume In % | |
| 7.096 | 0.00 | |
| 7.962 | 0.00 | |
| 8.934 | 0.00 | |
| 10.024 | 0.00 | |
| 11.247 | 0.00 | |
| 12.619 | 0.00 | |
| 14.159 | 0.00 | |
| 15.887 | 0.00 | |
| 17.825 | 0.00 | |
| 20.000 | 0.00 | |
| 22.440 | 0.00 | |
| | | |

25.179

28.251

31.698

35.566

39.905

44.774

50.238

0.19

| e in % | | Size (µm) | volume in % |
|--------|---|-----------|-------------|
| 0.00 | | 50.238 | 6.36 |
| 0.00 | | 56.368 | 11.50 |
| 0.00 | | 63.246 | 16.41 |
| | | 70.963 | |
| 0.00 | | 79.621 | 18.96 |
| 0.00 | | 89.337 | 18.09 |
| 0.00 | | 100.237 | 14.12 |
| 0.00 | | 112.468 | 8.58 |
| 0.00 | | 126.191 | 3.29 |
| 0.00 | | | 0.23 |
| 0.00 | | 141.589 | 0.00 |
| 0.00 | | 158.866 | 0.00 |
| 0.00 | | 178.250 | 0.00 |
| 0.00 | | 200.000 | 0.00 |
| 0.00 | | 224.404 | 0.00 |
| 0.00 | | 251.785 | 0.00 |
| 0.00 | ı | 202 500 | 0.00 |

316.979 355.656

| | _ | | |
|------------------------|---|----------------------------------|----------------------|
| e In % | | Size (µm) | Volume In % |
| 6.36 11.50 16.41 | | 355.656 399.052 447.744 | 0.00 0.00 0.00 |
| 18.96 | | 502.377 563.677 | 0.00 |
| 14.12 | | 632.456 709.627 | 0.00 |
| 3.29 | | 796.214 893.367 | 0.00 |
| 0.00 | | 1002.374 1124.683 | 0.00 |
| 0.00 | | 1261.915 1415.892 | 0.00 |
| 0.00 | | 1588.656 1782.502 2000.000 | 0.00 |
| 0.00 | | 2000.000 | |

Operator notes:

1.002 XLS7892 sieved, < 75 um fraction

0.796

0.893

0.00

0.00

0.00

0.00

0.00

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

0.112

0.126

Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545

0.00

0.00

File name: SHSVD WC samples Feb 2017.mea Record Number: 206 9/5/2017 3:40:28 PM





Sample Name:

NiCladWC->80 um-SN-1.1 - Average

Sample Source & type:

Sample bulk lot ref:

SOP Name:

Measured by:

D3M966

Monday, August 21, 2017 5:05:40 PM

Analysed:

Monday, August 21, 2017 5:05:41 PM

Result Source: Averaged

Particle Name: Tungsten powder Particle RI: 2.240

Dispersant Name: Water

Concentration: 0.1437 %Vol

Specific Surface Area: 0.0528 m²/g

Accessory Name: Hydro 2000G (A) Absorption:

0.1 Dispersant RI:

1.330

Span:

0.615 Surface Weighted Mean D[3,2]:

113.610 um Analysis model:

Single narrow mode (spherical)

Size range:

to 2000.000 0.020

Weighted Residual: 0.553

Uniformity: 0.194

Vol. Weighted Mean D[4,3]:

119.676

Sensitivity:

Obscuration:

Result units:

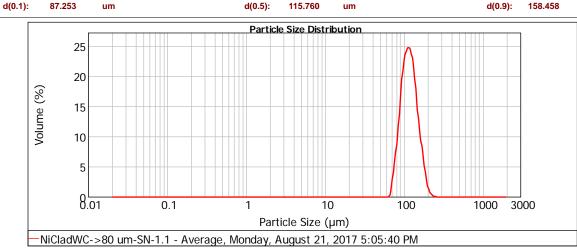
Volume

Result Emulation:

um

Enhanced

8.56



| | | | 3 - 1 | · · · | - 3 1 | | | | | | | |
|-----------|-------------|-----------|-------------|-----------|-------------|-----------|-------------|-----------|-------------|---|-----------|-------------|
| Size (µm) | Volume In % | Ī | Size (µm) | Volume In % |
| 0.020 | 0.00 | 0.142 | 0.00 | 1.002 | 0.00 | 7.096 | 0.00 | 50.238 | 0.00 | | 355.656 | 0.00 |
| 0.022 | 0.00 | 0.159 | 0.00 | 1.125 | 0.00 | 7.962 | 0.00 | 56.368 | 0.00 | | 399.052 | 0.00 |
| 0.025 | 0.00 | 0.178 | 0.00 | 1.262 | 0.00 | 8.934 | 0.00 | 63.246 | 0.00 | | 447.744 | 0.00 |
| 0.028 | | 0.200 | | 1.416 | | 10.024 | | 70.963 | | | 502.377 | |
| 0.032 | 0.00 | 0.224 | 0.00 | 1.589 | 0.00 | 11.247 | 0.00 | 79.621 | 4.12 | | 563.677 | 0.00 |
| 0.036 | 0.00 | 0.252 | 0.00 | 1.783 | 0.00 | 12.619 | 0.00 | 89.337 | 8.04 | | 632.456 | 0.00 |
| 0.040 | 0.00 | 0.283 | 0.00 | 2.000 | 0.00 | 14.159 | 0.00 | 100.237 | 14.90 | | 709.627 | 0.00 |
| 0.045 | 0.00 | 0.317 | 0.00 | 2.244 | 0.00 | 15.887 | 0.00 | 112.468 | 18.09 | | 796.214 | 0.00 |
| 0.050 | 0.00 | 0.356 | 0.00 | 2.518 | 0.00 | 17.825 | 0.00 | 126.191 | 18.53 | | 893.367 | 0.00 |
| 0.056 | 0.00 | 0.399 | 0.00 | 2.825 | 0.00 | 20.000 | 0.00 | 141.589 | 16.11 | | 1002.374 | 0.00 |
| 0.063 | 0.00 | 0.448 | 0.00 | 3.170 | 0.00 | 22.440 | 0.00 | 158.866 | 10.24 | | 1124.683 | 0.00 |
| 0.071 | 0.00 | 0.502 | 0.00 | 3.557 | 0.00 | 25,179 | 0.00 | 178,250 | 6.51 | | 1261.915 | 0.00 |
| 0.080 | 0.00 | 0.564 | 0.00 | 3.991 | 0.00 | 28.251 | 0.00 | 200.000 | 2.64 | | 1415.892 | 0.00 |
| 0.089 | 0.00 | 0.632 | 0.00 | 4.477 | 0.00 | 31.698 | 0.00 | 224.404 | 0.62 | | 1588.656 | 0.00 |
| 0.100 | 0.00 | 0.710 | 0.00 | 5.024 | 0.00 | 35.566 | 0.00 | 251.785 | 0.05 | | 1782.502 | 0.00 |
| 0.112 | 0.00 | 0.796 | 0.00 | 5.637 | 0.00 | 39.905 | 0.00 | 282.508 | 0.00 | | 2000.000 | 0.00 |
| 0.112 | 0.00 | 0.893 | 0.00 | 6.325 | 0.00 | 44.774 | 0.00 | 316.979 | 0.00 | | 2000.000 | |
| 0.120 | 0.00 | 1 002 | 0.00 | 7.096 | 0.00 | 50 238 | 0.00 | 355 656 | 0.00 | | | |
| | | | | | | | | | | | | |

