

LABORATORY SCALE VITRIFICATION OF LOW LEVEL RADIOACTIVE NITRATE SALTS AND SOILS FROM THE IDAHO NATIONAL ENGINEERING LABORATORY*

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ABSTRACT

The Idaho National Engineering Laboratory (INEL) currently has radiologically contaminated nitrate salt and soil waste stored above and below ground in Pad A and the Acid Pit at the Radioactive Waste Management Complex. Pad A contains uranium and transuranic contaminated potassium and sodium nitrate salts generated during operations at the Rocky Flats Plant. The Acid Pit was used to dispose of liquids containing waste mineral acids, uranium, nitrate, chlorinated solvents, and some mercury. Some of these wastes may be mixed hazardous low-level radioactive waste and may require treatment. Ex situ vitrification is an effective treatment that destroys nitrates and organics and immobilizes hazardous and radioactive metals. Laboratory scale melting of actual radionuclides containing INEL Pad A nitrate salts and Acid Pit soils was performed using crucibles in a bench-top furnace that simulated ex situ vitrification treatment. High sodium and borosilicate glasses were produced from melting actual Pad A salts (15 to 50 wt%) and Acid Pit soils (40 to 80 wt%) together at 1550°C. Glass oxidation-reduction and handling properties were controlled by using additives such as boric acid and carbon (0 to 10%). The salt/soil/additive ratios were varied to determine the range of glass compositions; maximize mass and volume reduction, durability, and immobilization of hazardous and radioactive metals; and minimize viscosity and off-gas generation for wastes prevalent at INEL and other U.S. Department of Energy sites. The resulting wasteforms after thermal processing occupied less volume (28 to 76% less) and mass (up to 42% less) than the wastes. Some mixtures were spiked with additional hazardous (0.7% lead) and radioactive (50 pCi/g ²³⁴U, 100 pCi/g ²⁴²Pu, 100 pCi/g ²⁴¹Am) metals. Representative glasses passed the Toxicity Characterization Leaching Procedure. No nitrate was found in TCLP leachates. Samples spiked with transuranic showed low nuclide leaching. Wasteform densities were 2.4 g/cc compared to 0.77 (salt) and 1.57 (soil). Recommended salt-to-soil ratio is 0.43 with a maximum alkali content of 25%. Thermally coprocessing soils and salts is an effective remediation method for destroying nitrate salts while stabilizing the radiological and hazardous metals they contain. The measured durability of these low-level waste glasses approached those of high-level waste glasses. Lab scale vitrification of actual INEL contaminated salts and soils was performed at General Atomics Laboratory as part of the INEL Waste Technology Development and Environmental Restoration within the Buried Waste Integrated Demonstration Program.

INTRODUCTION

The Idaho National Engineering Laboratory (INEL) Radioactive Waste Management Complex (RWMC) currently contains radiologically contaminated waste including about 4% of the nitrate salt waste stored at U.S. Department of Energy (DOE) defense facilities. (1) Many of these salts are on Pad A. (2) Soils containing radionuclides and heavy metals present at these facilities are typified by those at the Acid Pit. (3)

Pad A is a ground-level asphalt pad constructed for the aboveground disposal of radioactive contaminated wastes generated by the DOE Rocky Flats Plant (RFP) between 1972 and 1978. (2) These wastes are composed primarily of nitrate

salts, contain less than 10 nCi/g of transuranic radionuclides, and exhibit a dose rate of less than 200 mR/h at the surface of each container. (2) The salt waste is composed of about 90% sodium and potassium nitrate, with the balance of sodium and potassium sulfates, chlorides, hydroxides, and above background trace concentrations of chromium.

The Acid Pit contains liquid wastes that are primarily uranium-contaminated acids and chlorinated solvents generated by the INEL between 1954 and 1965. (3) The liquid wastes have been neutralized among layers of soil and lime. Extensive borehole sampling in 1991 showed some samples with above background concentrations of ²³⁸U, ¹³⁷Cs, transuranic, mercury, and chromium.

