REPORT

ROOT CAUSE ANALYSIS REPORT

September 9, 2014 Incident Investigation Pressurized Drum of Yellowcake at the Honeywell Uranium Refinery

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Golder Associates

EXECUTIVE SUMMARY

Golder Associates Inc. (Golder) was requested to assist Uranium One USA Inc. (“U1”) with the investigation and root cause analysis of an incident that occurred at the Honeywell Uranium Refinery in Metropolis, Illinois on September 9, 2014, involving a pressurized drum of dried uranium yellowcake produced from the Willow Creek Project and dried at the Irigaray Processing Plant, Wyoming. Upon opening of Drum 43 in Lot 51 to conduct sampling, the drum was found to be pressurized and the resulting escape of gas pressure from the drum lifted the drum lid and a small amount of dried yellowcake was expelled from the drum. The escaping dried yellowcake powder spilled on the floor and covered an area of approximately six feet in diameter. The two sampling operators that were exposed to the uranium dust were found to have received less than the allowable dose of yellowcake as a result of the incident.

A previous incident of pressurization of a drum that was shipped from the Willow Creek facility to the Cameco Blind River Refinery, Ontario, Canada in 2012 was found to be caused by a build-up of oxygen gas generated by the decomposition of residual uranyl peroxide hydrates and/or hydrogen peroxide in the dried yellowcake product.

Upon investigation it was found that the root cause of the September 9, 2014, incident was also decomposition of uranyl peroxide hydrates resulting in the generation of oxygen gas in the closed drum. The mechanism by which this gas generation occurred was likely due to the premature closure of a drum filled with dried yellowcake that was not allowed to properly cool and vent. A secondary gas generation mechanism was also investigated which involved the generation of oxygen in the drum due to the incursion of water into the drum after drum closure. This mechanism, while capable of causing oxygen gas generation, was deemed to be improbable since the route of entry of water into the closed drum would also have been a route for the gas to escape resulting in no significant pressurization.

Process changes to the packaging and drying operations at the Irigaray Processing Plant have been recommended and implemented that should prevent future pressurization of dried uranium yellowcake drums. These process changes include:

1. Improved Packaged Drum Venting and Cooling Procedures. The Irigaray site has instituted a revised cooling and venting procedure that involves the use of a temporary lid with a mesh opening to allow any build-up of gases to escape prior to drum closure. Longer cooling and venting times have been instituted to insure that the drummed yellowcake has stabilized prior to shipment.
2. Revision to the Packaged Drum Handling Procedures. A revised drum washing procedure has been developed to ensure that no water can enter a closed drum during decontamination of the drum exterior prior to shipment. The previous procedure involved the use of a high-pressure wash to remove any residual dust from the exterior of the drum. Irigaray has introduced an alternate washing procedure which will reduce the likelihood of water being introduced into a drum while still removing any uranium dust from the exterior to meet the surface contamination shipping requirements.

3. Dryer Rehabilitation Program. In August and September 2014, the Irigaray site completed a dryer optimization program which included reworking of the burner system and an overhaul of the system controls. This comprehensive dryer upgrade resulted in significantly improved burner operations and better temperature control of the dryer system. The upgrade enabled the Irigaray dryer to operate more efficiently and to produce a higher degree of drying with reduced amounts of amorphous uranyl oxide being produced.
4. Increase Dryer Temperature. It has been shown that increased dryer temperatures will eliminate the presence of amorphous uranyl oxide. The Irigaray operation has, therefore, increased its recommended dryer operating temperature from 1,200 ^{o}F to 1,300 ^{o}F. This 100 ^{o}F increase in temperature has virtually eliminated the presence of amorphous uranyl oxides in the dried yellowcake and thereby has reduced the potential for any reactive oxides to be present in the final packaged drum.
5. Pre-shipment Pressure Evaluation. U1 is also proposing to open each drum prior to shipping to ensure that no pressure exists in the drum and that no moisture has entered the drum while it is awaiting shipment. This will ensure that all drums shipped to a refinery will not have positive internal pressure. U1 will conduct this procedure until they feel confident that the other proposed changes which have been implemented are effectively ensuring that no pressure build-up in dried yellowcake drums is occurring.

The root cause of the drum pressurization incident was found to be consistent with the causes previously determined by the US Nuclear Regulatory Commission (“USNRC”) in their USNRC Information Notice 99-03 and the updated USNRC Information Notice 99-03 Rev.1 that was issued on March 24, 2014, as a result of intensive investigation by the USNRC and the industry. The revised Information Notice 99-03 (Rev.1) was initiated by the USNRC following the Blind River pressurization incident and the update was intended to provide additional technical information and recommendations to the entire uranium industry.

Table of Contents

List of Tables

List of Figures

List of Appendices

1.0 INTRODUCTION

Golder Associates Inc. (Golder) was requested to assist Uranium One USA Inc. (U1) with the investigation and root cause analysis of an incident involving a pressurized drum of dried uranium yellowcake produced from the Willow Creek Project and dried at the Irigaray Processing Plant, Wyoming, that occurred at the Honeywell Uranium Refinery, Metropolis, Illinois on September 9, 2014. Upon opening Drum 43 in Lot 51 to conduct sampling, the drum was found to be pressurized and the resulting escape of gas pressure from the drum lifted the drum lid and a small amount of dried yellowcake was expelled. The escaping dried yellowcake powder covered an area of approximately six feet in diameter. The two sampling operators in the area that were exposed to the uranium dust were found to have received less than the allowable dose of yellowcake as a result of the incident.

This report examines the possible causes of the over-pressurization of the drum and recommends prevention and mitigation measures that should be implemented to prevent future over-pressurization of uranium drums from the Willow Creek Irigaray Processing Plant (Willow Creek Plant).

Golder personnel have examined the Willow Creek plant operational records leading up to the production, drying, packaging, and shipping of the yellowcake drums to the Honeywell Uranium Refinery. In addition, Golder has conducted a site visit to the Irigaray plant, proposed certain sampling and analysis, investigated the root cause of the incident, and recommended preventative and mitigative measures which may be implemented to prevent gas build-up in future dried yellowcake shipping containers. The details of this proposed investigation were discussed with representatives of the USNRC on September 24, 2014, and a written proposed investigation plan was communicated to them on September 29, 2014.

2.0 DESCRIPTION OF THE INCIDENT

Uranium One USA Inc. (U1) was informed on September 9, 2014, by management staff at the Honeywell Uranium Refinery in Metropolis, Illinois, that an incident had occurred involving low-level pressurization of one of the drums of dried yellowcake product that was received from the Willow Creek Project.

During routine sampling at the Honeywell facility Sampling Plant, a dried yellowcake drum from U1 (Drum 43 of Lot WC 51 from the Willow Creek Project) was found to be pressurized. The sampling operator observed the prescribed Honeywell drum opening procedures and did not see any indication that the contents were under pressure prior to opening. With the ring bolt removed, the operator tapped the drum ring and the escaping gases lifted the drum lid and entrained some of the uranium product expelling uranium dust in the sampling area. Two operators were in the area but were not injured. Airborne radioactivity measurements in the area were taken and were below Honeywell's administrative level for respiratory protection. The bioassay sample results for both operators involved in the incident were below Honeywell administrative action levels for uranium of the 8 parts per billion (ppb) repeat level and the 25 ppb investigation level. Additional information received from Honeywell as reported by U1 indicated the following:

- Honeywell personnel involved in the sampling of U1 yellowcake shipments conveyed that none of the drums that were previously opened and sampled exhibited any drum pressurization. This includes 104 drums from Willow Creek Lots 52 and 54, plus approximately 20 drums opened in Lot 51 prior to Drum 43.
- Honeywell also indicated that none of the previously sampled drums (including Drum 43 of Lot 51) showed any physical signs of pressurization such as bulging lids.
- Honeywell confirmed that they had opened and sampled the remaining drums in Lot 51. The remaining drums in Lot 51 were drilled to release any potential pressure buildup, opened, and sampled on September 10, 2014. No signs of pressurization of the remaining 9 drums in Lot 51 could be confirmed. Honeywell at the time of the incident had 156 drums from Lots 53, 55, and 56 located in storage. Honeywell resumed sampling or processing of these drums with no incidents of further drum pressurization encountered.

After being informed of the incident, U1 management immediately informed the USNRC of this occurrence, and Willow Creek voluntarily discontinued any additional planned shipments of yellowcake to the Honeywell refinery. The USNRC understands that U1 is conducting an Incident Investigation and Root Cause Analysis to determine the cause for the pressurized drum. With completion of the preliminary root cause analysis in October, 2014, U1 resumed the shipment of existing inventories of dried yellowcake after the recommended site procedures in that report had been undertaken.

3.0 REVIEW OF INDUSTRY PRACTICES AND STANDARD OPERATING PROCEDURE

While the uranium industry has been packaging and shipping dried yellowcake to uranium refineries for many years without a significant number of incidents of pressure build-up in the 55-gallon drum shipping containers, there have been a few incidents of similar occurrences of low-level pressurization. These incidents have been thoroughly investigated and have been reported to the USNRC and appropriate state and federal agencies. The resolution of these incidents has led the USNRC to issue a guidance document that examined the potential causes for the build-up of pressure in shipping drums and provided recommendations for preventing and mitigating pressurization in yellowcake drums (USNRC Information Notice 99-03, issued on January 29, 1999; and the updated USNRC Information Notice 99-03 Rev.1, issued on March 24, 2014).

3.1 Review of USNRC Information Notice 99-03 and Information Notice 99-03 Rev.1

On March 24, 2014 the USNRC issued Rev.1 to the Information Notice 99-03 “Exothermic Reactions Involving Dried Uranium Oxide Powder (Yellowcake).” The development of this Information Notice was based on the analysis of similar incidents that occurred at four separate Wyoming and Texas in situ recovery (ISR) operations in 1998 and one in 2012 at the Cameco Blind River refinery. Following the pressurized drum incident in 2012, the USNRC assembled a panel of industry and USNRC experts to review the previous findings of the original Information Notice and to update the notice as appropriate.

The panel of experts described two conditions that could cause gas generation from decomposition reactions in yellowcake after drying and packaging.

1. Generation of oxygen derived from the decomposition of dried uranyl peroxide or hydrogen peroxide residuals from hydrogen peroxide precipitated yellowcake. This reaction was observed at three separate yellowcake processing facilities.
2. Generation of decomposition gases from the reaction of hydrocarbon contaminants in the dried yellowcake which can react with hydrogen peroxide precipitated yellowcake. This reaction was observed at two separate incidents at yellowcake drying and packaging facilities.

The USNRC staff concluded that pressurized drums of dried yellowcake produced from a hydrogen peroxide precipitation process resulted from the generation of oxygen from the decomposition of residual hydrogen peroxide or the decomposition of intermediate uranyl peroxide hydrates. It was also their conclusion that when organics were present in the dried product, the possibility of exothermic reactions of peroxide with the organic contaminants could result in the generation of heat and organic combustion gases, carbon monoxide (CO), and carbon dioxide ($CO_{2)}$.

As a result of the 2012 pressurized drum event at Blind River the Willow Creek Plant instituted a revision to their Standard Operating Procedure IR-12 “Drypack Yellowcake Drying and Drumming” to add a requirement that the filled yellowcake drums be allowed to cool and vent for at least 24 hours prior to lid closure. This cooling and venting period was increased from the previously recommended 3 hours (Information Notice 99-03) to 24 hours for additional cooling of the yellowcake and to allow cooling of the yellowcake product and to allow any gas generation to be dissipated prior to final closure. This revised procedure has since been followed at the Willow Creek Project site with all filled drums of yellowcake and has become the recommended industry standard for venting of filled uranyl peroxide yellowcake drums.

3.2 Drying, Packaging, Loading, and Shipping of Willow Creek Lot 51

The yellowcake dryer used at the Irigaray Processing Plant is a multiple rotating hearth dryer that was operated at a nominal 1,200^{o}F (650^{o}C) during the drying of the yellowcake drum (Lot 51, Drum 43) involved in this incident. Upon inspection of the records of the operation during this period the operation of the dryer during the drying of Lot 51 was found to be normal. No unusual circumstances were observed in the operators’ logs or from discussions with operating personnel. The site reported that the standard operating procedures for loading and shipping of dried yellowcake were also followed as evidenced by their operational logs.

The specific times of the drying, packaging, loading, and shipping of Lot 51 (52 drums) including Drum 43, were obtained from available site records and are shown in Appendix A. All drums in Lot 51 were heated to the proper drying temperature as evidenced by review of operational records and had a drum fill time in the range of five to seven hours. The specifics for Drum 43 are as follows.

Drum 43 filled	April 18, 2014
Drum Fill Time	5 hours, 25 Minutes
Top Hearth Temperature	1,130^{o}F
Middle Hearth Temperature	1,200^{o}F
Lower Hearth Temperature	1,201^{o}F
Drum Vent Time	28 Hours, 15 Minutes
Trailer shipped to Honeywell	June 23, 2014

At the time of shipment a visual drum inspection for signs of pressurization was conducted and no unusual drum bulging was observed in any of the filled drums. The visual inspection was enhanced by tapping the lid with a rubber hammer and pressing on the lid to detect any potential pressure. Irigaray operations personnel confirmed that there was nothing unusual about the processing of Lot 51. The “RSO Survey and Release Form” also confirmed that the shipment of drums from Lot 51 was below the required alpha count level for shipment and that the “Drum Inspection for Pressure Form” indicated that all of the drums were inspected for pressurization and all appeared to be normal with no signs of pressurization.

3.3 Investigation of Pre-dryer Yellowcake Processing and Dryer Operation

The process of operation of the precipitation circuit and the dryer operations were also reviewed. The operations of the elution circuit, the precipitation circuit, and the dryer operations were considered to be normal and followed standard plant operating procedures. There were no unusual process conditions encountered during processing of Lot 51.

4.0 ROOT CAUSE ANALYSIS

A root cause analysis plan was developed and proposed to the USNRC to investigate the Honeywell pressurized drum incident. This investigation was conducted in two phases. The first phase investigated the physical and operational conditions surrounding the uranyl peroxide processing, drying, packaging, shipping, and storage of the affected drum. The second phase involved a detailed analysis of the chemical characteristics of the dried yellowcake product to establish if any intermediate uranium oxides are being formed that could contribute to drum pressurization.

4.1 Physical Conditions and Operating Procedures

An evaluation of the physical and operating conditions at the Irigaray site was conducted to determine if any operational procedures could have contributed to the drum pressurization event.

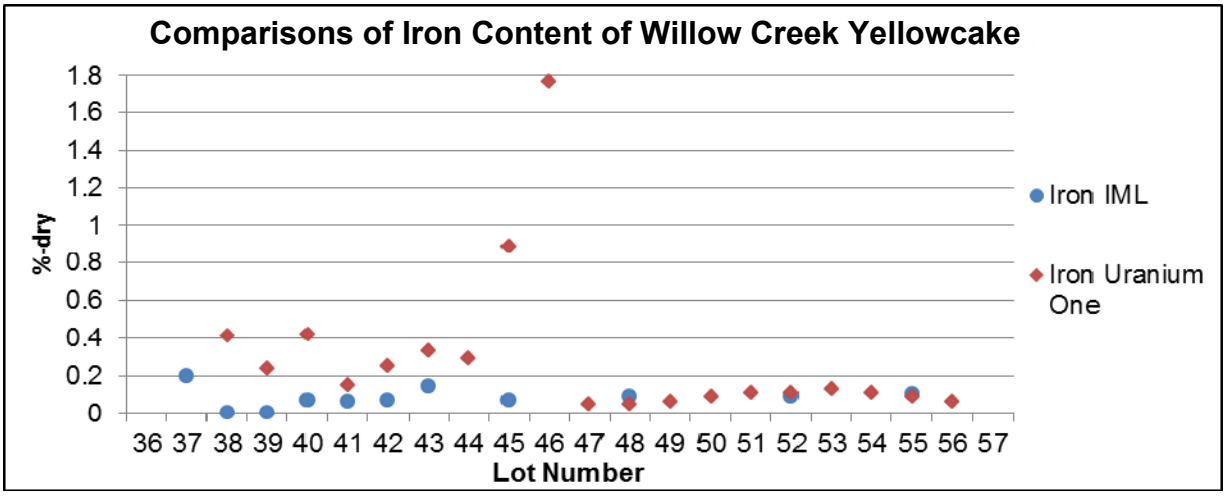
4.1.1 Evaluation of the Uranyl Peroxide Precipitation Process

The process operations of the precipitation circuit and the dryer operations were carefully examined. Based on review of operational logs and discussions with site operations personnel, the operations of the elution circuit, the precipitation circuit, and the yellowcake slurry operations were considered to be normal and followed standard plant operating procedures. There were no unusual process conditions encountered during the time period when Lot 51 and specifically Drum 43 were processed.

The possibility of organic contaminants giving rise to gas generation and potential drum pressurization was also reviewed. The operations staff at Irigaray has been trained to closely monitor any use of organic compounds, such as gear box seal oil or lubricants, to ensure that organic contaminants are not introduced in the precipitation circuit. This was confirmed in discussions with the operations personnel. They have been trained to look for any drips of gear box oils or other sources that might lead to organic contamination. No unusual events were discovered that could have given rise to organic contamination during processing of Lot 51.

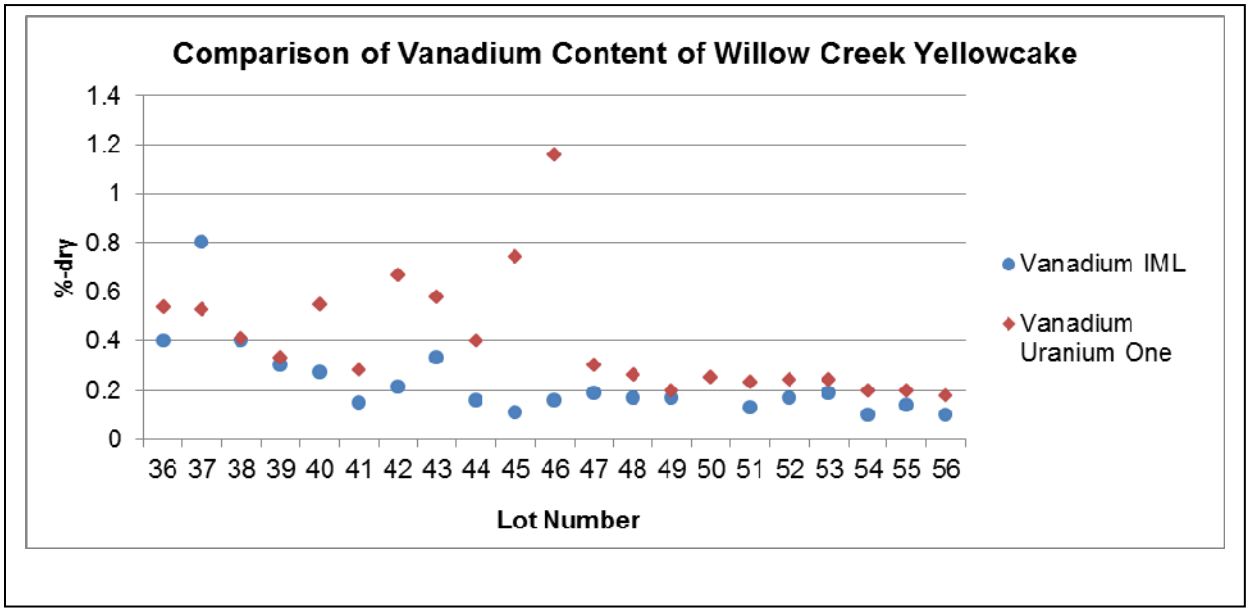
Additionally, the potential for unusual concentrations of inorganic contaminants contributing to the drum pressurization was also investigated. It is known that some trace metals can act as a catalyst in the decomposition of hydrogen peroxide and peroxide-containing compounds. Since iron and vanadium are the two most prevalent naturally occurring metals in the ore body at Willow Creek, chemical analyses for previously processed dryer lots were reviewed, focusing on vanadium and iron results. These results are shown in Figures 1 and 2 which indicate the vanadium and iron content on a percent dry weight basis for historical yellowcake shipments. The vanadium and iron concentrations in Lot 51 were not abnormal. In fact, the presence of these metals in the yellowcake was lower than they had been historically. It does not appear that inorganic trace metal levels in the processed yellowcake played any role in the pressurization event.

Figure 1: Iron Content of Willow Creek Yellowcake



IML = InterMountain Laboratory; an independent 3rd party laboratory

Figure 2: Vanadium Content of Willow Creek Yellowcake



In September of 2011, U1 switched from the use of hydrochloric acid (HCl) to sulfuric acid (H_2SO_4) in the precipitation circuit. Historically, the Irigaray Plant had used hydrochloric acid (HCl) for precipitation of the uranium. It is unlikely that this change would have had any influence in the generation of gases in the final product as many uranium processing plants use sulfuric acid as the acidification media in their precipitation circuit.

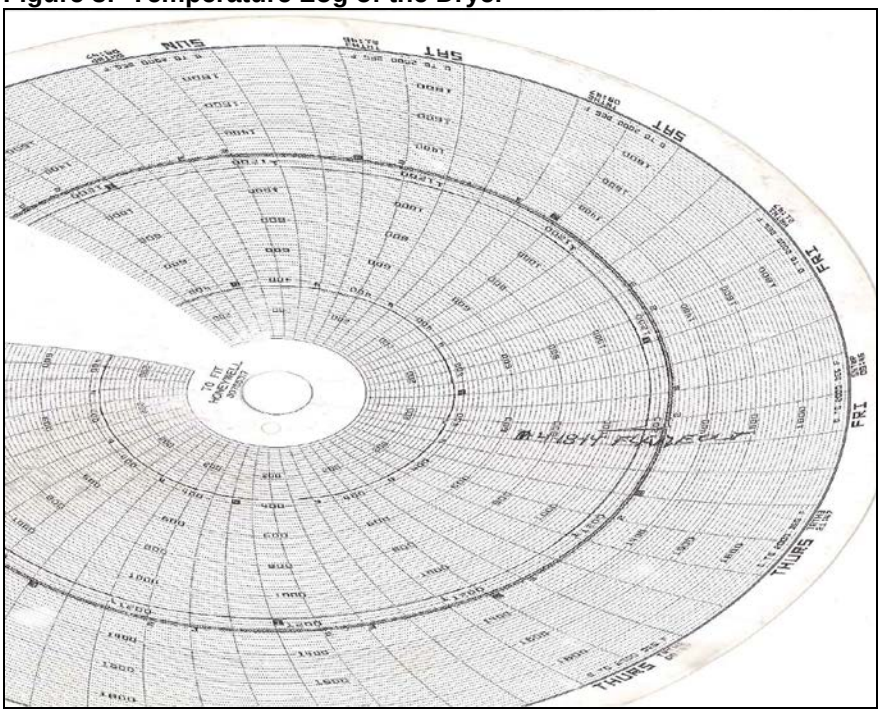
4.2 Evaluation of the Dryer and Dryer Operating Conditions

The yellowcake dryer used at the Irigaray Processing Plant is a multiple rotating hearth dryer operated at a nominal 1,200°F (650°C). Review of the operating conditions for the dryer during Lot 51 run was normal and no unusual circumstances were observed in the operators' logs or in discussions with operating personnel.

Records of the dryer operational parameters have been reviewed to ensure that no upset conditions or deviation from standard operating procedures were observed. WC Lot 51 drum 43 loading was completed on April 18, 2014, at 11:05 a.m. The total loading time for the drum was 5 hours and 25 minutes. The moisture content as measured at Irigaray was 1.1%. Once the drum was filled it was allowed to vent for 28 hours and 15 minutes. The venting procedure consisted of placing the lid on the top of the drum offsetting the lid enough to allow for gas and heat to escape the system. In conversations with operators the manner in which lid placement for venting was done had a considerable amount of variation. Some operators placed the lid on the drum and then loosely placed the ring closure on the drum while others left the ring off. Efforts to standardize the venting have been made and are more fully discussed in section 4.2.4, Drum Venting and Cooling Procedures.

During the time of filling WC 51 Drum 43 the temperature in the dryer was relatively steady as can be seen on the temperature log of the dryer shown in Figure 3. In the figure the only abnormal condition seen is the flameout on April 18, 2014. The event occurred at 11:55 a.m., which was 50 minutes after Drum 43 was filled.

Figure 3: Temperature Log of the Dryer



As part of the root cause analysis the physical condition of the dryer was inspected to determine if there were any abnormal conditions inside the dryer that could give rise to undried yellowcake passing into the packaging drum. This effort involved shutting the dryer down and physically looking inside each hearth to see if any unusual buildup or other mechanical phenomena could be observed which could give rise to undried material passing through the dryer. Photographs of each hearth were taken and an examination of these revealed that each hearth functioned as designed with the first hearth already drying the yellowcake slurry to a semi-dry powder consistency. Hearths 2 and 3 showed a consistent fine particle powder that was being passed from the grates to each hearth exit and to the final discharge. No unusual aspects of the dryer internals were otherwise observed.

Additionally the Irigaray site supervisor and the site radiation safety officer (RSO) reviewed the operators' daily logbooks to determine if there were any upset conditions around the time of the filling of WC51 Drum 43. The logbooks indicated there were no abnormal activities around the time period of March to April 18, 2014, that would lead to any process upsets or dryer malfunctions. Further, there were no major maintenance items performed in the time period that could have given rise to the introduction of oils or greases or any other organic contaminants.

During the months of August and September 2014 Irigaray site personnel conducted a detailed maintenance program and dryer upgrade program on the dryer burner system and the dryer controls. Table 1 provides a summary of maintenance activities performed during this period. This comprehensive dryer upgrade resulted in significantly improved burner operations and better temperature control of the dryer system. The upgrade enabled the Irigaray dryer to operate more efficiently and to produce a higher degree of drying with reduced amounts of amorphous uranyl oxide as will be discussed in another section of this report.