Operator notes:

XLS7892 sieved, >80 um fraction

Malvern Instruments Ltd. 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: 210 9/5/2017 3:40:28 PM





Sample Name:

NiCladWC->80 um-SN-1.2 - Average

Sample Source & type:

Sample bulk lot ref:

SOP Name:

Measured by: D3M966

Result Source:

Averaged

Monday, August 21, 2017 5:07:42 PM

Analysed:

Monday, August 21, 2017 5:07:43 PM

Particle Name: Tungsten powder Particle RI: 2.240

Dispersant Name:

Water

Concentration: 0.1502 %Vol

d(0.1):

Specific Surface Area: 0.0528 m²/g

87.153

Accessory Name: Hydro 2000G (A) Absorption:

Dispersant RI: 1.330

0.1

Span: 0.620

Surface Weighted Mean D[3,2]: 113.661 um

d(0.5):

Analysis model:

Single narrow mode (spherical)

Size range:

to 2000.000 0.020

Weighted Residual: 0.554

Uniformity: 0.198

Vol. Weighted Mean D[4,3]:

120.100

Result units:

Sensitivity:

Obscuration:

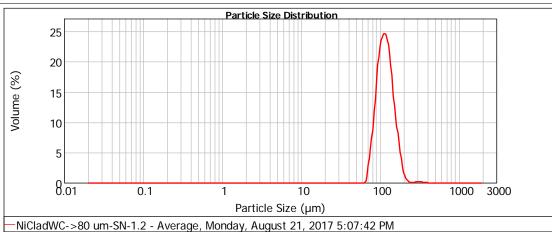
Result Emulation:

Enhanced

8.93

Volume

d(0.9): 158.845 um



115.688

Size (µm) Size (µm) Volume In % Volume In % 0.00 0.00 0.00 0.00 0.00 0.04 0.022 0.159 1.125 7.962 56.368 399.052 0.00 0.00 0.025 0.178 1.262 8.934 63.246 447.744 0.00 0.00 0.00 0.00 0.17 0.00 502.377 0.028 0.200 1.416 10.024 70.963 0.00 0.00 0.00 0.00 4.16 0.00 0.032 563.677 0.224 1.589 11.247 79.621 0.00 0.00 0.00 0.00 8.08 0.00 0.036 0.252 1.783 12.619 89.337 632.456 0.00 0.00 0.00 0.00 14.92 0.00 0.040 0.283 2.000 14.159 100.237 709.627 0.00 0.00 0.00 0.00 18.07 0.00 0.045 0.317 2.244 15.887 112.468 796.214 0.00 0.00 0.00 0.00 18.47 0.00 0.050 0.356 2.518 17.825 126,191 893,367 0.00 0.00 0.00 0.00 16.01 0.00 0.056 0.399 2 825 20.000 141 589 1002 374 0.00 0.00 0.00 10.13 1124.683 0.063 0.448 3.170 22,440 158.866 0.00 0.00 0.00 0.00 6.41 0.00 0.071 0.502 3.557 25.179 178.250 1261.915 0.00 0.00 0.00 0.00 2.59 0.00 0.080 3.991 28.251 200.000 1415.892 0.564 0.00 0.00 0.00 0.00 0.61 0.00 0.089 0.632 4.477 31.698 224.404 1588.656 0.00 0.00 0.00 0.00 0.08 0.00 0.100 0.710 5.024 35.566 251.785 1782.502 0.00 0.00 0.03 0.00 0.00 0.00 0.112 0.796 5.637 39.905 282.508 2000.000 0.00 0.00 0.00 0.00 0.13 0.126 0.893 6.325 44.774 316.979 0.00 7.096 355.656 1.002 50.238

Operator notes:

XLS7892 sieved, >80 um fraction





Sample Name:

NiCladWC->80 um-SN-1.3 - Average

Sample Source & type:

Sample bulk lot ref:

SOP Name:

Measured by: D3M966

Result Source: Averaged

Monday, August 21, 2017 5:09:37 PM

Analysed:

Monday, August 21, 2017 5:09:38 PM

Particle Name: Tungsten powder Particle RI: 2.240

Dispersant Name: Water

Concentration: 0.1492 %Vol

Specific Surface Area: 0.0529 m²/g

Accessory Name: Hydro 2000G (A) Absorption:

Dispersant RI: 1.330

0.1

Span:

0.614 Surface Weighted Mean D[3,2]:

113.497 um Analysis model:

Single narrow mode (spherical)

Size range:

Uniformity:

to 2000.000 0.020

Weighted Residual: 0.563

0.194 Vol. Weighted Mean D[4,3]:

119.547

d(0.1):

87.175

d(0.5):

115.674

d(0.9):

158.194

Sensitivity:

Obscuration:

Result units:

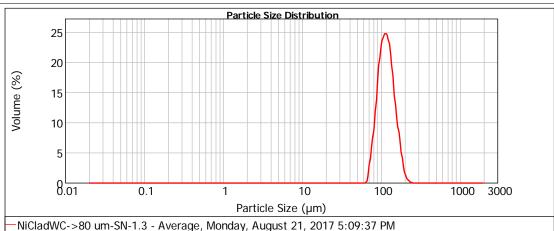
Volume

Result Emulation:

Enhanced

8.88

um



| Size (µm) | Volume In % | Size (µm) | Volume In % |
|----------------|-------------|----------------|-------------|
| 0.020 | 0.00 | 0.142 | 0.00 |
| 0.022 | 0.00 | 0.159 | 0.00 |
| 0.025 | 0.00 | 0.178 | 0.00 |
| 0.028 | 0.00 | 0.200 | 0.00 |
| 0.032 | 0.00 | 0.224 | 0.00 |
| 0.036 | 0.00 | 0.252 | 0.00 |
| 0.040 | 0.00 | 0.283 | 0.00 |
| 0.045 | 0.00 | 0.317 | 0.00 |
| 0.050 | 0.00 | 0.356 | 0.00 |
| 0.056 0.063 | 0.00 | 0.399 0.448 | 0.00 |
| 0.063 | 0.00 | 0.502 | 0.00 |
| 0.071 | 0.00 | 0.564 | 0.00 |
| 0.089 | 0.00 | 0.632 | 0.00 |
| 0.100 | 0.00 | 0.032 | 0.00 |
| 0.112 | 0.00 | 0.796 | 0.00 |
| 0.126 | 0.00 | 0.893 | 0.00 |
| 0.142 | 0.00 | 1.002 | 0.00 |

| Size (µm) | Volume In % |
|-----------|-------------|
| 1.002 | 0.00 |
| 1.125 | 0.00 |
| 1.262 | 0.00 |
| 1.416 | |
| 1.589 | 0.00 |
| 1.783 | 0.00 |
| 2.000 | 0.00 |
| 2.244 | 0.00 |
| 2.518 | 0.00 |
| 2.825 | 0.00 |
| | 0.00 |
| 3.170 | 0.00 |
| 3.557 | 0.00 |
| 3.991 | 0.00 |
| | |