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Vitrification is a process that produces glassy solids from wastes, and glass forms by melting the materials at high temperature. Off-gases from waste vitrification processes are treated to remove acids, particulates, and other potentially hazardous constituents. Vitrification has been used for a wide range of waste types, from high-level radioactive waste (ex situ) to contaminated soil still in the ground (in situ). (1,4) Ex situ vitrification, using joule heating, was the selected method for the coprocessing of the soil and salt wastes described in this paper. (4)

Experimental Description

To attain anticipated vitrification temperatures of 1500 to 1600°C for testing melt mixtures, a high temperature furnace was used. The melt furnace, a modified Deltech, has working space within the melting unit (closed) of 24×24×28 cm in height and is capable of a maximum temperature of 1800°C. A Blue M muffle furnace was used as an annealing oven and maintained at 660°C to relieve cast glass stress. Crucibles used in these experiments were of the fused silica type. These crucibles are highly resistant to thermal shock and are preferred for charging of glass batches and have a service temperature of 1600°C.

Soil was prepared by screening out the +25 mesh fraction (greater than 0.7 mm) and grinding the stones and pebbles in a Wiley mill equipped with a 10-mesh screen. The ground material was recombined with the -25 mesh fraction, and samples were split from the batches for each melt. Nonwaste additives used were technical or reagent grade sand (SiO₂), limestone (CaCO₃), boric acid (H₃BO₃), fluor spar (CaF₂), carbon (activated charcoal, flour or lamp black), lead (Pb), zinc oxide (ZnO) and titanium oxide (TiO₂). Two milliliters of the radionuclide spike standard containing 16,050 pCi/ml of ²⁴²Pu; 15,922 pCi/ml of ²⁴¹Am; and 8,282 pCi/ml of ²³⁴U were added to three batches.

Each melt of the wastes used the same procedure. Personnel calculated the required salt/soil/additive amounts. Prepared raw materials were weighed out and homogenized by mixing, splitting, and sieving. The batch was weighed and packed into a tared ceramic crucible. A small amount of water (10 ml) was added to each batch during mixing to avoid segregation when the batch was poured into the crucible.

The melt furnace was heated to 1500–1575°C, opened, and the crucible inserted. Each batch was held in the furnace for about 60 minutes. The crucible was then removed from the furnace, and the molten glass poured into a graphite mold. During pouring, the senior glass technologist estimated the glass viscosity by eye. The color, presence of a separate metallic phase, or other inhomogeneities were noted. Each piece of poured glass was allowed to solidify in the mold for about 5 minutes before it was transferred to the annealing furnace at 660°C for at least one half hour, then cooled slowly.

The cooled glass was weighed, viewed under the microscope, photographed, and archived. To provide data for glass density calculation, a piece from each of the glass pours was weighed separately, and its volume measured by displacement of sand. Since the inside of each crucible was coated with glass after pouring, the weight of the crucible was also determined to allow calculation of the percent of the glass that poured out of the crucible. The appearance of the crucible was observed, noting the presence of corrosion, foamover, slag, or a separate metallic phase.

Compatibility of candidate electrode materials, molybdenum, chromium, and graphite was tested during a glass melt of medium (0.43) salt-to-soil ratio and 21% alkali. Candidate electrode material coupons were measured, weighed, and covered by the placed raw material mix in the crucibles. The batches were melted at 1500°C for 4 hours, maintained at 1000°C for 13 hours, raised to 1500°C for an additional 4 hours, and removed from the furnace, annealed, and cooled. The coupons were cleaned, weighed, measured, and described visually as to the physical condition of the coupons. Molybdenum was more resistant than chrome with no apparent corrosion or corrosion related weight loss. Graphite electrodes completely oxidized and no conclusions are possible due to inappropriate test conditions.

Two types of leach testing were performed: Environmental Protection Agency (EPA) Method 1311 Toxicity Characteristic Leach Procedure (TCLP) tests on lead and transuranic spiked glass samples and Nuclear Regulatory Commission (NRC) Product Characterization Test (PCT) leach tests on glass forms with a wide range of alkali contents. Glass samples were crushed for leach testing. A particle size of <9.5 mm is required for the TCLP and <0.2 mm for the PCT. Room temperature was used for 24 hours TCLP leaching, and the temperature was raised to 90°C for 7 days in the PCT. The TCLP method requires a high pH sodium acetate leach solution, and the PCT uses distilled water.