Table 1: Irigaray Dryer Maintenance Performed During August/September 2014

Date	Description	Implications	RWP #
8/5/14 through 8/14/14	Dryer is down for routine maintenance. The ID fan was cleaned, acidized, and rebalanced. The scrubber, demister screens, and scrubber settling tank were cleaned and acidized. Lastly the mixed air and burners were calibrated.	If the ID fan is not cleaned it creates too much stress on the motor and excessive vibration. If the demister screens, scrubber, and settling tank are not cleaned the draft requirements are not met.	62/14, 63/14, 67/14, 68/14
8/16/14 through 8/19/14	Replace bearings, motor, and delumper shaft.	The delumper breaks up any clumped material prior to packaging.	71/14, 73/14, 74/14
8/26/14	Second hearth main burner would not stay lit. Replaced burner actuator controller, discovered the wiring for all three burner actuators was brittle, so all wiring was replaced.	Replacing the wire allows for more accurate temperature readings.	76/14
9/9/14	Replaced inner lower sand seal material in dryer.	Prevents airborne uranium and keeps the material in the dryer.	77/14
9/10/14	Rebuilt all six burner regulators with new internal parts. Replaced thermocouples in all three hearths. Two of the three thermocouples were shorted out and all three were too long.	The regulators help in controlling the flame and therefore the temperature. The new thermocouples allow for more accurate burner control.	78/14
9/12/14	Calibrate all burner controls and air controls.	Runs smoother and allows for proper air mixture for combustion.	81/14
9/17/14	Replace pilot solenoid on burner 2.	Prevents flameout.	82/14
9/18/14	Draft differential valve would not move. Upon investigation the valve flap was bent and the piping was ¾ full of black cake. Repaired the damper valve.	Cleaned the draft pipe and improved air flow through the dryer. Allowed for the bag house to operate more efficiently.	84/14, 85/14
9/22/14	Replaced fire eye sensor for the second hearth, and the heat seal coupling.	Sight glass that allows for operator to see flame.	86/14
9/25/14	Adjusted the high and low fires for burner control because of better draft through the furnace.	With the draft differential valve repaired an increase in the furnace draft required for burner control adjustment.	88/14
9/29/14	Cleaned ID fan and checked balance, also opened up each hearth to inspect beds and clean burner ports inside the hearths.	Helps motor run smoother which relieves excessive vibration of the fan.	91/14
9/30/14	Readjusted the high fires back down because the high end temperature was climbing.	With increased draft the burners were getting too much air on the high side which caused the temperature to climb.	92/14
10/1/14	Had to adjust the Honeywell controllers to slow down the valve actuators for the burner.	This allows for a slower and better response control of the burners.	93/14

4.2.1 Dryer Temperature Profile Evaluation

While the newly installed thermocouples located in each of the hearths in the dryer showed that the operating temperatures of each hearth was being accurately measured, the final temperature of the dried yellowcake as it entered the drum still appeared considerably lower than the exit temperature of the bottom of the dryer. To better understand this temperature change from the final hearth to the drummed product, two thermocouples were installed in the dryer discharge system to determine what the drop in temperature would be as the dried yellowcake passed through the discharge system. Figures 4 and 5 show the discharge system and the location of the two additional thermocouples located at the first available point in the discharge pipe after the delumper and one after the star feeder which passes the dried material into the drum. Thermocouple probe CT1 was installed just after the delumper and above the star feeder and represents the temperature of the product as it is coming out of the dryer. The distance between the first thermocouple location and the exit of the dryer is roughly seven feet. Thermocouple DT1 was installed such that the probe hangs inside the drum being filled and measures the product temperature as it is being placed in the drum. The distance between the drum lid and the dryer exit is approximately 11 feet.

The discharge system from the dryer to the drum passes through two mechanical devices (the delumper and star feeder) which contribute to some of the observed temperature drop. In addition, a vent system is connected to the drum head which provides for the collection of dust as the drum head is being removed. All of these contribute to the large temperature drop that is seen from the dryer discharge to the drum. The thermocouple placed in the discharge line is believed to be reading the temperature of the bulk air stream in the line rather than the actual dried product temperature.

Figure 4: Schematic of Hearth Dryer and Thermocouple Locations

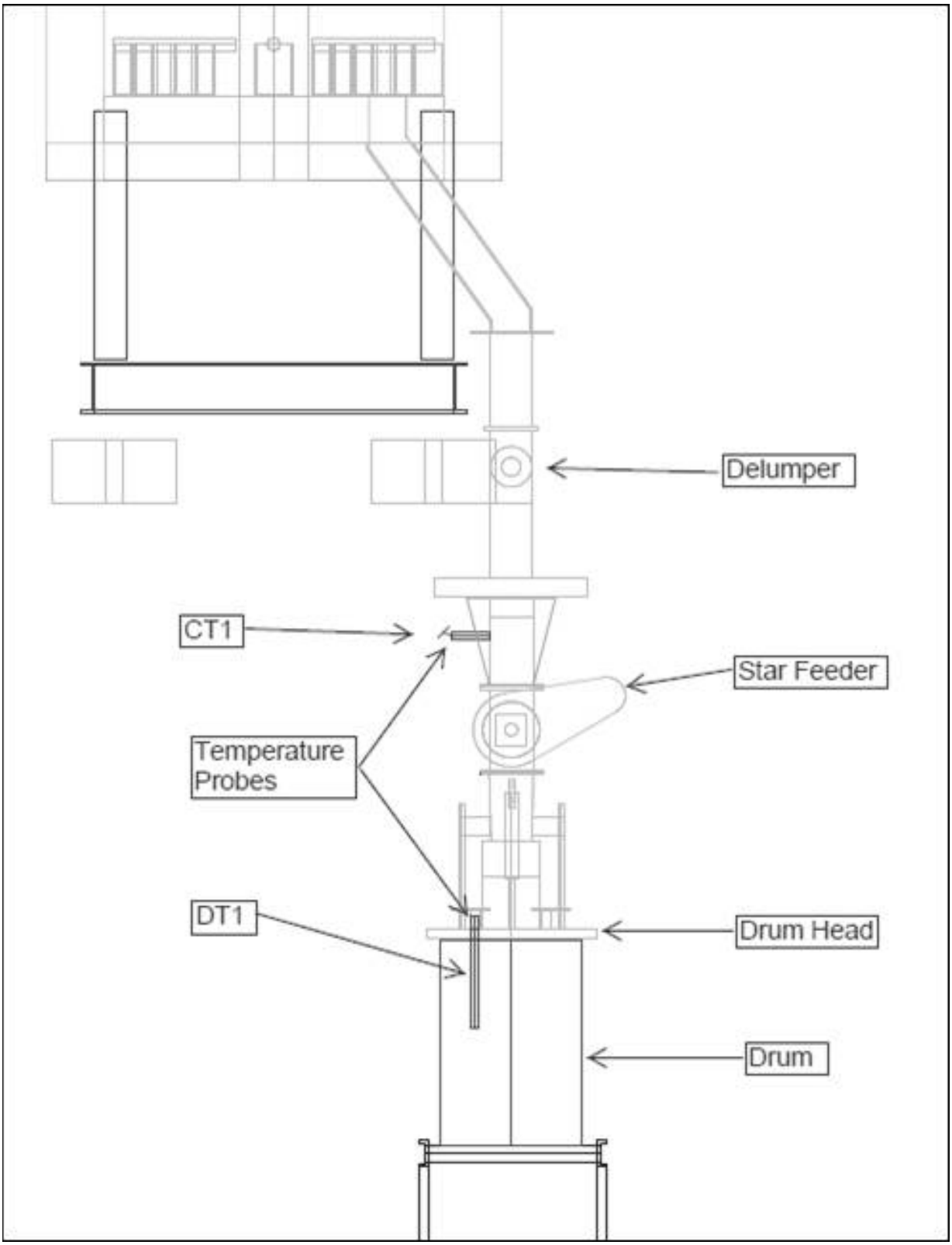
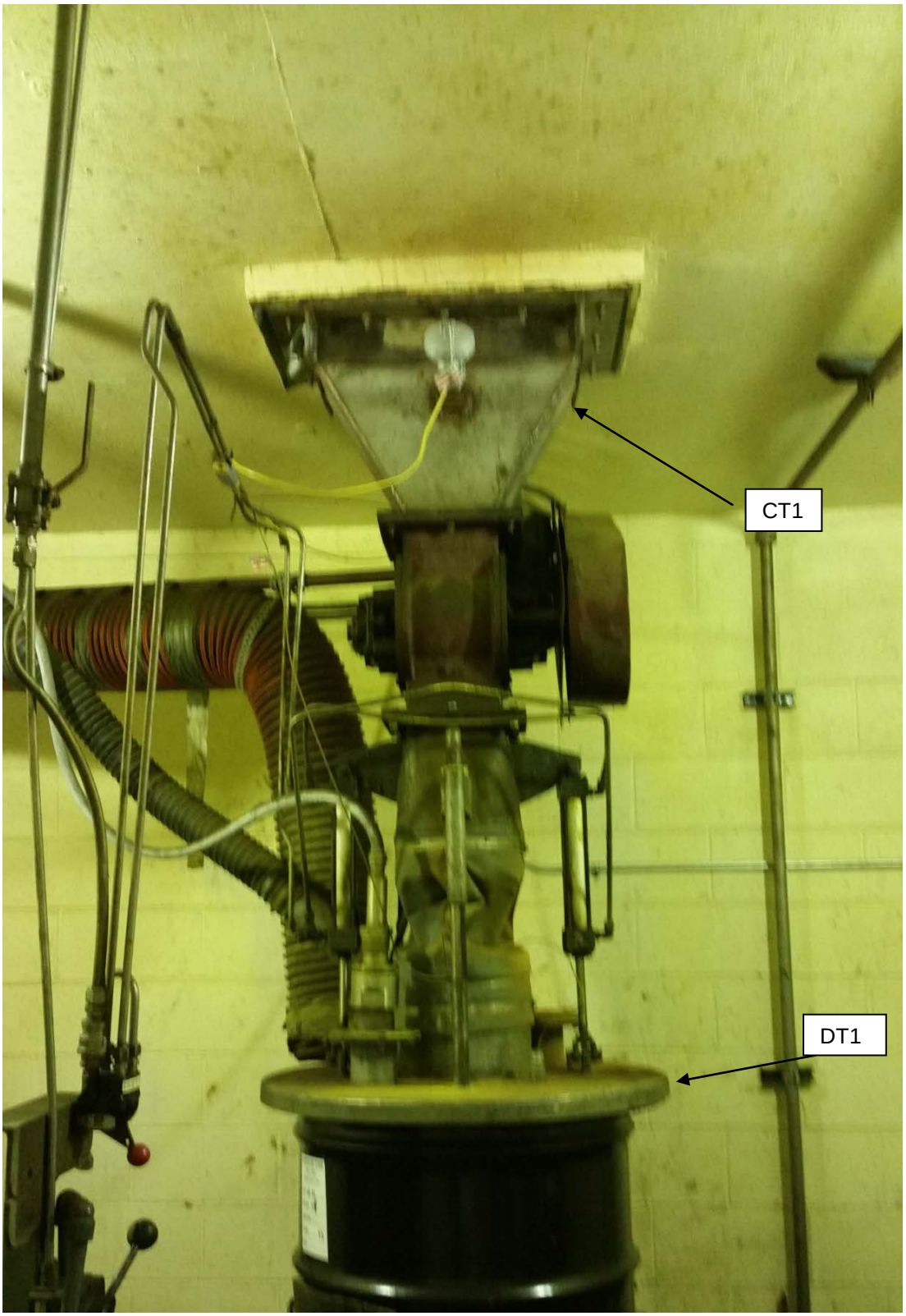
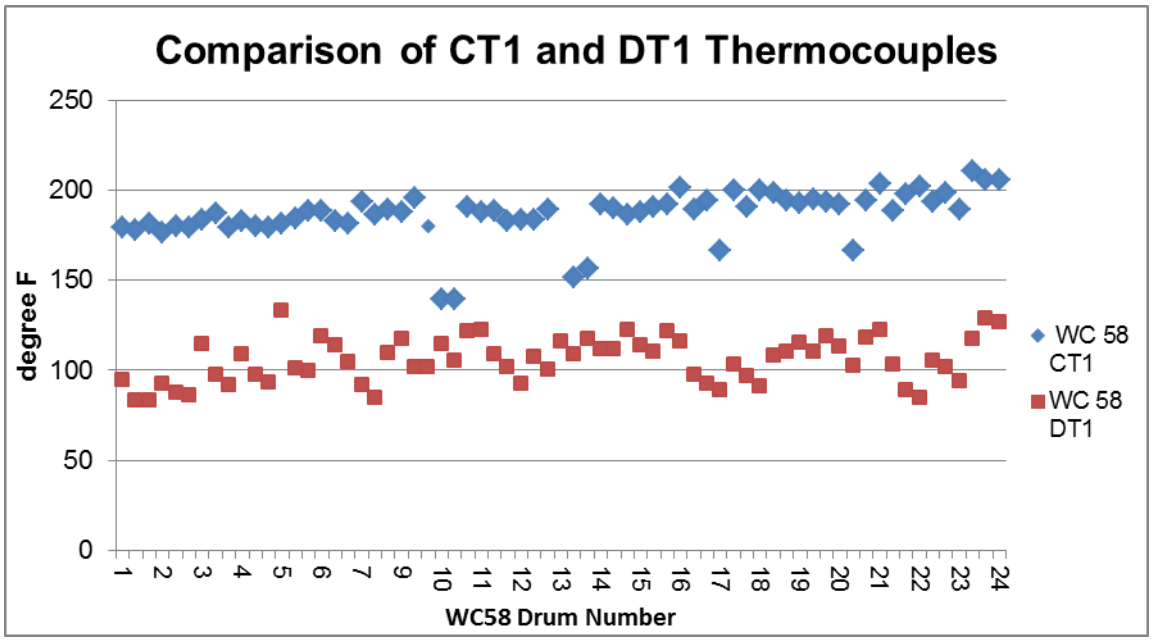


Figure 5: Picture of Thermocouple Locations



During this phase of the investigation operators were required to record three temperature measurements of the drum while it was being loaded. Figure 6 shows the temperature readings of both thermocouples for all of the drums monitored. As can be noted in the graph the thermocouple above the star feeder provides more consistent measurements. The thermocouple inside the drum has more variance possibly because it was measuring the head space temperature of the drum. It is evident that the temperature in the drum head space, not the dried yellowcake, was being measure by DT1 until the hot yellowcake covered the probe. As can be seen in the drum temperature profiles given in the next section (Figures 7 – 9), once a thermocouple is fully immersed in the yellowcake product the temperature was found to be higher.

Figure 6: Comparison of the Thermocouples



4.2.2 Packaged Drum Temperature Profile Evaluation

Temperature profiles were taken of the yellowcake inside drums as they were allowed to vent. The profiles were created by placing stationary temperature probes into 3 yellowcake drums (WC58 Drum 17, WC58 Drum 18, and WC58 Drum 19) and recording measurements every 2 hours over a monitoring period of at least 76 hours as is shown by Figure 7. It was noted that the temperatures inside the drum appeared to be highly dependent on the location of the probe within the drum. Due to the insulating properties of the yellowcake, a thermal gradient was observed inside the drums such that temperature was a function of depth and closeness to the outside edges of the container.

The thermal gradient was quantified by measuring temperatures at two-inch increments in the center of a drum (WC58 Drum 16) and recording the temperature measurements. The drum contained approximately

31 inches of yellowcake and the measurements were taken over a 10-minute time frame. The results are shown in Figure 8.

Additionally, to further identify the temperature profile, operators recorded initial, after 12 hours, and after 24 hours temperature measurements of the yellowcake product. Operators performed these measurements in drums WC58 Drum 7 through WC58 Drum 48 for a total of roughly 74 measurements. These measurements were then plotted against the temperature profiles that were created as shown in Figure 9.

Figure 7: Drumming Temperature over Time

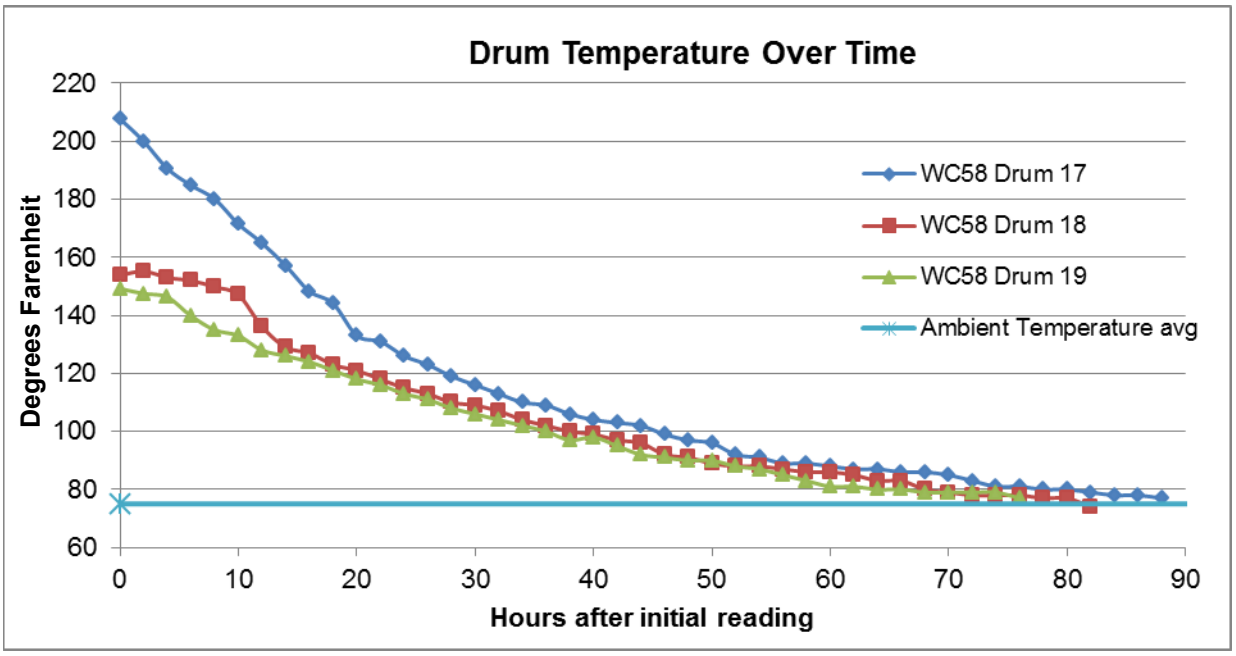


Figure 8: Temperature Variance in WC 58 Drum 16

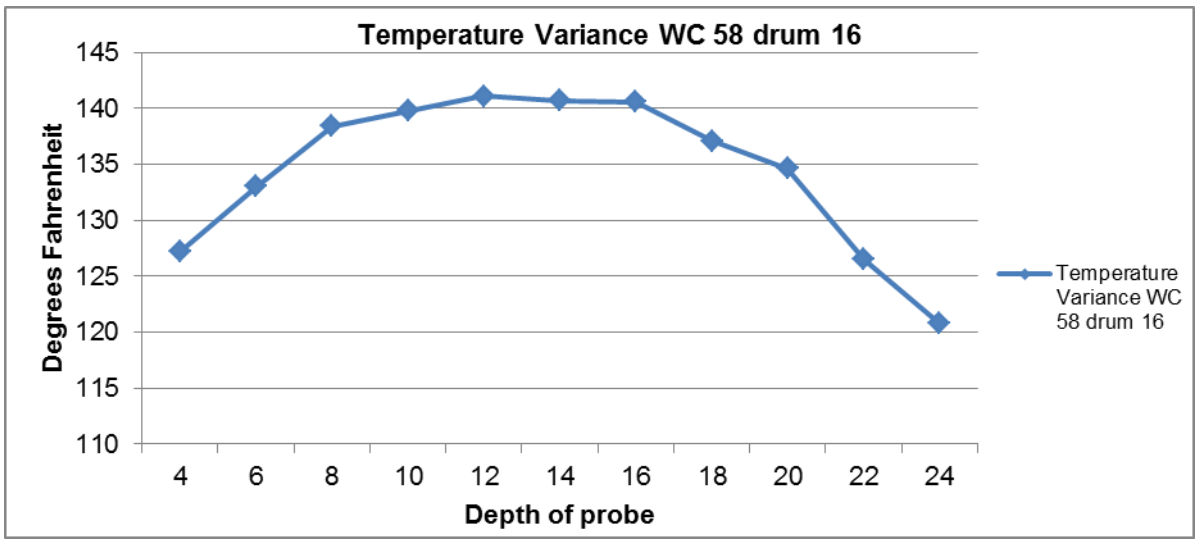
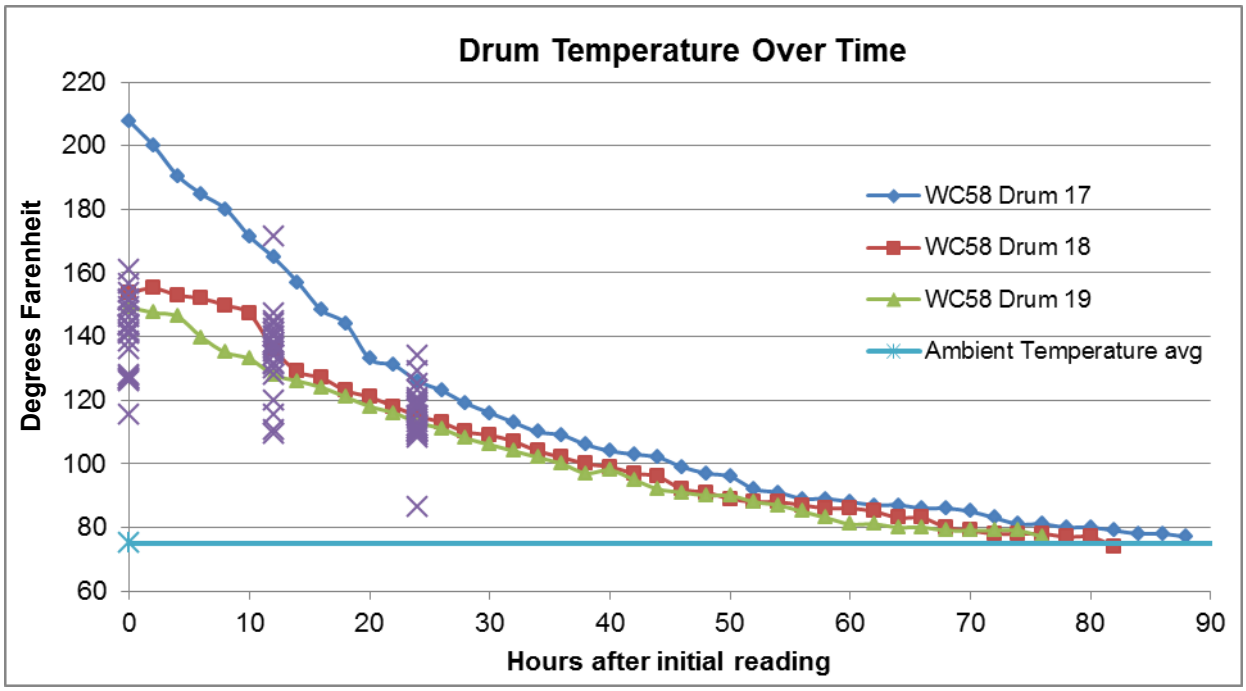


Figure 9: Temperature Measurements in Comparison with Temperature Profiles



4.2.3 Packaged Drum Pressure Testing

Pressurization tests were performed on dried product drums to verify that no pressure was building up in the production runs. As shown in Figure 10, four drums were monitored, (WC57 Drum 9, WC57 Drum 10, WC57 Drum 49, and WC57 Drum 48). Drums WC57 Drum 9 and WC57 Drum 10 were allowed to vent for 24 hours and then were sealed with a pressure test lid. The lid was equipped with a zero to five pound-per-square-inch (psi) gauge and a bleed-off valve. The temperature at the time of sealing the drums was approximately 100°F (38°C). Two additional drums WC57 Drum 47 and WC57 Drum 48 that had already been sealed and were in storage were re-opened and pressure lids were installed. The temperature of the product when pressure lids were installed was 80°F (27°C). Each of the drums was monitored for at least eight days and no pressurization was observed. Additionally, a pressure test was performed on a drum that was vented using the old procedure of placing the lid loosely on the drum. To represent a possible worst case scenario a lid with a pressure gauge was placed directly on the drum and the closure ring was loosely placed on the drum as it was allowed to vent for 24 hours. The pressure gauge was then monitored for 24 hours with no observable pressure build-up.

Figure 10: Picture of Selected Drums for Pressurization Test

4.2.4 Drum Venting and Cooling Procedures

The procedure for cooling and venting of filled dried yellowcake drums at the time of drying Lot 51 required that a lid be loosely set on the drums to allow cooling and venting for 24 hours to avoid pressurization of the drum. To prevent the possibility of yellowcake becoming airborne, the lid was placed on the drum and was offset to leave a gap sufficient for gas venting. Through conversation with operators it became apparent that there were multiple ways in which lids were being placed on drums, including some operators placing the lid and loosely attaching the closure ring around the drum with no offset gap. One of U1's concerns is that a drum lid with a closure ring loosely installed would not allow for adequate ventilation. It was also a concern that the heat from the drum could possibly seal the lid to the drum rim. This was seen as a possible cause of pressure being held in the drum during the venting cycle. When the prescribed period of venting was concluded and the closure ring was bolted on the drum the potential of sealing the drum with some positive internal pressure could occur.

These issues were discussed with site personnel and an alternate method of venting was proposed. A new temporary lid was designed, replacing the solid lid with a fine mesh screen lid. This temporary lid, when placed on the drum, allows the drum to fully vent for the entire cooling and venting period while still preventing the airborne escape of uranium oxide dust. Once the cooling and venting period is complete, the temporary mesh drum lid is removed and the final lid, ring, and bolt are attached.

Figure 11 is a picture of the temporary drum lid with the inserted open screens. Three sizes of mesh screens were evaluated; 200, 400, and 600 micron sieve screens. All of these mesh sizes allowed for release of gas and heat while also preventing yellowcake from becoming airborne.

Measurements using a four-gas meter indicated that the screens allow for proper ventilation of any gas residuals in the drum. The meter was used with the measurement tube placed inside a drum underneath the screened lids. These measurements were performed on four drums and all drums had oxygen levels at or close to background (within 0.1 percent) indicating that any uranyl peroxide decomposition reaction that could take place is either not occurring, or occurring to a very small degree. Using the mesh screened lids allows for more complete ventilation and cooling of the packaged yellowcake.

