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October 13, 2016

To: E. J. Freed, 704-S

From: C. L. Crawford, 773-42A

Results for the Third Quarter Calendar Year 2016 Tank 50H Salt Solution Sample

Approved by: _____
A. D. Cozzi, Technical Reviewer, per E7, 2.60 _____ Date

E. N. Hoffman, EPD Manager _____ Date

SUMMARY

In this memorandum, the chemical and radionuclide contaminant results from the Third Quarter Calendar Year 2016 (CY16) sample of Tank 50H salt solution are presented in tabulated form. The Third Quarter CY16 Tank 50H samples (a 200 mL sample obtained 6" below the surface (HTF-5-16-63) and a 1 L sample obtained 66" from the tank bottom (HTF-50-16-64)) were obtained on July 14, 2016 and received at Savannah River National Laboratory (SRNL) on the same day.¹ Prior to obtaining the samples from Tank 50H, a single pump was run at least 4.4 hours and the samples were pulled immediately after pump shut down.¹ The information from this characterization will be used by Defense Waste Processing Facility (DWPF) & Saltstone Facility Engineering for the transfer of aqueous waste from Tank 50H to the Saltstone Production Facility, where the waste will be treated and disposed of in the Saltstone Disposal Facility. This memorandum compares results, where applicable, to Saltstone Waste Acceptance Criteria (WAC) limits and targets.² Data pertaining to the regulatory limits for Resource Conservation and Recovery Act (RCRA) metals will be documented at a later time per the Task Technical and Quality Assurance Plan (TTQAP) for the Tank 50H saltstone task.³ The chemical and radionuclide contaminant results from the characterization of the Third Quarter CY16 sampling of Tank 50H were requested by Savannah River Remediation (SRR) personnel⁴ and details of the testing are presented in the SRNL TTQAP.⁵ This memorandum is part of Deliverable 2 from SRR request.⁴

The following facts pertaining to the WAC are drawn from the analytical results provided in this memorandum:

- WAC targets or limits were met for all analyzed chemical and radioactive contaminants for which the detection limits are below the WAC targets or limits.
- Norpar 13 and Isopar L have higher detection limits⁶ compared with the Saltstone WAC. The data provided in this memorandum is based upon the Norpar 13 and Isopar L concentrations from the sample obtained 6" below the surface. Due to the limited solubility of these materials in aqueous solution and the limited mixing of the tank by a single pump for 4.4 hours before sampling, Norpar 13 and Isopar L concentrations may not represent the overall concentrations of the analytes in Tank 50H.
- Minimum detection limits are reported for ⁹⁴Nb, ²⁴⁷Cm, ²⁴⁹Cf, and ²⁵¹Cf as determined from the minimum detectable activity associated with the radiochemical methods used for these radionuclides. The reported detection limits are above the requested SRR target minimum detection limit concentrations.⁷ However, the reported minimum detection limits reported for the Third Quarter CY16 Tank 50H sample for these four radionuclides are all lower than the estimated detection limits initially established by SRNL in 2009.⁸ Thus per guidance from SRR,⁷ SRNL continues to achieve as low as practical detection limits for these radionuclides.
- The measured values for ⁹⁰Sr and the plutonium isotopes of ²³⁸Pu, ²³⁹Pu and ²⁴⁰Pu for the Third Quarter CY16 Tank 50H sample are higher than previous First Quarter CY16 Tank 50H sample⁹ by 84X, 64X, 46X and 46X, respectively. However they are still below the WAC Limit (⁹⁰Sr) and WAC Targets (²³⁸Pu, ²³⁹Pu and ²⁴⁰Pu). This increase is due to the discontinued use of monosodium titanate (MST) in the actinide removal process (ARP) near the start of 2016.