Characterization of salts, soils, wasteforms, and leachates was completed by a variety of techniques. Atomic Absorption (AA) was used for mercury, potassium, and sodium. Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) was used for all metals but mercury. Instrumental Neutron Activation Analysis (INAA) was used for potassium, sodium, and TCLP metals except lead. Alpha spectrometry was used for salt and wasteform concentrations of ²³⁹Pu, ²³⁸Pu, ²⁴¹Am, ²³⁴U, and ²³⁸U. Gamma spectrometry was used for determining uranium, thorium, americium, and potassium in salts, soils, and wasteforms. Anion concentrations in the soluble portion of the salts and soils and verifying destruction of nitrate was done by Ion Chromatography (IC). Gravimetric determinations include waste and wasteform, volume, mass, density, and moisture. These results are used to determine waste minimization. Leachate results help establish long term durability, and processability is determined from apparent viscosity and operational observations. An actual joule melter with a cold cap is required to determine destruction of nitrous gases and metals volatilization.

Results and Discussion

Thirty-four glass wasteforms were prepared using the above procedure. Fifteen samples were prepared with surrogate salts and sand or INEL soil. Nineteen samples were prepared from melting actual Pad A salts (15 to 50 wt%) and Acid Pit soils (40 to 80 wt%) together at 1550°C. Ten were leach tested. Six high alkali (40 to 60% salt, 22 to 39% alkali) and 19 glasses of a normally more durable type (6% borosilicate, 5 to 15% calcium or 65-70% silicon) were produced.

The color of the glass produced from this waste was black-green to very dark black somewhat like iron rich basalt. Glass color depends on how reduced the melt was primarily by type and amount of carbon added to the batch. Less reduced glasses were green, more reduced were black. Soil mineral matter was incorporated in the melt with metal phases. Some slag (metallic iron) was apparent in reduced

melts. Devitrification (crystallization) in some melts with ZnO_2 and TiO_2 was noted.

Both glass oxidation-reduction and handling properties were controlled by using additives such as boric acid, fluor-spar and carbon (0 to 10%). Adding carbon reduced the nitrate, decreased foaming and probably nitrous gas emissions and greatly reduced crucible attack. Viscosities were influenced most by total alkali and boron content. Melt formulations with salt contents below 17% were not pourable. Salt contents of 17 to 22% were at the limit of processing with viscosities near 700 to 900. Borosilicate glasses gave workable viscosities with low salt (<20%) loadings. Normally more durable glasses with other additives to increase calcium and silicon but lower sodium were difficult to pour. The very high alkali and high alkali glasses had some of the lowest viscosities and were easily processed.

Melting reduced the volume of the wastes by eliminating void volume and removing nitrates, water, and carbon with final vitrified densities averaging 2.30 ± 0.5 compared to bulk density of the soil at 1.57 and salt at 0.77. The volume and mass reductions for each melt were measured using masses and bulk densities of both input salts and soils and the final glass wasteform. Other properties measured that had bearing on volume reduction were moisture content, total organic carbon, and loss on ignition.

High amounts of salts, 25 to 35%, gave the greatest volume and mass reduction. These high alkali glasses resulted in mass reductions up to 41% and 77% in volume. A more durable glass with less alkali but no additives had a salt/soil ratio of 0.43, reduced mass up to 27%, and volume of 68%. Borosilicate glasses actually showed slight mass increases with volume reductions up to 56%. Durable glasses with other additives reduced the mass up to 19% and reduced volume up to 63%. High silicon (sand) and calcium or boron additives gave the most durable waste product. A highly durable glass that passed the PCT test had a silicon dioxide content of 64 wt%. Ideally for soda lime glasses a 65 to 70% is desirable. Glasses with 59% silicon dioxide passed TCLP. Glasses with alkali contents above 26% and silicon dioxide below 55% gave an observable increase in silicon leaching but still may be acceptable for LLW.

Pad A nitrate salt, Acid Pit soil, and the final glass waste form characterization analysis results are given in Table I. Three melts using INEL salt and soil were spiked and tested using the TCLP. These melts were spiked with lead, uranium, plutonium, and americium to provide a higher source concentration than is found in the waste and soil. The leachate passed the TCLP toxic metals test. Leachates results are listed in Table II. Results from elemental and nitrate analysis of the TCLP leachates are also presented in Table II. Potassium was detected in the leachate indicating some sample breakdown. Trace arsenic and chromium metal were detected, but all TCLP metals were at least 50% below limits.