4.477

5.024

5.637

6.325 7.096

| 2017 5: | 09:37 PM | |
|-----------|-------------|--|
| Size (µm) | Volume In % | |
| 7.096 | 0.00 | |
| 7.962 | 0.00 | |
| 8.934 | 0.00 | |
| 10.024 | 0.00 | |
| 11.247 | 0.00 | |
| 12.619 | 0.00 | |
| 14.159 | 0.00 | |
| 15.887 | 0.00 | |
| 17.825 | | |
| 20.000 | 0.00 | |
| 22 440 | 0.00 | |

0.00

0.00

0.00

0.00

0.00

25.179

28.251

31.698

35.566

39.905

44.774

50.238

| Size (µm) | Volume In % |
|-----------|--------------|
| 50.238 | 0.00 |
| 56.368 | 0.00 |
| 63.246 | 0.18 |
| 70.963 | 4.15 |
| 79.621 | 4.15 8.05 |
| 89.337 | 0.00 |
| 100.237 | 14.91 |
| 112.468 | 18.11 |
| 126.191 | 18.55 |
| 141.589 | 16.12 |
| 159 966 | 10.21 |

2.60

0.59

0.05

0.00

0.00

0.00

178.250

200.000

224.404

251.785

282.508

316.979 355.656

| | Size (µm) | Volume In % |
|--|-----------|-------------|
| | 355.656 | 0.00 |
| | 399.052 | 0.00 |
| | 447.744 | 0.00 |
| | 502.377 | |
| | 563.677 | 0.00 |
| | 632,456 | 0.00 |
| | 709.627 | 0.00 |
| | 796.214 | 0.00 |
| | 893.367 | 0.00 |
| | 1002.374 | 0.00 |
| | 1124.683 | 0.00 |
| | 1261.915 | 0.00 |
| | | 0.00 |
| | 1415.892 | 0.00 |
| | 1588.656 | 0.00 |
| | 1782.502 | 0.00 |
| | 2000.000 | |
| | | |

Operator notes:

XLS7892 sieved, >80 um fraction

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

Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545

0.00

0.00

0.00

File name: SHSVD WC samples Feb 2017.mea Record Number: 218 9/5/2017 3:40:28 PM





Sample Name:

NiCladWC-75-80 um-1.1-Average

Sample Source & type:

Sample bulk lot ref:

SOP Name:

Measured by: D3M966

Result Source:

Averaged

Monday, August 21, 2017 4:32:11 PM

Analysed:

Thursday, August 31, 2017 4:48:41 PM

Sensitivity:

Obscuration:

Result units:

Volume

Result Emulation:

Normal

9.44

Particle Name: Tungsten powder Particle RI: 2.240

Dispersant Name: Water

Concentration: 0.1267 %Vol

Specific Surface Area: 0.066 m²/g

Accessory Name: Hydro 2000G (A) Absorption: 0.1

Dispersant RI: 1.330

Span: 0.638

Surface Weighted Mean D[3,2]: 90.891 um

Analysis model: General purpose Size range:

0.020

0.201

0.672

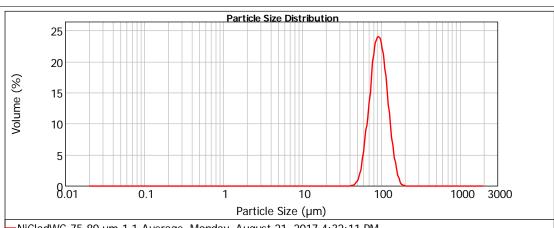
to 2000.000 um Weighted Residual:

Uniformity:

Vol. Weighted Mean D[4,3]:

96.360 um

d(0.1): 68.116 um d(0.5): 93.583 d(0.9): 127.868 um



-NiCladWC-75-80 um-1.1-Average, Monday, August 21, 2017 4:32:11 PM

| Size (µm) | Volume In % | Size (µm) | Volume In % | Size (µm) | Volume In % | Size (µm) | Volume In % | Size (µm) | Volume In % | Size (µm) | Volume In % |
|-----------|---|--|--|---|--|---|--|---|--|--|---|
| Size (µm) | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | 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 0.710 0.796 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | 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 5.637 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | Size (µm) 7.096 7.096 8.934 10.024 11.247 12.619 15.887 17.825 20.000 22.440 25.179 28.251 31.698 35.566 39.905 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | 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 | 1.11 3.93 7.75 12.50 17.13 18.13 16.31 11.84 6.95 3.12 0.92 0.07 0.00 0.00 0.00 0.00 | Size (µm) 355.656 399.052 447.744 502.377 563.677 632.456 709.627 796.214 893.367 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:

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

Malvern Instruments Ltd. 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





Sample Name:

NiCladWC-75-80 um-1.2-Average

Sample Source & type:

Sample bulk lot ref:

SOP Name:

Measured by: D3M966

Result Source:

Averaged

Monday, August 21, 2017 4:34:06 PM

Analysed:

Thursday, August 31, 2017 4:49:59 PM

Sensitivity:

Obscuration:

Result units:

Result Emulation:

Normal

10.06

Volume

um

Particle Name: Tungsten powder Particle RI: 2.240

Dispersant Name:

Water

Concentration: 0.1352 %Vol Specific Surface Area:

0.066 m²/g **Accessory Name:** Hydro 2000G (A) Absorption:

0.1 Dispersant RI: 1.330

Span: 0.639

Surface Weighted Mean D[3,2]: 90.847 um

Analysis model: General purpose Size range:

to 2000.000 0.020 Weighted Residual:

0.675

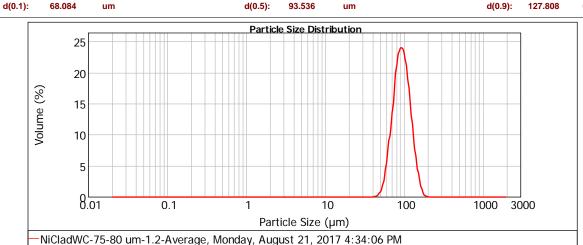
Uniformity:

Vol. Weighted Mean D[4,3]:

96.313

d(0.5): 93.536 d(0.9): 127.808 um

0.201



| Size (µm) | Volume In % | Size (µm) | Volume In % | Size (µm) | Volume In % | Size (µm) | Volume In % | Size (µm) | Volume In % | Size (µm) | Volume In % |
|---|--|--|---|--|---|---|---|--|---|--|---|
| 0.020 0.022 0.025 0.028 0.032 0.036 0.040 0.045 0.050 0.056 0.063 0.071 0.080 0.089 0.100 0.112 0.126 | 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 0.00 | 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 0.893 1.002 | 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 6.325 7.096 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | 7.096 7.962 8.934 10.024 11.247 12.619 14.159 15.887 17.825 20.000 22.440 25.179 28.251 31.698 35.566 39.905 44.774 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | 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 | 1.12 3.94 7.77 12.53 17.14 18.13 16.29 11.82 6.93 3.11 0.92 0.07 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 |

Operator notes:

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

Malvern Instruments Ltd. 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





Monday, August 21, 2017 4:36:01 PM

to 2000.000

Result Analysis Report

Sample Name:

NiCladWC-75-80 um-1.3-Average

Sample Source & type:

Sample bulk lot ref:

Particle Name:

Particle RI:

2.240

Water

0.1362

0.066

Tungsten powder

Dispersant Name:

Concentration:

SOP Name:

Measured by: D3M966

Result Source:

Accessory Name:

Hydro 2000G (A)

Absorption:

Dispersant RI:

0.1

1.330

Span:

0.639

90.877

Averaged

Analysed: Thursday, August 31, 2017 4:50:58 PM

Analysis model: General purpose

Size range:

0.020

Weighted Residual:

0.669

Uniformity: 0.201

Vol. Weighted Mean D[4,3]:

96.351

d(0.1):

68.099

%Vol

m²/g

Specific Surface Area:

d(0.5):

Surface Weighted Mean D[3,2]:

um

93.571

d(0.9):

um

Sensitivity:

Obscuration:

Result units:

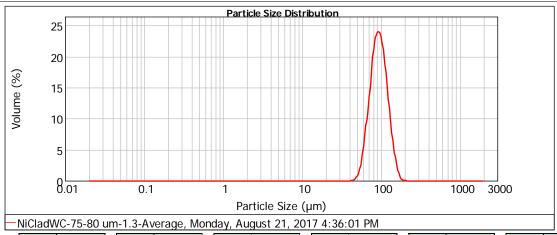
Result Emulation:

Normal

10.12

Volume

127.873 um



| Size (µm) 0.020 0.022 0.025 0.028 0.032 0.036 0.040 | 0.00 0.00 0.00 0.00 0.00 | Size (µm) 0.142 0.159 0.178 0.200 0.224 0.252 0.283 | Volume In % 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0 | Size (µm) 1.002 1.125 1.262 1.416 1.589 1.783 2.000 | Volume In % 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0 | Size (µm) 7.096 7.962 8.934 10.024 11.247 12.619 14.159 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | Size (µm) 50.238 56.368 63.246 70.963 79.621 89.337 100.237 | Volume in % 1.11 3.93 7.76 12.51 17.12 18.12 16.30 | Size (µm) 355.656 399.052 447.744 502.377 563.677 632.456 709.627 | Volume In % 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0. |
|---|--|---|---|---|---|--|---|---|---|--|---|
| 0.056 0.063 0.071 0.080 0.089 0.100 0.112 0.126 0.142 | 0.00 0.00 0.00 0.00 0.00 0.00 | 0.399 0.448 0.502 0.564 0.632 0.710 0.796 0.893 1.002 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | 2.825 3.170 3.557 3.991 4.477 5.024 5.637 6.325 7.096 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | 20.000 22.440 25.179 28.251 31.698 35.566 39.905 44.774 50.238 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | 141.589 158.866 178.250 200.000 224.404 251.785 282.508 316.979 355.656 | 6.95 3.13 0.93 0.07 0.00 0.00 0.00 0.00 0.00 | 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 |

Operator notes:

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

Malvern Instruments Ltd. 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





Sample Name:

NiCladWC-<75 um-1.1-Average

Sample Source & type:

Sample bulk lot ref:

SOP Name:

Measured by: D3M966

Result Source: Averaged

Monday, August 21, 2017 4:49:01 PM

Analysed:

Thursday, August 31, 2017 4:52:16 PM

Sensitivity:

Obscuration:

Result units:

Volume

Result Emulation:

um

Normal

7.89

um

Size (µm) Volume In %

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

6.90

11.54

15.82

17.85

16.71

12.96

8.16

3.98

1.45

0.23

0.00

0.00

0.00

0.00

0.00

0.00

Particle Name: Tungsten powder Particle RI: 2.240

Dispersant Name: Water

Concentration: 0.0851 %Vol

Specific Surface Area: 0.0811 m²/g

Accessory Name: Hydro 2000G (A) Absorption:

0.1 Dispersant RI:

1.330

Span: 0.648

Surface Weighted Mean D[3,2]: 74.005 um

Analysis model: General purpose Size range:

to 2000.000 0.020 Weighted Residual:

0.528

Uniformity:

Vol. Weighted Mean D[4,3]: 78.664

um

'olume In %

0.00

0.00

0.00

0.00

0.00

0.00

0.00

0.00

0.00

0.00

0.00

0.02

0.16

1.06

d(0.1): 55.432 d(0.5): 76.373 d(0.9): 104.950 Particle Size Distribution 25 20 Volume (%) 15 10 5 8.01 0.1 100 1000 3000 Particle Size (µm) NiCladWC-<75 um-1.1-Average, Monday, August 21, 2017 4:49:01 PM

| Size (µm) | Volume In % | Size (µm) | Volume In % | Size (µm) | Volume In % | Size (µm) | ٧ |
|-----------|-------------|-----------|-------------|-----------|-------------|-----------|---|
| 0.020 | 0.00 | 0.142 | 0.00 | 1.002 | 0.00 | 7.096 | |
| 0.022 | 0.00 | 0.159 | 0.00 | 1.125 | 0.00 | 7.962 | |
| 0.025 | 0.00 | 0.178 | 0.00 | 1.262 | 0.00 | 8.934 | |
| 0.028 | 0.00 | 0.200 | 0.00 | 1.416 | 0.00 | 10.024 | |
| 0.032 | 0.00 | 0.224 | 0.00 | 1.589 | 0.00 | 11.247 | |
| 0.036 | 0.00 | 0.252 | 0.00 | 1.783 | 0.00 | 12.619 | |
| 0.040 | 0.00 | 0.283 | 0.00 | 2.000 | 0.00 | 14.159 | |
| 0.045 | 0.00 | 0.317 | 0.00 | 2.244 | 0.00 | 15.887 | |
| 0.050 | 0.00 | 0.356 | 0.00 | 2.518 | 0.00 | 17.825 | |
| 0.056 | 0.00 | 0.399 | 0.00 | 2.825 | 0.00 | 20.000 | |
| 0.063 | 0.00 | 0.448 | 0.00 | 3.170 | 0.00 | 22.440 | |
| 0.071 | 0.00 | 0.502 | 0.00 | 3.557 | 0.00 | 25.179 | |
| 0.080 | 0.00 | 0.564 | 0.00 | 3.991 | 0.00 | 28.251 | |
| 0.089 | 0.00 | 0.632 | 0.00 | 4.477 | 0.00 | 31.698 | |
| 0.100 | 0.00 | 0.710 | 0.00 | 5.024 | 0.00 | 35.566 | |
| 0.112 | 0.00 | 0.796 | 0.00 | 5.637 | 0.00 | 39.905 | |
| 0.126 | 0.00 | 0.893 | 0.00 | 6.325 | 0.00 | 44.774 | |
| 0.142 | 0.00 | 1.002 | 0.00 | 7.096 | 0.00 | 50.238 | |

Operator notes:

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

Malvern Instruments Ltd. 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

Size (µm)

399.052

447.744

502.377

563.677

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

0.00

0.00

0.00

0.00

0.00

0.00

0.00





Sample Name:

NiCladWC-<75 um-1.2-Average

Sample Source & type:

Sample bulk lot ref:

SOP Name:

Measured by: D3M966

Result Source:

Averaged

Monday, August 21, 2017 4:51:22 PM

Analysed:

Thursday, August 31, 2017 4:53:02 PM

Particle Name: Tungsten powder Particle RI: 2.240

Dispersant Name:

Concentration:

Water

0.0889

0.0812

Accessory Name: Hydro 2000G (A) Absorption: 0.1

Dispersant RI:

1.330

Span:

0.649

Surface Weighted Mean D[3,2]:

73.883 um Analysis model: General purpose

Size range:

to 2000.000 0.020 um Weighted Residual:

0.527

Uniformity:

Vol. Weighted Mean D[4,3]:

78.538

d(0.1):

55.334

%Vol

m²/g

Specific Surface Area:

d(0.5):

76.245

d(0.9):

104.785

Sensitivity:

Obscuration:

Result units:

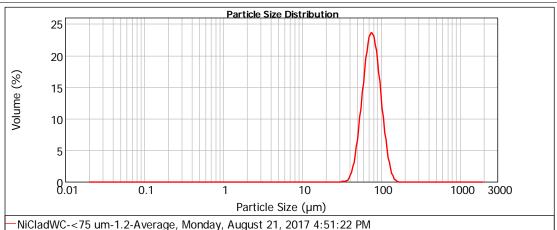
Volume

Result Emulation:

Normal

8.24

um



| Si | ze (µm) 0.020 0.022 0.025 0.028 0.032 0.036 0.040 0.045 0.050 0.056 0.063 0.071 0.080 0.089 0.100 0.112 0.126 | 0.00 | 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 0.710 0.796 0.893 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | 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 5.637 6.325 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | Size (µm) 7.096 7.962 8.934 10.024 11.247 12.619 14.159 15.887 17.825 20.000 22.440 25.179 28.251 31.698 35.566 39.905 44.774 | Volume in % 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0 | 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 | Volume in % 6.96 11.61 15.87 17.85 16.67 12.89 8.10 3.93 1.43 0.22 0.00 0.00 0.00 0.00 0.00 0.00 | 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 | | 1.08 3.21 | | 0.00 | 2000.000 | |

Operator notes:

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

Malvern Instruments Ltd. 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





Sample Name:

NiCladWC-<75 um-1.3-Average

Sample Source & type:

Sample bulk lot ref:

Particle Name:

Particle RI:

2.240

Water

0.0901

0.0812

d(0.1):

Tungsten powder

Dispersant Name:

Concentration:

%Vol

m²/g

55.356

Specific Surface Area:

SOP Name:

vanic.

Measured by: D3M966

Result Source: Averaged

Analysed:

Thursday, August 31, 2017 4:54:04 PM

Monday, August 21, 2017 4:53:20 PM

Accessory Name: Hydro 2000G (A) Absorption:

0.1

Dispersant RI:

1.330

Span: 0.649

Surface Weighted Mean D[3,2]:

d(0.5):

73.911 um

Analysis model: General purpose

Size range: 0.020 to 2

0.020 to 2000.000 um Weighted Residual:

0.524 %

Uniformity:

Vol. Weighted Mean D[4,3]:

78.569 um

um

d(0.9): 1

104.835 um

Sensitivity:

Obscuration:

Result units:

Volume

Result Emulation:

Normal

8.35

Particle Size Distribution

25
20
(%) 15
8.01 0.1 1 1 10 100 1000 3000
Particle Size (μm)

NiCladWC-<75 um-1.3-Average, Monday, August 21, 2017 4:53:20 PM

76.271

| _ I, | viciauv | /C- 5 um-</th <th>1.3-Ave</th> <th>rage, ivionida</th> <th>ay, Augu</th> <th>JSL 21, 2017</th> <th>4.55.20</th> <th>J PIVI</th> <th></th> <th></th> <th></th> <th></th> | 1.3-Ave | rage, ivionida | ay, Augu | JSL 21, 2017 | 4.55.20 | J PIVI | | | | |
|------|-----------|---|-----------|----------------|-----------|--------------|-----------|-------------|-----------|-------------|-----------|-------------|
| | Size (µm) | Volume In % | Size (µm) | Volume In % | Size (µm) | Volume In % | Size (µm) | Volume In % | Size (µm) | Volume In % | Size (µm) | Volume In % |
| | 0.020 | 0.00 | 0.142 | 0.00 | 1.002 | 0.00 | 7.096 | 0.00 | 50.238 | 6.95 | 355.656 | 0.00 |
| | 0.022 | 0.00 | 0.159 | 0.00 | 1.125 | 0.00 | 7.962 | 0.00 | 56.368 | 11.60 | 399.052 | 0.00 |
| | 0.025 | 0.00 | 0.178 | 0.00 | 1.262 | 0.00 | 8.934 | 0.00 | 63.246 | 15.86 | 447.744 | 0.00 |
| | 0.028 | 0.00 | 0.200 | 0.00 | 1.416 | 0.00 | 10.024 | 0.00 | 70.963 | 17.85 | 502.377 | 0.00 |
| | 0.032 | 0.00 | 0.224 | 0.00 | 1.589 | 0.00 | 11.247 | 0.00 | 79.621 | 16.67 | 563.677 | 0.00 |
| | 0.036 | 0.00 | 0.252 | 0.00 | 1.783 | 0.00 | 12.619 | 0.00 | 89.337 | 12.90 | 632.456 | 0.00 |
| | 0.040 | 0.00 | 0.283 | 0.00 | 2.000 | 0.00 | 14.159 | 0.00 | 100.237 | 8.11 | 709.627 | 0.00 |
| | 0.045 | 0.00 | 0.317 | 0.00 | 2.244 | 0.00 | 15.887 | 0.00 | 112.468 | 3.95 | 796.214 | 0.00 |
| | 0.050 | 0.00 | 0.356 | 0.00 | 2.518 | 0.00 | 17.825 | 0.00 | 126.191 | 1.44 | 893.367 | 0.00 |
| | 0.056 | 0.00 | 0.399 | 0.00 | 2.825 | 0.00 | 20.000 | 0.00 | 141.589 | 0.22 | 1002.374 | 0.00 |
| | 0.063 | 0.00 | 0.448 | 0.00 | 3.170 | 0.00 | 22.440 | 0.00 | 158.866 | 0.00 | 1124.683 | 0.00 |
| | 0.071 | 0.00 | 0.502 | 0.00 | 3.557 | 0.00 | 25.179 | 0.00 | 178.250 | 0.00 | 1261.915 | 0.00 |
| | 0.080 | 0.00 | 0.564 | 0.00 | 3.991 | 0.00 | 28.251 | 0.00 | 200.000 | 0.00 | 1415.892 | 0.00 |
| | 0.089 | 0.00 | 0.632 | 0.00 | 4.477 | 0.00 | 31.698 | 0.02 | 224.404 | 0.00 | 1588.656 | 0.00 |
| | 0.100 | 0.00 | 0.710 | 0.00 | 5.024 | 0.00 | 35.566 | 0.16 | 251.785 | 0.00 | 1782.502 | 0.00 |
| | 0.112 | 0.00 | 0.796 | 0.00 | 5.637 | 0.00 | 39.905 | 1.07 | 282.508 | 0.00 | 2000.000 | |
| | 0.126 | 0.00 | 0.893 | 0.00 | 6.325 | 0.00 | 44.774 | 3.20 | 316.979 | 0.00 | | |
| | 0.142 | | 1.002 | | 7.096 | | 50.238 | | 355.656 | | | |

Operator notes:

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

Malvern Instruments Ltd.
Malvern, UK
Tel := +[441 (0) 1684-892456 Fax +[441 (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





Sample Name:

NiCladWC->80 um-1.1-Average

Sample Source & type:

Sample bulk lot ref:

SOP Name:

Measured by:

D3M966

Monday, August 21, 2017 5:05:40 PM

Analysed:

Thursday, August 31, 2017 4:54:58 PM

Result Source: Averaged

Particle Name: **Accessory Name:** Tungsten powder Hydro 2000G (A) Particle RI: Absorption: 0.1

um

2.240 **Dispersant Name:** Dispersant RI:

Water Concentration:

%Vol

m²/g

84.689

Specific Surface Area:

0.1428

0.0531

d(0.1):

1.330

Span: 0.660

Surface Weighted Mean D[3,2]:

d(0.5):

112.980 um

Analysis model: General purpose

Size range:

to 2000.000 0.020 um

0.540

Sensitivity: Normal

Obscuration: 8.56

Result units:

Volume

Weighted Residual: Result Emulation:

d(0.9):

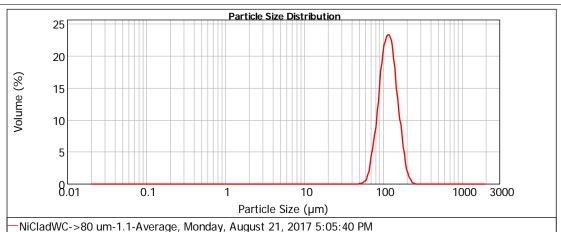
Uniformity: 0.207

Vol. Weighted Mean D[4,3]:

120.188

um

161.639



116.543

| 0.02 0.02 0.02 0.03 0.03 0.04 0.04 0.05 0.05 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | Size (µm) 0.142 0.159 0.178 0.200 0.224 0.252 0.283 0.317 0.356 0.399 0.448 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | Size (µm) 1.002 1.125 1.262 1.416 1.589 1.783 2.000 2.244 2.518 2.825 3.170 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | Size (µm) 7.096 7.962 8.934 10.024 11.247 12.619 14.159 15.887 17.825 20.000 22.440 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | Size (µm) 50.238 56.368 63.246 70.963 79.621 89.337 100.237 112.468 126.191 141.589 | Volume In % 0.03 0.30 1.59 4.63 7.82 13.49 16.68 17.61 15.91 10.69 7.07 | Size (µm) 355.656 399.052 447.744 502.377 563.677 632.456 709.627 796.214 893.367 1002.374 | Volume In % 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0 |
|--|---|---|--|---|--|---|--|---|--|--|---|
| 0.03 0.04 0.04 0.05 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | 0.252 0.283 0.317 0.356 0.399 | 0.00 0.00 0.00 0.00 0.00 | 1.783 2.000 2.244 2.518 2.825 | 0.00 0.00 0.00 0.00 0.00 | 12.619 14.159 15.887 17.825 20.000 | 0.00 0.00 0.00 0.00 0.00 | 89.337 100.237 112.468 126.191 141.589 | 13.49 16.68 17.61 15.91 10.69 | 632.456 709.627 796.214 893.367 1002.374 | 0.00 0.00 0.00 0.00 0.00 |

Operator notes:

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

Malvern Instruments Ltd. 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





Sample Name:

NiCladWC->80 um-1.2-Average

Sample Source & type:

Sample bulk lot ref:

SOP Name:

Measured by: D3M966

Result Source: Averaged

Monday, August 21, 2017 5:07:42 PM

Analysed:

Thursday, August 31, 2017 4:56:05 PM

Sensitivity:

Obscuration:

Result units:

Volume

Result Emulation:

Normal

8.93

um

Particle Name: Tungsten powder Particle RI: 2.240

Dispersant Name: Water

Concentration: 0.1491 %Vol

Specific Surface Area: 0.0532 m²/g

Accessory Name: Hydro 2000G (A) Absorption: 0.1

Dispersant RI:

1.330

Span: 0.661

Surface Weighted Mean D[3,2]: 112.845 um

Analysis model: General purpose Size range:

to 2000.000 0.020 Weighted Residual:

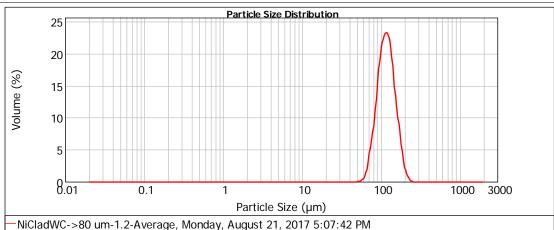
0.557

Uniformity: 0.207

Vol. Weighted Mean D[4,3]:

120.051

d(0.1): 84.564 d(0.5): 116.406 d(0.9): 161.469 um



Operator notes:

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

Malvern Instruments Ltd. 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





Sample Name:

NiCladWC->80 um-1.3-Average

Sample Source & type:

Sample bulk lot ref:

SOP Name:

Measured by: D3M966

Result Source: Averaged

Monday, August 21, 2017 5:09:37 PM

Analysed:

Thursday, August 31, 2017 4:56:50 PM

to 2000.000

Sensitivity:

Obscuration:

Result units:

161.450

um

Volume

Result Emulation:

Normal

8.88

um

d(0.9):

Particle Name: Tungsten powder Particle RI: 2.240

84.590

Dispersant Name: Water

Concentration: 0.1483 %Vol

Specific Surface Area: 0.0532 m²/q

d(0.1):

Accessory Name: Hydro 2000G (A) Absorption:

Dispersant RI: 1.330

0.1

Span: 0.660

Surface Weighted Mean D[3,2]: 112.862 um

d(0.5):

Analysis model: General purpose Size range:

0.020 Weighted Residual:

0.541

Uniformity: 0.207

Vol. Weighted Mean D[4,3]: 120.059

Particle Size Distribution 25 20 15

Particle Size (µm)

116.425

/olume (%) 10 5 8.01 0.1 100 1000 3000

NiCladWC->80 um-1.3-Average, Monday, August 21, 2017 5:09:37 PM

Size (µm) Size (µm) Volume In % Size (µm) | Volume In % Volume In % 0.00 0.00 0.00 0.04 0.00 0.00 0.022 0.159 1.125 7.962 56.368 399.052 0.00 0.31 0.025 0.178 1.262 8.934 63.246 447.744 0.00 0.00 0.00 0.00 1.61 0.00 0.028 0.200 1.416 10.024 70.963 502.377 0.00 0.00 0.00 0.00 4.66 0.00 563.677 0.032 0.224 1.589 11.247 79.621 0.00 0.00 0.00 0.00 7.85 0.00 0.036 0.252 1.783 12.619 89.337 632.456 0.00 0.00 0.00 0.00 13.53 0.00 0.040 0.283 2.000 14.159 100.237 709.627 0.00 0.00 0.00 0.00 16.70 0.00 0.045 0.317 2.244 15.887 112.468 796.214 0.00 0.00 0.00 0.00 17.61 0.00 0.050 0.356 2.518 17.825 126,191 893,367 0.00 0.00 0.00 0.00 15.89 0.00 0.056 0.399 2 825 20.000 141 589 1002 374 0.00 0.00 10.65 0.00 1124.683 0.063 0.448 3.170 22,440 158.866 0.00 0.00 0.00 0.00 7.03 0.00 0.071 0.502 3.557 25.179 178.250 1261.915 0.00 0.00 0.00 0.00 3.08 0.00 0.080 200.000 1415.892 0.564 3.991 28.251 0.00 0.00 0.00 0.00 0.93 0.00 0.089 0.632 4.477 31.698 224.404 1588.656 0.00 0.00 0.00 0.00 0.12 0.00 0.100 0.710 5.024 35.566 251.785 1782.502 0.00 0.00 0.00 0.00 0.00 0.00 0.112 0.796 5.637 39.905 282.508 2000.000 0.00 0.00 0.00 0.00 0.00 0.126 0.893 6.325 44.774 316.979 0.00 355.656 50.238

Operator notes:

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

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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 Master-sizer 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.