TABLES CONTAINING RESULTS

Unless otherwise stated, all of the concentrations presented in the tables (except upper limits) are averages based on triplicate analyses of the Third Quarter CY16 Tank 50H samples. The standard deviation of each average is also presented. Several of the contaminants were either not detected in the slurry samples or detected at values below the method reporting limit (MRL). For contaminants not detected or detected below the MRL, the result is preceded by a "<", which indicates the result is an upper limit based on the sensitivity of the method used to analyze the individual analyte. If only one value out of the triplicate analysis is above the detection limit, then that single value is reported and noted in the tables. Also, if only two values out of the triplicate analysis are above the detection limit, then the average of those two values is reported and noted in the tables. Data reported for atomic absorption (AA), cold-vapor atomic absorption (CVAA), inductively coupled plasma emission spectroscopy (ICP-ES) and inductively coupled plasma mass spectroscopy (ICP-MS) are derived from the digested Tank 50H supernates by the aqua regia method. All analytical methods shown by the acronyms in the tables for this memorandum have been previously defined in the TTQAP.⁵

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Mercury (Hg) speciation data shown in Table 1, Table 2 and Table 5 are taken from previous work.¹⁰ These species include elemental Hg (Hg(0)), monomethyl mercury, ethyl mercury and dimethyl mercury. The concentration values shown for monomethyl mercury, ethyl mercury and dimethyl mercury represent the concentrations of these organomercury species. They are calculated from the reported values for monomethyl mercury, ethyl mercury and dimethyl mercury on a 'mg Hg/L' basis from the Hg speciation data.¹⁰ As a sample calculation, from reference 10, the reported monomethyl concentration on a mg Hg/L basis is 57.5 mg Hg/L. This value is then multiplied by the formula weight of monomethyl mercury from the WAC² (215.62 g monomethyl mercury/mol) divided by the molecular weight of Hg (200.6 g Hg/mol). Thus the calculated concentration of the species monomethyl mercury is $57.5 \text{ mg Hg/L} \times (215.62 \text{ g monomethyl mercury/mol} / 200.6 \text{ g Hg/mol}) = 61.8 \text{ mg monomethyl mercury/L}$.

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Table 1. Chemical Contaminants from Third Quarter CY16 Tank 50H Samples and Saltstone WAC, Revision 16, Attachment 8.1 Limits²

<u>Chemical Name (Formula)</u>	<u>Method</u>	<u>Average Concentration (mg/L)</u>	<u>Std. Dev.</u>	<u>WAC Limit (mg/L)</u>
Aluminate (Al(OH) ₄ ⁻)	ICP-ES	1.34E+04 ^a	5.04E+01	4.08E+05
Ammonium (NH ₄ ⁺)	IC	< 1.00E+01	NA	2.12E+02
Carbonate (CO ₃ ²⁻)	TIC	1.73E+04 ^b	5.77E+01	1.20E+05
Chloride (Cl ⁻)	IC	3.78E+02	2.52E+00	7.95E+03
Fluoride (F ⁻)	IC	< 1.00E+02	NA	4.07E+03
Free Hydroxide (OH ⁻)	Total Base	3.48E+04 ^b	1.96E+02	1.58E+05
Nitrate (NO ₃ ⁻)	IC	1.14E+05	1.15E+03	4.37E+05
Nitrite (NO ₂ ⁻)	IC	3.16E+04	2.08E+02	2.14E+05
Oxalate (C ₂ O ₄ ²⁻)	IC	3.99E+02	4.00E+00	2.72E+04
Phosphate (PO ₄ ³⁻)	IC	3.98E+02	2.21E+01	2.94E+04
Sulfate (SO ₄ ²⁻)	IC	5.52E+03	6.66E+01	5.69E+04
Arsenic (As)	AA	<1.12E-01	NA	2.30E+01
Barium (Ba)	ICP-ES	< 6.17E-01	NA	6.19E+02
Cadmium (Cd)	ICP-ES	< 2.63E+00	NA	3.10E+02
Chromium (Cr)	ICP-ES	5.62E+01	2.58E-01	1.24E+03
Lead (Pb)	ICP-MS	4.27E-01	1.23E-02	6.19E+02
Total Mercury (Hg)	CVAA	9.64E+01	1.52E+01	3.25E+02
Elemental Mercury (Hg(0))	CVAFS	2.52E+00	1.08E-01	1.82E+01
Monomethyl Mercury (CH ₃ Hg)	CVAFS w/ Distillation	6.18E+01	1.93E+00	3.50E+02
Ethyl Mercury (C ₂ H ₅ Hg)	CVAFS w/ Distillation	<1.01E+00	NA	3.73E+02
Selenium (Se)	AA	<2.24E-01	NA	4.46E+02
Silver (Ag)	ICP-ES	<2.49E+00	NA	6.19E+02
Aluminum (Al)	ICP-ES	3.80E+03	1.43E+01	1.16E+05
Potassium (K)	AA	3.92E+02	2.58E+00	3.03E+04
n-Butanol (C ₄ H ₉ OH)	VOA	< 5.00E-01 ^c	NA	7.73E+00
i-Butanol (C ₄ H ₉ OH)	VOA	< 5.00E-01 ^c	NA	7.73E+00
i-Propanol (C ₃ H ₇ OH)	VOA	< 2.50E-01 ^c	NA	1.88E+00
Phenol (C ₆ H ₅ OH)	SVOA	< 1.00E+01 ^c	NA	7.50E+02
Isopar L (----)	SVOA	< 2.66E+01 ppm ^{c,d}	NA	1.10E+01 ppm
Total Organic Carbon (----)	TOC	2.77E+02 ^b	2.31E+00	5.00E+03
Tetraphenylborate [TPB anion] (B(C ₆ H ₅) ₄ ⁻)	HPLC	< 5.00E+00	NA	5.00E+00

