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Symbol: DIR-15-127
LA-UR: 15-26713

August 31, 2015

Secretary Ryan Flynn
New Mexico Environment Department
Harold L. Runnels Building
1190 St. Francis Drive, Suite N4050
Santa Fe, New Mexico 87505

Subject: Self-Disclosure of Non-Compliances Resulting From the Extent of Condition Review, Los Alamos National Laboratory Hazardous Waste Facility Permit No. NM0890010515

Dear Secretary Flynn:

This letter voluntarily reports instances of non-compliance to the New Mexico Environment Department (NMED) and is submitted by the U.S. Department of Energy (DOE) and Los Alamos National Security, LLC (LANS), collectively, the Permittees, to meet reporting requirements of Section 1.9.14 of the Los Alamos National Laboratory (LANL) Hazardous Waste Facility Permit (the Permit).

The instances of non-compliance identified in the enclosed report (Enclosure 1) were first addressed with NMED on July 31, 2015, when the Permittees met with NMED to provide preliminary information on the non-compliances. As discussed at this meeting, these non-compliances were discovered in the course of a comprehensive Extent of Condition (EOC) review focused on determining the scope of non-compliances resulting from the use of inadequate waste management procedures similar to those used to process the nitrate salt-bearing waste stream associated with the February 14, 2014, WIPP incident and issuance of Administrative Compliance Order (ACO) HWB-14-20. These non-compliances resulted from processing other legacy mixed transuranic (MTRU) wastes under the same procedures that have been determined to be deficient

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and were used to remediate the nitrate salt-bearing waste stream associated with the WIPP incident.

Our staffs have completed a technical evaluation of these non-compliances and concluded that they do not present a credible safety concern to workers or the public and do not pose a threat to human health or the environment (*see* Los Alamos National Laboratory Report, *Characterization of Potential Hazards Associated with Potential RCRA Treatment Non-Compliances*, LA-UR-15-26595, Drs. D. Funk and D. Clark (Aug. 2015)) (Enclosure 1, Attachment A). Importantly, the non-compliances addressed in the enclosed report constitute the culmination of an extensive review of all legacy non-nitrate salt waste containers processed under deficient waste management procedures at LANL. We have taken a conservative approach in identifying the containers at issue and believe that after additional evaluation and testing the numbers of containers will decrease.

The EOC process resulting in this disclosure has been of critical importance to the lab and DOE, and both of us personally have been involved in reviewing the strategy for the EOC and have been frequently updated on the results included in this report. While we anticipate follow-up exchanges of information regarding these wastes, we believe that this EOC review completes the investigation into non-compliances associated with processing the non-nitrate legacy MTRU wastes at LANL. We are confident that the non-compliances identified in this report fully capture the deficiencies associated with legacy non-nitrate salt MTRU wastes processed and remediated at LANL under deficient waste management procedures.

We continue to be available to discuss with you at your convenience this self-disclosure report. If you have questions, please contact Michael T. Brandt (LANS) at (505) 667-4218, or Dave Nickless (DOE) at (505) 665-6448.

Sincerely,

Charles F. McMillan
Director

Kimberly Davis Lebak,
Field Office Manager

Enclosure: (1) Self-Disclosure of Non-Compliances Resulting From the Extent of Condition Review (FY 2006-2014), Los Alamos National Laboratory Hazardous Waste Facility Permit

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ENCLOSURE 1

Self-Disclosure of Non-Compliances Resulting From the Extent of Condition Review (FY 2006-2014)

Los Alamos National Laboratory (LANL)
Hazardous Waste Facility Permit

DIR-15-127

LAUR-15-26713

Date: AUG 3 1 2015.

Self-Disclosure of Non-Compliances Resulting From the Extent of Condition Review, Los Alamos National Laboratory Hazardous Waste Facility Permit

August 31, 2015

1. Introduction

This report has been prepared by the U.S. Department of Energy (DOE) and Los Alamos National Security, LLC (LANS), collectively the Permittees, to report to the New Mexico Environment Department (NMED) instances of non-compliance with the Los Alamos National Laboratory (LANL) Hazardous Waste Facility Permit (the Permit), as required by Permit Section 1.9.14. On July 31, 2015, the Permittees met with NMED to discuss the results of the Extent of Condition (EOC) review and provide preliminary information on the non-compliances. As a follow-up, this report is being submitted to provide further information.