Figure 11: Picture of Screened Lid

4.2.5 Physical Evaluation of the Shipping Drums

The quality of the refurbished drums used for packaging yellowcake was also investigated to determine if any possible contaminants could be present which could give rise to chemical reactions with the dried yellowcake. Each drum is examined prior to use as a packaging and shipping container for the dried yellowcake product. There have been occasions where some film has been observed in the lids of some of the refurbished drums. While this is a rare occurrence, it was decided to sample and analyze this material to determine if this material is organic in nature. Analysis of the material on the lid revealed that there were no volatile organics on the lid and that it contained ten milligrams (mg) of n-hexane extractable

material. It was concluded that this very small amount of film could not be responsible for chemical reactions leading to drum pressurization. Conversation with the drum refurbishing company indicates that the lids also have an epoxy phenolic lining. This lining material was also considered to be a negligible contributor to any potential gas generation reactions.

The lid gasket was also evaluated to determine if it was possible for the gasket material to form a seal on the drum rim when exposed to high temperature conditions as might be experienced when a drum is filled with hot yellowcake. The gasket is made of an open cell natural rubber sponge material which has been the industry standard for drum seal material and has been used for many years in the packaging of dried yellowcake. It was sometimes observed that the gasket material tended to stick to the rim of the drum indicating a partial melting of the gasket to the rim of a hot drum. While partial gasket melting is seldom observed, it is possible that during cooling and venting of the drum, a lid could become sealed on the hot drum rim, preventing the release of heat and gases prior to final closure. This could be especially true if the lid and ring closure was added to the drum during cooling and venting. The new cooling and venting procedure proposed by U1 using a temporary screened lid will prevent partial gasket melting and inadvertent sealing of the drum during the venting cycle.

The lid gasket was additionally evaluated to determine if water could penetrate the seal during normal washing operations or when exposed to rain when stored outdoors at the receiving refinery. The normal decontamination procedure for the drum exterior was to power wash the drum with a high pressure spray. This was done to ensure that the alpha survey of the drums shows no appreciable exterior contamination.

Three experiments to determine if power washing or rainfall could result in water intrusion into the drum were performed. During the first test, an empty sealed drum was power washed using normal washing procedure. The drum exterior was then carefully dried and the drum opened. The interior of the drum was found to be completely dry. A second empty drum was sealed and subjected to a very aggressive power wash which included directly spraying the drum closure mechanism with the high pressure jet. After careful drying of the exterior, the drum was opened and several drops of moisture were observed. A third test was conducted with a sealed empty drum to simulate a rainfall event. The drum was placed under the flow of an emergency shower for a period of twelve hours. After drying the exterior, the drum was opened and the interior was found to be completely dry.

Based on the results of the experiments, U1 eliminated the aggressive power washing step in their procedure and incorporated a less aggressive washing step. This new process will insure that no moisture can enter a drum during the decontamination operations prior to shipping.

4.2.6 Theoretical Calculations of Pressure Changes Due to Environmental Conditions

Calculations were performed to determine if a change in ambient temperature or altitude could be responsible for pressure build-up in a shipping drum. The elevation of the Irigaray drying facility at approximately 4,400 ft. is a higher elevation than both of the yellowcake refineries used by U1 (Honeywell and Blind River). If a drum was shipped having an internal atmospheric pressure as found at the Irigaray site, then shipping to either of the refineries would exert a higher atmospheric pressure on the drum. A slight negative pressure would exist within the drum at the receiving refinery. For example, shipping a sealed drum from an elevation of approximately 4,400 ft. (Irigaray) to the Honeywell site at an elevation of 350 ft. would result in a negative pressure (internal drum pressure relative to ambient) of approximately 1.8 psi. Accordingly, shipping drums from Irigaray to the Honeywell or Blind River sites could not result in the build-up of internal drum pressure due to a change in altitude.

Another calculation was performed to determine if a significant change in temperature could result in pressure build-up in the drum. If a drum initially closed at a temperature of 100^{o}F and containing 20% free head space were to be subjected to a temperature rise of 25^{o}F and 50^{o}F it would be possible that this change in drum temperature could respectively give rise to a 0.6 psi and 1.3 psi change in the internal drum pressure. However, since the altitude change from Irigaray to the refinery sites resulted in a negative pressure, even raising the temperature of the drum by 50^{o}F would not result in any significant increase in the internal pressure of the drum.

4.2.7 Organic Contamination of the Yellowcake.

A careful examination of the potential for organic contamination in the yellowcake was also undertaken. Each operation in the plant where lubricants were used was evaluated and it was confirmed that no organics could have entered the yellowcake product. Confirming gas analysis samples from dried Willow Creek yellowcake drums showed elevated levels of oxygen (O_2) and nitrogen. Analysis of gas samples for CO_2 and CO did not show levels which would indicate decomposition gases of an organic reaction. The elevated levels of oxygen and nitrogen along with normal levels of CO and CO_2 is a clear indication that the gas in the pressurized drum was generated by a reaction of decomposing uranyl peroxide hydrates and that no organics were involved in the gas generation for the over-pressurized drum.

4.3 Uranyl Peroxide Chemical Considerations

The chemical conversion of uranyl peroxide at elevated temperatures to uranium oxides was investigated by independent laboratories utilizing a series of analytical tests to determine what uranium crystalline structure (intermediate oxides or peroxides) could exist in the dried Willow Creek yellowcake. This investigation primarily involved the use of x-ray diffraction (“XRD”) and thermogravimetric analysis (TGA) on various dried yellowcake products from the Irigaray drying operation.

4.3.1 XRD Analysis of Dried Uranyl Peroxide

Various samples of yellowcake dried in the Irigaray dryer at approximately 1,200°F (704°C) were sent to Evans Analytical Group, a laboratory specializing in the analysis of solid materials using XRD and to Dr. Peter Burns, Director, Energy Frontier Research Center *Materials Science of Actinides* at Notre Dame University. Dr. Burns is a well-respected uranium researcher who has conducted significant work in the behavior of uranium oxides and peroxides.

Evans Analytical was initially sent three samples of the production run from Lot 57 (WC57 Drum 28, WC57 Drum 37, WC57 Drum 39) that was dried during late August, September and into early October 2014, and a sample of the affected drum (WC51 Drum 43) that was taken from a sample received from the Honeywell refinery. The samples from Lot 57 represent several aspects of the production of dried yellowcake before and after the dryer refurbishments. WC57 Drum 28 is product that was taken before the refurbishment and calibration of the dryer was completed. WC57 Drum 37 and 39 represent dried yellowcake that was produced at 1,200°F (650°C) after the completion of the dryer refurbishment. WC51 Drum 43 is a sample from Honeywell that was taken from the pressurized drum. In addition, Evans Analytical was sent a recent (January 2015) sample of dried yellowcake that was produced at 1,300°F.

Six samples were sent to Dr. Burns at the Energy Frontier Research Center *Materials Science of Actinides* at Notre Dame University for analysis. Four of these samples were composites of drums from Lots WC51, WC53, WC55, and WC56. The other two samples were from the WC Lot 57 production run (WC57 Drum 42) and the other was from the pressurized drum (WC51 Drum 43) as received from Honeywell.

4.3.1.1 Evans Analytical Group XRD Study

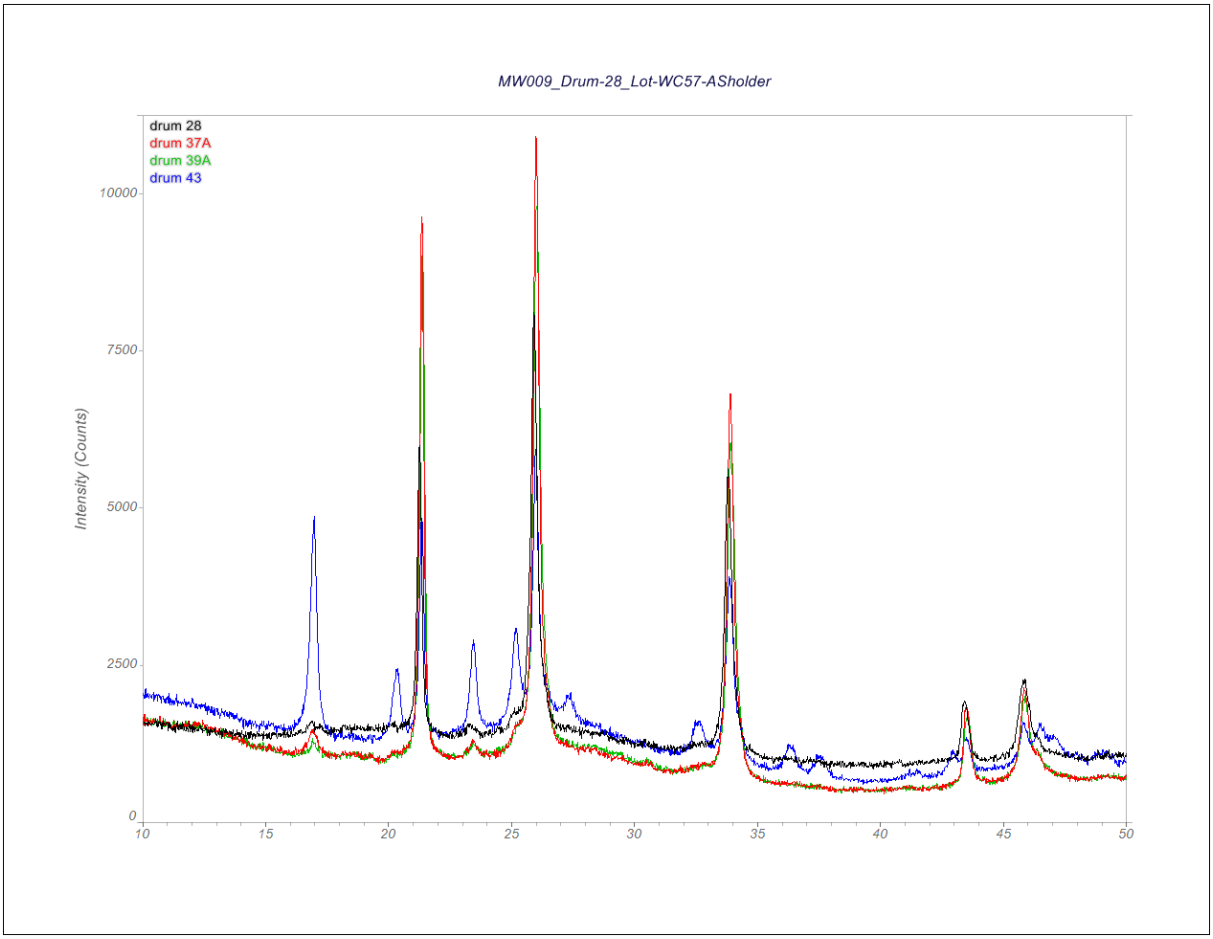
Evans Analytical performed XRD analysis on the four initial yellowcake samples as described above and one sample taken from the most recent drying of yellowcake at 1,300°F (January 2015). The results of the four XRD analyses are summarized in Table 2 and the comparison XRD spectra for the samples is shown in Figure 12. The difference in the spectra from WC57 Drum 28 and the two other spectra from Lot 57 (WC57 Drum 37 and WC57 Drum 39) is that there is a difference in the peak shape and the presence of a few low angle peaks. The XRD data from WC51 Drum 43 is considerably different than the other samples as shown in Figure 12. WC51 Drum 43 has the highest percentage of amorphous uranyl peroxide (metastudite heated above 230°C). A copy of the full Evans Report is included in Appendix B.

Table 2: Evans Analytical Result Summary –Initial XRD Samples

Sample ID	Phases Identified	Concentration wt% (+/- 5%)
Sample 1 (WC57 Drum 28) Air-sensitive holder	UO_3 – Uranium Oxide Hexagonal P-3m1 PDF# 00-031-1416	24.5
	$UO_4.2H_2O$ – Metastudtite Orthorhombic Immm PDF# 00-035-0571	1.1
	Amorphous materials	74.4[a]
Sample 2 (WC57 Drum 37A) After exposure to air/moisture	UO_3 – Uranium Oxide Hexagonal P-3m1 PDF# 00-031-1416	38.4
	$UO_4.2H_2O$ – Metastudtite Orthorhombic Immm PDF# 00-035-0571	3.5
	$(UO_2)4O(OH)_6(H_2O)_5$ – Metaschoepite?? Orthorhombic Pbcn PDF# 04-011-3920	1.4
	Amorphous materials	56.7[a]
Sample 3 (WC57 Drum 39A) After exposure to air/moisture	UO_3 – Uranium Oxide Hexagonal P-3m1 PDF# 00-031-1416	32.5
	$UO_4.2H_2O$ – Metastudtite Orthorhombic Immm PDF# 00-035-0571	0.9
	$(UO_2)4O(OH)_6(H_2O)_5$ – Metaschoepite?? Orthorhombic Pbcn PDF# 04-011-3920	0.2
	Amorphous materials	66.4[a]
Sample 4 (WC51 Drum 43) After exposure to air/moisture	UO_3 – Uranium Oxide Hexagonal P-3m1 PDF# 00-031-1416	15.3
	$UO_4.2H_2O$ – Metastudtite Orthorhombic Immm PDF# 00-035-0571	13.8
	$(UO_2)4O(OH)_6(H_2O)_5$ – Metaschoepite?? Orthorhombic Pbcn PDF# 04-011-3920	0.4
	Amorphous materials	70.5[a]

(a) The amorphous material in the sample has not been clearly identified. These results are uncertain due to unknown density/stoichiometry of the sampled material.

Figure 12: Evans Analytical Yellowcake XRD Analysis Comparison

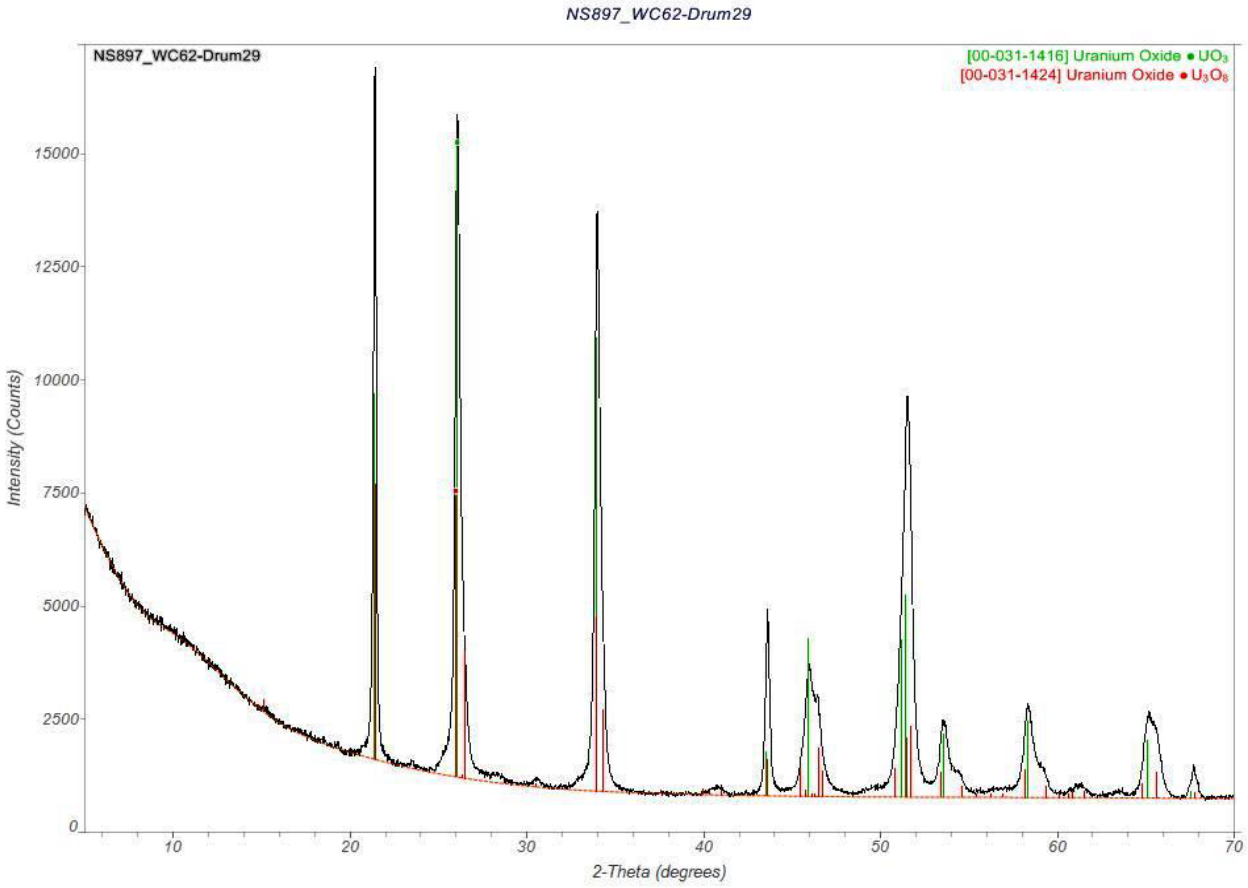


A more recent sample of yellowcake from Lot 62 that was dried to 1,300°F in January of 2015 showed that the presence of any residual amorphous uranyl oxide is virtually gone. This indicates that the material has all converted to UO3/U3O8 and should be stable. Table 3 summarizes the XRD analysis for this sample and the XRD pattern for this sample is shown in Figure 13.

Table 3: Evans Analytical Result Summary – High Temperature XRD WC62 Drum 29 Sample

Sample ID	Phases Identified	Concentration wt % (+/- 5%)
WC62 Drum 29	UO_3 – Uranium Oxide Hexagonal P-3m1 PDF# 00-031-1416	77.3
	U_3O_8 – Uranium Oxide Orthorhombic C2mm PDF# 00-031-1424	22.7

Figure 13: XRD Phase Identification for Sample WC62 Drum 29



4.3.1.2 Notre Dame XRD Study

Dr. Peter Burns. Professor of Civil and Environmental Engineering and Director, Energy Frontier Research Center *Materials Science of Actinides* at Notre Dame University was also consulted to evaluate the XRD patterns of the yellowcake from various samples of the Lot 57 production run, historic production runs and a sample of the pressurized drum, WC51 Drum 43. The results of the XRD spectra from his analysis are given in Appendix C Notre Dame XRD Data.

Dr. Burns and his team of researchers found similar XRD results to those obtained by the Evans Analytical Group for all of the initial samples that were analyzed. The interpretation of the data by both groups varied, however, as each interpreted various spectra in a different manner. The Notre Dame Study provides the following summary conclusions:

- WC51 Drum 43 showed the highest level of metastudtite and amorphous uranyl oxide (metastudtite heated above 230°C) indicating that the possibility of a lower temperature of drying was experienced for this drum.

- The three composite samples from historic dryer campaigns, (WC53, WC55, and WC56) all showed much lower levels of metastudtite and increased amorphous uranyl oxide (metastudtite heated above 230°C).
- The sample from Lot 57 production run (WC57 Drum 42) showed negligible levels of metastudtite and significant amounts of amorphous uranyl oxide. As in the Evans study, all samples showed a significant level of amorphous uranyl oxide.
- The higher temperature (1,300°F) sample (WC62 Drum 29) was not analyzed by the Notre Dame lab.

The Notre Dame study also identified the possibility that amorphous uranyl oxide (metastudtite heated above 230°C) could be involved in a gas generation reaction that produces oxygen gas if the material is allowed to come into contact with water. This is also more fully explained in the discussion section below.

4.3.2 Discussion of the Chemical Characteristics of Uranyl Peroxide and Uranium Oxides

4.3.2.1 Uranyl Peroxide and Uranium Oxide Mineralogy.

It is well known that uranyl peroxide hydrates produced from hydrogen peroxide precipitated uranium will decompose under high temperature conditions to produce lower forms of the uranyl hydrate and uranium oxides. The nature of this heat driven decomposition is the basis of the dryer operations at the high temperature dryer at the Irigaray drying facility. The uranium minerals of interest in these drying and decomposition reactions can be summarized in the following table.

Table 4: Uranium Minerals Involved in Yellowcake (YC) Drying

Compound Name	Chemical Composition	Generic Designation	Drying Range
Studtite	$(UO_2)(O_2)(H_2O)_2(H_2O)_2$	$UO_4 \bullet 4H_2O$	YC Slurry and YC Dried to ~ 130°C
Metastudtite	$(UO_2)(O_2)(H_2O)_2$	$UO_4 \bullet 2H_2O$	~130°C to ~230°C
Amorphous Uranyl Peroxide (Hydrate)	$UO_2(O_2) \bullet (H_2O?)$ to $UO_3(O_x) \bullet (H_2O?)$ where x is thought to be 0.5 – 1.0 and some water of hydration may be retained in the lower temperature range	UO_{3+x} (Note 1)	~230°C to ~ 520°C
Uranium Trioxide	UO_3	UO_3	~520°C to ~ 650°C
Triuranium Octoxide	U_3O_8	U_3O_8	650°C +

Note 1: The form of the amorphous uranyl peroxide and its hydrates is not fully understood. However, it has been shown that metastudtite decomposes gradually in the ~230°C to ~ 520°C temperature range with the loss of both water(s) of hydration and a gradual loss of oxygen to form the stable UO_3.

As can be seen from this table, once the uranyl peroxide tetrahydrate (studtite) is formed from the precipitation of uranium with hydrogen peroxide it will decompose in the temperature range of ~130°C to

~230°C to the uranyl peroxide dihydrate (metastudtite). When metastudtite is heated in the range of ~130°C to ~230°C it dehydrates and, in the range of 230°C - 520°C, it will form an intermediate amorphous uranyl oxide compound (UO_{3+x}). This intermediate amorphous uranyl oxide may still retain some water of hydration but thermogravimetric analysis indicates that most of the water of hydration is liberated early in this temperature range (<230°C). At the upper end of this temperature range the amorphous compound will further decompose to fully dehydrate with the release of oxygen gas to form the stable UO_3. Temperature increases above 520°C begins the transition from UO_3 to form the stable U_3O_8. In the drying of yellowcake at elevated temperatures a mixture of the oxides of UO_3 and U_3O_8 is typically the composition of the dried material.

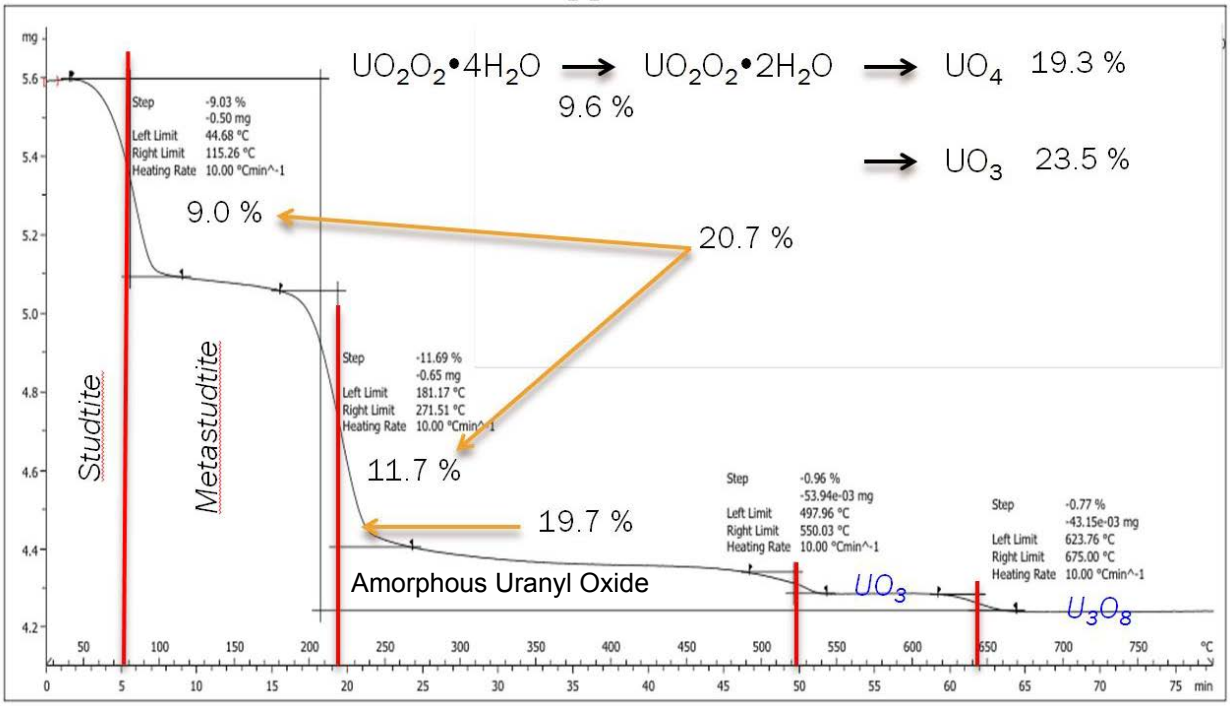
The phenomena of an intermediate uranyl oxide formation that is produced from the decomposition of metastudtite upon heating the dihydrate in the 230°C to 520°C range has previously been reported in the literature (Ref. 1, 2, 3, 4, 5) although no definitive determinations of this compound have been made. Earlier literature reports that this compound is an “amorphous” uranyl hydrate compound often designated in these studies as, UO_x where x is said to be between 3 and 3.5. More recent studies of this compound at the Notre Dame laboratory have more clearly shown that this intermediate material is an amorphous uranyl oxide with a poorly defined crystalline structure that results from the nearly complete dehydration of the uranyl peroxide dihydrate around 230°C and the decomposition of the resulting uranyl oxide to a variety of oxide forms (UO_x) which have not been clearly identified.

Thermogravimetric analyses of the dehydration reactions of studtite and metastudtite have been investigated by Rey *et al.* (2009) (Ref 6) and by Burns (2014) (Ref 7). In both cases the starting material was verified as studtite (tetrahydrate) by powder XRD. The heating rates of the two studies differed, but each showed the following progression of phases:

Studtite → metastudtite → amorphous uranyl oxide → UO_3/U_3O_8

The transition temperatures and mass losses are shown in Figure 14. The theoretical and measured mass losses for the first two transitions are in very good agreement. Furthermore, X-ray diffraction data confirm the presence of the four phases in the appropriate samples (studtite, metastudtite, amorphous uranyl oxide and UO_3/U_3O_8).