Radionuclide content of the salts, soils, and wasteforms are summarized in Table III. Both nondestructive large sample (>50 g) gamma spectrometry and destructive small sample (<5 g) alpha spectrometry were used. Results are fairly consistent considering the differences in sample size and sampling and analytical techniques. Apparent losses of spiked ^{234}U and ^{241}Am probably result from analytical error because of the higher density of the glass than either soil or salt. The ^{241}Am is detected by a lower energy gamma than the uranium

and is more attenuated when there is a density difference between the standard and sample.

Radionuclide leaching results are summarized in Table II. The TCLP leachates were analyzed for gross alpha (proportional counting) and gamma spectrometry. Alpha radionuclide content leaching from the spiked wasteforms was low and similar to the alpha and gamma spectroscopy results. Gross alpha measurement was performed on the leachates; total activity (transuranic) of the leachates was very low and gave a similar leach index over 10 times lower than the tightly bound chromium. Gamma counting was performed on the leachates, and no gamma emitters were detected including the spiked ^{241}Am . The leaching behavior of ^{241}Am seemed similar to other heavy metals.

Six melts with alkali (sodium and potassium) oxide contents ranging from 15 to 39% were tested with a high-level waste PCT. The results are summarized in Table IV. Leachate was measured for material loss (aluminum, boron, barium, calcium, cadmium, titanium, nickel, chromium, iron, potassium, magnesium, manganese, sodium, phosphorous, silicon, and titanium) by ICP-AES. Table IV provides most structural elements leached in proportion to the alkali content in the wasteform. Metals leaching was significant in high alkali wasteforms but low in durable wastes. Only the most durable wasteform tested with 23% salt and 49% soil (15% alkali oxide, 6% boron oxide, 7% calcium oxide, 9% aluminum oxide, and 59% silicon oxide) "passed" the PCT, that is it is similar in leaching to a high-level waste glass.

Quality control was exercised in sample handling, sample melting, and wasteform testing. The waste to be processed was homogenized extensively. The melt temperature was controlled within 1%. The entire procedure was reproducible as evidenced by the similarity of viscosities and final densities of replicate batches [5% relative percent difference (RPD) in densities for similar melts]. Experimental variations were most pronounced in glass pouring.

Analytical results were verified through use of different analytical techniques, spikes, duplicates, National Institute of Standards and Technology (NIST) standards, interlaboratory comparisons, and mass balance. For example, the potassium content in Pad A salt was measured by ICP, INAA, AAS, and gamma counting at three different laboratories. Results agreed within the limits of the sample inhomogeneity, which was about 25%. NIST standards were used in the ICP analysis of PCT leachates and glass wasteforms with an average 6% RPD. Most measurements on the salt and soil wastes were performed twice on different samples to ensure validity of data, analytical method, and homogeneity of samples. Generally, the largest uncertainty in analysis is in the sampling and sample inhomogeneity rather than in the specific analytical technique.

CONCLUSIONS AND RECOMMENDATIONS

This laboratory-scale test simulated ex situ vitrification treatment of actual radionuclide contaminated INEL Pad A nitrate salts and soils from the Acid Pit. Salt/soil coprocessing was studied in three areas: waste processability, waste form durability, and waste minimization. Processability was measured by qualitative viscosity measurement, crucible and electrode corrosion, and off-gas and reactivity observations. Durability was determined by EPA TCLP and NRC PCT leach testing of high alkali, low alkali, toxic metal, and radionuclides spiked wasteforms. Waste minimization was