As a result of previous reporting and Administrative Compliance Order No. HWB-14-20 (ACO), the Permittees conducted an EOC review to assess the scope of non-compliance stemming from the deficient procedures used to process nitrate salt-bearing wastes associated with the February 14, 2014 incident at WIPP and issuance of the ACO. During that review, the Permittees examined processing of other legacy mixed transuranic (MTRU) wastes remediated at LANL and found non-compliances similar to those identified in the self-disclosures associated with nitrate salt-bearing wastes reported in 2014.[1] The specific waste streams and non-compliant procedures are discussed in greater detail below. It is important to note that the Permittees have completed a technical evaluation of these non-compliances and determined that they do not present a credible safety concern to workers or the public and do not pose a threat to human health or the environment (*see* Los Alamos National Laboratory Report, *Characterization of Potential Hazards Associated with Potential RCRA Treatment Non-Compliances*, LA-UR-15-26595, Drs. D. Funk and D. Clark (Aug. 2015)) (Attachment A). Further, and equally important, the Permittees are confident that the non-compliances identified in this report constitute the full set of deficiencies associated with legacy non-nitrate salt MTRU wastes processed and remediated at LANL under deficient waste management procedures.

2. Summary of Non-Compliances

The EOC review addressed in this report focused on the following four legacy MTRU waste streams described in Central Characterization Project (CCP) Acceptable Knowledge Summary Reports[2] as follows: drummed debris waste (LA-MHDxx), oversized box debris waste (LA-

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[1] In July and October 2014, the Permittees reported non-compliances under the Permit associated with the processing of the nitrate-salt bearing mixed transuranic waste (MTRU) stream inventory at the Waste Characterization, Reduction, and Repackaging Facility (WCRRF). These self-disclosures resulted in receipt of an NMED-issued Administrative Compliance Order No. HWB-14-20 (ACO) dated December 6, 2014.

[2] *See* CCP-AK-LANL-004 (http://www.epa.gov/radiation/docs/wipp/ccp-ak-lanl-004-rev7.pdf) and CCP-AK-LANL-006 (http://energy.gov/sites/prod/files/2015/05/f22/CCP-AK-LANL-006-Revision%2013.pdf).

MHDxx), inorganic wastewater treatment sludge (LA-MINxx), and cemented waste (LA-CINxx).[3] The review determined that the same type of procedural deficiencies identified with the remediation of the nitrate salt-bearing wastes at WCRRF existed with regard to these four waste streams, i.e., procedures that specified unpermitted treatment through the improper use of "organic" kitty litter and/or neutralizers. Based on conservative assumptions, the non-compliances include the following:

- unpermitted treatment by use of absorbents in an impermissible manner;
- unpermitted treatment by neutralization;
- mixing potentially incompatible materials with waste in containers; and
- inadequate waste characterization; failure to manage and label waste containers, and meet generator requirements for off-site transportation of potentially ignitable (D001) and corrosive (D002) hazardous wastes (LA-CIN01).

The fourth bullet, identified above, is reported as a result of new information received from preliminary analytical results from liquid in one container in a subset of the cemented MTRU waste stream (LA-CIN01) indicating the possible presence of chemical oxidizing properties (D001). Although these test results did not indicate that the liquids are corrosive (D002) based on pH, further testing will be conducted to evaluate the potential for the corrosivity characteristic. For these reasons, this waste stream is currently being re-evaluated and re-characterized to confirm or deny the applicability of the ignitability (D001) and corrosivity (D002) characteristics. As a conservative measure, however, the Permittees are reporting potential non-compliances that arise if subsequent re-characterization efforts (e.g., analytical and sampling results) confirm the presence of the D001 or D002 characteristic. These non-compliances, and the status of the re-characterization efforts, are discussed below in Section 4.

3. Overview of Extent of Condition (EOC) Review

This report follows several non-compliance disclosures by the Permittees as a result of the February 14, 2014 incident at WIPP and issuance of the ACO. The initial EOC review, following the WIPP incident, focused on the inventory of nitrate salt-bearing MTRU waste containers and the associated safety issues. This resulted in two self-disclosures made to NMED in July and October 2014.

The EOC review was continued to determine if other legacy MTRU waste streams had been treated and/or managed in a manner that involved the same type of deficient procedures and non-compliances identified in the self-disclosures associated with the processing of nitrate salt-bearing MTRU wastes at WCRRF reported in 2014. The review included legacy MTRU waste streams (other than nitrate salt-bearing waste) and associated waste processing procedures and documentation used to "remediate" approximately 10,000 parent containers from the beginning of Fiscal Year 2006 (October 2005) until May 2014.[4] This time period was chosen because FY 2006

[3] The "xx" indicates that each of these CCP-AK waste streams includes containers in several sub-waste streams.