- a. Result is calculated from the measured Al concentration assuming all the Al is present as the OH compound.
b. Measurement performed on filtered supernate samples.
c. Measurement performed on duplicate samples rather than triplicate samples.
d. Result is calculated from the reported concentration of < 33 mg/L and the density of the slurry sample listed in Table 8.

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Table 2. Chemical Contaminants from Third Quarter CY16 Tank 50H Samples and Saltstone WAC, Revision 16, Attachment 8.2 Targets²

<u>Chemical Name (Formula)</u>	<u>Method</u>	<u>Average Concentration (mg/L)</u>	<u>Std. Dev.</u>	<u>WAC Target (mg/L)</u>
Boron (B)	ICP-ES	5.81E+01	1.24E-01	7.43E+02
Cobalt (Co)	ICP-MS	<2.04E-02	NA	1.75E+02
Copper (Cu)	ICP-ES	< 8.92E+00	NA	7.43E+02
Iron (Fe)	ICP-ES	7.48E+00	2.20E+00	4.95E+03
Lithium (Li)	ICP-ES	1.39E+01	2.48E-01	7.43E+02
Manganese (Mn)	ICP-ES	< 1.25E+00	NA	7.43E+02
Molybdenum (Mo)	ICP-ES	1.56E+01	1.73E+00	7.43E+02
Nickel (Ni)	ICP-ES	< 1.72E+01	NA	7.43E+02
Silicon (Si)	ICP-ES	3.29E+01 ^a	6.13E-01	1.07E+04
Strontium (Sr)	ICP-ES	< 1.73E-01	NA	7.43E+02
Zinc (Zn)	ICP-ES	1.17E+01	8.70E-02	8.03E+02
Benzene (C₆H₆)	VOA	< 1.50E-01 ^a	NA	3.10E+02
Methanol (CH₃OH)	VOA	b	NA	1.88E+00
Dibutylphosphate [DBP] (C₈H₁₉O₄P)	IC	< 2.50E+02	NA	3.47E+02
Tributyl Phosphate [TBP] ((C₄H₉O)₃PO)	SVOA	< 7.50E-01 ^a	NA	7.50E+00
Toluene (C₆H₅CH₃)	VOA	< 1.50E-01 ^a	NA	3.10E+02
EDTA (C₁₀H₁₂N₂O₈⁴⁻)	HPLC	< 1.00E+02	NA	3.10E+02
NORPAR 13 (C_nH_{2n})	SVOA	< 7.50E-01 ^a	NA	7.5E-01
Dimethyl Mercury ((CH₃)₂Hg)	CVAFS	2.06E-01	1.68E-02	1.00E+00

a. Measurement from average of two detectable values from the analyzed triplicate set.

b. Measurement performed on duplicate samples rather than triplicate samples

c. Currently, a routine method for detecting this species does not exist in Analytical Development (AD).