Figure 14: Uranyl Peroxide Thermogravimetric Analysis



The decomposition of metastudtite in the temperature range of 230°C – 520°C is shown on X-ray diffraction patterns as peak broadening and overall reduction of the quality of the diffraction character. As such, mixtures of various oxide states of uranyl peroxide can be said to be poorly crystalline, although they do have a definite structure and composition within a defined range. The point at which one decides to call a poorly crystalline material "amorphous" is arbitrary in a case such as this. However, it is notable that one does not need to invoke the existence of an unknown phase, amorphous or otherwise, to explain the diffraction, spectroscopic, and thermogravimetric data collected by Burns (Ref 7, 2014).

As the uranyl peroxide tetrahydrate is heated, the following reactions will occur. Of interest in the evaluation of potential gas generation is the third equation. In this reaction the decomposition of the amorphous uranyl oxide can give rise to the formation of oxygen gas:

Up to ~130°C:	$UO_4•4H_2O$ + Heat → $UO_4•2H_2O + 2H_2O$	(1)
~130°C to ~230°C:	$UO_4•2H_2O$ + Heat → UO2(O2) $+2H_2O$	(2)
~230°C to ~520°C:	$UO_2(O_2)$+ Heat → UO_{3-4} + ~ ½O_2	(3)
Above 520°C:	Gradual conversion of UO_3 to U_3O_8	(4)

It has been shown that the conversion to the amorphous phase will not occur at temperatures below ~230°C, and this is the basis of low temperature vacuum drying to produce a dried yellowcake that is stable once it is cooled and vented after packaging. With the use of a high temperature dryer, the temperatures within the dryer drive the metastudtite to the amorphous oxide phase where increases in temperature can give rise to oxygen release as the material is converted to UO_3 (Equation 3). The conversion of the amorphous phase to the stable UO_3 and U_3O_8 occurs gradually as the temperature is raised to ~520°C and above. While this reaction occurs at high temperatures during the drying of the yellowcake, it does not occur at lower temperatures as in packaged yellowcake that has been sufficiently cooled and vented.

A review of the XRD data from both the Evans study and the Notre Dame study found that some of the dried Irigaray yellowcake still contained some amount of the amorphous uranyl oxide (with the exception of Lot 62). While continued heating of this amorphous phase will result in the release of oxygen gas, this reaction will not occur as the temperature is decreased. The reaction to decompose any residual amorphous material will not occur at the temperatures of a typical packaged drum if it is properly cooled and all gases are allowed to vent. This has been the basis for the industry standard to allow dried yellowcake to cool and vent to prevent capture of decomposition gases in the packaged yellowcake drum. As the industry has shown through the packaging and shipping of literally hundreds of thousands of dried yellowcake drums, proper cooling and venting prior to drum closure stabilizes the dried yellowcake even if it contains some compounds that could be capable of oxygen gas generation. The converse is also true, however, in that a drum improperly cooled and vented can capture decomposition gases which can result in drum pressurization.

4.3.2.2 Reaction of Metastudtite Heated above 230°C with Water

One of the additional questions that was evaluated in the root cause analysis was the possibility of a reaction of the dried yellowcake with water. It has recently been reported by Rogers, et al (Ref.8) that gas generation can occur when uranyl peroxide dihydrate is heated to intermediate temperatures (~230°C to ~520°C) and then comes in contact with water. A series of tests were conducted at the Notre Dame laboratory to determine if any of the Irigaray dried material could react with water to give rise to gas generation. All of the samples that were sent to Notre Dame were exposed to water and some had a reaction with water to produce noticeable gas generation. This was especially true for WC51 Drum 43 (the pressurized drum). Upon investigation of this result it was determined that the following reaction can take place with amorphous uranyl oxide and water to produce oxygen gas and a stable metaschoepite.

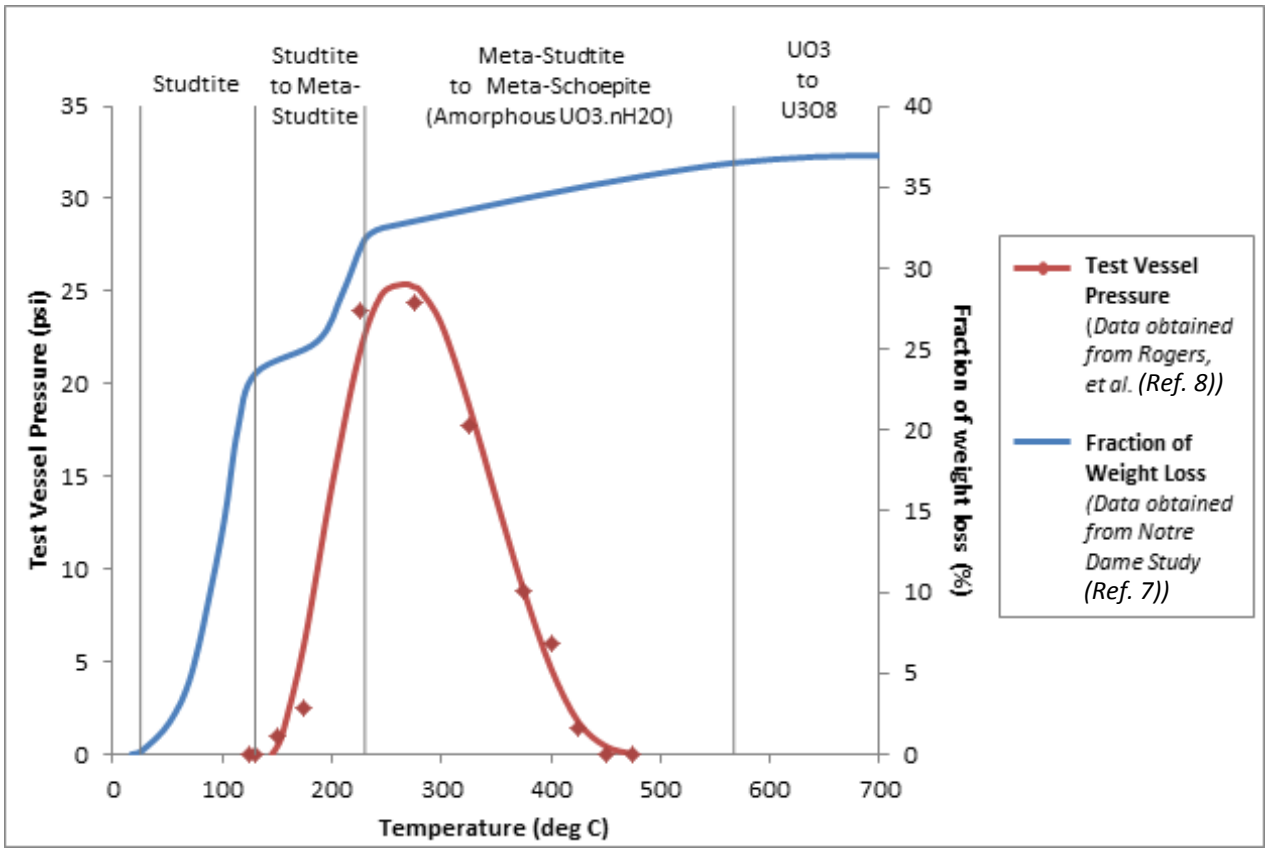
$$UO_{3+x}\,(\text{amorphous}) + 2H_2O \rightarrow UO_3 \bullet 2H_2O + \sim\tfrac{1}{2}O_2 \qquad (5)$$

Since some of the Irigaray samples contained some residual amorphous uranyl oxide it is possible that this reaction can take place when the material is exposed to water. This could be especially true for

WC51 Drum 43 which exhibited the highest amount of amorphous uranyl oxide as evidenced by the XRD analysis of both the Evans Analytical and Notre Dame studies. This reaction was observed by visual confirmation of gas evolution upon taking Irigaray dried yellowcake and exposing it to a significant amount of water. The evolving gas was found to be oxygen, indicating that the decomposition reaction of amorphous uranyl oxide when combined with water can occur without the need for high temperature uranyl oxide decomposition. It may be possible that this amorphous phase could release oxygen under conditions other than contact with liquid water such as contact with humid air. Once the metaschoepite, $UO_3 \bullet 2H_2O$, is formed it is a stable species and will not further react with water as was demonstrated in experiments conducted at the Notre Dame laboratory.

In order to verify the results that were presented by Rogers, et al, their data on pressure generation was superimposed on the general uranyl peroxide decomposition curve as reported in the Notre Dame study. These results are shown in Figure 15. This graph shows that once amorphous uranyl oxide begins to be formed we also see the beginnings of gas generation when this material is mixed with water. As the temperature increases in the range of 130 - 230°C, the metastudtite begins conversion to the amorphous oxide form and release of oxygen occurs when mixed with water which results in increased pressure. Further increases in temperature drive the amorphous uranyl oxide to UO_3 leading to a decrease in pressure as the amorphous uranyl oxide is decomposed to the more stable oxide form. Once mixed with water the amorphous uranyl oxide forms stable metaschoepite or other oxide forms which do not produce any oxygen generation as the rehydration occurs This indicates that water will not react with any other species once the amorphous uranyl oxide is decomposed.

Figure 15: Reaction of Amorphous Uranyl Oxide with Water



This relationship of water with amorphous uranyl oxide is an indication that water should not be allowed to come into contact with the dried product if the dried yellowcake still contains some amount of amorphous uranyl oxide.

The postulation of water contact with the dried Irigaray yellowcake, while possible, is considered to be unlikely. If water were somehow to enter the drum through a breach in the seal of the drum or the drum lid, then it is logical to assume that gas generated in the drum could escape via the same route. Since it is typically more difficult to pass a liquid through a leaking seal or breach of the container, gas release should be much easier to envision if it were to be generated from the reaction of water with the yellowcake. The only mechanism by which water could get into a drum without gas getting out is if the water were to enter the container just prior to the opening of the drum and there was not sufficient time for the gas to escape. U1 has checked with the operations personnel of the Honeywell facility and established that the drums are not washed or exposed to water upon entering the sampling room.

While it seems unlikely that water was involved in this pressurization incident, it is still important to note that the dried yellowcake should contain as little as possible of the reactive amorphous uranyl oxide species. As previously noted, U1 has refurbished the burners and control system of the Irigaray dryer to

increase dryer efficiency and have recently (January 2015) begun to operate the dryer at higher temperatures (1,300°F vs 1,200°F) to ensure that the potentially gas producing oxides of uranium are fully decomposed. When a comparison is made of the XRD data for WC51 Drum 43 with the XRD data from the recent production run (WC62 Drum 29), it is clear that the recently dried yellowcake has no amorphous uranyl oxide and that WC51 Drum 43 (the pressurized drum) had a significant amount of this material in the dried product.

5.0 CONCLUSIONS AND RECOMMENDATIONS

It is concluded that the fundamental root cause of the drum pressurization was a build-up of oxygen gas generated by the decomposition of residual uranyl oxides in the dried yellowcake product. The conditions that could have caused this situation are as follows:

1. Premature closure of the drum lid during the cooling and venting period. As previously described, a drum lid could have been placed on the drum with a loose ring closure attached. The ring closure could have exerted sufficient pressure on the lid seal to cause some of the venting gas to remain in the drum prior to the final closure of the locking bolt. It is also possible that the lid gasket sufficiently softened, due to the hot drum rim, causing the lid gasket to stick to the drum rim. This could have prevented the drum from properly venting. U1 has re-evaluated its cooling and venting procedures and will continue using specially designed temporary lids with a mesh opening to allow unimpeded cooling and venting, while preventing airborne release of uranium product prior to final closure of the drum lid. This screen mesh procedure has been incorporated into Irigaray Standard Operating Procedure (SOP) IR12. SOP IR12 has also been modified to require that all drums be allowed to cool and vent for a minimum of 48 hours, or until product temperature is approximately 90°F, prior to placement of the solid lid closure on the drum.
2. Potential Contact of the Dried Yellowcake with Water. It has been shown that dried yellowcake with a significant amount of residual amorphous uranyl oxide can react with water to produce oxygen gas. The mechanism by which this can occur has been explored; however, it has not been identified how contact with water can occur without the concurrent release of the generated gas. This is, however, a sufficient concern to the U1 operations and a new drum decontamination procedure has been implemented which will eliminate the possibility of water entering a drum prior to shipment.
3. Non-Optimal Dryer Operation. U1 has completed a dryer rehabilitation program that included a rework of the burner system and an upgrade to the dryer controls. This upgrade has enabled the dryer to operate at optimum levels to reduce the amount of un-decomposed metastudtite and amorphous uranyl oxide in the final dried product. As evidence of the effectiveness of this dryer maintenance program, it has been demonstrated by the use of XRD data that subsequent performance of the dryer produced a dried yellowcake product that showed reduced amounts of the amorphous uranyl oxide and higher amounts of stable uranium oxides. U1 will also commit to conducting a quarterly dryer inspection (in addition to the required annual inspection) to insure that the dryer is operating at optimum conditions.
4. Low Dryer Temperature. It has been shown that increased dryer temperatures will eliminate the presence of amorphous uranyl oxide. The Irigaray operation has, therefore, increased its recommended dryer operating temperature from 1,200°F to 1,300°F. This 100°F increase in temperature has eliminated the presence of amorphous uranyl oxides in the dried yellowcake and thereby has eliminated the potential for any reactive oxides to be present in the final packaged drum.
5. Pre-shipment Drum Pressure Buildup. U1 has considered the possibility that pressure could be captured in a closed drum prior to shipment. While it is difficult to postulate that this could occur for drums that are properly cooled and vented, U1 is proposing to open each drum prior to shipping to ensure that no pressure exists in the drum and that no moisture has entered the drum while it is awaiting shipment. This will ensure that all drums shipped to a refinery will not have positive internal pressure. U1 will conduct this procedure until they feel confident that the other proposed changes which have been implemented are effectively ensuring that no pressure build-up in dried yellowcake drums is occurring.

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APPENDIX A
LOT 51 DRYING AND PACKAGING RECORDS

DRUM_ID	Date/Time	Drumming Time	Gross	Tare	Net	%U	Net LBS U	LBS of U3O8	Init. Temp F	IR Moisture	CR Moisture	To Date Gross	To Date Net	Est Net Lbs U_3O_8
WC51-1	3/20/14 18:15	26:15:00	788	45	743	80.717%	590.9	696.8		1.4%		788	743	686.9
WC51-2	3/21/14 15:02	20:47:00	918	44	874	80.717%	695.0	819.6		1.0%	1.0%	1,706	1,617	808.0
WC51-3	3/27/14 14:10	143:08:00	920	46	874	80.717%	695.0	819.6		1.0%		2,626	2,491	808.0
WC51-4	4/2/14 6:50	136:40:00	877	45	832	80.717%	661.6	780.2		1.0%	1.0%	3,503	3,323	769.2
WC51-5	4/2/14 11:35	4:45:00	849	47	802	80.717%	637.8	752.1		1.5%		4,352	4,125	741.5
WC51-6	4/2/14 17:15	5:40:00	862	37	825	80.717%	656.1	773.7		1.2%	1.8%	5,214	4,950	762.7
WC51-7	4/2/14 23:35	6:20:00	833	47	786	80.717%	625.1	737.1		1.0%		6,047	5,736	726.7
WC51-8	4/3/14 7:25	7:50:00	913	45	868	80.717%	690.3	814.0		1.2%	1.6%	6,960	6,604	802.5
WC51-9	4/4/14 7:15	23:50:00	791	42	749	80.717%	595.6	702.4		1.0%		7,751	7,353	692.5
WC51-10	4/4/14 12:50	5:35:00	839	37	802	80.717%	637.8	752.1		1.6%	1.8%	8,590	8,155	741.5
WC51-11	4/7/14 20:05	79:15:00	858	36	822	80.717%	653.7	770.9		2.6%		9,448	8,977	760.0
WC51-12	4/10/14 16:30	68:25:00	835	44	791	80.717%	629.0	741.8		0.9%	1.1%	10,283	9,768	731.3
WC51-13	4/10/14 23:40	7:10:00	836	47	789	80.717%	627.5	739.9		0.6%		11,119	10,557	729.5
WC51-14	4/11/14 6:10	6:30:00	816	46	770	80.717%	612.3	722.1		0.6%	1.0%	11,935	11,327	711.9
WC51-15	4/11/14 13:00	6:50:00	845	45	800	80.717%	636.2	750.2		0.7%		12,780	12,127	739.6
WC51-16	4/11/14 19:40	6:40:00	868	43	825	80.717%	656.1	773.7		0.8%	1.2%	13,648	12,952	762.7
WC51-17	4/12/14 1:50	6:10:00	848	45	803	80.717%	638.6	753.0		0.6%		14,496	13,755	742.4
WC51-18	4/12/14 7:45	5:55:00	831	44	787	80.717%	625.9	738.0		0.7%	1.0%	15,327	14,542	727.6
WC51-19	4/12/14 14:20	6:35:00	924	45	879	80.717%	699.0	824.3		0.8%		16,251	15,421	812.7
WC51-20	4/12/14 20:45	6:25:00	906	45	861	80.717%	684.7	807.4		1.0%	1.3%	17,157	16,282	796.0
WC51-21	4/13/14 2:40	5:55:00	860	37	823	80.717%	654.5	771.8		1.3%		18,017	17,105	760.9
WC51-22	4/13/14 8:35	5:55:00	820	46	774	80.717%	615.5	725.9		0.7%	1.0%	18,837	17,879	715.6
WC51-23	4/13/14 14:30	5:55:00	879	47	832	80.717%	661.6	780.2		1.0%		19,716	18,711	769.2
WC51-24	4/13/14 20:35	6:05:00	892	46	846	80.717%	672.8	793.4		1.5%	1.8%	20,608	19,557	782.2
WC51-25	4/14/14 2:00	5:25:00	829	46	783	80.717%	622.7	734.3		1.4%		21,437	20,340	723.9
WC51-26	4/14/14 7:40	5:40:00	855	46	809	80.717%	643.4	758.7		1.2%	1.8%	22,292	21,149	747.9
WC51-27	4/14/14 13:30	5:50:00	880	43	837	80.717%	665.6	784.9				23,172	21,986	773.8
WC51-28	4/14/14 19:40	6:10:00	922	45	877	80.717%	697.4	822.4		1.3%	1.9%	24,094	22,863	810.8
WC51-29	4/15/14 1:30	5:50:00	889	45	844	80.717%	671.2	791.5		1.3%		24,983	23,707	780.3
WC51-30	4/15/14 7:25	5:55:00	899	45	854	80.717%	679.1	800.9		1.1%	1.4%	25,882	24,561	789.5
WC51-31	4/15/14 12:50	5:25:00	854	45	809	80.717%	643.4	758.7		1.7%		26,736	25,370	747.9
WC51-32	4/15/14 18:20	5:30:00	848	42	806	80.717%	641.0	755.9		1.4%	1.9%	27,584	26,176	745.2
WC51-33	4/16/14 0:15	5:55:00	890	37	853	80.717%	678.3	799.9		1.4%		28,474	27,029	788.6
WC51-34	4/16/14 6:05	5:50:00	872	45	827	80.717%	657.7	775.6		1.2%	1.9%	29,346	27,856	764.6
WC51-35	4/16/14 11:35	5:30:00	858	45	813	80.717%	646.5	762.4		1.0%		30,204	28,669	751.6
WC51-36	4/16/14 17:35	6:00:00	859	45	814	80.717%	647.3	763.4		1.0%	0.7%	31,063	29,483	752.6

WC51-37	4/16/14 23:00	5:25:00	846	43	803	80.717%	638.6	753.0		1.3%		31,909	30,286	742.4
WC51-38	4/17/14 5:15	6:15:00	915	45	870	80.717%	691.9	815.9			1.7%	32,824	31,156	804.3
WC51-39	4/17/14 11:35	6:20:00	917	45	872	80.717%	693.5	817.8				33,741	32,028	806.2
WC51-40	4/17/14 17:40	6:05:00	919	39	880	80.717%	699.8	825.3		1.4%	1.5%	34,660	32,908	813.6
WC51-41	4/17/14 23:45	6:05:00	871	46	825	80.717%	656.1	773.7		1.2%		35,531	33,733	762.7
WC51-42	4/18/14 5:40	5:55:00	861	48	813	80.717%	646.5	762.4		1.5%	1.8%	36,392	34,546	751.6
WC51-43	4/18/14 11:05	5:25:00	858	44	814	80.717%	647.3	763.4		1.1%		37,250	35,360	752.6
WC51-44	4/18/14 16:15	5:10:00	840	45	795	80.717%	632.2	745.5		1.1%	2.0%	38,090	36,155	735.0
WC51-45	4/18/14 21:30	5:15:00	814	44	770	80.717%	612.3	722.1		1.4%		38,904	36,925	711.9
WC51-46	4/19/14 3:05	5:35:00	851	37	814	80.717%	647.3	763.4		1.5%	1.6%	39,755	37,739	752.6
WC51-47	4/19/14 9:00	5:55:00	842	35	807	80.717%	641.8	756.8		1.0%		40,597	38,546	746.1
WC51-48	4/19/14 15:05	6:05:00	881	35	846	80.717%	672.8	793.4		1.1%	1.7%	41,478	39,392	782.2
WC51-49	4/19/14 20:50	5:45:00	867	45	822	80.717%	653.7	770.9		1.3%		42,345	40,214	760.0
WC51-50	4/20/14 2:45	5:55:00	858	45	813	80.717%	646.5	762.4			1.4%	43,203	41,027	751.6
WC51-51	4/20/14 8:55	6:10:00	864	45	819	80.717%	651.3	768.1		1.3%		44,067	41,846	757.2
WC51-52	4/20/14 14:50	5:55:00	868	46	822	80.717%	653.7	770.9		1.4%	1.5%	44,935	42,668	760.0

Drum	Date/Time Start Vent	Date/Time Stop Vent	Total Vent Time
WC51-1	3/20/14 18:15	3/22/14 4:30	34:15:00
WC51-2	3/21/14 15:02	3/27/14 14:20	143:18:00
WC51-3	3/27/14 14:10	4/2/14 12:00	141:50:00
WC51-4	4/2/14 6:50	4/3/14 7:40	24:50:00
WC51-5	4/2/14 11:35	4/4/14 7:25	43:50:00
WC51-6	4/2/14 17:15	4/4/14 7:30	38:15:00
WC51-7	4/2/14 23:35	4/4/14 7:35	32:00:00
WC51-8	4/3/14 7:25	4/4/14 13:00	29:35:00
WC51-9	4/4/14 7:15	4/7/14 3:45	68:30:00
WC51-10	4/4/14 12:50	4/7/14 3:45	62:55:00
WC51-11	4/7/14 20:05	4/9/14 14:01	41:56:00
WC51-12	4/10/14 16:30	4/11/14 19:45	27:15:00
WC51-13	4/10/14 23:40	4/12/14 2:00	26:20:00
WC51-14	4/11/14 6:10	4/12/14 16:05	33:55:00
WC51-15	4/11/14 13:00	4/12/14 16:08	27:08:00
WC51-16	4/11/14 19:40	4/12/14 20:55	25:15:00
WC51-17	4/12/14 1:50	4/13/14 2:55	25:05:00
WC51-18	4/12/14 7:45	4/13/14 14:35	30:50:00
WC51-19	4/12/14 14:20	4/13/14 14:37	24:17:00
WC51-20	4/12/14 20:45	4/13/14 20:50	24:05:00
WC51-21	4/13/14 2:40	4/14/14 5:50	27:10:00
WC51-22	4/13/14 8:35	4/14/14 20:00	35:25:00
WC51-23	4/13/14 14:30	4/14/14 20:10	29:40:00
WC51-24	4/13/14 20:34	4/15/14 1:45	29:11:00
WC51-25	4/14/14 2:00	4/15/14 18:30	40:30:00
WC51-26	4/14/14 7:40	4/15/14 18:40	35:00:00
WC51-27	4/14/14 13:30	4/15/14 18:50	29:20:00
WC51-28	4/14/14 19:40	4/16/14 0:20	28:40:00
WC51-29	4/15/14 1:30	4/16/14 6:20	28:50:00
WC51-30	4/15/14 7:30	4/16/14 11:50	28:20:00
WC51-31	4/15/14 12:50	4/16/14 17:45	28:55:00
WC51-32	4/15/14 18:20	4/17/14 5:30	35:10:00
WC51-33	4/16/14 0:15	4/17/14 5:33	29:18:00
WC51-34	4/16/14 6:05	4/17/14 11:20	29:15:00
WC51-35	4/16/14 11:35	4/17/14 11:45	24:10:00
WC51-36	4/16/14 17:35	4/17/14 17:45	24:10:00
WC51-37	4/16/14 23:00	4/18/14 5:45	30:45:00
WC51-38	4/17/14 5:15	4/18/14 5:45	24:30:00
WC51-39	4/17/14 11:35	4/18/14 16:25	28:50:00
WC51-40	4/17/14 17:40	4/18/14 17:40	24:00:00
WC51-41	4/17/14 23:45	4/19/14 3:10	27:25:00
WC51-42	4/18/14 5:40	4/19/14 8:50	27:10:00
WC51-43	4/18/14 11:05	4/19/14 15:20	28:15:00
WC51-44	4/18/14 16:15	4/20/14 6:00	37:45:00

WC51-45	4/18/14 21:30	4/20/14 6:00	32:30:00
WC51-46	4/19/14 3:05	4/20/14 6:00	26:55:00
WC51-47	4/19/14 9:00	4/20/14 9:15	24:15:00
WC51-48	4/19/14 15:05	4/20/14 15:15	24:10:00
WC51-49	4/19/14 20:50	4/21/14 2:15	29:25:00
WC51-50	4/20/14 2:45	4/21/14 3:05	24:20:00
WC51-51	4/20/14 8:55	4/21/14 13:45	28:50:00
WC51-52	4/20/14 14:50	4/21/14 19:40	28:50:00
			0:00:00
			0:00:00
			0:00:00
			0:00:00
Total Vent Time			1836:23:00
Average Vent Time			35:18:54

APPENDIX B
EVANS ANALYTICAL XRD REPORTS

APPENDIX B-1
EVANS ANALYTICAL XRD REPORT: INITIAL SAMPLES

C

ISO 17025

Testing Cert. #2797.01

X-RAY DIFFRACTION (XRD)
ANALYSIS REPORT
08 Oct 2014

JOB NUMBER C0EMW009
PO NUMBER Credit Card

for

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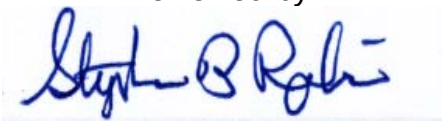

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Requester: Ryan Schierman
Job Number: C0EMW009
Analysis Date: 08 Oct 2014

X-RAY DIFFRACTION ANALYSIS REPORT

Purpose: Use x-ray diffraction to determine the phases present in four Uranium oxide powder samples and quantify these phases using Whole Pattern Fitting (WPF). The samples were identified as indicated in the summary table below.