[4] "Remediate" refers to the repackaging and processing of LANL MTRU waste to remove liquids from "parent" containers through the addition of absorbents (e.g., Swheat kitty litter), and in some cases, with the addition of neutralizers, that are repackaged into drums or waste boxes (called "daughter" containers). This remediation process

is when LANL started waste remediation operations under procedures that involved the use of kitty litter and/or neutralizer in waste containers with free liquids. Waste remediated prior to FY 2006 involved sorting and segregation activities only (i.e., no absorbents and/or neutralizers were added to the waste during the process). These remediation efforts ceased in approximately May 2014. As a result, the four MTRU waste streams described above in Section 2 were identified as having been remediated and processed at LANL under deficient procedures.

3.1 EOC Review Approach

One of the primary factors contributing to the non-compliances associated with the nitrate salt-bearing waste stream was deficient repackaging procedures used at WCRRF which enabled the improper use of use of an "organic" kitty litter and neutralizer (*see*, WCRRF Procedure, R.36 and R.37.10.3). As with nitrate salt-bearing waste processing, these same procedures effectively enabled impermissible treatment for other legacy MTRU waste streams (*see* discussion below at Sections 4.A and 4.B).

The EOC team reviewed 13 waste management procedures used to process and remediate MTRU waste streams from October 2005 to May 2014. Due to changes to these procedures during this time frame, the team reviewed a total of 201 different versions of the procedures in order to identify which procedures (and associated timeframes) may have enabled potentially unpermitted treatment activities. The review disclosed that certain procedures, similar to those used to remediate nitrate salt-bearing waste at WCRRF, inappropriately specified processes outside the RCRA permit exemptions including: (1) the addition of absorbents directly to a parent container that previously held waste; (2) the addition of neutralizing agents to liquid waste; and (3) in some cases, specified the use of an "organic" kitty litter, which, in turn could create a potential incompatibilities.

After reviewing these procedures and identifying the specific time frames during which impermissible treatment activities could have occurred, the review team then conducted a detailed examination of individual container operating records to further identify specific containers that may have held free liquids. This information allowed the team to identify those specific containers that held free liquids and were remediated under deficient procedures that most likely involved impermissible absorption and/or neutralization.[5]

4. Non-Compliance Determination

This Section discusses the non-compliances identified in the EOC that were associated with the processing of the four MTRU waste streams under deficient procedures. Subsection A describes unpermitted treatment by absorption in 154 waste containers in a manner that did not comply with

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was intended to remove free liquids from parent containers to meet the WIPP waste acceptance criteria (WIPP cannot accept containers with free liquids greater than 1% volume of the waste).

[5] The EOC review also identified an additional four nitrate salt containers that were originally incorporated into the LA-MIN-04 waste stream, rather than the LA-MIN-02 nitrate-salt bearing waste stream. This discovery was verbally reported to NMED on March 3, 2015, and the containers were promptly incorporated into LA-MIN-02 nitrate salt-bearing waste inventory and Revised Nitrate Salt-Bearing Waste Container Isolation Plan (2014).

the treatment permit exemption. Subsection B describes unpermitted treatment by neutralization in 58 waste containers, where the use of neutralizing agents did not qualify for a treatment permit exemption. Subsection C describes, based on conservative assumptions, potentially incompatible mixtures in up to 354 waste containers due to the addition of organic kitty litter and/or neutralizers to the waste. Subsection D describes, based on conservative assumptions, up to 440 LA-CIN01 waste containers at LANL that may possess the ignitability (D001) and/or corrosivity (D002) characteristic.[6]

A. Unpermitted Treatment by Absorption (40 CFR §264.l(g)(10))

The addition of absorbents to a container is exempt from the RCRA treatment permit requirement if it meets the requirements of 40 CFR §§270.1(c)(2)(vii) and 264.1(g)(10) which provide, among other things, that (1) the absorbent be added to a container "at the first time waste is placed in a container" (i.e., the timing requirement); and (2) the absorbent is "compatible" with the container and the waste.