TABLE I
Composition of Pad A Salt, Acid Pit Soil, and Final Glass Wasteform

Element	Pad A Salt	Acid Pit Soil	Vitrified Wasteform (35% Salt)
	Concentration in ppm		
Arsenic	< 15 ^a	< 9.2 ^a	< 7 ^a
Barium	< 75 ^a	1315 ± 25% ^b	490 ^a
Cadmium	< 10 ^c	< 4.6 ^a	< 22 ^a
Chromium	325 ± 32% ^c	39 ± 25% ^b	180 ± 4% ^a
Mercury	< 0.5 ^d	18 ^a	< 1 ^a
Selenium	< 3.4 ^a	< 2.8 ^d	< 3 ^a
Silver	< 1.4 ^a	< 2.2 ^d	< 2 ^a
Lead	< 150 ^b	20 ^b	< 1000 ^c
Sodium	200,000 ^a ± 0.5%	10,300 ± 0.5% ^a	91,300 ± 0.5% ^a
Potassium	75,600 ^a ± 24%	20,400 ± 14% ^a	47,000 ± 18% ^a
Silicon	340 ^f	330,000 ± 3% ^{bc}	249,000 ± 3% ^c
Iron	640 ^f	16,900 ± 3% ^{bc}	14,900 ± 18% ^c
Aluminum	880 ± 4% ^d	53,600 ± 4% ^{bc}	44,700 ± 4% ^c
Magnesium	20 ^f	6,600 ± 25% ^b	4,200 ± 37% ^c
Calcium	2,610 ± 13% ^d	1,740 ± 4% ^{bc}	8,130 ± 10% ^c
Lithium	1,000 ^f	NA ^g	NA
Nitrate (NO ₃)	549,000 ± 7% ^h	2,000 ± 50% ^h	< 0.1 ⁱ
Chloride	20,900 ± 43% ^h	NA	NA
Fluoride	6,080 ± 57% ^h	NA	NA
Sulfate (SO ₄)	36,000 ± 51% ^h	2,000 ± 50% ^h	< 1,000 ⁱ
Phosphate (PO ₄)	13,720 ± 33% ^h	2,000 ± 25% ^h	10,000 ± 21% ^c
Carbon	9640 ± 18% ^j	24,500 ± 20% ^j	< 0.1 ⁱ
Water	18,000 ± 5% ^k	110,000 ± 5% ^k	< 0.1 ⁱ

- a. Instrumental Neutron Activation Analysis on actual Pad A salt, Acid Pit soil, and wasteform from test; uncertainty is analytical counting.
- b. ICP analysis on a similar soil adjacent to the core section used in test; uncertainty includes analytical and sample inhomogeneity.
- c. Inductively Coupled Plasma analysis on actual salt used in test; uncertainty is analytical and sampling.
- d. Activation Analysis on actual Pad A salt and an adjacent soil; uncertainty includes analytical and sample inhomogeneity.
- e. ICP analysis of 28% salt wasteform, uncertainty is analytical and sampling.
- f. AA and Emission Spectrometry on a grab sample of feed for salt; uncertainty unknown.
- g. NA = Not Attempted.
- h. Ion Chromatography analysis of dissolved Pad A salt and dissolved adjacent soil; uncertainty is analytical and sampling.
- i. Assumed concentration after high temperature vitrification based on compound destruction, gaseous release, and solubility in glass.
- j. Total Organic Carbon analysis of dissolved Pad A salt and Loss of Ignition of adjacent soil; uncertainty is analytical and sampling.
- k. Gravimetric analysis of Pad A salt and similar adjacent soil; uncertainty is analytical and sampling

measured by the mass and volume reductions of the waste and final density increase.

The leachates contained no nitrates, passed TCLP, and had low matrix disintegration if the alkali content was under 25%. Waste minimization of up to 77% by volume and 41% by mass with wasteform densities of 2.4 g/cc were produced (compared to 0.77 and 1.57 for the original salt and soil). Destructive examination revealed some mass loss, primarily sodium and potassium. Off-gas was not analyzed so the conversion of nitrous oxide to nitrogen is unknown. Melt formulations using carbon and boron and calcium minimized sample foaming and corrosion, yet produced glass with viscosity low enough for commercial melter operation. Future pilot-scale

testing should start with a salt-to-soil ratio of 0.43, an alkali content of no more than 25%, sufficient carbon to chemically reduce the available nitrate, 4 to 7%, and 2 to 3% boron.

Coprocessing of soils and salts is an effective remediation method for destroying nitrate salts while stabilizing the radiologically contaminated salts. The test provided the range of salt/soil/additive compositions that can be efficiently processed by a high temperature melter that will fix radiological contaminants, destroy nitrates, and minimize overall volume and mass.

These data will be applicable to other buried wastes, residuals, and soils at the RWMC, and other DOE sites containing nitrate salts, sludges, and contaminated soils.