Summary:

Sample ID	Phases Identified	Concentration wt % (+/- 5%)
Sample 1 (Drum 28) Air-sensitive holder	UO_3 – Uranium Oxide Hexagonal P-3m1 PDF# 00-031-1416	24.5
	$UO_4 \cdot 2H_2O$ – Metastudtite Orthorhombic Immm PDF# 00-035-0571	1.1
	Amorphous materials	74.4[a]
Sample 2 (Drum 37A) After exposure to air/moisture	UO_3 – Uranium Oxide Hexagonal P-3m1 PDF# 00-031-1416	38.4
	$UO_4 \cdot 2H_2O$ – Metastudtite Orthorhombic Immm PDF# 00-035-0571	3.5
	$(UO_2)4O(OH)_6(H_2O)_5$ – Metaschoepite?? Orthorhombic Pbcn PDF# 04-011-3920	1.4
	Amorphous materials	56.7[a]

Sample 3 (Drum 39A) After exposure to air/moisture	UO_3 – Uranium Oxide Hexagonal P-3m1 PDF# 00-031-1416	32.5
	$UO_4 \cdot 2H_2O$ – Metastudtite Orthorhombic Immm PDF# 00-035-0571	0.9
	$(UO_2)4O(OH)_6(H_2O)_5$ – Metaschoepite?? Orthorhombic Pbcn PDF# 04-011-3920	0.2
	Amorphous materials	66.4[a]
Sample 4 (Drum 43) After exposure to air/moisture	UO_3 – Uranium Oxide Hexagonal P-3m1 PDF# 00-031-1416	15.3
	$UO_4 \cdot 2H_2O$ – Metastudtite Orthorhombic Immm PDF# 00-035-0571	13.8
	$(UO_2)4O(OH)_6(H_2O)_5$ – Metaschoepite?? Orthorhombic Pbcn PDF# 04-011-3920	0.4
	Amorphous materials	70.5[a]

[a] – high uncertainty due to unknown density/stoichiometry

Table 1: Phase identification and Quantitative analysis results

Results and Interpretations: The as-received samples were packed into a bulk sample holder and pressed with a glass slide for analysis. To check whether these samples were reacting to moisture in the air, data from sample Drum 28 was acquired in both an air-sensitive holder and in a normal bulk sample holder after exposure to air. The Drum 28 sample was prepared in a plastic glove bag under flowing dry Nitrogen. The powder from this sample was removed from the vial and was deposited in the center of a sample holder making up the bottom part of a special air-sensitive sample holder. The powder was pressed flat with a glass slide and the top part of the holder was sealed in place with an o-ring prior to removal from the glove bag. XRD data was collected by a coupled Theta:2-Theta scan on a Rigaku Ultima-III

diffractometer equipped with Copper x-ray tube with Ni beta filter, parafocusing optics, computer-controlled slits, and a D/Tex Ultra 1D strip detector.

Figure 1 compares the XRD patterns for the Drum 28 sample both in the air-sensitive holder and normal bulk holder. There are differences in the peak shape and the presence of a few low angle peaks between these two different conditions of collecting data. In particular, the weaker peaks are stronger in the sample that has been exposed to air/moisture. The strong, high angle peaks in the air-sensitive holder data are from the air-sensitive holder itself (see below).

Figure 2 overlay the XRD raw data in air sensitive holder from the Drum 28 sample on top of the blank sample holder that was acquired under the same conditions as the unknown sample. This blank sample consists of an empty air-sensitive holder employing a Beryllium window for collection of XRD from highly reactive materials. The y-axis is in square root (counts) to emphasize the weaker peaks. All of the crystalline peaks in the blank sample that are due to the presence of Beryllium and Beryllium oxide are also observed in the drum 28 sample. The broad peaks near 27 and 50 degrees two-Theta are due to the sample and indicate that it is contains some amorphous material.

Figure 3 shows the data overlaid between the Drum 28 sample in air sensitive holder (after removal of the peaks due to the holder) and other three samples measured in normal bulk sample holder. While the patterns of samples Drum 28, Drum 37A and Drum 39A are quite similar, the pattern from sample Drum 43 is considerably different.

Figure 4 shows the best matches for the Drum 28 sample obtained by comparing the background-subtracted experimental data (after removing diffraction peaks due to the Be sample holder) with the ICDD/ICSD diffraction database. This sample is composed of Uranium Oxide (UO_3) and amorphous material. Metastudtite ($UO_4.2H_2O$) is observed as a trace phase in the sample as well. This is the phase whose peaks increased after air/moisture exposure.

The phase identification results for sample Drum 37A, Drum 39A and Drum 43 are shown in Figure 6, Figure 8 and Figure 10, respectively. Uranium Oxide (UO_3) is detected as the minor phase in all samples. Metastudtite ($UO_4.2H_2O$) trace phase is also present with greatest amounts in sample Drum 43. Metaschoepite ($(UO_2)4O(OH)_6(H_2O)_5$) appears to be present in these samples, but this should be considered a speculative match because most of its diffraction peaks are overlapped with the amorphous peak near 27 degrees two-Theta. In addition, all of these three samples contain significant amounts of amorphous material.

Semiquantitative analysis was performed using WPF (whole pattern fitting), which is a subset of Rietveld refinement that accounts for all intensity under above background. This technique requires that either the structure factors and atomic locations or the reference intensity ratio (a way of comparing the diffracting power of different phases) are known for all phases identified. During this process, structure factor (which relates to concentration), lattice parameters (which relate to peak position), peak width and peak shape are refined for each phase to minimize the R value – an estimate of the agreement between the model and the experimental data over the entire pattern.

To obtain quantitative results from the samples that consist of amorphous material, the density of the amorphous has to be assigned in order to determine how much amorphous material is present. It is not clear from the location of the amorphous peaks what this amorphous material is. From the location of the amorphous peaks, it is assumed that it is probably a Uranium Hydrate form and has density of approximately 4.7 g/cm^3. Any error in this density translates to an error in the concentration of the amorphous material. In this case, the relative concentrations of the crystalline phases will be correct, but the absolute concentrations will be off by an amount proportional to the error in the amorphous concentration. In addition, there is no RIR value for the Metastudtite phase and its concentration is calculated based on the RIR of other orthorhombic Uranium hydrate form phases from the ICSD database. Note that the WPF results for all four samples only extend to 50 degrees 2θ because the Metastudtite reference pattern stops at 51º 2θ.

Figure 5, Figure 7, Figure 9 and Figure 11 show the WPF results for four samples. The concentrations are listed in Table 1. The R values for these refinements varied from 6.4% to 8.3%. All of these R values are quite good. The difference curve on the top of each figure show the major sources of error in all cases are due the differences in relative peak intensities between the experimental data and the reference patterns.

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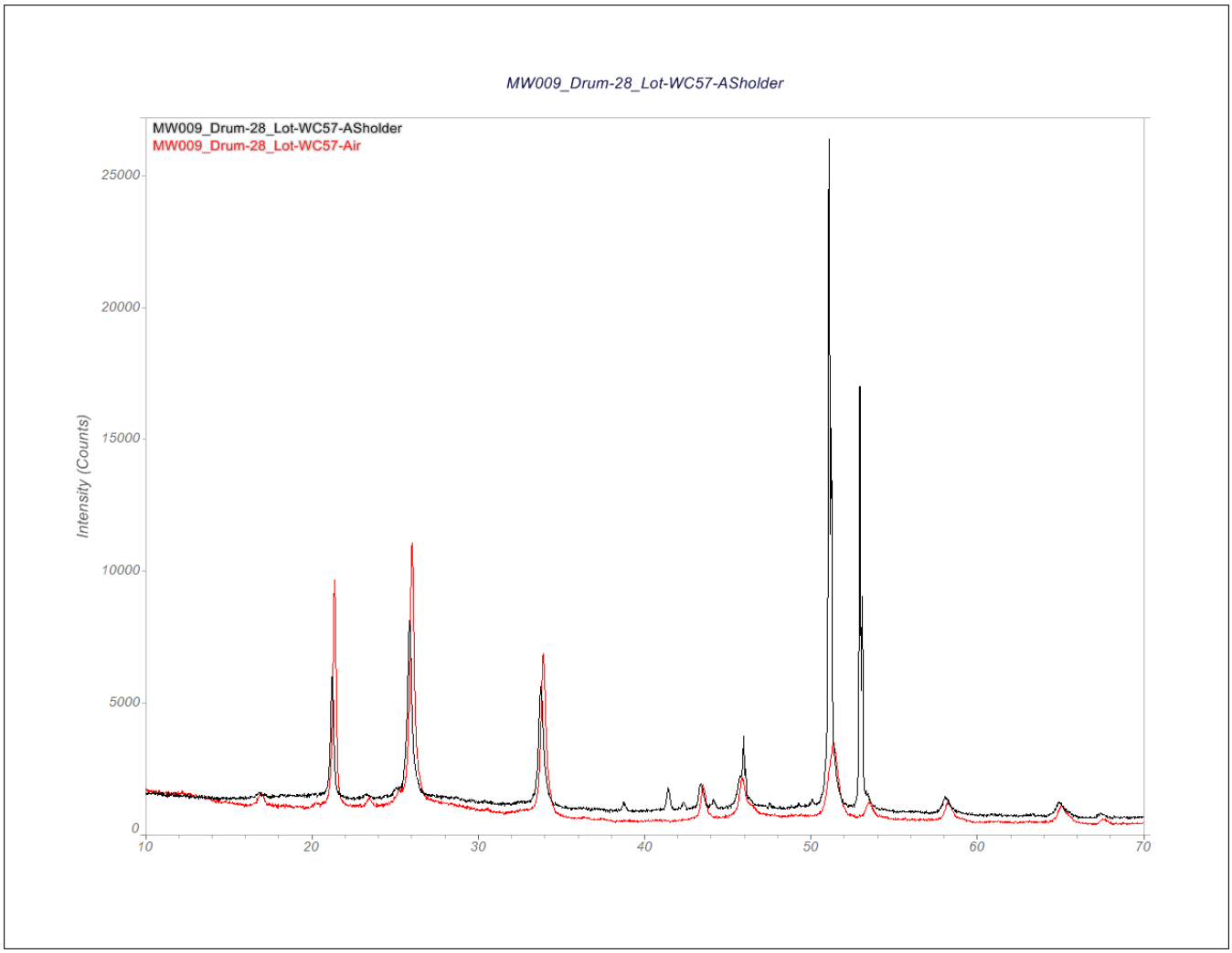

Figure 1: Comparison the raw data between the Drum 28 sample in both air sensitive holder (black) and bulk sample holder (red)

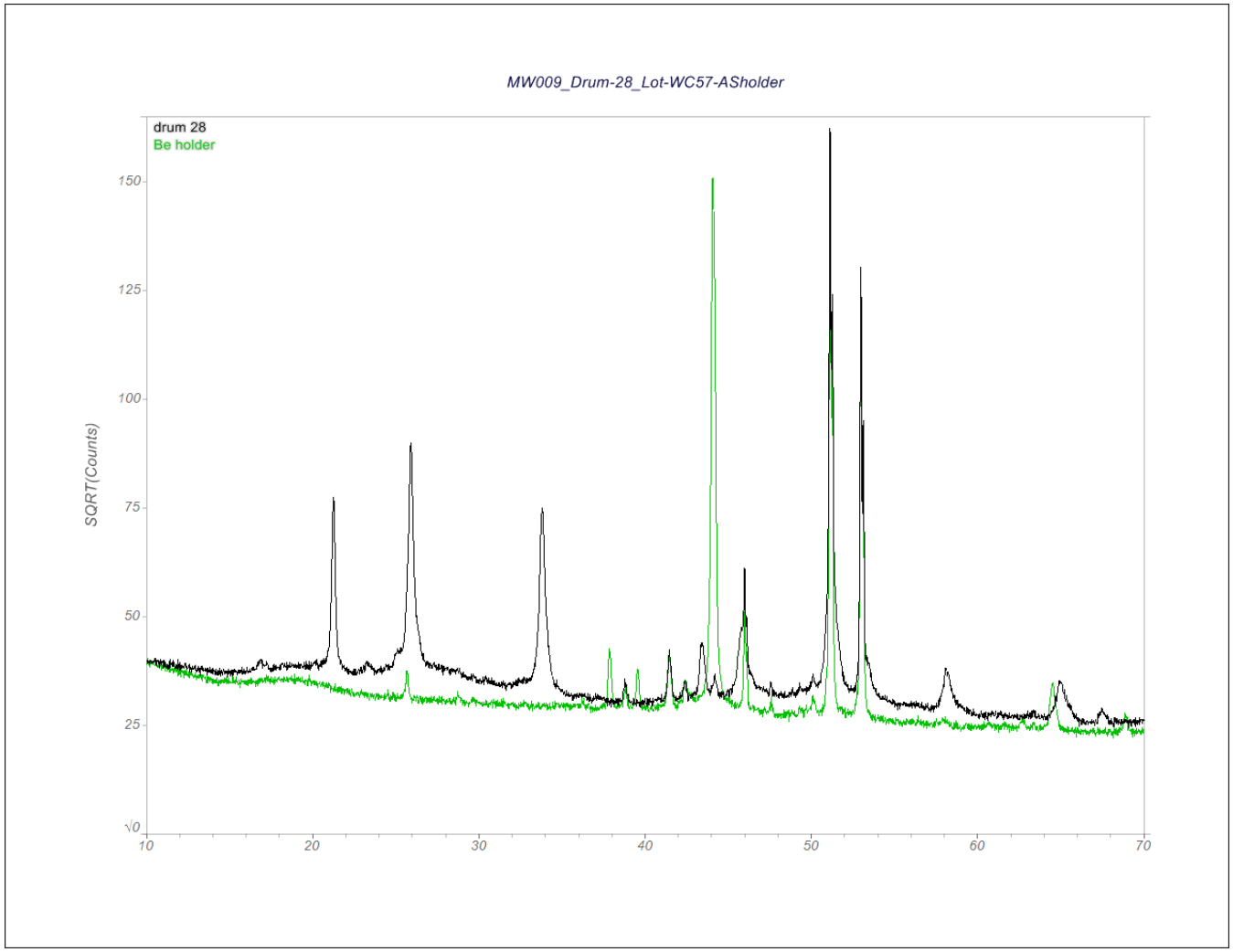

Figure 2: Comparison the raw data between the Drum 28 sample (black) and the blank sample consisting of an empty air-sensitive sample holder (green)

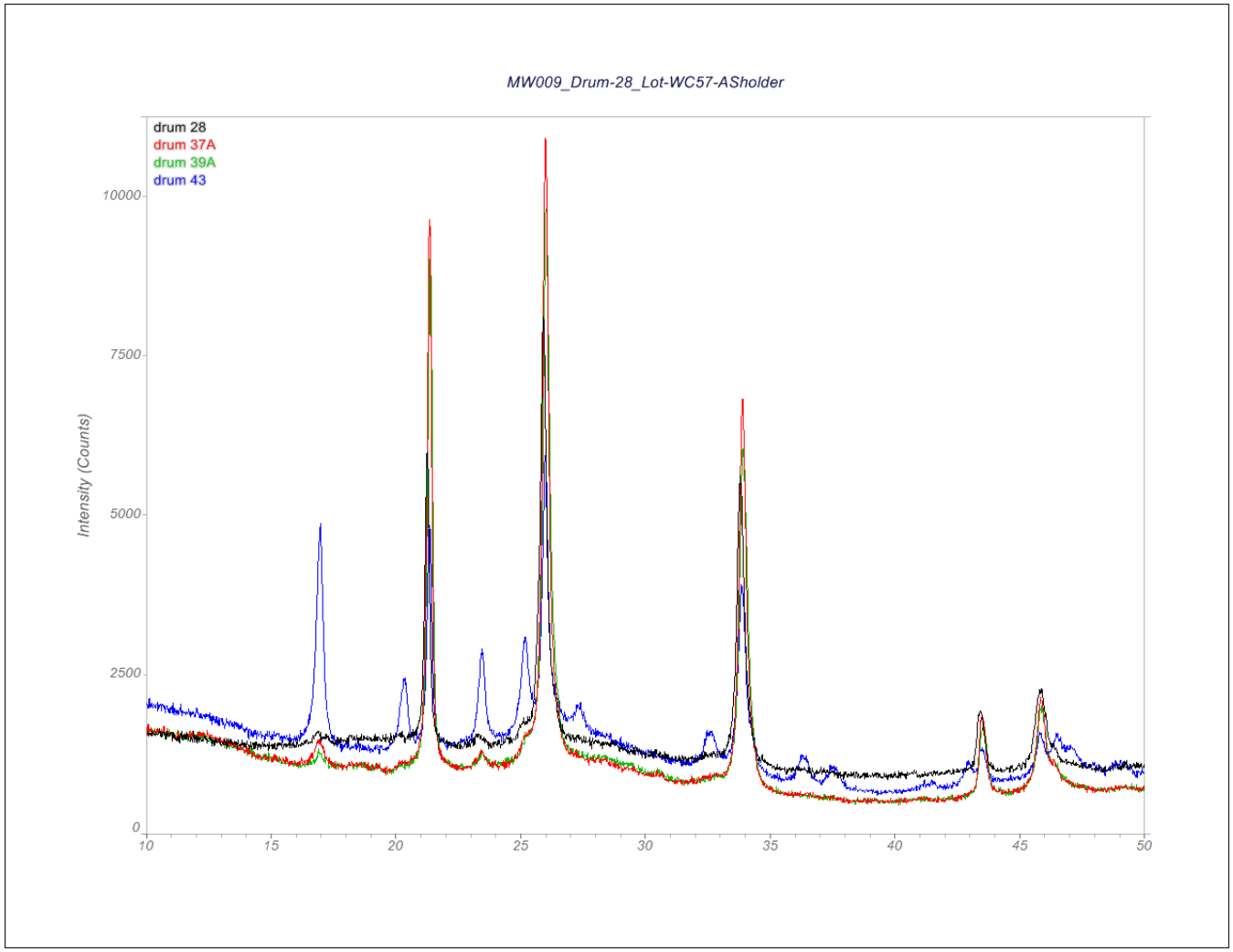

Figure 3: Comparison between sample Drum 28 in air sensitive holder and three other samples in normal bulk sample holder

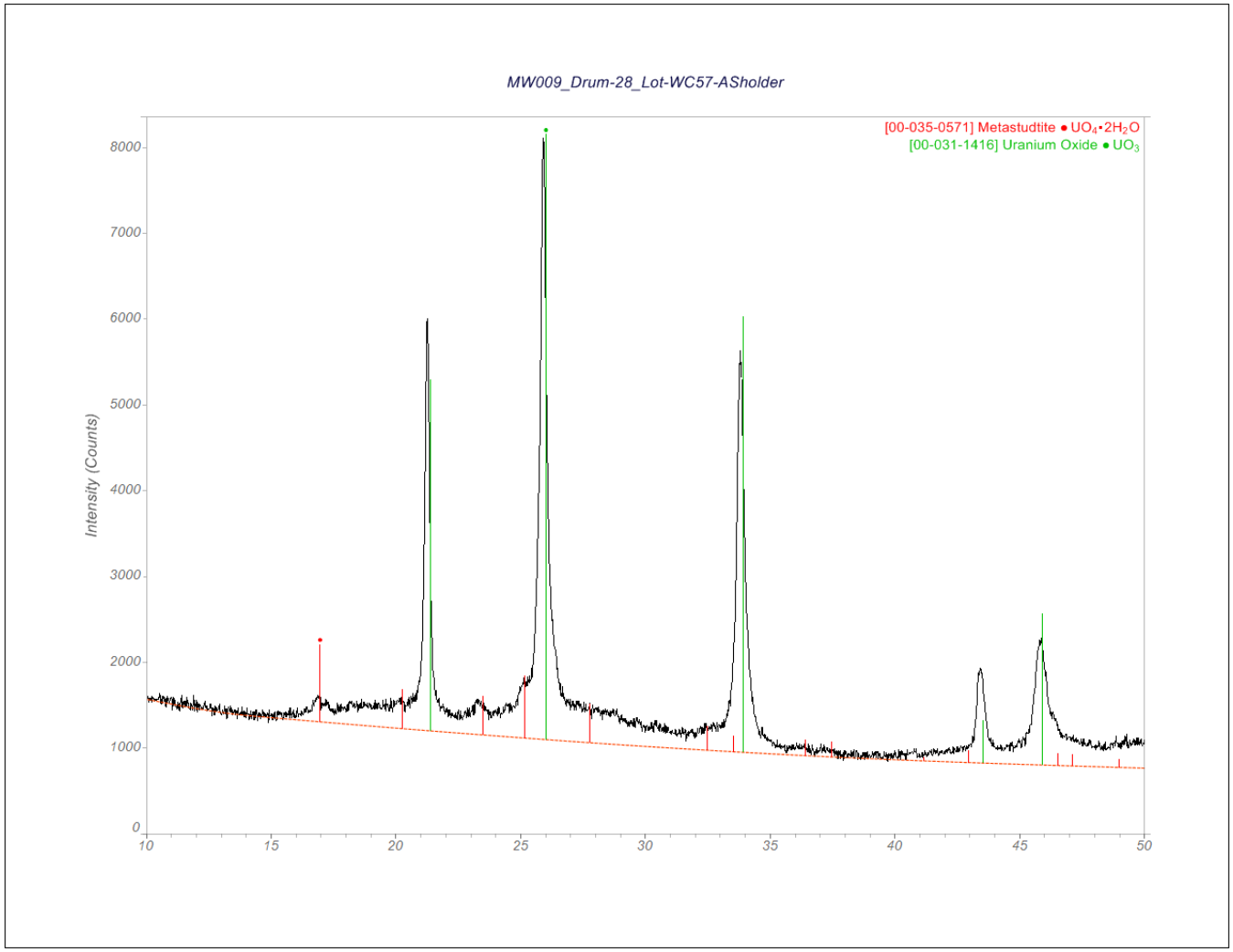

Figure 4: Phase identification for sample 1 (Drum 28)

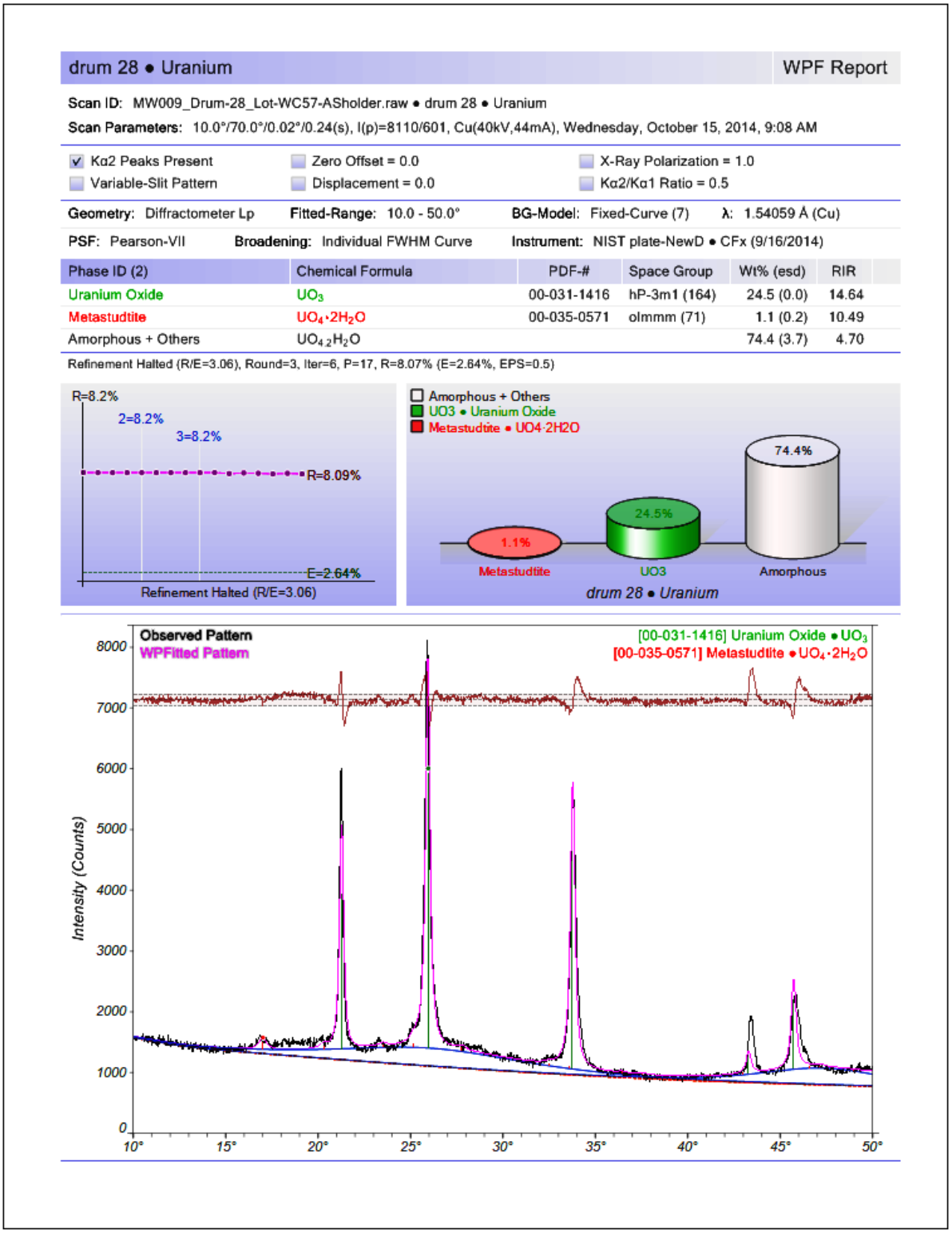

Figure 5: Quantitative analysis for sample 1 (Drum 28)