Between October 2005 and May 2014, the Permittees processed MTRU waste drums and waste boxes belonging to the four waste streams described above to remove small amounts of free liquids. The remediation process involved decanting liquids from "parent" containers (drums), adding absorbents to sorb free liquids, and repackaging the contents of the "parent" containers into new "daughter" containers. This process occurred at three LANL facilities: the WCRRF, a permitted storage unit located at Technical Area 50, Building 69 (TA-50-69); a Perma-Con® enclosure in Dome 231; and a soft sided containment enclosure in Dome 412. These facilities are permitted for storage only.

The EOC team identified two waste management procedures used to remediate MTRU waste that impermissibly specified the addition of absorbents directly into a parent container that held waste during remediation (wastewater treatment sludge (LA-MIN-03) and oversized boxes (LA-MHDxx). As with the nitrate-salt bearing waste stream, these procedures were deficient and did not comply with the RCRA treatment permit exemption requirement that waste be added "at the time the waste was first placed in the container" (*see* 40 CFR §264.l(g)(10)). A detailed review of the first procedure (for the LA-MIN-03 waste stream) addressing wastewater treatment sludge showed that this deficient process was in effect from September 2006 through June 2007. The second procedure (for the LA-MHDxx waste stream) addressed the Box Line operations at TA-54, and the deficient process was in effect from October 2011 through May 2014. As part of the EOC review, documentation for all of the containers remediated during the relevant time periods under these procedures were evaluated for impermissible absorption issues.

With regard to the second permit exemption criterion (e.g., use of compatible absorbents), the EOC team identified procedures that, like the nitrate salt-bearing waste processed at WCRRF, specified the use of "organic" kitty litter as an absorbent. As a result, the EOC team considered whether the organic kitty litter used during remediation of the waste stream was potentially incompatible with the waste. This issue is discussed further below (*see* Section 4.C).

[6] The containers identified in Section 4 may have multiple non-compliances (e.g., one waste container could have been treated with both absorbents and/or neutralizers).

Finally, the EOC team reviewed procedures to assess whether the procedure specified the mixing of absorbents with hazardous waste for the purpose of rendering the mixture less hazardous or non-hazardous. This process, if specified, would also constitute impermissible treatment under 40 CFR §§264.1(g)(1) and 270.1(c)(2)(vii). The reviewers examined all of the procedures and remediation records for the four EOC waste streams and identified no instances of potential treatment of this type.

In summary, the EOC review identified the same type of non-compliances regarding the unpermitted treatment by absorption previously identified in the self-disclosures for the nitrate salt-bearing wastes reported in 2014. Based on the EOC review of extensive documentation and best efforts, the Permittees believe that this non-compliance applies to 154 waste containers that were remediated under deficient procedures.

B. Unpermitted Treatment by Neutralization (40 CFR §264.1(g)(6))

The EOC review revealed that, like the deficient procedures used to remediate the nitrate salt-bearing wastes at WCRRF, the four MTRU waste streams were also remediated under procedures that specified neutralization of liquids during a two-year time period, from 2013 to 2014. As with the self-disclosures for the nitrate salt-bearing wastes reported in 2014, the Permittees believe that the addition of neutralizing agents to liquids in containers found in these four waste streams did not qualify for the elementary neutralization treatment permit exemption at 40 CFR §264.1(g)(6) because the waste streams were assigned multiple EPA Hazardous Waste Numbers (HWNs) D and F, and were not hazardous wastes solely due to the corrosivity characteristic (D002). Based on the EOC review of extensive documentation and best efforts, the Permittees believe that this non-compliance applies to 58 waste containers that were remediated under deficient procedures.

C. Mixing of Potentially Incompatible Organic Absorbents/Neutralizers with the Waste

The Permittees also considered possible instances of mixing incompatible organic kitty litter and neutralizers in these four waste streams.[7] The EOC review identified no more than 354 waste containers in these waste streams that had organic kitty litter mixed with the waste. As discussed below, the Permittees do not believe that the mixture of organic kitty litter and/or neutralizer within waste containers would present a significant (if any) incompatibility for these four waste streams. The Permittees comprehensively assessed the hazards associated with the use of potentially incompatible organic absorbents and/or neutralizers in each of the four waste streams. Using conservative assumptions that organics were used to absorb and neutralize liquids, along with review of CCP Acceptable Knowledge (AK) documentation for the waste streams, the Permittees concluded that there was no credible safety hazard that would pose a risk to the worker or the public. In addition, it is not expected that the non-compliances would generate or produce uncontrolled flammable fumes, gases, extreme heat, pressure, fire, explosions, or violent reactions (*see* Los Alamos National Laboratory Report, *Characterization of Potential Hazards Associated with Potential RCRA Treatment Non-Compliances*, LA-UR-15-26595, Drs. D. Funk and D. Clark (Aug. 2015)) (Attachment A).