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Table 3. Radionuclide Contaminants from Third Quarter CY16 Tank 50H Samples and Saltstone WAC, Revision 16, Attachment 8.3 Limits²

<u>Radionuclide</u>	<u>Method</u>	<u>Average Concentration</u> (pCi/mL)	<u>Std. Dev.</u>	<u>WAC Limit</u> (pCi/mL)
Tritium (³H)	Tritium counting	1.44E+03	8.19E+01	5.63E+05
Carbon-14 (¹⁴C)	C-14 Liquid scintillation	3.86E+02	2.46E+01	1.13E+05
Nickel-63 (⁶³Ni)	Ni-59/63	< 6.94E+00	NA	1.13E+05
Strontium-90 (⁹⁰Sr)	Sr-90 Liquid scintillation	1.45E+05	9.20E+03	3.15E+06
Technetium-99 (⁹⁹Tc)	Tc-99 Liquid scintillation	4.04E+04	9.38E+01	2.11E+05
Iodine-129 (¹²⁹I)	I-129 (w/ separation) Liquid scintillation	3.53E+01	2.70E+00	6.30E+01
Cesium-137 (¹³⁷Cs)	Gamma Scan	2.72E+05	5.38E+04	3.96E+06
Uranium-233 (²³³U)	ICP-MS	< 1.98E+02	NA	1.13E+04
Uranium-235 (²³⁵U)	ICP-MS	2.80E-01	5.56E-03	1.13E+02
Plutonium-241 (²⁴¹Pu)	Pu238/241 Liquid scintillation	9.25E+03	8.38E+02	8.38E+05
Total Alpha	Liquid Scintillation Counting	5.34E+04	2.87E+03	2.13E+05

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Table 4. Radionuclide Contaminants from Third Quarter CY16 Tank 50H Samples and Saltstone WAC, Revision 16, Attachment 8.4 Targets²

