

November 14, 2014

Mr. Ron Linton, Project Manager Decommissioning & Uranium Recovery Licensing Directorate Division of Waste Management & Environmental Protection Office of Federal & State Materials & Environmental Management Programs U.S. Nuclear Regulatory Commission 11545 Rockville Pike Mail Stop T7-E18 Rockville, Maryland 20852-2738

Re: Honeymoon Yellowcake Re-dying – Submittal of Test Results from Lot #1 Materials License SUA-1341, Electronic Submission Only

Dear Mr. Linton:

Please find attached information pertaining to the first lot of Honeymoon re-dried yellowcake pursuant to the requirements of Condition 10.22 of SUA-1341. License Condition 10.22 identifies a number of tests required to be completed and the results submitted to the NRC for review and verification before any re-dried Honeymoon yellowcake is allowed to be transported off site.

Uranium One has successfully re-dried Lot 1 of the Honeymoon yellowcake and is providing (attached) the results of the test work required by Condition 10.22. The results of the Lot 1 testing have verified that the redrying effectively removes organics from the yellowcake and that the resulting material is U_3O_8 . We currently have 2.5 lots of re-dried material on site ready for transport to the converter. Your timely review and verification that shipments of this material may commence will be greatly appreciated.

If you have any questions or need additional clarification on any information provided, please do not hesitate to contact me at 307-233-6330.

Sincerely, OMM ON atou

Scott Schierman Sr. HS&E Specialist Uranium One Americas

cc: Linda Gersey – NRC Region IV w/attachment Donna Wichers – Uranium One USA, Inc. w/attachment Ryan Schierman – Uranium One USA, Inc. w/attachment

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On February 28, 2014 Uranium One USA, Inc. (U1) requested an amendment to Materials License No. SUA-1341 to allow re-drying of approximately 570,000 pounds of yellowcake material from the Honeymoon Project in Australia at Willow Creek's Irigaray Central Processing facility. On October 23, 2014, U1 received approval to re-dry the Honeymoon yellowcake through Amendment 3 to Materials License SUA-1341.

Amendment 3 allows for the re-drying of the Honeymoon yellowcake material at the Willow Creek facility with certain testing procedures to be completed, submitted and verified by NRC prior to shipment of the re-dried material to a conversion facility. License condition 10.22 as stated below outlines the tests that are required:

"10.22 Re-dried Honeymoon yellowcake shall be tested prior to shipment offsite. In addition to testing for successful removal of organics, the licensee shall test the first Lot of re-dried Honeymoon yellowcake as specified below. A lot is defined as a group of drums containing between 20,000 lbs and 50,000 lbs of yellowcake that are intended for shipment.

The licensee shall provide the following test information for the first Lot of Honeymoon re-dried yellowcake:

- Residual organics
- Information on how the optimal temperature and drying time was determined including dryer temperature profile, including (if available) minimum and maximum temperatures, time at designated temperature, and copies of temperature charts
- Yellowcake product temperature, color, and consistency as drummed
- An estimate of time the yellowcake is physically in the dryer with a technical basis for determining that estimate
- Information on individual drum venting and cooling times, and drum pressurization tests conducted on site
- Samples for yellowcake analysis, including results for UO4.2H2O, amorphous UOX, where (3 <x>3.5 (refer to Information Notice 1999-03, Rev. 1), and crystalline UO3 and lower oxides, will be taken during each significant change in dryer temperature, the speed of the dryer rake arm, or yellowcake feed rate to the dryer as recommended in RAI-2 (b) response Recommendations for Drying Operating Conditions" Nos. 1-4 (MI14192B247, ML 14275A443) during the re-drying of the first Lot, as the optimal drying conditions are established.

Results from the testing of the first lot of the re-dried material will be submitted in writing to the NRC for review and written verification. Re-dried Honeymoon yellowcake shall not be shipped offsite until written verification is received from the NRC headquarters staff."

The purpose of this report is to present the NRC with the data required by Condition 10.22 for NRC to review and provide written verification that the re-dried Honeymoon yellowcake may be shipped.