[7] Section 2.8 of the Permit addresses the placement of incompatible wastes and materials in the same container, and imposes special precautions for waste management.

The EOC team assessed potential incompatibility based on use of an "organic" kitty litter mixed into waste containers. A number of procedures were identified that specified the use of an "organic" kitty litter for the four waste streams remediated in FY13 and FY14.[8] The Permittees assessed potential incompatibility using the method outlined at 40 CFR Part 264, Appendix V, EPA's Hazardous Waste Compatibility Chart[9] and the U.S. Department of Transportation (DOT) requirements at 49 CFR Section 177.848. Using EPA's Chemical Compatibility Chart, and associated guidance, the EOC team identified several dozen chemicals based on CCP's AK documentation that, if present in a pure form or as a majority constituent in the waste, may be incompatible with organic kitty litter when mixed with the waste. The use of an organic neutralizer, likewise, would have the same type of potential incompatibility issue.

The Permittees do not believe, however, that the mixture of organic kitty litter and/or neutralizer within waste containers would present an actual incompatibility for three of the four waste streams. For the two debris waste streams, the organic kitty litter used as an absorbent would be compatible – not incompatible – with the combustible primary waste form of these waste streams that include "organic" types of cellulosic materials (e.g., paper, rags, plastic, and wood-based high efficiency particulate air filters (LA-MDHxx (debris drums and oversized box wastes)). Regarding the third waste stream, which consists primarily of metal hydroxides and oxides (LA-MINxx (wastewater sludges)), an organic absorbent would also be compatible with these majority constituents. The fourth waste stream (the cemented waste stream (LA-CIN01)) consists of several waste types from the TA-55 nitric acid process that have been immobilized in gypsum or Portland cement. Although the cemented materials cannot react with the cellulosic absorbent, the Permittees are re-evaluating this waste stream due to new analytical information received regarding the presence of nitrate-bearing liquids (see Section 4.D, below).

Further, the CCP AK process is conservatively driven and assigns a chemical to an entire waste stream – not an individual container – based on the possible presence of such chemicals. Even chemicals at trace levels can trigger CCP to list the chemical for an entire waste stream, as opposed to verified chemical amounts or concentrations. CCP's AK process may assign a chemical to a waste stream based on (1) its reference as feed or process chemicals, (2) interviews with personnel performing operations (who sometimes stated only that a given chemical may have been used during a certain time period), or (3) lack of sufficient documentation to confirm the absence of the suspect chemical in the waste stream. For this reason, the CCP AK documentation alone would not provide a sufficient basis to support a finding of incompatibility.

Finally, incompatibilities may be expected from chemicals in pure form (or as major constituents), which are not expected in these waste streams. Pure form chemicals (or as major constituents) are not expected because these legacy wastes have undergone treatment processes prior to placement in the parent waste containers. Some wastes underwent liquid waste treatment processes and dewatering prior to placement in the original parent container, and others were cemented or had cement added to solidify them. All these processes would substantially reduce the potential

[8] During this time period, Swheat® kitty litter was prevalent at the locations the four waste streams were remediated (although small quantities of other organic kitty litters were used including Tidy Cat® and Yesterday's News ®). For this reason, the EOC team conservatively assumed that documented use of absorbent included the use of an organic kitty litter mixed with the waste.

[9] A Method for Determining Compatibility of Hazardous Wastes, EPA-600/2-80-76, Hatayama et. al., 1980.

incompatibility of the waste materials, decreasing the likelihood of a potential reaction with organic kitty litter such as use of Swheat® during remediation.

D. LA-CIN01 Waste Stream (D001, D002)

The Permittees are currently re-evaluating and re-characterizing a subset of the cemented MTRU waste stream (LA-CIN01) due to new information obtained from preliminary analytical results from one container that revealed chemical constituents in the liquids that may possess chemical oxidizing properties (D001). Although the test results did not indicate that the liquid was corrosive for pH, further testing will also be conducted to evaluate the waste stream for the corrosivity characteristic (D002). This re-evaluation and re-characterization effort, required by LANL Permit Condition 2.4.7, consists of additional analytical sampling and testing and is expected to take approximately three months.