Radionuclide	Method	Average Concentration (pCi/mL)	Std. Dev.	WAC Target (pCi/mL)
Aluminum-26 (²⁶Al)	Gamma scan (Cs removed)	< 2.05E-01	NA	2.88E+03
Cobalt-60 (⁶⁰Co)	Gamma scan (Cs removed)	< 3.23E-01	NA	9.75E+02
Potassium-40 (⁴⁰K)	Gamma scan (Cs removed)	< 3.42E+00	NA	1.00E+02
Nickel-59 (⁵⁹Ni)	Ni-59/63	< 5.86E+00	NA	1.13E+03
Selenium-79 (⁷⁹Se)	Se-79	9.16E+01	2.83E+01	1.90E+04
Yttrium-90 (⁹⁰Y)	Secular Equilibrium w/ Sr-90	1.45E+05	9.20E+03	3.15E+06
Zirconium-93 (⁹³Zr)	ICP-MS	< 5.13E+01	NA	1.00E+05
Niobium-94 (⁹⁴Nb)	Nb-94	< 5.86E-01	NA	1.53E+02
Rhodium-106 (¹⁰⁶Rh)	Secular Equilibrium w/ Ru-106	< 4.64E+00	NA	1.13E+06
Ruthenium-106 (¹⁰⁶Ru)	Gamma scan (Cs removed)	< 4.64E+00	NA	1.13E+06
Antimony-125 (¹²⁵Sb)	Gamma scan (Cs removed)	1.29E+01	1.31E+00	7.99E+03
Tellurium-125m (^{125m}Te)	Secular Equilibrium w/ Sb-125	1.29E+01	1.31E+00	1.83E+03
Tin-126 (¹²⁶Sn)	Gamma scan (Cs removed)	3.97E+02	8.98E+00	1.80E+04
Cesium-134 (¹³⁴Cs)	Gamma Scan	< 6.17E+01	NA	1.82E+04
Cesium-135 (¹³⁵Cs)	ICP-MS	< 2.35E+01	NA	2.50E+02
Barium-137m (^{137m}Ba)	Calculation (Secular Equilibrium w/ 94.6% of Cs-137)	2.57E+05	5.09E+04	3.75E+06
Cerium-144 (¹⁴⁴Ce)	Gamma scan (Cs removed)	< 1.05E+01	NA	1.13E+05
Promethium-147 (¹⁴⁷Pm)	Pm-147/Sm-151 Liquid scintillation	<3.27E+01	NA	5.63E+06
Samarium-151 (¹⁵¹Sm)	Pm-147/Sm-151 Liquid scintillation	5.30E+01	1.01E+01	2.25E+04
Europium-154 (¹⁵⁴Eu)	Gamma scan (Cs removed)	< 1.11E+00	NA	1.62E+03
Europium-155 (¹⁵⁵Eu)	Gamma scan (Cs removed)	< 4.77E+00	NA	1.13E+04
Radium-226 (²²⁶Ra)	Ra-226	<1.35E+00	NA	1.00E+03
Radium-228 (²²⁸Ra)	Gamma scan (Cs removed)	< 2.45E+00	NA	1.00E+04
Actinium-227 (²²⁷Ac)	Th-229/230	<9.55E-03	NA	1.00E+04
Thorium-229 (²²⁹Th)	Th-229/230	<1.99E-02	NA	1.63E+05
Thorium-230 (²³⁰Th)	Th-229/230	<1.53E-02	NA	6.26E+03
Thorium-232 (²³²Th)	ICP-MS	< 2.24E-03	NA	2.88E+03
Protactinium-231 (²³¹Pa)	Pa-231	< 6.26E-01	NA	1.00E+03
Uranium-232 (²³²U)	U-232 ^a	2.72E+00	2.65E+00	9.06E+03
Uranium-234 (²³⁴U)	ICP-MS	< 1.28E+02	NA	1.13E+04
Uranium-236 (²³⁶U)	ICP-MS	< 1.32E+00	NA	1.13E+04
Uranium-238 (²³⁸U)	ICP-MS	5.89E+00	1.15E-01	1.13E+04
Neptunium-237 (²³⁷Np)	ICP-MS	<1.44E+01	NA	1.00E+04

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Table 4. Radionuclide Contaminants from Third Quarter CY16 Tank 50H Samples and Saltstone WAC, Revision 16, Attachment 8.4 Targets², continued

Radionuclide	Method	Average Concentration (pCi/mL)	Std. Dev.	WAC Target (pCi/mL)
Plutonium-238 (²³⁸Pu)	Pu238/241 Pu alpha PHA	2.54E+04	2.10E+03	2.13E+05
Plutonium-239 (²³⁹Pu)	Pu238/241 Pu alpha PHA	6.11E+02	9.66E+01	2.13E+05
Plutonium-240 (²⁴⁰Pu)	Pu238/241 Pu alpha PHA	6.11E+02	9.66E+01	2.13E+05
Plutonium-242 (²⁴²Pu)	ICP-MS	< 7.80E+01	NA	2.13E+05
Plutonium-244 (²⁴⁴Pu)	ICP-MS	< 3.62E-01	NA	7.02E+04
Americium-241 (²⁴¹Am)	Am/Cm	4.03E+00	8.82E-01	2.13E+05
Americium-242m (^{242m}Am)	Am/Cm	<2.65E-01	NA	4.50E+05
Americium-243 (²⁴³Am)	Am/Cm	<5.23E-01	NA	2.13E+05
Curium-242 (²⁴²Cm)	Am/Cm	<2.19E-01	NA	1.13E+04
Curium-244 (²⁴⁴Cm)	Am/Cm	1.04E+00	3.36E-01	2.13E+05
Curium-245 (²⁴⁵Cm)	Am/Cm	<3.74E+00	NA	2.25E+05

a. Measurement from average of two detectable values from the analyzed triplicate set.