Sample Name: 10003C-1.1 - Average

Sample Source & type: NIST

Sample bulk lot ref:

SOP Name:

Measured by:

Wednesday, June 14, 2017 6:17:16 PM

Analysed:

Wednesday, June 14, 2017 6:17:17 PM

Result Source: Averaged

D3M966

Particle Name: Glass beads (typical) Particle RI:

%Vol

m²/g

1.520

Concentration:

Dispersant Name:

Specific Surface Area:

Water

0.0448

0.0773

Accessory Name: Hydro 2000G (A) Absorption:

Dispersant RI:

1.330

Span:

0.661

Surface Weighted Mean D[3,2]: 31.699 um

Analysis model:

Single narrow mode (spherical)

Size range:

to 2000.000 0.020

Weighted Residual:

0.305

Uniformity: 0.209

Vol. Weighted Mean D[4,3]:

33.758

d(0.1):

23.411

um

d(0.5):

32.985

d(0.9):

45.209

Sensitivity:

Obscuration:

Result units:

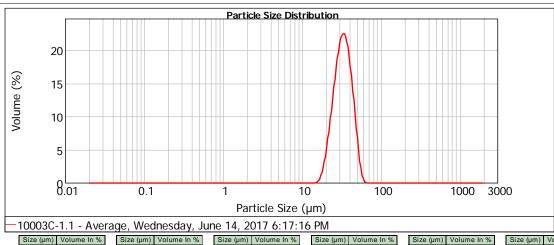
Volume

Result Emulation:

Enhanced

9.74

um



| Size (µm) | Volume In % | | Size (µm) | Volume In % |
|-----------|-------------|---|-----------|-------------|
| 0.020 | 0.00 | | 0.142 | 0.00 |
| 0.022 | 0.00 | | 0.159 | 0.00 |
| 0.025 | 0.00 | | 0.178 | 0.00 |
| 0.028 | 0.00 | | 0.200 | 0.00 |
| 0.032 | | | 0.224 | |
| 0.036 | 0.00 | | 0.252 | 0.00 |
| 0.040 | 0.00 | | 0.283 | 0.00 |
| 0.045 | 0.00 | | 0.203 | 0.00 |
| | 0.00 | | | 0.00 |
| 0.050 | 0.00 | | 0.356 | 0.00 |
| 0.056 | 0.00 | | 0.399 | 0.00 |
| 0.063 | 0.00 | | 0.448 | 0.00 |
| 0.071 | 0.00 | | 0.502 | 0.00 |
| 0.080 | | | 0.564 | |
| 0.089 | 0.00 | | 0.632 | 0.00 |
| 0.100 | 0.00 | | 0.710 | 0.00 |
| 0.112 | 0.00 | | 0.796 | 0.00 |
| 0.112 | 0.00 | | 0.796 | 0.00 |
| | 0.00 | | | 0.00 |
| 0.142 | | l | 1.002 | |

| JI. | IE 14, Z | 017 0.17. |
|-----|-----------|-------------|
| | Size (µm) | Volume In % |
| | 1.002 | 0.00 |
| | 1.125 | 0.00 |
| | 1.262 | 0.00 |
| | 1.416 | |
| | 1.589 | 0.00 |
| | 1.783 | 0.00 |
| | 2.000 | 0.00 |
| | 2.244 | 0.00 |
| | 2.518 | 0.00 |
| | 2.825 | 0.00 |
| | 3.170 | 0.00 |
| | 3.557 | 0.00 |
| | | 0.00 |
| | 3.991 | 0.00 |

4.477

5.024

5.637

6.325 7.096

| 6 | PM | | |
|---|-----------|-------------|--|
| | Size (µm) | Volume In % | |
| | 7.096 | 0.00 | |
| | 7.962 | 0.00 | |
| | 8.934 | 0.00 | |
| | 10.024 | 0.00 | |
| | 11.247 | 0.00 | |
| | 12.619 | 0.00 | |
| | 14.159 | 0.00 | |
| | 15.887 | 0.45 | |
| | 17.825 | 2.06 | |
| | 20.000 | 4.80 | |
| | 22 440 | 4.60 | |

12.53

15.75

17.03

15.79

12.21

251.785

282.508

316.979 355.656

25.179

28.251

31.698

35.566

39.905

44.774

50.238

| i | Size (µm) | Volume In % | |
|---|-----------|-------------|----|
| Ì | 50.238 | 2.95 | |
| | 56.368 | 0.35 | |
| | 63.246 | 0.00 | |
| | 70.963 | | |
| | 79.621 | 0.00 | |
| | 89.337 | 0.00 | |
| | 100.237 | 0.00 | |
| | 112.468 | 0.00 | |
| | 126.191 | 0.00 | |
| | 141.589 | 0.00 | ١. |
| | 158.866 | 0.00 | ١. |
| | | 0.00 | |
| | 178.250 | 0.00 | |
| | 200.000 | 0.00 | 1 |
| | 224.404 | 0.00 | 1 |

0.00

0.00

0.00

0.00

| Size (µm) | Volume In % |
|-----------|-------------|
| 355.656 | 0.00 |
| 399.052 | 0.00 |
| 447.744 | 0.00 |
| 502.377 | |
| 563.677 | 0.00 |
| 632.456 | 0.00 |
| 709.627 | 0.00 |
| 796.214 | 0.00 |
| 893.367 | 0.00 |
| 1002.374 | 0.00 |
| 1124.683 | 0.00 |
| 1261.915 | 0.00 |
| 1415.892 | 0.00 |
| | 0.00 |
| 1588.656 | 0.00 |
| 1782.502 | 0.00 |
| 2000.000 | |
| | |

Operator notes:

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Mastersizer 2000 Ver. 5.60 Serial Number : MAL1019545

0.00

0.00

0.00

0.00

File name: Standards LSL2-2015.mea Record Number: 52 9/7/2017 1:33:45 PM





Sample Name: 1003c-1.1 - Average

Sample Source & type:

Sample bulk lot ref:

Glass beads (typical)

Dispersant Name:

Concentration:

Particle Name:

Particle RI:

1.520

Water

0.0723

d(0.1):

Averaged

D3M966

SOP Name:

Measured by:

Result Source:

Accessory Name:

Hydro 2000G (A)

Dispersant RI:

Surface Weighted Mean D[3,2]:

um

1.330

Span:

31.128

0.667

Absorption:

Tuesday, August 22, 2017 6:32:08 PM

Analysed:

0.020

0.339

Uniformity: 0.209

Vol. Weighted Mean D[4,3]:

33.176

Analysis model:

Single narrow mode (spherical)

Size range:

to 2000.000 Weighted Residual:

Tuesday, August 22, 2017 6:32:07 PM

Result units:

Sensitivity:

Obscuration:

Result Emulation:

Enhanced

15.50

Volume

Specific Surface Area: 0.0787 m²/g

22.969

%Vol

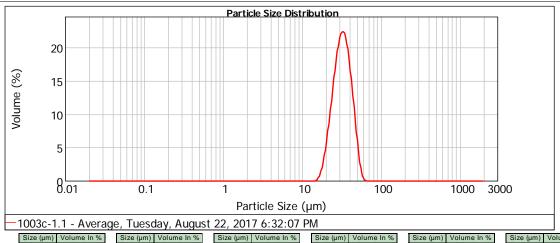
um

d(0.5): 32.369

d(0.9):

44.544

um



| 0.020 0.022 0.025 0.028 0.032 0.036 0.040 0.045 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 0.0 | 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 0.710 0.796 | Volume In % 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0 | 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 5.637 | 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0 | Size (µm) 7.096 7.962 8.934 10.024 11.247 12.619 14.159 15.887 17.825 20.000 22.440 25.179 28.251 31.698 39.905 | Volume In % 0.00 0.00 0.00 0.00 0.00 0.00 0.01 0.63 2.44 5.38 9.25 13.15 16.06 16.88 15.21 11.40 | \$\size (\text{\text{\text{wn}}}\) 50.238 56.368 63.246 63.246 70.963 79.621 89.337 100.237 112.468 126.191 141.889 158.866 178.250 200.000 224.404 251.785 282.508 | 2.54 0.27 0.00 0.00 0.00 0.00 0.00 0.00 0.00 | Size (µm) 355.656 399.052 447.744 502.377 563.677 632.456 709.627 796.214 893.367 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. |
|--|---|---|---|---|---|---|---|---|--|--|---|
| | 0.00 | | 0.00 0.00 0.00 | | 0.00 | | 15.21 11.40 6.79 | | 0.00 0.00 0.00 | | 0.00 |

Operator notes:

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





Sample Name: 1003C-3.2 - Average

SOP Name:

Wednesday, September 06, 2017 4:27:55 PM

Sample Source & type:

Measured by: D3M966

Analysed:

Analysis model:

Size range:

0.020

0.391

Wednesday, September 06, 2017 4:27:56 PM

Single narrow mode (spherical)

Sample bulk lot ref:

Result Source:

Averaged

Particle Name: Glass beads (typical)

Particle RI: 1.520

Concentration:

Dispersant Name:

Water

0.0441

0.0788

Accessory Name: Hydro 2000G (A) Absorption:

Dispersant RI:

1.330

Span:

0.614

Surface Weighted Mean D[3,2]:

31.071 um

Uniformity: 0.194

Weighted Residual:

Vol. Weighted Mean D[4,3]:

32.848 um

d(0.1):

23.410

%Vol

m²/g

Specific Surface Area:

d(0.5):

32.114

to 2000.000

43.123

Sensitivity:

Obscuration:

Result units:

Volume

Result Emulation:

Enhanced

9.78

um

d(0.9): Particle Size Distribution 25 20 Volume (%) 15 10 5 8.01 0.1 100 1000 3000 Particle Size (µm) 1003C-3.2 - Average, Wednesday, September 06, 2017 4:27:55 PM Volume In %

| Size (µm) | Volume In % | Size (µm) | Volume In % | Size (µm) | |
|-----------|-------------|-----------|-------------|-----------|--|
| 0.020 | 0.00 | 0.142 | 0.00 | 1.002 | |
| 0.022 | 0.00 | 0.159 | 0.00 | 1.125 | |
| 0.025 | 0.00 | 0.178 | 0.00 | 1.262 | |
| 0.028 | 0.00 | 0.200 | 0.00 | 1.416 | |
| 0.032 | 0.00 | 0.224 | 0.00 | 1.589 | |
| 0.036 | 0.00 | 0.252 | 0.00 | 1.783 | |
| 0.040 | 0.00 | 0.283 | 0.00 | 2.000 | |
| 0.045 | 0.00 | 0.317 | 0.00 | 2.244 | |
| 0.050 | | 0.356 | | 2.518 | |
| 0.056 | 0.00 | 0.399 | 0.00 | 2.825 | |
| 0.063 | 0.00 | 0.448 | 0.00 | 3.170 | |
| 0.071 | 0.00 | 0.502 | 0.00 | 3.557 | |
| 0.080 | 0.00 | 0.564 | 0.00 | 3.991 | |
| 0.089 | 0.00 | 0.632 | 0.00 | 4.477 | |
| 0.100 | 0.00 | 0.710 | 0.00 | 5.024 | |
| 0.112 | 0.00 | 0.796 | 0.00 | 5.637 | |
| 0.126 | 0.00 | 0.893 | 0.00 | 6.325 | |
| 0.142 | 0.00 | 1.002 | 0.00 | 7.096 | |

| Size (µm) | Volume In % | Size (µm) | ٧ |
|-----------|-------------|-----------|---|
| 1.002 | 0.00 | 7.096 | |
| 1.125 | 0.00 | 7.962 | |
| 1.262 | | 8.934 | |
| 1.416 | 0.00 | 10.024 | |
| 1.589 | 0.00 | 11.247 | |
| 1.783 | 0.00 | 12.619 | |
| 2.000 | 0.00 | 14.159 | |
| 2.244 | 0.00 | 15.887 | |
| 2.518 | 0.00 | 17.825 | |
| 2.825 | 0.00 | 20.000 | |
| 3.170 | 0.00 | 22.440 | |
| | 0.00 | | |
| 3.557 | 0.00 | 25.179 | |
| 3.991 | 0.00 | 28.251 | |
| 4.477 | 0.00 | 31.698 | |
| | | | |

0.00

0.00

0.00

44.774

50.238

| | | | | | | _ |
|----------|-------------|-----------|-------------|-----|-----------|---|
| ize (µm) | Volume In % | Size (µm) | Volume In % | | Size (µm) | Γ |
| 7.096 | 0.00 | 50.238 | 2.08 | | 355.656 | Ī |
| 7.962 | 0.00 | 56.368 | 0.00 | | 399.052 | ı |
| 8.934 | 0.00 | 63.246 | 0.00 | | 447.744 | ı |
| 10.024 | 0.00 | 70.963 | 0.00 | | 502.377 | ı |
| 11.247 | | 79.621 | | | 563.677 | ı |
| 12.619 | 0.00 | 89.337 | 0.00 | | 632.456 | ı |
| 14.159 | 0.00 | 100.237 | 0.00 | | 709.627 | ı |
| 15.887 | 0.00 | 112.468 | 0.00 | | 796.214 | ı |
| 17.825 | 0.33 | 126.191 | 0.00 | | 893.367 | ı |
| 20.000 | 1.88 | 141.589 | 0.00 | | 1002.374 | ı |
| 22,440 | 4.91 | 158.866 | 0.00 | | 1124.683 | ı |
| 25,179 | 9.31 | 178.250 | 0.00 | | 1261.915 | ı |
| 28.251 | 13.99 | 200.000 | 0.00 | | 1415.892 | ı |
| 31.698 | 17.50 | 224.404 | 0.00 | | 1588.656 | ı |
| 35.566 | 18.07 | 251.785 | 0.00 | | 1782.502 | l |
| 39.905 | 15.86 | 282.508 | 0.00 | | 2000.000 | ı |
| 00.000 | 10.80 | 202.000 | 0.00 | 1 1 | 2000.000 | ı |

316.979

355,656

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 0.00

0.00

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