1.0 Residual Organics

The purpose of re-drying the Honeymoon yellowcake is to burn off the residual organic content within the yellowcake by feeding the material through the Willow Creek dryer to meet specifications of the uranium conversion facilities for Total Organic Carbon (TOC) of <0.1%.

TOC concentrations of the re-dried material were determined on site using a Leco SC-144 carbon analyzer. The carbon analyzer operates by placing a known amount of sample inside a pure oxygen environment that is regulated roughly at 1350°. The sample undergoes complete

combustion releasing the sample's carbon as carbon dioxide (CO₂) gas. The amount of CO_2 detected is then represented as percent carbon.

Re-drying of the Honeymoon yellowcake began continuously on October 27, 2014 (a trial run was performed on October 24 to test equipment but is not considered part of the processing of Lot 1). The range of TOC levels for drums in Lot 1 ranged from 0.03% to 0.321% with an average TOC concentration of 0.071%. These results are provided in Figure 1. As the target TOC concentration is <0.1% for converter acceptance, the re-drying process is quite effective at removing the organics.



Figure 1: Total Organic Carbon Results Re-dried Honeymoon Yellowcake Lot 1

2.0 Optimal Temperature and Drying Time Determinations

U1 conducted a re-drying trial on October 24, 2014 to test the drum tipper and conveyor equipment with Honeymoon yellowcake and to obtain initial TOC results, pursuant to U1's June 28, 2014 RAI response package to NRC [RAI 2(b): "... initial test run of the dryer will be operated at 300C or 572F on a single drum to demonstrate that residual organics are decomposed ... Temperature increases of 100F increments will be conducted until a temperature is established that produces a yellowcake product with negligible residual organic levels. A dryer residence time for the initial test will be set at 2 hours which was the maximum time shown in the bench testing that showed complete decomposition of residual organics."] The 5.5 hour trial run started with an initial dryer temperature of 572°F (300°C). TOC samples were taken every thirty minutes for analysis to determine if the <0.1% organic target was met. After almost 2 hours of drying at this temperature the TOC results remained unchanged at 0.8%, above the <0.1% target. The temperature was raised at this point to 675°F (357°C) and remained at 675°F for the remainder of the test. As a result of the temperature increase, TOC levels quickly responded to below 0.1%.

The feed rate to the dryer during the trial was initially set at 250 lbs/hr to test the conveyance equipment. Increases in the feed rate were made to 350 lbs/hr and 424 lbs/hr, at which point the trial ended. The feed rate of 424 lbs/hr provides a dryer retention time for the yellowcake of 2.5 hours. No changes were made to the dryer rake speed during the trial. The rake speed was set at 25.5 Hz at the beginning and was maintained at that level throughout the test.

As a result of the trial run, it was determined that a dryer feed rate of 424 lbs per hour and a dryer temperature of 675°F was sufficient to remove the organics to a level that met converter specifications (see Table 1). The dryer rake speed of 25.5 Hz would also be utilized for the

continued processing. Continuous reprocessing of the Honeymoon material was initiated on 10/27/14 using these established settings. The one drum of material that was re-dried during the trial was set aside to be further processed to reduce organics. XRD analysis was not completed for this drum.

Date	Time	Temperature	Feed Rate	TOC Analysis %C
10-24-2014	1305	572	250 lbs/hr	0.8245 @14:14
10/24/2014	14:25	572	325 lbs/hr	0.8080 @14:30
10-24-2014	14:48	675	325 lbs/hr	0.0957 @16:08 0.0785 @16:54
10-24-2014	17:00	675	424 lbs/hr	0.0699 @17:49 0.0699 @18:25
10-24-2014	Test Stopped 18:25	675	424lbs/hr	

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2.1 Temperature Evaluations

The temperature of the dryer is recorded through the use of a Honeywell chart graph that keeps a continuous record of the temperatures for each hearth. Temperatures are regulated using thermocouples within each hearth that are connected to instrumentation whereby temperatures may be set and regulated. The instrumentation also provides a digital readout of the instantaneous reading of the air temperature for each hearth.