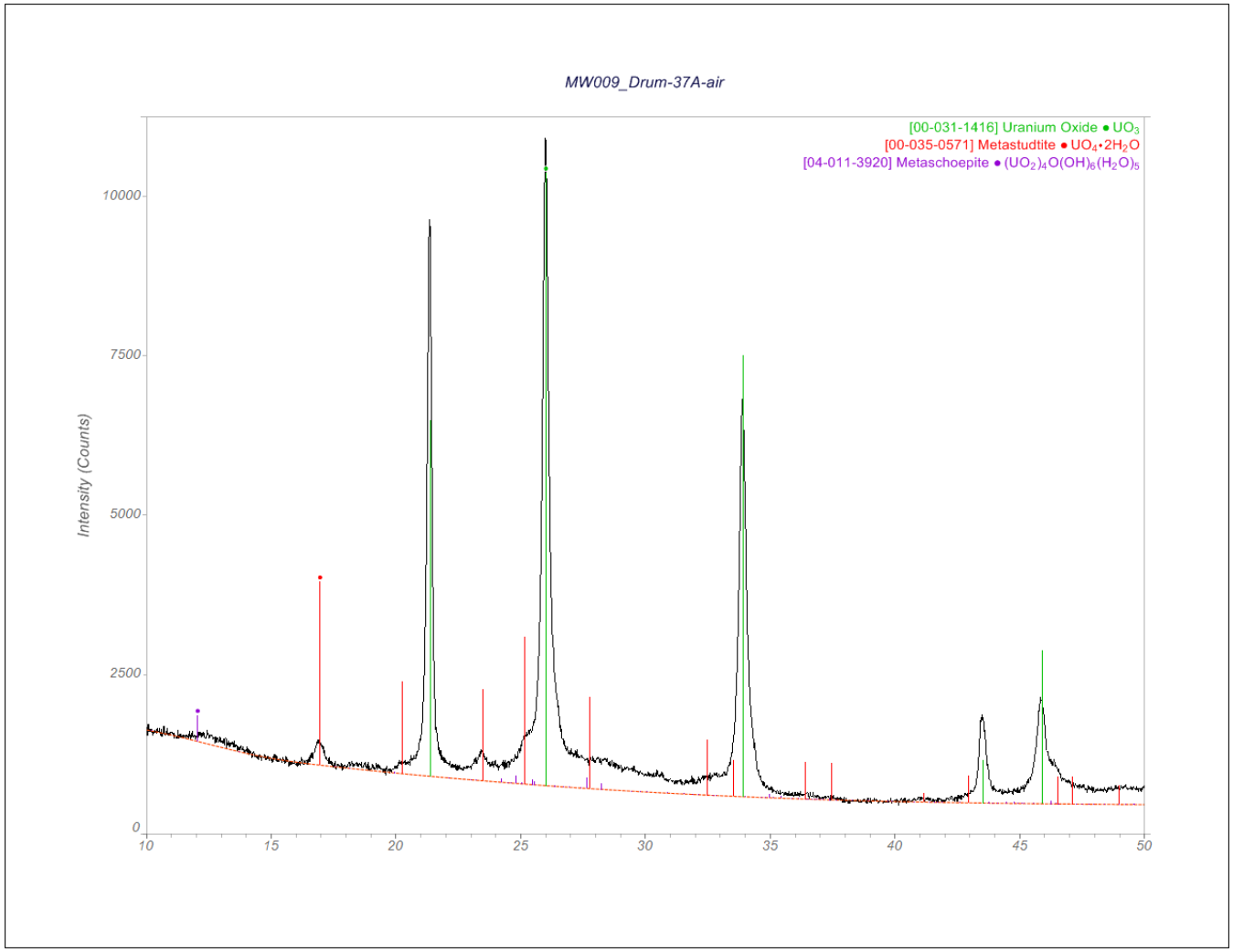

Figure 6: Phase identification for sample 2 (Drum 37A)

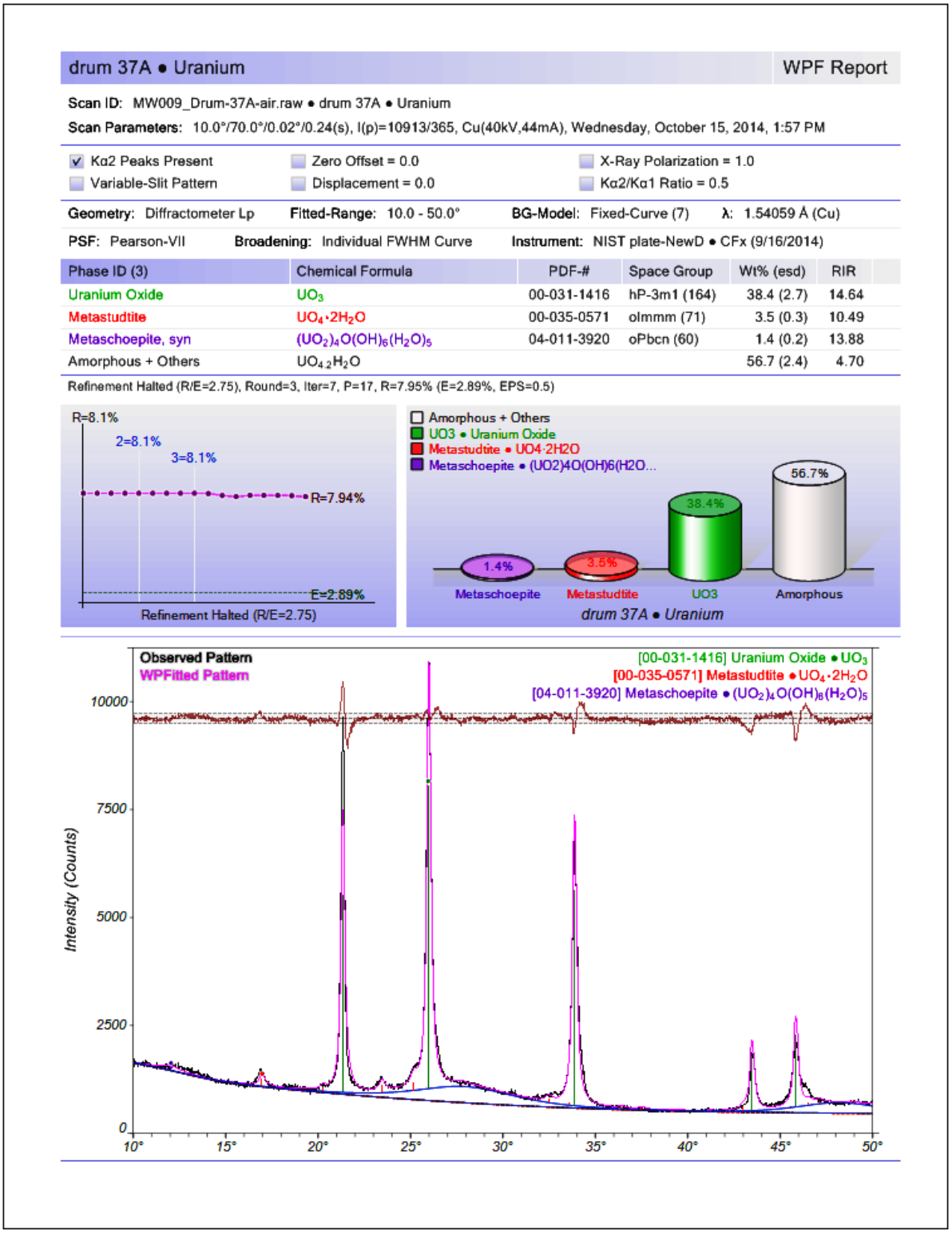

Figure 7: Quantitative analysis for sample 2 (Drum 37A)

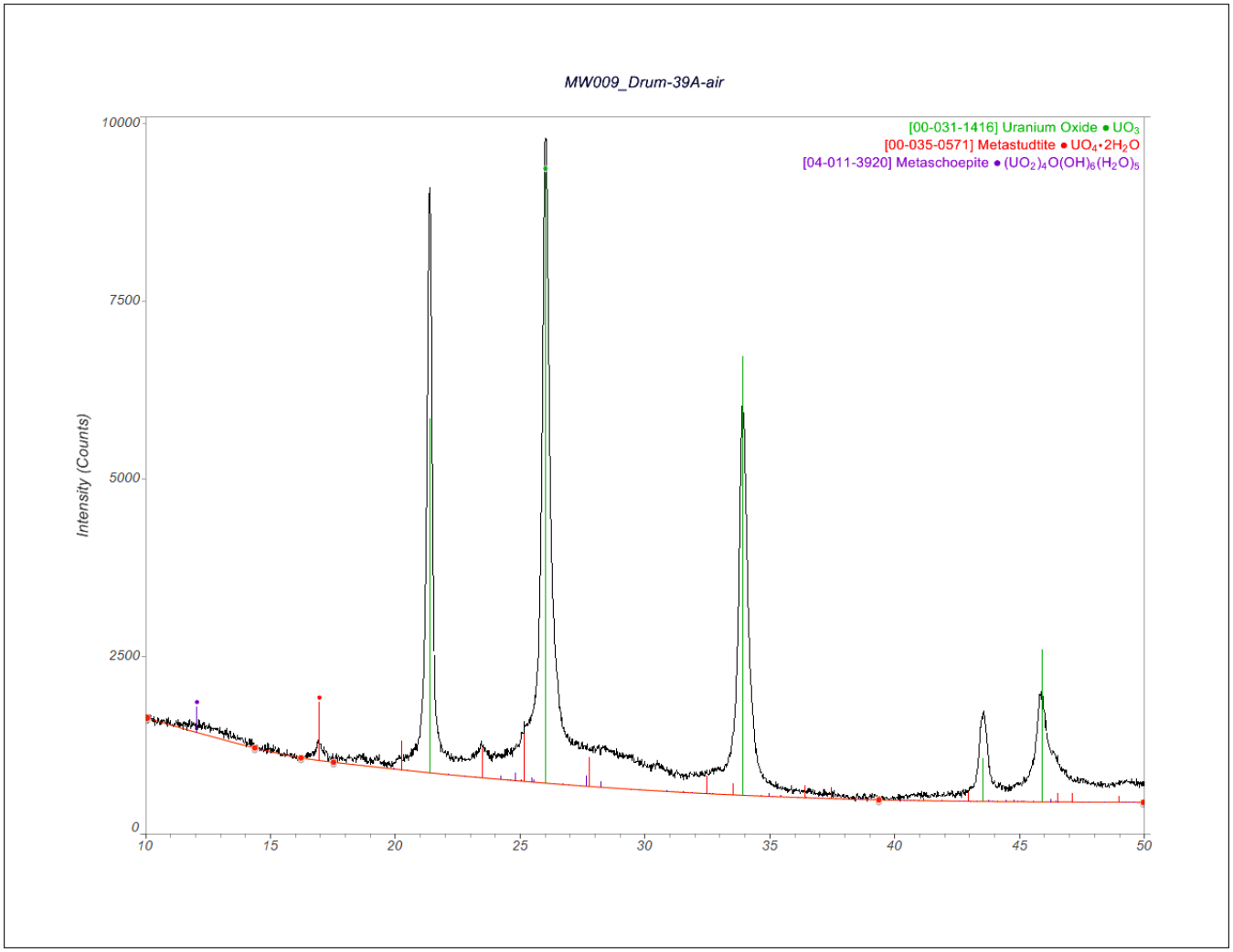

Figure 8: Phase identification for sample 3 (Drum 39A)

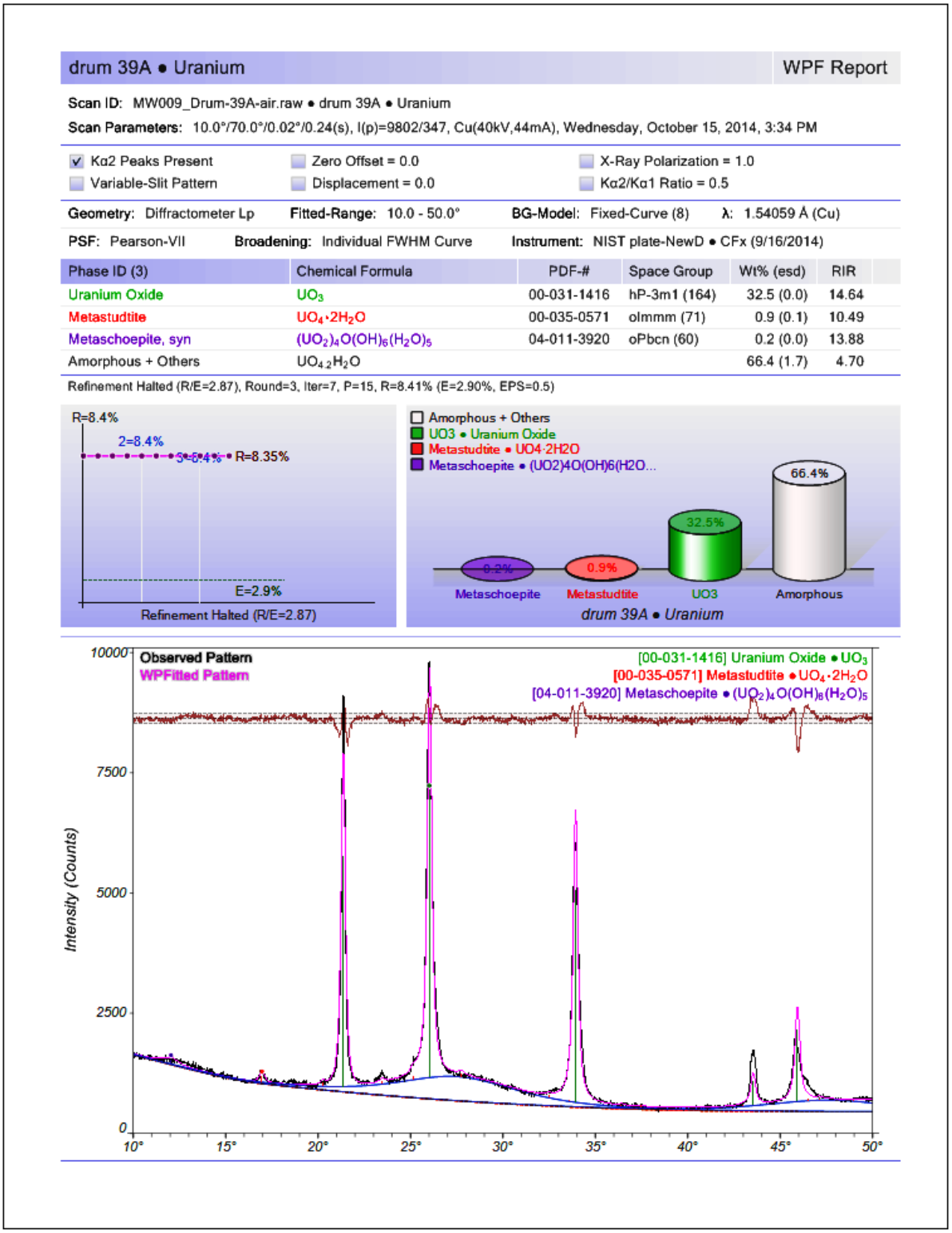

Figure 9: Quantitative analysis for sample 3 (Drum 39A)

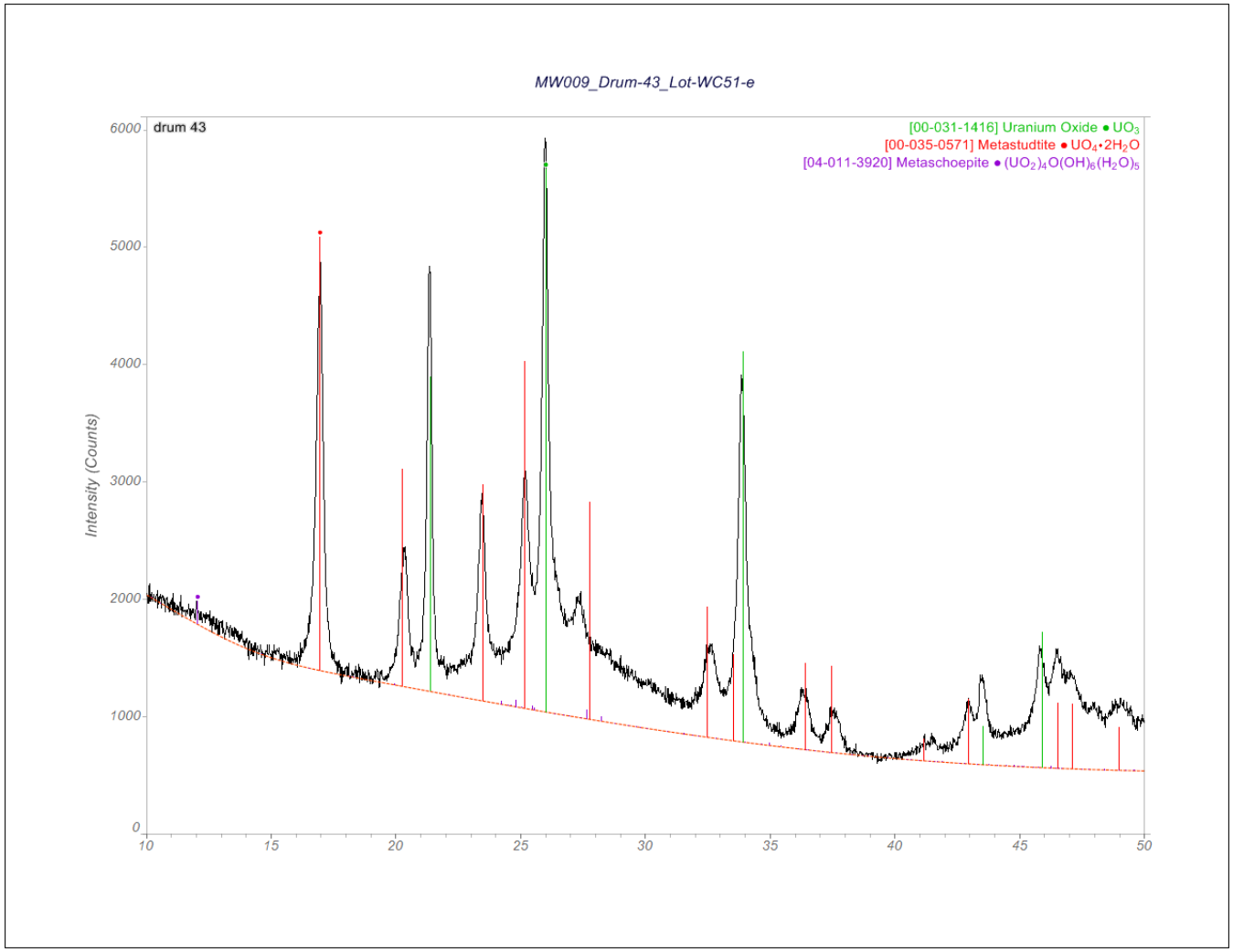

Figure 10: Phase identification for sample 4 (Drum 43)

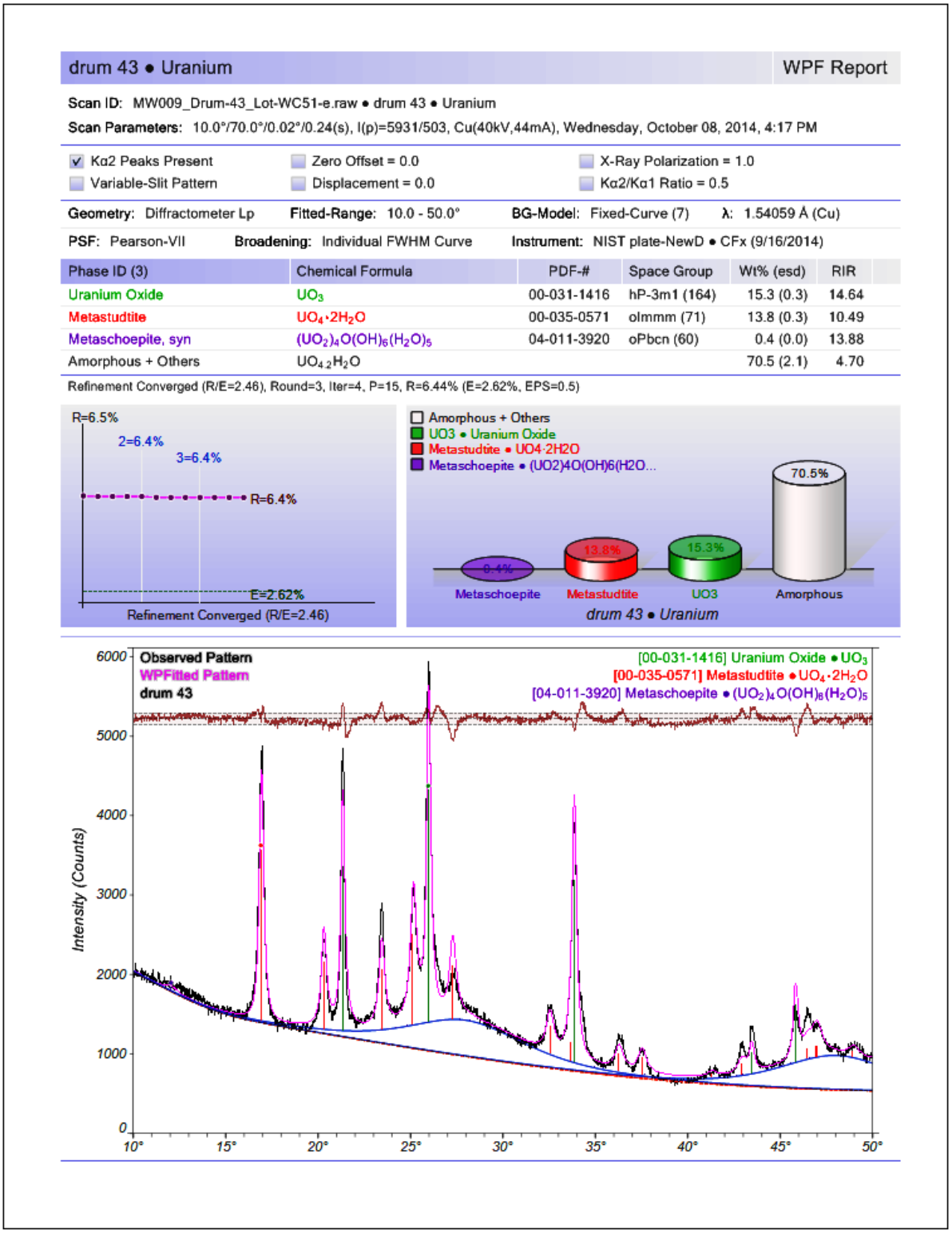

Figure 11: Quantitative analysis for sample 4 (Drum 43)

Appendix

Measurement Uncertainty:

There are two types of uncertainty in XRD analysis; uncertainty in the number of x-ray counts at a particular angle and uncertainty in the diffraction angle. Because the arrival of X-ray quanta in the detector is random with respect to time, the accuracy of X-ray counting rate measurements is governed by the laws of probability. In particular, the size of the one sigma standard deviation in an X-ray measurement is equal to the square root of the number of X-rays counted. A conservative criterion for the detection of a weak peak in a XRD pattern must have amplitude of greater than three standard deviations above background. As a result, the more slowly a measurement is made, the lower the relative standard deviation in the number of counts measured and the more likely is detection of trace diffraction peaks. If X-ray data is acquired at a constant speed, the relative standard deviation for the major diffraction peaks in a pattern will be on the order of a few percent or less while the relative standard deviation for the weaker peaks in a pattern will be on the order of tens of percent or more. This also implies that the uncertainty in the concentrations of the major phases in a sample will be lower than for the trace phases. Please note that there are a number of sample related factors that can influence peak intensity. These include (but are not limited to): average crystallite size, preferred orientation (texture), strain, and absorption.

Uncertainty in the position of X-ray diffraction peaks is due to both instrumental and sample effects. Instrumental position uncertainty is primarily due to diffractometer misalignment. Repeat measurements of NIST standard reference materials has shown that the maximum positional uncertainty is less than +/- 0.05 degrees 2-Theta and is typically much less than that. Positional uncertainty due to sample effects are related to sample displacement (displacement of the sample surface either above or below the diffractometer focusing circle) and sample transparency (the effect gets larger as the sample matrix becomes more transparent to the incident X-rays. Through careful sample preparation, the uncertainty due to these two sample effects should be less than +/- 0.03 degrees 2-Theta. Please note that in addition to these factors, solid solution effects, where one element is partially substituted for another within a given crystal structure, can produce significant shifts in measured peak positions. Unlike sample and instrumental peak position effects, solid solution effects can result in phase misidentification.

APPENDIX B-2
EVANS ANALYTICAL XRD REPORT: JANUARY 27, 2015 SAMPLE

ISO 17025

Testing Cert. #2797.01

X-RAY DIFFRACTION (XRD) ANALYSIS REPORT
27 Jan 2015

JOB NUMBER C0FNS897
PO NUMBER

for

Ryan Schierman
Uranium One
907 No. Poplar Street, Suite 260
Casper, WY 82601

Prepared by:

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Evans Analytical Group
810 Kifer Rd
Sunnyvale, CA 94086 USA

Requester: Ryan Schierman
Job Number: C0FNS897
Analysis Date: 27 Jan 2015

X-RAY DIFFRACTION ANALYSIS REPORT

Purpose: Use x-ray diffraction to determine the phases present in a Uranium oxide powder sample and quantify these phases using Whole Pattern Fitting (WPF).

Summary:

Best Matches from the ICDD/ICSD data bases

Sample ID	Phases Identified	Concentration wt % (+/- 5%)
WC62 Drum 29	UO_3 – Uranium Oxide Hexagonal P-3m1 PDF# 00-031-1416	77.3
	U_3O_8 – Uranium Oxide Orthorhombic C2mm PDF# 00-031-1424	22.7
	Unknown phase(s)	?

Table 1: Phase identification and Quantitative analysis results

Results and Interpretations: The as-received powder sample was pressed into a bulk special sample holder with a glass slide for analysis. XRD data was collected by a coupled Theta:2-Theta scan on a Rigaku Ultima-III diffractometer equipped with copper x-ray tube, parafocusing optics, computer-controlled slits, and a D/Tex Ultra 1D strip detector.