TABLE II
Metals and Radionuclides Leached From a 35% Pad a Salt
Glass Wasteform During TCLP Test

Metal	TCLP (ppm)	Leachate Concentration (ppm)	Weight Fraction Leached
Arsenic	5.0	0.02	0.07
Barium	100.0	< 0.7	< 0.02
Cadmium	1.0	< 0.1	< 0.1
Chromium	5.0	0.05	0.005
Mercury	0.2	< 0.01	< 0.1
Selenium	1.0	< 0.03	< 0.2
Silver	5.0	< 0.02	< 0.2
Lead ^a	5.0	< 1	< 0.1
Potassium	-	1.46	0.7
Americium ²⁴¹ Am	4 ^b	< 0.44	< 0.08
pCi/ml ^c			
Total Alpha pCi/ml ^d	5 ^b	0.017	0.0004

a. Spiked to 0.7 wt% with lead shavings
b. NRC 10 CFR 61 limit for release to surface water.
c. Spiked to 100 pCi/g with standard ²⁴¹Am solution.
d. Spiked to 260 pCi/g alpha with standard ²⁴¹Am, ²⁴²Pu and ²³⁴U solution.

TABLE III
Radionuclide Content of Pad A Salt, Acid Pit Soil, and Glass Wasteforms

Radionuclide	Activity in pCi/g		Wasteform 35% Salt	Wasteform Theoretical
	Pad A Salt	Acid Pit Soil		
²³⁸ U, ²³⁴ U	58±11% ^a	18±9% ^a	46±20% ^{bc}	33
²³⁸ U, ²³⁴ U ^d			75±33% ^e	88
²³⁵ U ^b	1.5±12%	2.6±17%	1.9±12%	2.1
Th	0.7±6% ^e	1.3±12% ^e	1.1±21% ^{bc}	1.1
²³⁸ Pu	0.42±32% ^c	< 10 ^b	4±50% ^c	< 8
²³⁹ Pu	0.27±19% ^c	< 100 ^c	0.5±50% ^c	< 60
²⁴¹ Am	0.28±13% ^{cd}	< 0.2 ^c	0.2±20% ^c	< 0.23
²⁴¹ Am ^d			72±4% ^d	106
¹³⁷ Cs ^b	0.1±9%	< 0.1	0.14±10%	< 0.1
⁴⁰ K ^b	86±2%	20±3%	44±6%	44

a. Gamma Spectrometry; nondestructive, average based on daughters ²²⁶Ra and ²³⁴Th; uncertainty is analytical.
b. Gamma Spectrometry; nondestructive; uncertainty is analytical and sample inhomogeneity.
c. Alpha Spectrometry destructive; uncertainty is analytical and sample inhomogeneity.
d. Spike added as liquid to salt/soil mixture, wasteform as analyzed by Gamma Spectrometry; nondestructive uncertainty analytical and sample inhomogeneity.
e. Gamma Spectrometry; nondestructive, average based on daughters ²¹²Pb, ²⁰⁸Tl, and ²²⁸Ac; and uncertainty is analytical.

TABLE IV
Metal Leaching From Glass Wasteforms During PCT Test

% Pad A Salt	41		28		23	
Metal	Leachate Conc. (ppm)	Weight Fraction Leached	Leachate Conc. (ppm)	Weight Fraction Leached	Leachate Conc. (ppm)	Weight Fraction Leached
Aluminum	33.7	0.013	9.7	0.0035	2.8	0.0013
Boron	0.1	0.0019	<0.1	<0.003	2.4	0.0028
Barium	<0.01	<0.0004	0.014	0.00035	<0.01	<0.0003
Calcium	2.1	0.0047	0.8	0.0015	3.0	0.0015
Chromium	0.5	0.059	<0.1	<0.01	<0.1	<0.01
Iron	1.1	0.0018	0.88	0.00094	<1	<0.001
Potassium	125	0.042	14	0.0048	6	0.0025
Magnesium	<0.5	<0.004	0.3	0.0011	<0.5	<0.005
Manganese	<0.02	<0.001	0.02	0.00067	<0.02	<0.001
Sodium	585	0.095	59	0.0135	16	0.0050
Silicon	187	0.014	53	0.0039	29	0.0022
Titanium	<0.1	<0.001	0.13	0.00083	<0.1	<0.001

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