2.1.1 Dryer Temperature Profile

Operators check and record the digital temperature readings of the dryer hearths multiple times during a shift. Table 2 shows a summary of the dryer temperatures recorded including the minimum and maximum temperatures for each hearth, as well as the hearth temperature setting, during the re-drying of Lot 1. In order to operate the dryer at lower temperatures, the burners on the top hearth of the dryer are not energized. The top hearth temperature readings are ambient conditions that are influenced by the heating occurring in the middle and bottom hearths.

YC Lot #1 Production	Minimum – Maximum Hearth Temperature Dryer Hearth Temperature Setting					
Date	Тор	Dryer °F	Middle	Dryer °F	Bottom	Dryer °F
10-27-2014	446-544	NA**	663-678	675	532-683	675
10-28-2014	494-583	NA	674-686	750	679-701	750
10-29-2014*	541-634	NA	765-826	800	725-800	800
10-30-2014	546-655	NA	760-805	800	790-805	800
10-31-2014	593-730	NA	796-837	750	800-817	800
11-1-2014	515-725	NA	795-843	750	816-852	750
11-2-2014	487-505	NA	765-803	750	795-848	750
11-4-2014	468-605	NA	768-804	850	779-892	800

 * Significant Change Occurred as defined in RAI-2(b) and License Condition 10.22

**NA – burners are not energized on the top hearth

2.1.2 Time at Designated Temperature

The time the yellowcake spends at a designated temperature is dependent on the yellowcake calculated residence which is discussed later in Section 4.

2.1.3 Temperature Charts

Copies of the Honeywell temperature log charts are provided in Appendix A.

3.0 Yellowcake Properties Analysis (Temperature, Color and Consistency)

3.1 **Product Temperatures**

The re-dried yellowcake product temperature was determined using a thermocouple placed inside each drum immediately after removal from the packaging room. The temperature taken at this point is the initial temperature recorded for the cooling and venting times. Operators recorded three temperature measurements of the product within each drum during the fill cycle. These readings are displayed in Figure 2. As can be seen from Figure 1, the re-dried yellowcake product temperatures ranged from over 100°F to over 600°F with an overall average of 382°F.



Figure 2: Temperature Readings of Re-Dried Yellowcake Product

3.2 Re-Dried Yellowcake Product Color and Consistency

The Honeymoon yellowcake changed in both color and consistency as a result of the redrying. Operators recorded both the color and consistency of the yellowcake in Lot 1 before and after processing. The overall color of the Honeymoon material prior to re-drying was a light yellow, almost cream color, with a consistency of a fine powder (see Figure 3). The light yellow color is consistent with the uranyl peroxide product generated by the Honeymoon low temperature vacuum dryer. After re-drying, the Honeymoon yellowcake changed to a dark olive green which is a color consistent with uranium that is converted to U_3O_8 (see Figure 4). The consistency of the re-dried material was also a fine powder, but was not as prone to dusting as the light yellow product. Figure 3: Honeymoon Yellowcake



Figure 4: Re-dried Honeymoon Yellowcake



4.0 Retention Time of Yellowcake in Dryer

As previously reported to NRC, the maximum time in the laboratory bench tests for complete decomposition of organic residuals in the Honeymoon yellowcake was two hours. It was noted in the laboratory report that the decomposition of the organics occurs rapidly and that lower dryer residence times could give acceptable results for the decomposition of the organics. Thus while it does require a certain time period in the dryer to decompose the organics the true indicator of adequate drying times is the TOC measurement and the XRD analysis. That being said, residence times were calculated for each drum using the same methodology that was submitted to the NRC in the U1 document dated September 4, 2013.

The residence times (RT) for the Honeymoon Lot 1 drums were calculated using the equation:

$$RT_{Dryer} = \frac{V_d}{F_{YC}}$$

Where

 V_d = Volume of the yellowcake in the dryer (ft³) F_{YC}= Average yellowcake feed rate (ft³/hr)

Because the internals of the dryer do not completely fill with yellowcake during the drying operation, a calculation of the active yellowcake dryer hold-up must be determined in order to calculate the dryer residence time. The Willow Creek dryer is a direct fired multiple hearth dryer with an internal rake drive mechanism. The rake mechanism is the device internal to the dryer that moves the yellowcake from the top of the dryer through the multiple heated hearths to the dryer discharge and into the receiving drum. The dryer internals have the following specifications:

Number of Hearths=3 Surface area of each Hearth=13.50 ft^2 Area occupied by the Rake mechanism on each hearth = 0.27 ft^2 Depth of yellowcake on each Hearth = 2.5 in (0.208 ft)

The active yellowcake volume internal to the dryer is:

$$V_d = A_H \times H_{YC}$$

Where:

 A_{H} = Area of Dryer Hearths H_{YC} = Height of yellowcake on the hearths For the Willow Creek dryer this calculates to be:

$$V_d(ft^3) = (13.50 ft^2 - 0.27 ft^2) \times 3 Hearths \times 0.208 ft$$

 $V_d = 8.27 ft^3$ of yellowcake hold up internal to the dryer

The average yellowcake feed rate for the Honeymoon Lot 1 material was kept at a consistent 424 pounds per hour. To convert the lbs per hour to cubic feet per hour one must use the density of the yellowcake. In previous retention time calculations the density of 131 pounds per cubic foot for yellowcake was used. Experiments on site have shown that the Honeymoon material has a similar density and therefore 131 pounds per cubic foot will be used. The conversion is shown below:

$$F_{YC} = \frac{424lbs}{hr} \times \frac{ft^3}{131\,lbs} = 3.21\,\frac{ft^3}{hr}$$

Therefore using this feed rate the residence time for the Honeymoon material should be:

$$RT_{Dryer} = \frac{V_d}{F_{YC}} = \frac{8.27ft^3}{\frac{3.21ft^3}{hr}} = 2.6 \text{ hours}$$

The above residence time is based on the assumption that variables such as feed rate, density, drying temperature, and dryer rake speed remain constant.

Residence time for the Lot 1 reprocessed yellowcake varied significantly due to material bridging in the hopper not allowing a constant feed rate of material to the dryer. Once this problem was resolved and a consistent feed rate was established to the dryer, yellowcake residence times corresponded closely with feed rate calculations. Lot 1 residence times varied due to the feed issues, but consistently ranged between 2 to 4 hours. The average retention times since dryer feed issues were resolved and a consistent feed rate established to the dryer are approximately 2.5 hours.

A check of this value is to calculate retention time using the drum fill time and the drum weight. An example of this calculation is:

RT (retention time in hours) = (8.27 ft³ x 131 lbs/ft³) *(1.5 hrs/800 lbs) = 2.2 hours where the average fill time of an 800 lbs drum is 1.5 hrs

5.0 Drum Venting/Cooling Time and Pressurization Tests

As is discussed further in Section 6, x-ray diffraction (XRD) analysis of the re-dried Honeymoon material has identified the yellowcake as U_3O_8 which is a non gas forming and stable uranium compound. Therefore the venting and cooling criteria that are critical for uranyl peroxide products are not necessary to alleviate gas production and potentially pressurized drums. However, pursuant to condition 10.22 and our site SOPs, the Lot 1 drums were cooled and vented until they reached a temperature of approximately 90°F. Due to the higher temperature of the Lot 1 drums, this process has taken up to 3 to 5 days per drum. The average temperatures of the Lot 1 drums and cooling times are provided in Figure 5.



Figure 5: Cooling Profile for Lot 1 Honeymoon Re-dried Yellowcake

Pressurization of drums containing the U_3O_8 compound has not been identified as an issue within the uranium industry because U_3O_8 is not a gas forming compound that would lead to pressurization (NRC IN 99-03). However U1 conducted pressurization experiments to verify that no pressure could be observed pursuant to condition 10.22. Drum lids equipped with zero to 5 psi pressure gauges and bleed off valves were placed and tightened down on three drums during their cooling process (Lot 1 Drums 10, 11 and 12). The temperature at the time the lid was attached and sealed to each drum was 200F, 206F and 213F respectively. Each of the drums was monitored for a 7 day period and no pressure was observed, confirming that venting time is not an issue for U_3O_8 product.

6.0 Significant Changes and XRD Analysis

Samples for yellowcake analysis were taken during each significant change in dryer temperature and yellowcake feed rate during the re-drying of the Honeymoon Lot 1. No changes were made to the dryer rake speed so this is not addressed. As agreed with NRC, a significant change consists of a change in the dryer temperature of greater than 100 degrees, and a change in the feed rate in 100 lbs/hr increments.