Figure 1 compares the best matches between the background-subtracted experimental data to the ICDD/ICSD diffraction database for sample WC62 Drum 29. The sample is composed of a mixture of the hexagonal UO_3 and orthorhombic U_3O_8 phases. In addition, there is a weak peak near 28º 2-Theta that could not be identified in the sample.

Semiquantitative analysis was performed using WPF (whole pattern fitting), which is a subset of Rietveld refinement that accounts for all intensity above the background. This technique requires that either the structure factors and atomic locations or the reference intensity ratio (a way of comparing the diffracting power of different phases) are known for all phases identified. During this process, structure factor (which relates to concentration), lattice parameters (which relate to peak position), peak width and peak shape are refined for each phase to minimize the

R value – an estimate of the agreement between the model and the experimental data over the entire pattern.

Figure 2 shows the WPF results for sample WC62 Drum 29 and the concentrations are listed in Table 1. The R value for the refinement, at 9.6%, is quite good. The difference curve on the top of the figure shows the major sources of error is due to the differences in relative peak intensities between the experimental data and the reference patterns.

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http://www.eaglabs.com/main-survey.html?job=C0FNS897

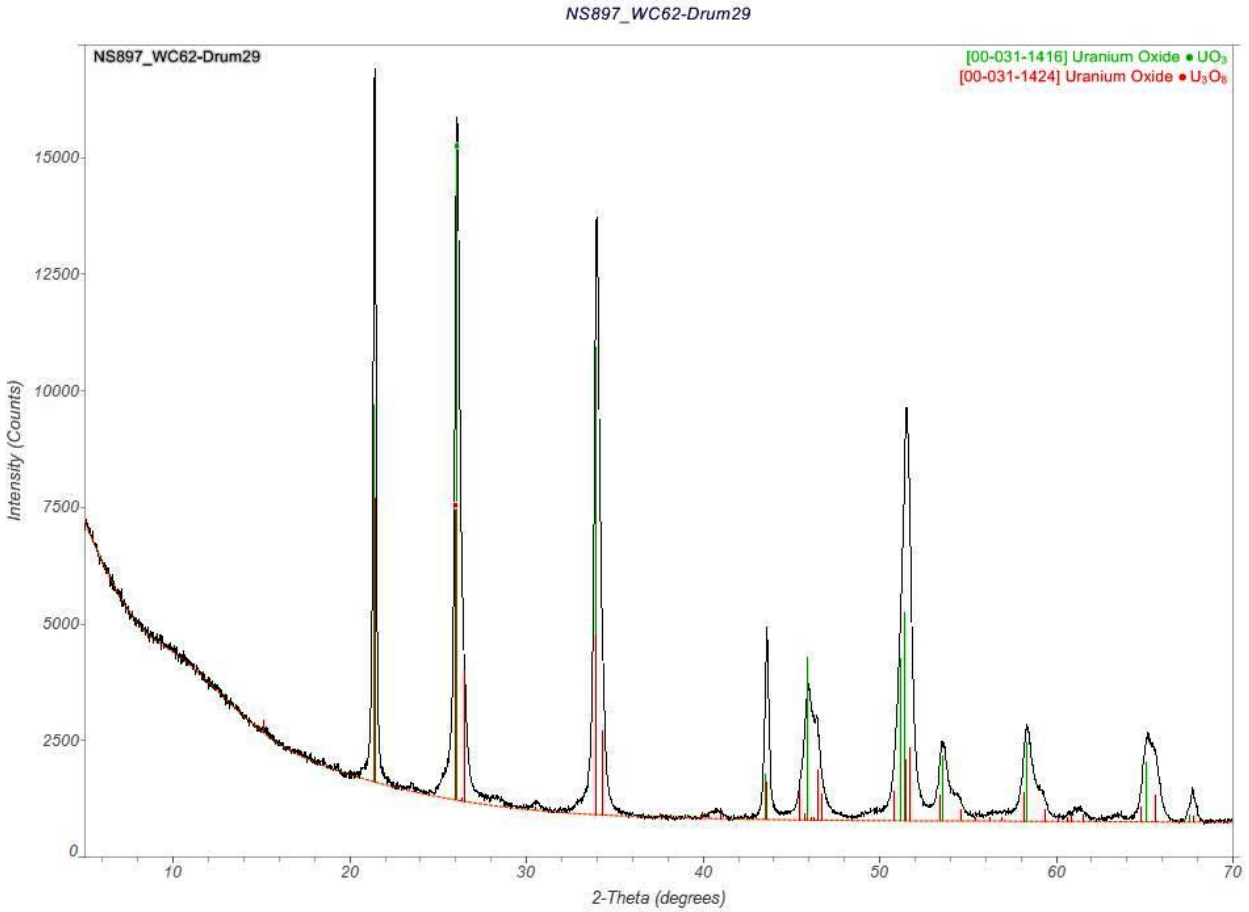

Figure 1: Phase identification for sample WC62 Drum 29

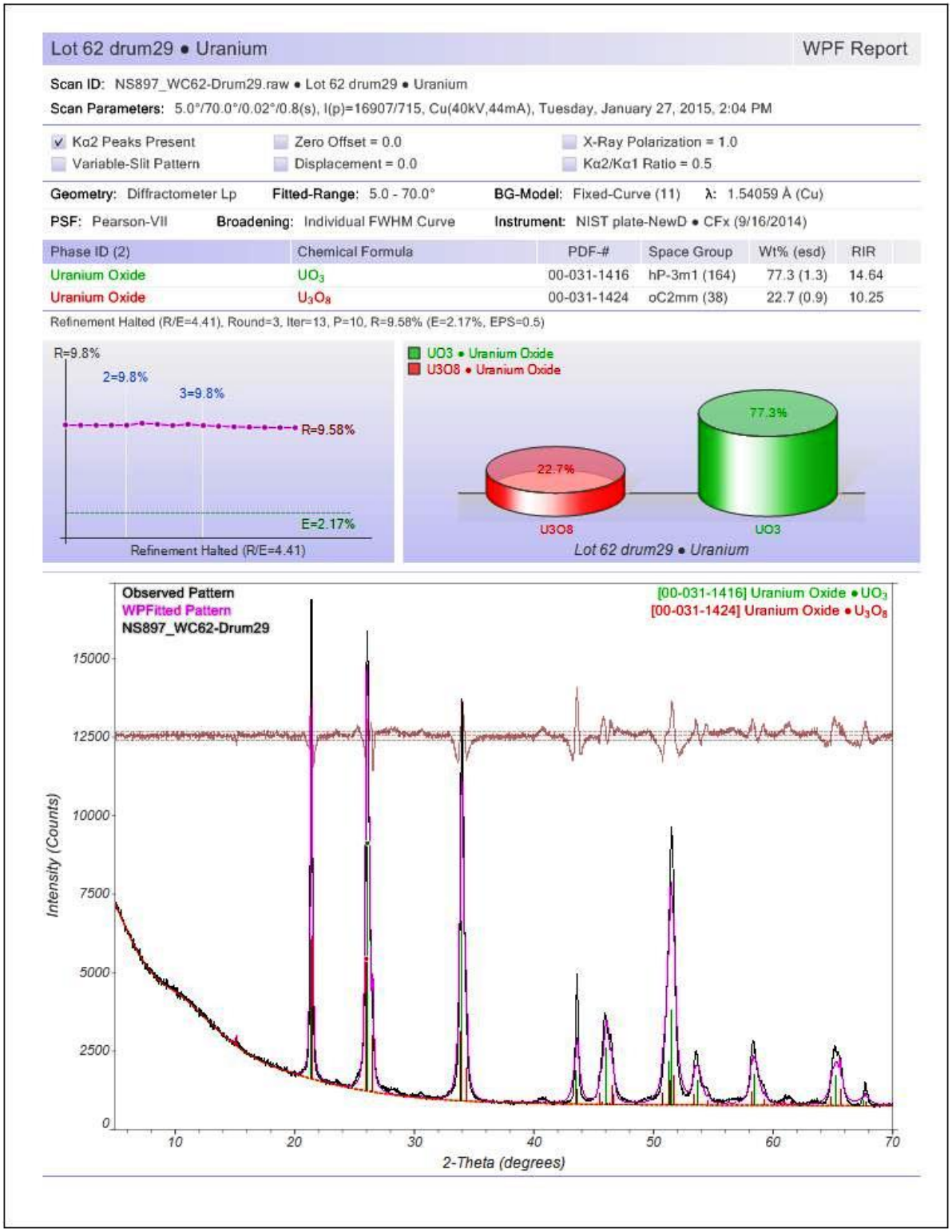

Figure 2: Quantitative analysis for sample WC62 Drum 29

Appendix

Measurement Uncertainty:

There are two types of uncertainty in XRD analysis; uncertainty in the number of x-ray counts at a particular angle and uncertainty in the diffraction angle. Because the arrival of X-ray quanta in the detector is random with respect to time, the accuracy of X-ray counting rate measurements is governed by the laws of probability. In particular, the size of the one sigma standard deviation in an X-ray measurement is equal to the square root of the number of X-rays counted. A conservative criterion for the detection of a weak peak in a XRD pattern must have amplitude of greater than three standard deviations above background. As a result, the more slowly a measurement is made, the lower the relative standard deviation in the number of counts measured and the more likely is detection of trace diffraction peaks. If X-ray data is acquired at a constant speed, the relative standard deviation for the major diffraction peaks in a pattern will be on the order of a few percent or less while the relative standard deviation for the weaker peaks in a pattern will be on the order of tens of percent or more. This also implies that the uncertainty in the concentrations of the major phases in a sample will be lower than for the trace phases. Please note that there are a number of sample related factors that can influence peak intensity. These include (but are not limited to): average crystallite size, preferred orientation (texture), strain, and absorption.

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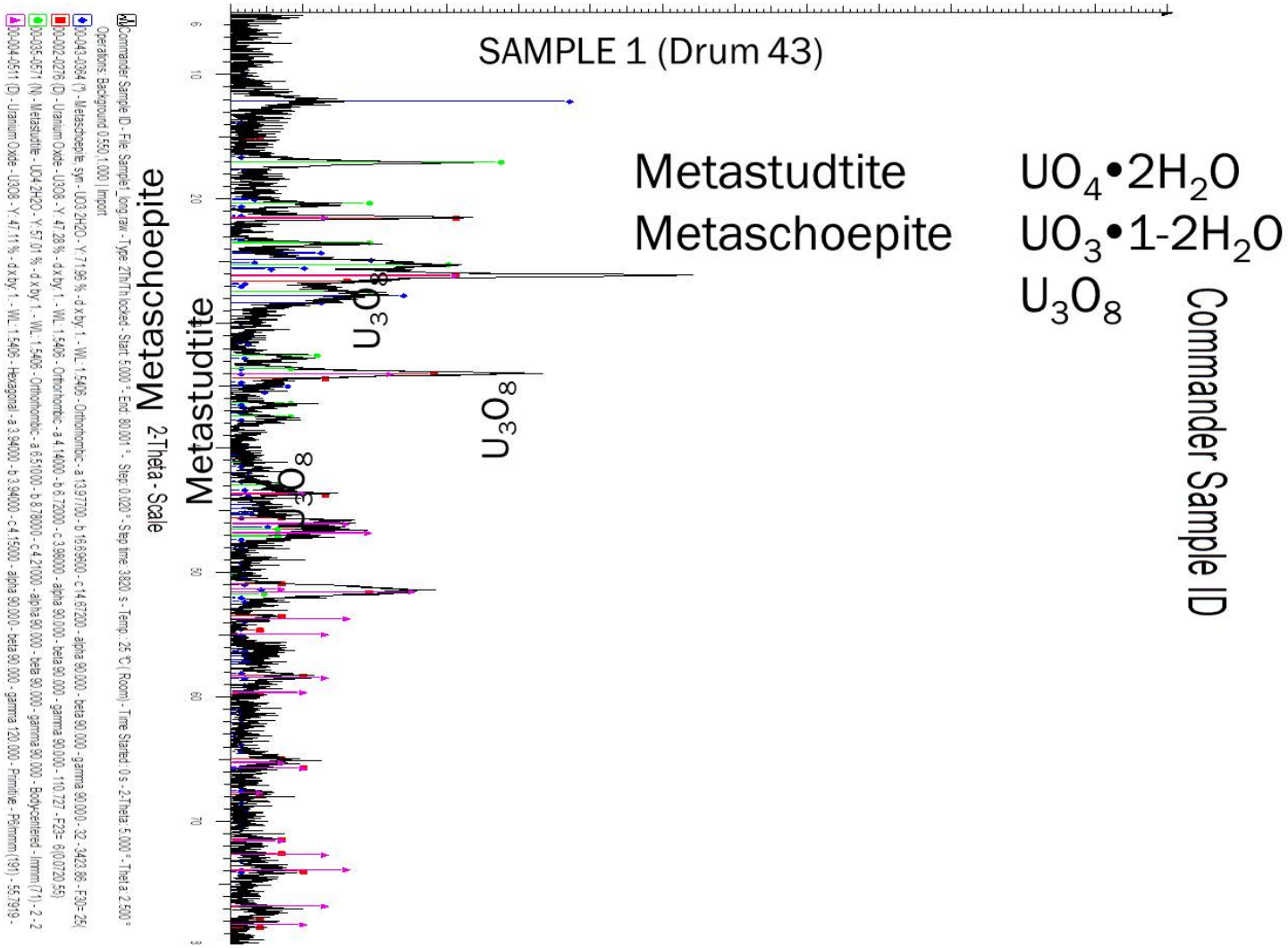
APPENDIX C
NOTRE DAME XRD STUDY RESULTS

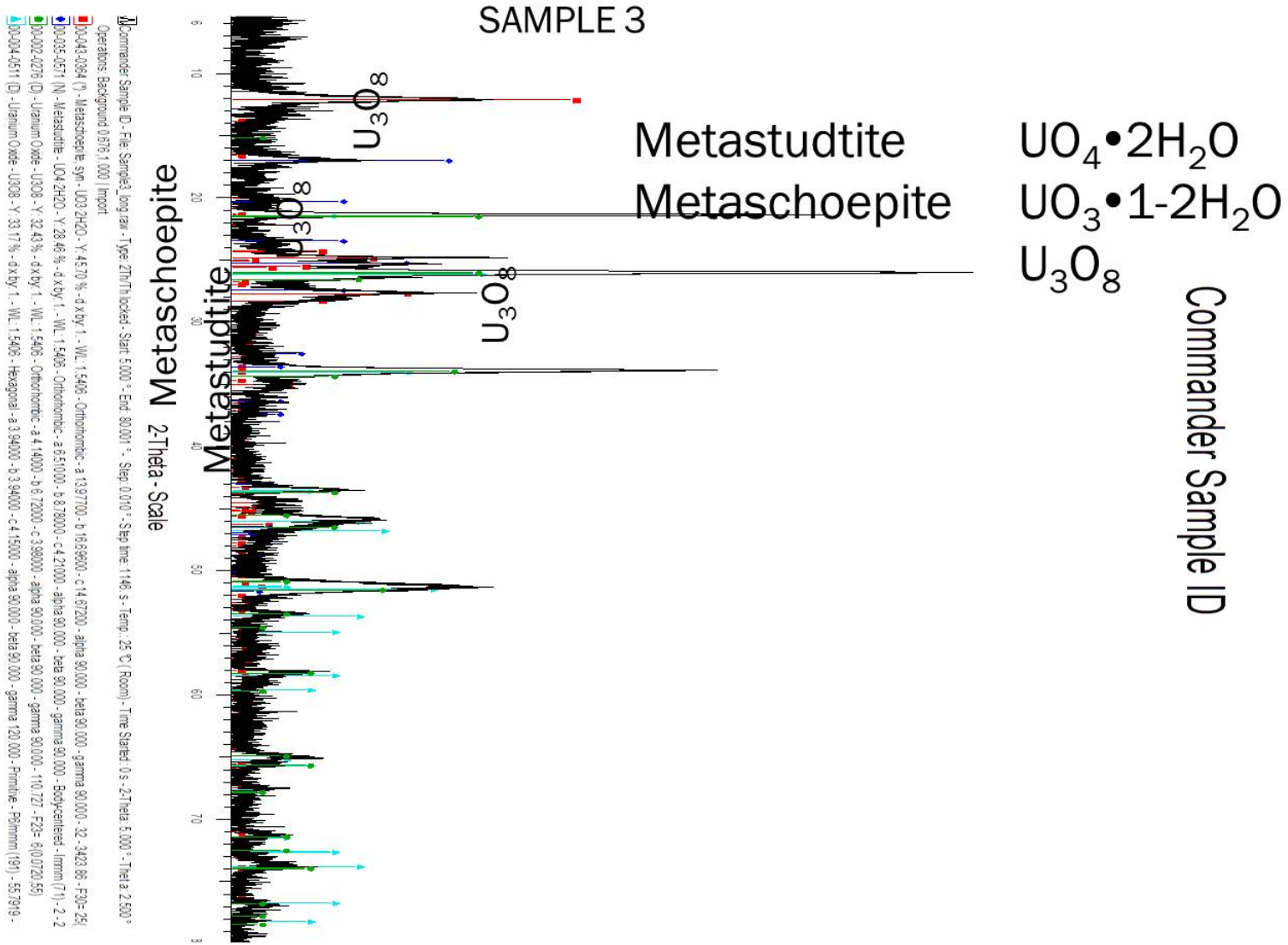
APPENDIX C1
NOTRE DAME DRIED YELLOWCAKE MINERALOGY STUDY
X-RAY DIFFRACTION SPECTRA

This appendix presents the XRD Spectra of the initial Irigaray dried yellowcake samples that were sent to Dr. Peter Burns at Notre Dame University for XRD analysis. The samples included several composites from previously dried lots, one from a subsequent lot (Lot WC57) and a sample from the pressurized drum WC51 Drum 43.

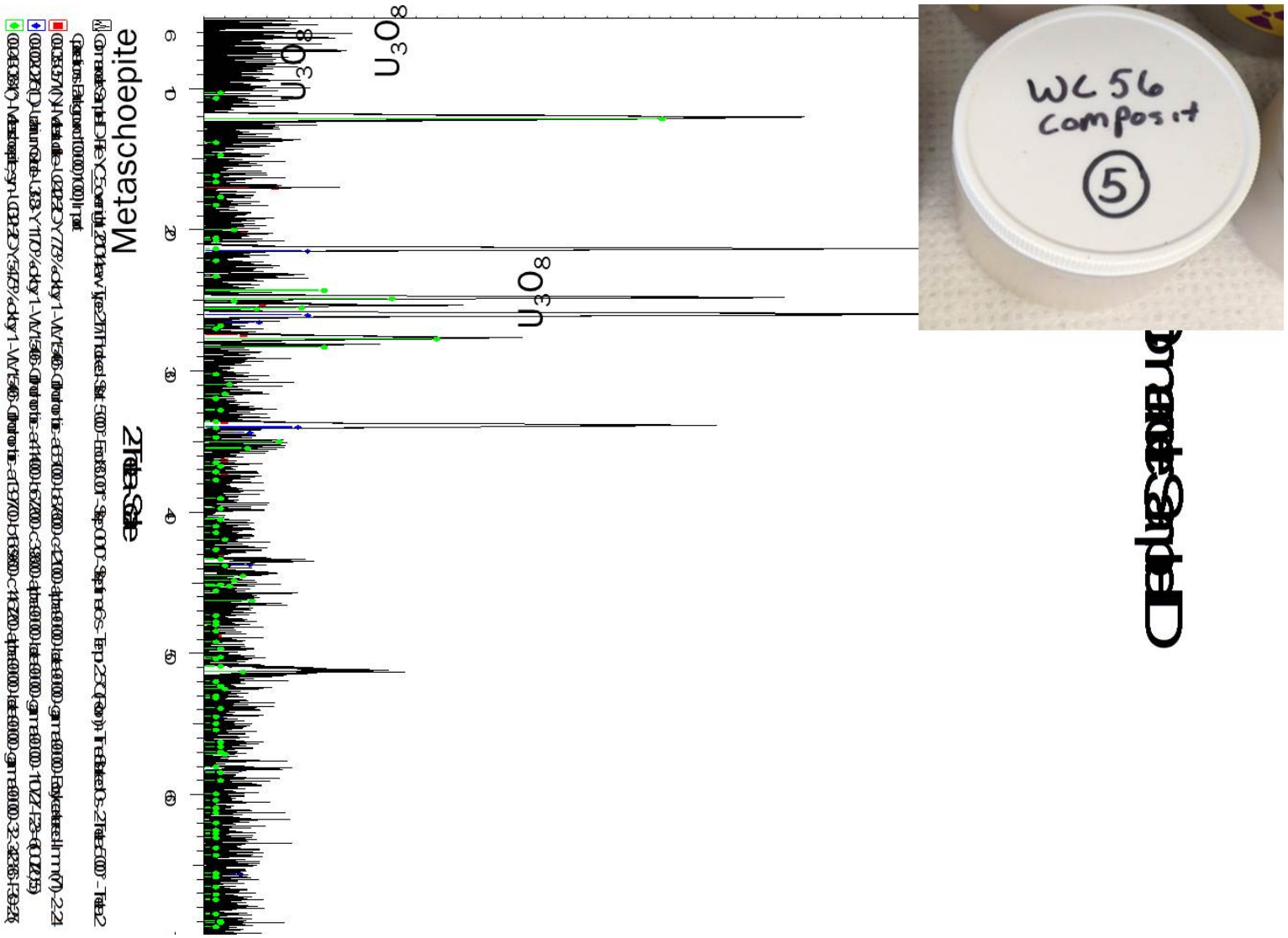
Sample 1 represents the XRD spectra from a sample taken from WC51 Drum 43 which is the drum that was found to be pressurized. This shows the highest percentage of metastudtite and amorphous uranyl oxide found in all of the samples tested. The metaschoepite (UO_3. 1-2 H_2O) is also a significant contributor to the composition of the matrix.



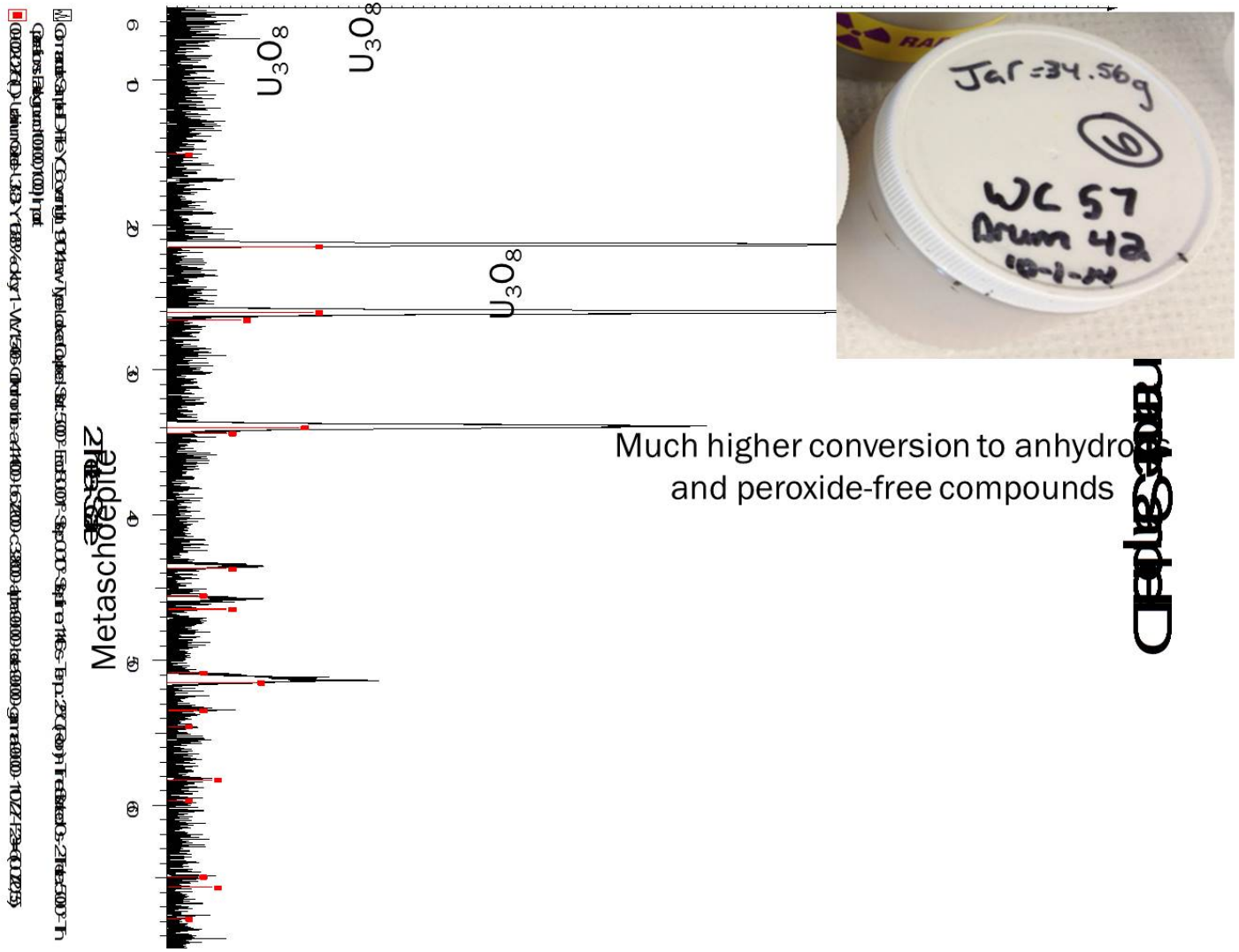
Sample 3 is from the composite of Lot 53 which was processed after Lot 51 Drum 43. It shows lower levels of metastudtite and amorphous uranyl oxide and higher levels of metaschoepite, indicating that this sample received a higher degree of drying.



Sample 5 is from the composite of Lot 56 which was processed after Lot 51 Drum 43. It shows much lower levels of metastudtite and amorphous uranyl oxide and higher levels of metaschoepite and uranium oxides, indicating that this sample received a higher degree of drying.