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Table 5. Chemical Contaminants Impacting Saltstone Disposal Unit (SDU) Flammability from Third Quarter CY16 Tank 50H Samples and Saltstone WAC, Revision 16, Table 2 Limits and Targets²

<u>Chemical Name</u>	<u>Method</u>	<u>Average Concentration</u> (mg/L)	<u>Std. Dev.</u>	<u>WAC</u> <u>Limit/Target</u>
Isopar L	SVOA	< 2.67E+01 ppm ^{a,b}	NA	1.10E+01 ppm (Limit)
Tetraphenylborate (TPB)	HPLC	< 5.00E+00	NA	5.00E+00 mg/L (Limit)
Ammonium	IC	< 1.00E+01	NA	2.12E+02 mg/L (Limit)
Total Mercury	CVAA	9.64E+01	1.52E+01	3.25E+02 mg/L (Limit)
Monomethyl Mercury	CVAFS w/ Distillation	6.18E+01	1.93E+00	3.25E+02 mg/L (Limit)
Dimethyl Mercury	CVAFS	2.06E-01	1.68E-02	1.00E+00 mg/L (Target)

- a. Measurement performed on duplicate samples rather than triplicate samples.
 b. Result is calculated from the reported concentration of < 33 mg/L and the density of the slurry sample listed in Table 8.

Table 6. Other Organics Impacting SDU Flammability from Third Quarter CY16 Tank 50H Samples and Saltstone WAC, Revision 16, Table 3 Concentrations²

<u>Chemical Name</u>	<u>Method</u>	<u>Average Concentration</u> (mg/L)	<u>Std. Dev.</u>	<u>WAC</u> <u>Concentrations</u> (mg/L)
Butanol	VOA	< 5.00E-01	NA	0.75
Tributylphosphate	SVOA	< 7.50E-01	NA	1.0
Isopropanol	VOA	< 2.50E-01	NA	0.25
Methanol	a	NA	NA	0.05
NORPAR 13	SVOA	< 7.50E-01	NA	0.75

- a. Currently, a routine method for detecting this species does not exist in AD.

Table 7. Processing Constituents from Third Quarter CY16 Tank 50H Samples and Saltstone WAC, Revision 16, Table 4 Limits²

<u>Processing Constituents</u>	<u>Method</u>	<u>Value</u>	<u>Std. Dev.</u>	<u>WAC Limit</u>
pH	Calculated	> 13	NA	> 10
Sodium Concentration	ICP-ES / AA	4.73 M	5.50E-01	2.5 M < [Na+] < 7.0 M
Total Insoluble Solids	Calculated	~0 wt%	NA	< 15 wt%

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Table 8. Additional Measured Constituents³

<u>Constituent</u>	<u>Method</u>	<u>Average Value</u>	<u>Std. Dev.</u>
Density (slurry)	Measured (21.9°C)	1.2390 g/mL	0.0003
Specific Gravity	a	1.2418	0.0003
Total Solids	Measured	29.15 wt%	0.19
Total Beta	LSC	6.40E+05 pCi/mL	9.01E+03
Total Gamma	b	2.58E+05 pCi/mL	2.94E+04 ^c
Thorium-228 (²²⁸Th)	Gamma scan (Cs removed)	< 2.56E+01 pCi/mL	NA
Curium-247 (²⁴⁷Cm) ^d	Am/Cm	<5.14E+00 pCi/mL	NA
Californium-249 (²⁴⁹Cf) ^d	Am/Cm	<5.36E+00 pCi/mL	NA
Californium-251 (²⁵¹Cf) ^d	Am/Cm	<4.14E+00 pCi/mL	NA
Beryllium (Be) ^e	ICP-ES	< 1.29E-01 mg/L	NA

a. Calculated from the measured density of slurry and density of water at 22.3 °C.¹¹

b. Calculated from the sum of gamma emitters (Sb-126, Sn-126, Sb-125, Am-241, and Ba-137m).

c. Value is the “standard error of the mean” rather than the standard deviation of the measurements since its calculation involves multiple radionuclides.

d. Reported values are all below the estimated detection limits of 90.1 pCi/mL established by SRNL.⁸

e. Beryllium requested by DWPF & Saltstone Facility Engineering personnel.¹

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Name:	
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J. P. Arnold	C. A. Langton
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M. N. Borders	K. B. Martin
H. P. Boyd	J. A. McCrary
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