During the re-drying of Lot 1 that began on 10-27-14, one significant change from the original test conditions was implemented. The temperature in the dryer was increased on 10-29-14 from the initial 675°F to 800°F after two days of processing. The temperature increase was made due to a perceived increasing trend in TOC levels. When a significant change in the re-drying process occurs, U1 was required by condition 10.22 to take a sample of the re-dried material and perform XRD analysis on the sample. A summary of the dryer temperature settings, feed rate, rake speed and when samples for XRD analysis were taken during the drying of Lot 1 is provided in Table 3.

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Date	Dryer Temperature (°F)	Feed Rate (lbs/hr)	Rake Speed (Hz)	Drums Packaged Under These Operational Settings	XRD Analysis Performed
10/27/2014	675	424	25.5	WCHM1 Drums 1-5	Drum 3 and 4
10/28/2014 (17:00)	750	424	25.5	WCHM1 Drums 6-7	
10/29/2014 (11:30)*	800	424	25.5	WCHM1 Drum 8	Drum 8
10/29/2014	800	424	25.5	WCHM1 Drums 9	Drum 9
11/3/2014 (14:00)	800	500	25.5	WCHM1 Drums 39-40	
11/3/2014 (17:00)	800	469	25.5	WCHM1 Drum 41	
11/3/2014 (18:30)	800	440	25.5	WCHM1 Drum 41-52	

 Table 3:
 Summary of Significant and Other Changes

*Significant change occurred as defined in RAI-2(b) and License Condition 10.22

Samples of the re-dried yellowcake taken during the significant changes were submitted to two outside laboratories to characterize the final uranium product using XRD analysis. Four samples of the re-dried yellowcake were sent to Dr. Peter Burns Director, Energy Frontier Research Center Materials Science of Actinides at Notre Dame University. Two samples were sent to Evans Analytical Group, a California laboratory specializing in the analysis of solid materials using XRD.

6.1 Notre Dame Analysis Results

The four samples sent to Notre Dame included a sample of Honeymoon yellowcake prior to re-drying, plus re-dried material from Lot 1, Drums 3, 4, and 8. The results of the XRD analysis show that the Honeymoon material <u>prior to re-drying</u> is predominantly Metastudtite $(UO_2)(O_2)(2H_2O)$ with trace amounts of Studtite, as is shown in Figure 6. All the samples after the re-drying showed that the predominant uranium phase in the material is the uranium oxide U_3O_8 (see Figures 7 through 10). Figures 8 and 9 show that Drum 4 may have a trace amount of the Metastudite.



Figure 7: XRD Spectra for Lot 1, Drum 3





Figure 8: XRD Spectra for Lot 1, Drum 4

Figure 9: XRD Spectra for Lot 1, Drum 4 with Metastudtite Diffraction Peaks Displayed







6.2.1 Evans Analytical Analysis

Evans Analytical performed XRD analysis on the Lot 1, Drum 3 and Drum 9 samples of redried yellowcake. The results of the XRD analysis are summarized in Table 4.

Sample ID	Phases Identified	Concentration wt% (+/- 5%)
	U ₃ O ₈ – Uranium Oxide Hexagonal P-62m PDF# 00-031-1425	99.8
	UO _{2.25} – Uraninite?? Cubic Fm-3m PDF# 00-020-1344	0.2
WCHM1 #0	U₃O ₈ – Uranium Oxide Hexagonal P-62m PDF# 00-031-1425	98.2
	UO _{2.25} – Uraninite Cubic Fm-3m PDF# 00-020-1344	1.8

Table 4:	Evans Summary	y of XRD Analysis
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The spectra for the XRD results of Lot 1, Drum 3 and Lot 1, Drum 9 are shown in Figures 11 and 12. The spectra from the two samples indicate that uranium oxide U_3O_8 is the primary component of the samples. Additionally, Evans attributes a peak near 28° two-theta to be a match for uraninite ($UO_{2.25}$). Evans mentions it is a speculative match because it is only based on a single weak peak. The important information to extract from these reports is that the major component is uranium oxide U_3O_8 .