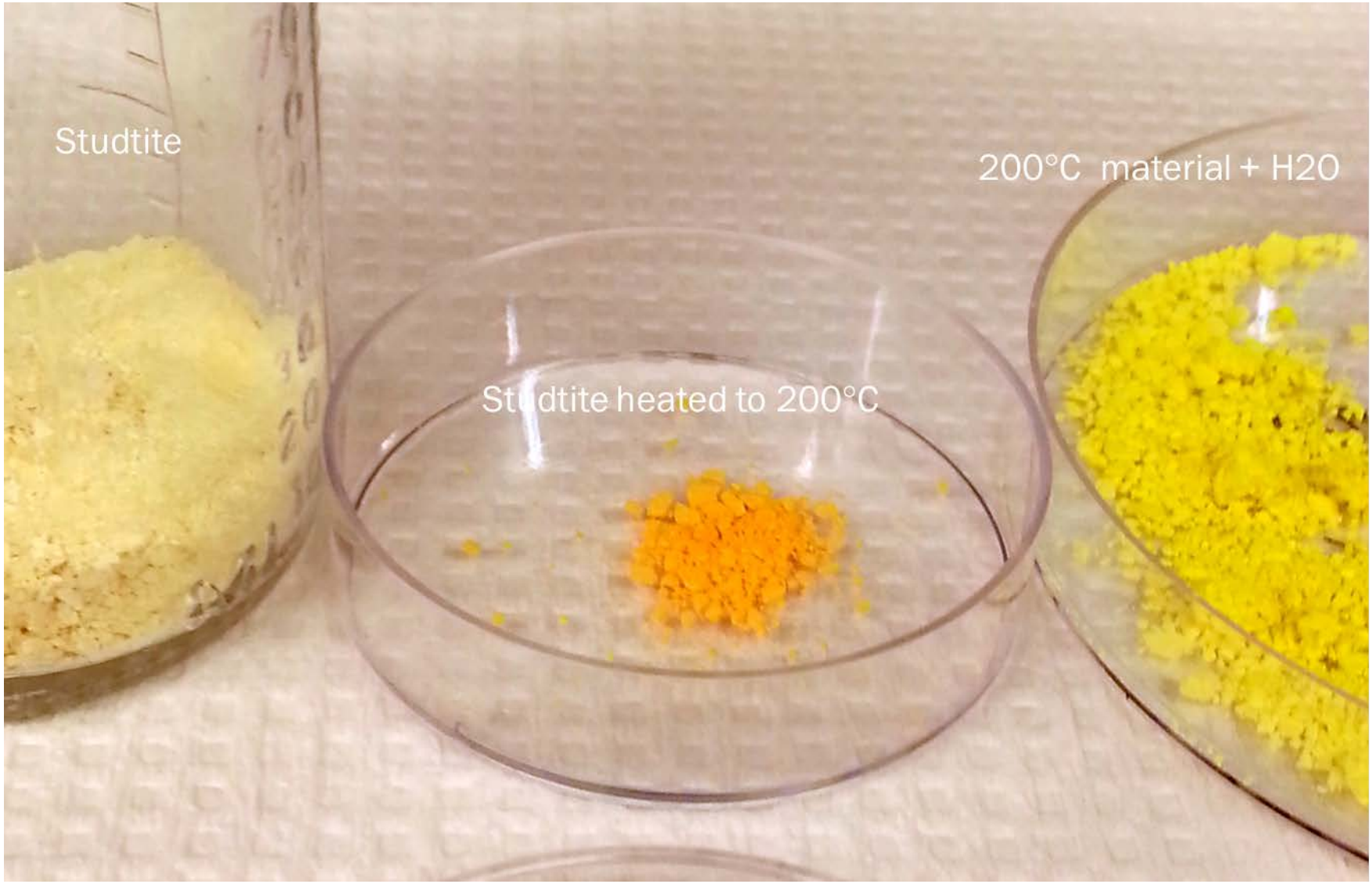
Sample 6 is from the composite of Lot 57 which was processed after Lot 51 Drum 43. It shows negligible levels of metastudtite and amorphous uranyl oxide and higher levels of metaschoepite and uranium oxides and no peroxide containing compounds. This material was recently processed after all of the dryer upgrades had been completed and essentially indicates that this sample received a higher degree of drying.



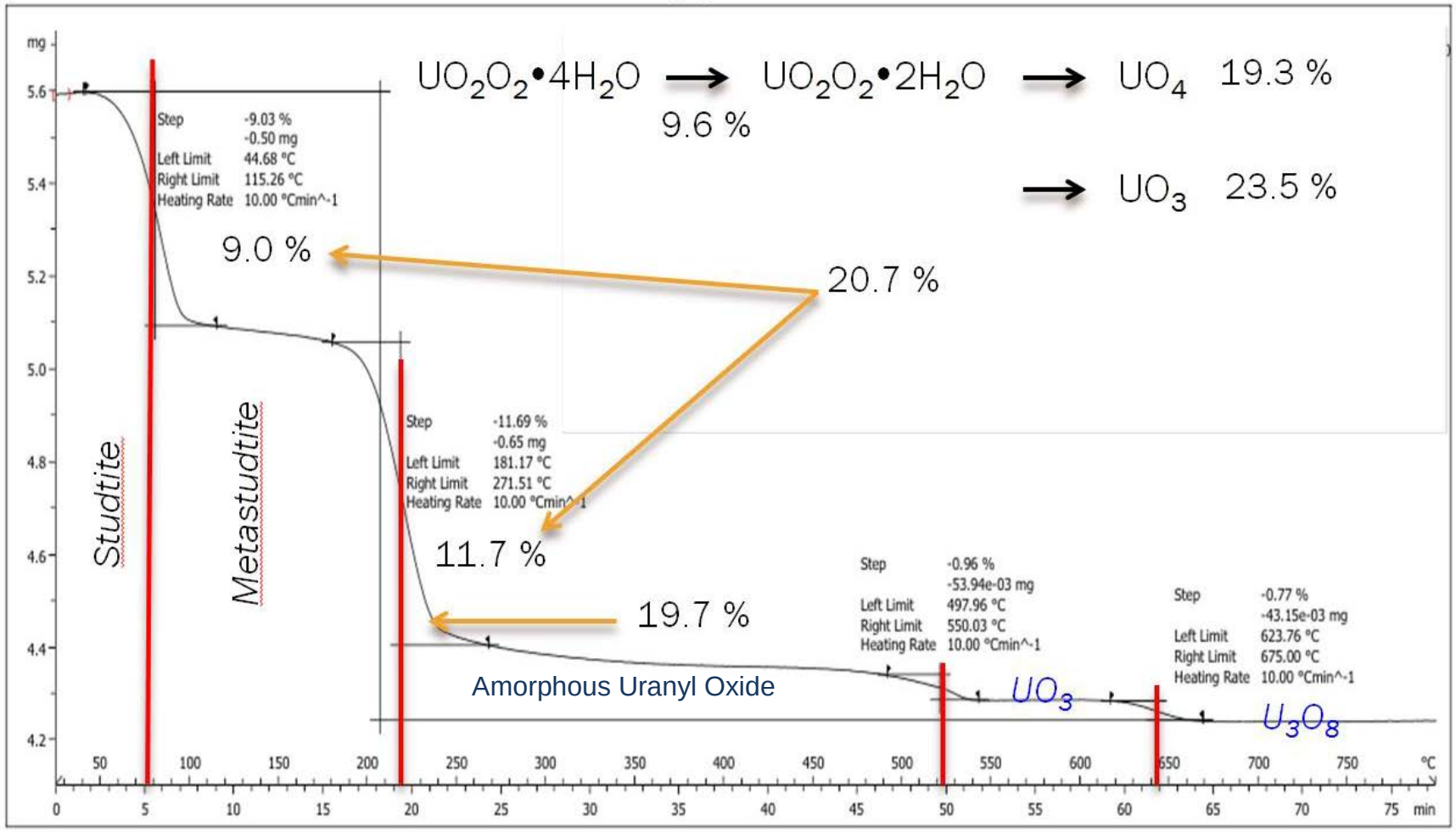
APPENDIX C2
NOTRE DAME DRIED YELLOWCAKE MINERALOGY STUDY
THERMOGRAVIMETRIC EVALUATIONS OF URANYL PEROXIDE

This appendix presents the results of thermogravimetric evaluations of uranyl peroxide that were performed by Dr. Peter Burns at Notre Dame University. This study shows the temperature ranges to be expected for the various species that are formed upon heating of uranyl peroxide.

Samples of studtite, metastudtite heated to 200°C (initial conversion to amorphous uranyl oxide) and then reacted with water at room temperature to form another species, namely metaschoepite (UO_3.1-2H_2O).

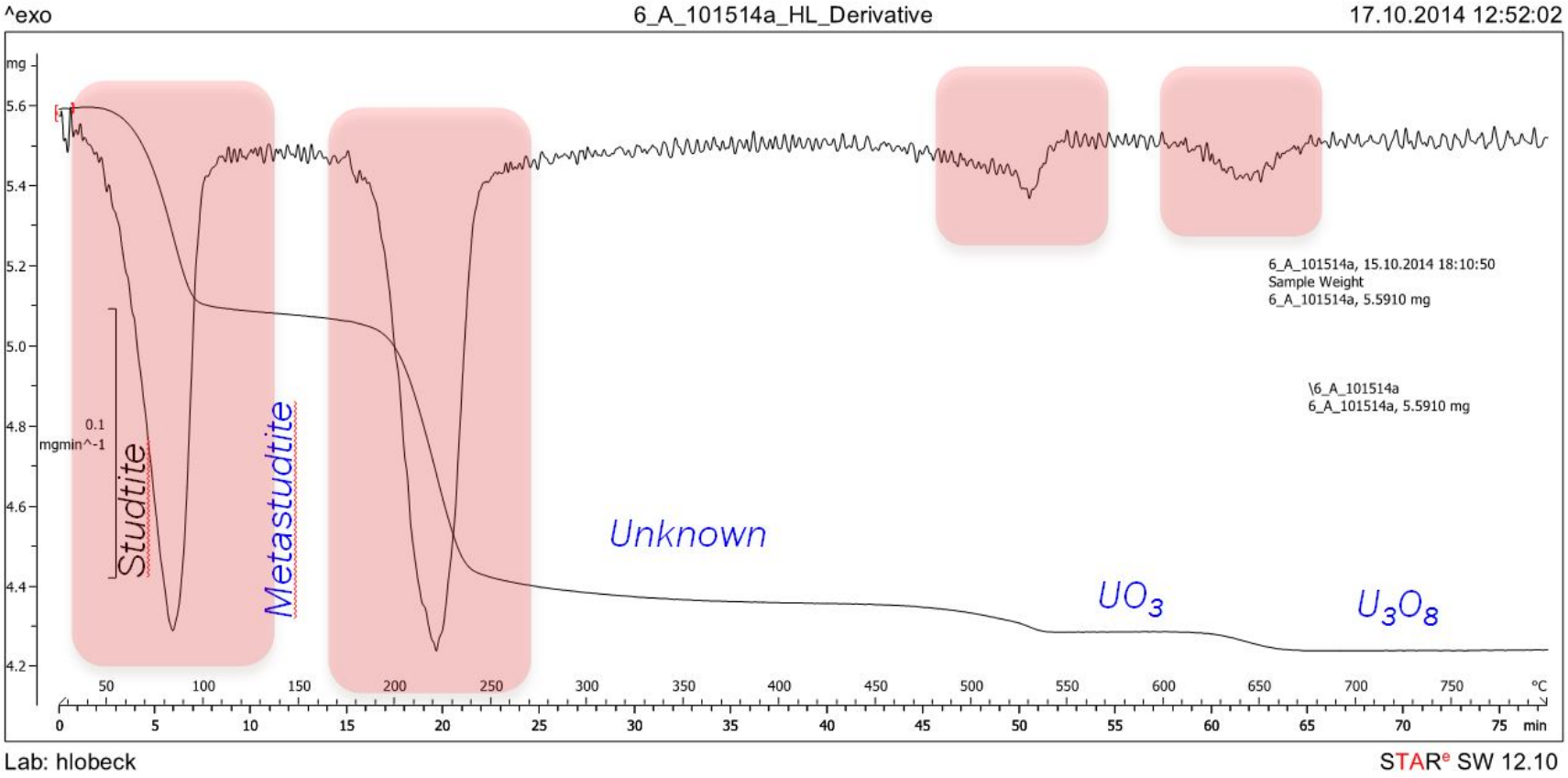


Thermogravimetric curve of the decomposition of uranyl peroxide. Of interest is that the heating rate that was used to develop this curve showed the conversion of studtite to metastudtite to begin at a lower temperature than has historically been reported in the literature. The dehydration of the metastudtite occurs over a very short temperature range with the formation of the amorphous uranyl oxide over a very broad temperature range prior to the conversion to UO_3.

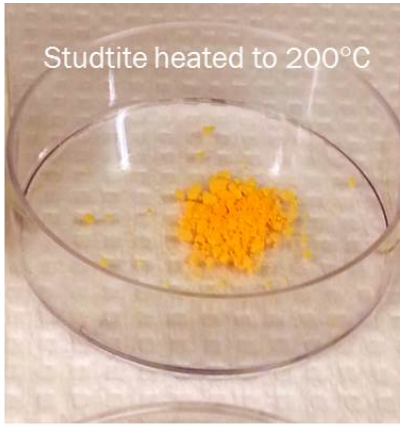


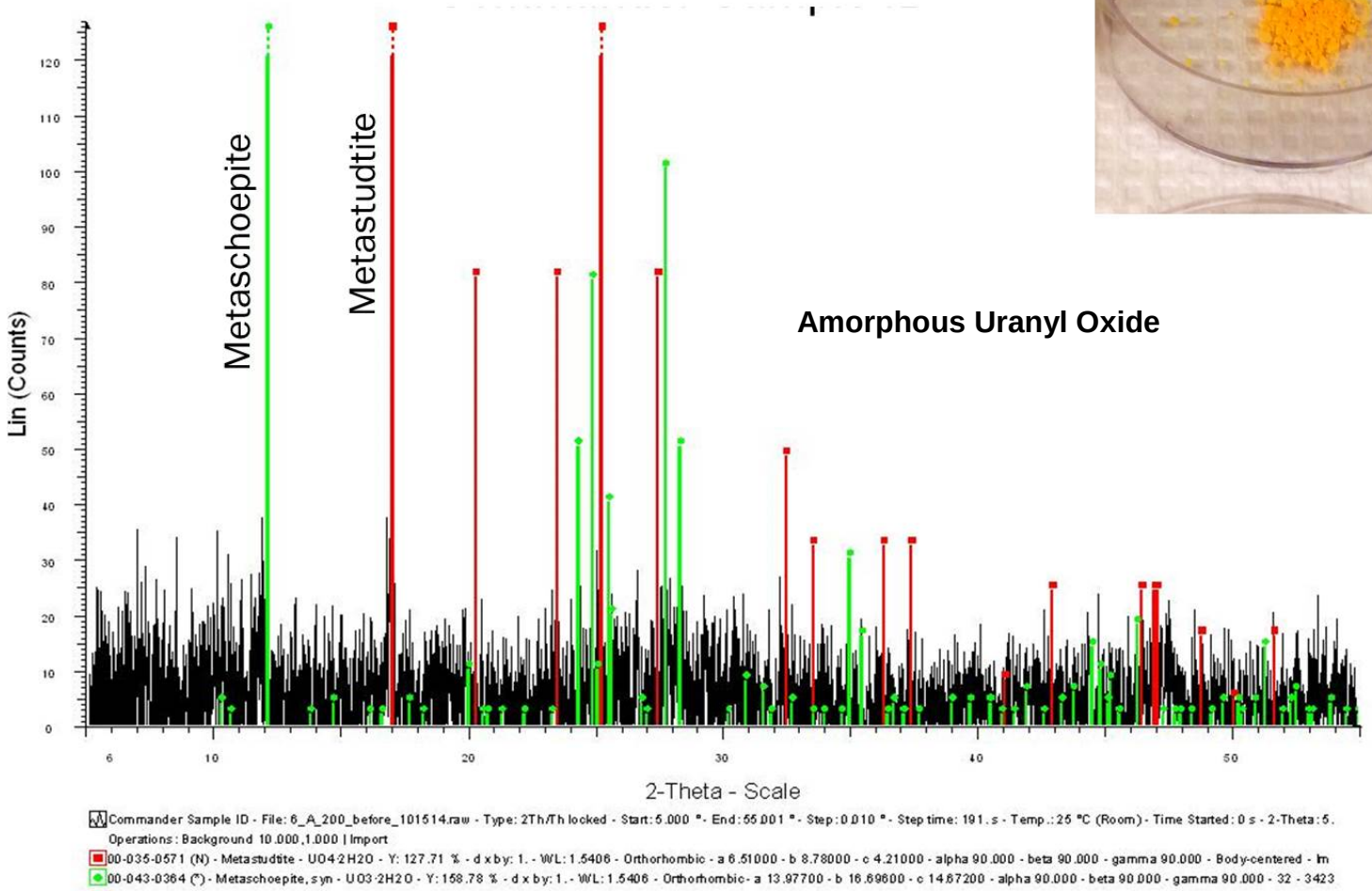
This graph is the first derivative curve of the mass-loss curve indicating the phase change transitions.

First derivative of mass-loss curve (indicates phase transitions)

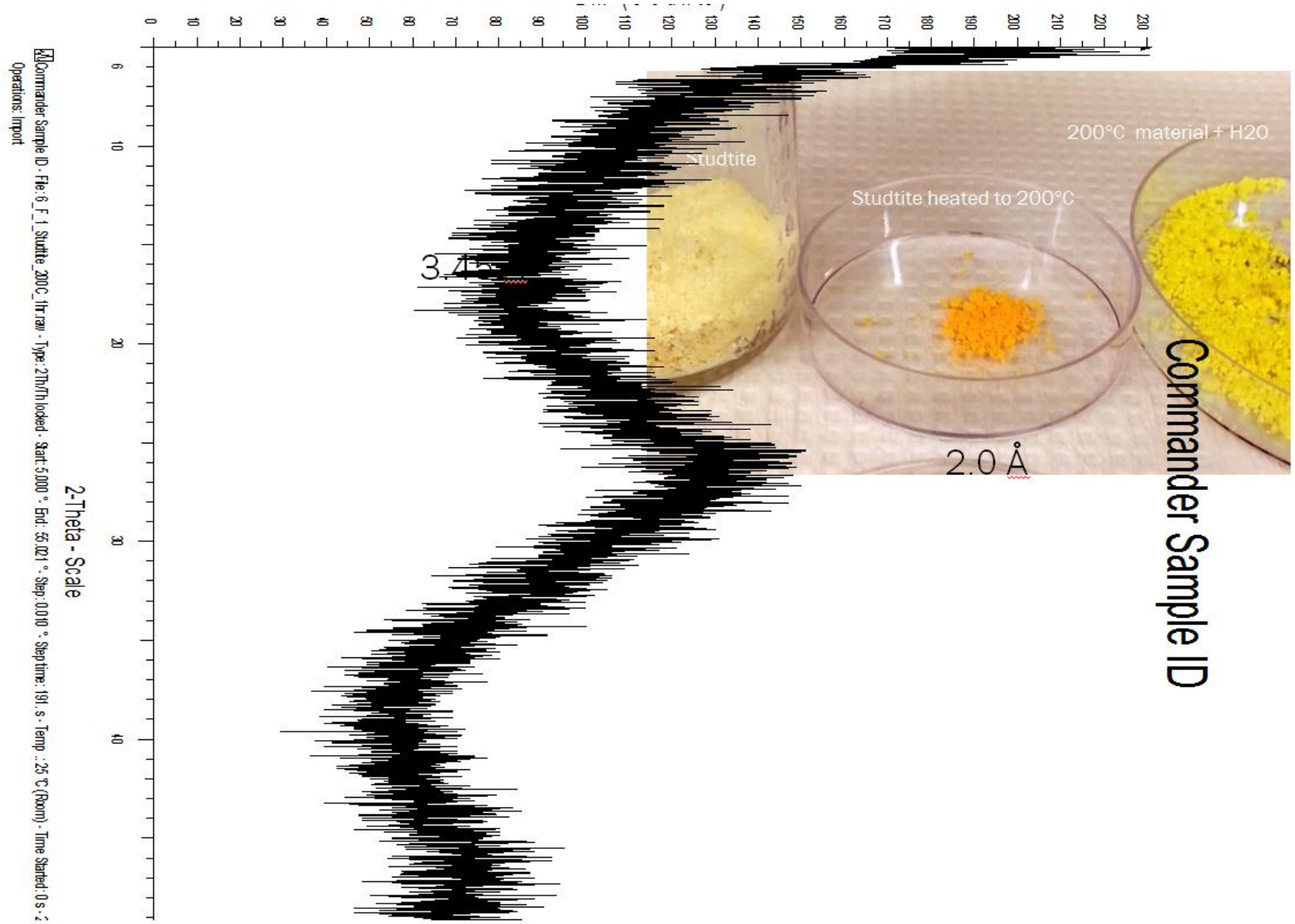


XRD spectra of 200°C heated metastudtite showing the metastudtite peaks and the development of amorphous uranyl oxide and mixed phases



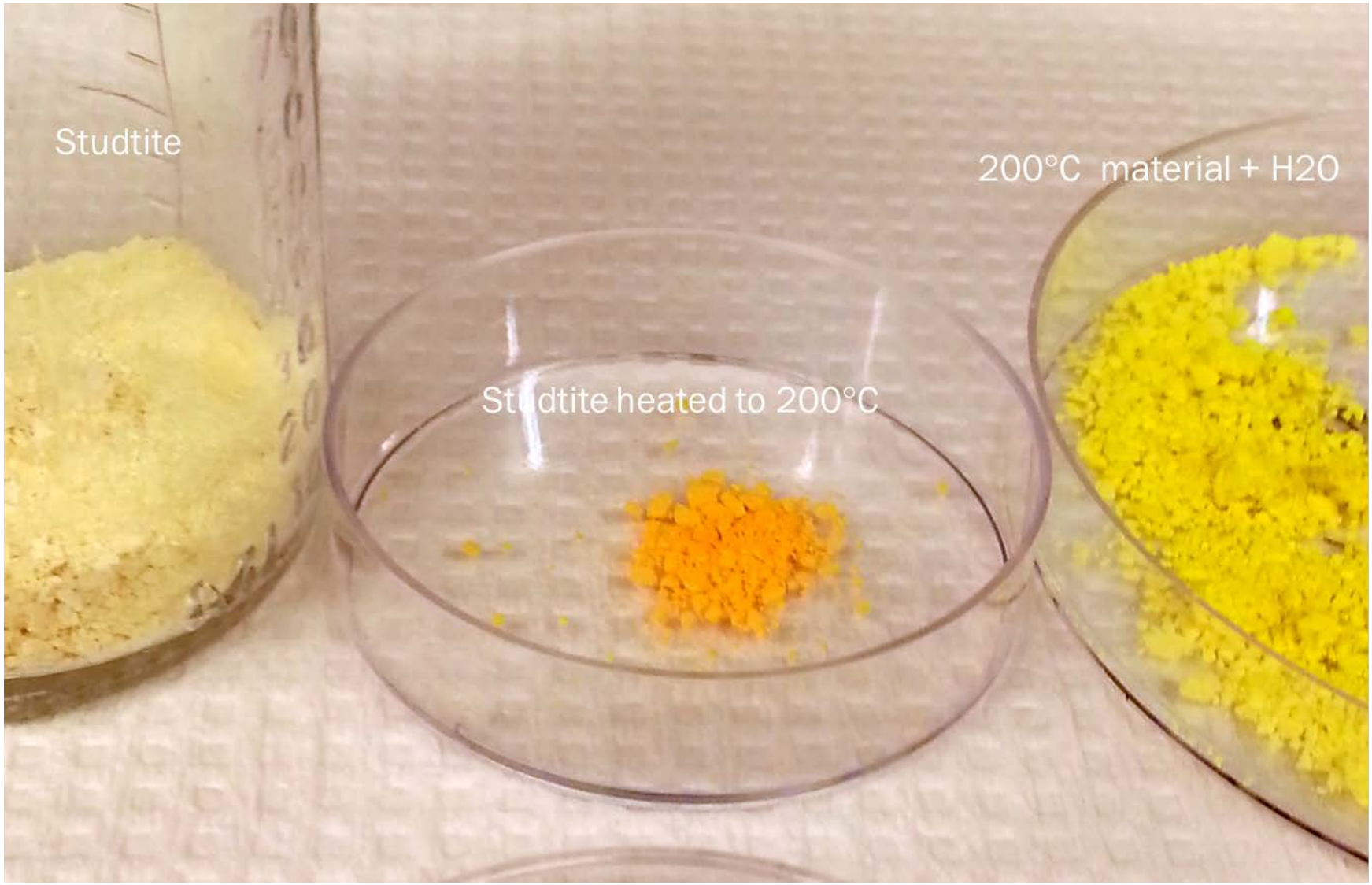


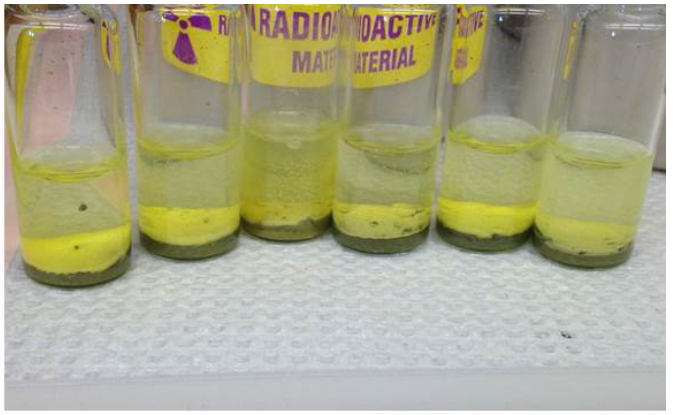
XRD Spectra of metastudtite heated to 200° which shows the amorphous nature of the uranyl oxide that is formed.



APPENDIX C3
NOTRE DAME DRIED YELLOWCAKE MINERALOGY STUDY
REACTION OF METASTUDTITE WITH WATER

This appendix presents the data from the reaction of metastudtite heated to above 200^{o}C (amorphous uranyl oxide) with water. As can be seen in the XRD spectra, all of the amorphous uranyl oxide is converted to the stable metaschoepite (UO_3.1-2H_2O).upon reaction with water. A similar result was seen for the initial Irigaray samples in that the reaction with water converted the amorphous uranyl oxide fraction to metaschoepite.


Studtite
200°C material + H2O
Studtite heated to 200°C



Reaction with water results in conversion to metaschoepite