As a conservative measure, and to provide a fully inclusive disclosure, the Permittees are reporting potential non-compliances that arise if subsequent re-characterization efforts (e.g., analytical and sampling results) confirm the presence of the D001 or D002 characteristic. These potential non-compliances may apply to no more than 440 waste containers at LANL and include the following: failure to characterize waste (40 CFR §262.11); failure to label and manage containers as a D001 and/or D002 hazardous waste (LANL Permit Sections 2.8.1, 2.8.2 and Permit Section 3.6(a)); and, failure to meet generator requirements associated with off-site transportation of potential D001 and/or D002 hazardous waste (40 CFR §§262.30-31). If subsequent re-characterization efforts confirm the absence of the D001 or D002 characteristic, then the Permittees will notify NMED in writing, institute orderly measures to remove container labels, and correct the next annual non-compliance report to reflect the removal of the non-compliances as required by Permit Section 1.9.14.

The subset of the cemented MTRU waste stream consists of homogeneous cemented legacy waste generated prior to 1991 resulting from the cementation of evaporator liquids from TA-55 nitric acid plutonium recovery operations.[10] Unlike the unconsolidated nitrate salt-bearing waste, this waste stream has been treated through cementation processes thereby substantially deactivating the chemicals in this waste stream. Real-time radiography (RTR) of the LA-CIN01 waste stream identified the presence of free liquids, which required the use of absorbents to resolve this free liquid in order to meet the WIPP waste acceptance criteria.

Ignitability (D001). As discussed with NMED, including at the July 31, 2015 meeting, some waste containers from the subset of the LA-CIN01 waste stream include small quantities of dewatered liquids with the potential for containing nitrate compounds. The liquid is believed to have originated from dewatering of the cemented waste over time. Preliminary sample results from free liquid in one unremediated cemented waste drum (No. S811785, LA-CIN01.001) was analyzed and found to contain oxidizing compounds, specifically nitrate in the ~34% wt. range. The applicable technical reference document states that 45 wt. % sodium nitrate solution is an example

[10] The evaporator liquids remained after nitrate salts were precipitated and segregated (eventually becoming the LA-MIN-02 stream, the nitrate salt-bearing waste stream). This liquid was cemented and became a subset of the LA-CIN01 waste stream.

of a Packing Group III, Division 5.1 oxidizer[11](*see* Appendix B Los Alamos National Laboratory Report, *Characterization of Potential Hazards Associated with Potential RCRA Treatment Non-Compliances*, LA-UR-15-26595, Drs. D. Funk and D. Clark (Aug. 2015)) (Attachment A). To determine if the sample liquid carries the ignitability characteristic (D001), the Permittees prepared a liquid surrogate (derived from the analytical results) and tested it by an EPA-certified testing laboratory for ignitability (D001, oxidizer). On August 24, 2015, the Permittees received test results indicating that the surrogate is not a Division 5.1 oxidizer (*see* Attachment C). This surrogate was based upon one LA-CIN01 sample analysis and further sampling, analysis, and testing is being conducted to fully evaluate the free liquid (and this liquid absorbed with organic kitty litter) for the ignitability characteristic (D001).

Corrosivity (D002). The preliminary sample result from the nitrate solution, discussed above, also showed that the liquid was basic (pH of 10.9) and does not meet the pH corrosivity characteristic (D002). However, additional sampling, analysis, and testing is being conducted to fully evaluate the free liquid for the corrosivity characteristic (D002).[12]

Discussion. As a result of its comprehensive review, the Permittees have identified no more than 440 waste containers stored at LANL that contain free liquids or liquids that were absorbed with organic kitty litter (118) or are awaiting RTR review for the presence of free liquids (322). The RTR process is on-going, and it is expected that the number of waste containers (440) will decrease. However, as a conservative measure, the Permittees have decided to label and manage these 440 waste containers in the interim as ignitable (D001) and corrosive (D002) hazardous waste pending completion of RTR results confirming absence of liquids and analytical oxidizer test results. The Permittees are implementing a sampling and analysis effort to analyze these waste containers to confirm or deny the applicability of the ignitability (D001) and corrosivity (D002) characteristic. In addition to the one container discussed above, approximately seven waste containers are planned to be sampled and analyzed to confirm the chemical composition of their drum contents. Analytical results will then be used to determine if additional testing is necessary. Concurrently, the Permittees have reviewed existing RTR data (available for most of the LA-CIN01 waste containers), and will schedule RTR analysis for the remaining containers without RTR data, or pre-screen data, as soon as practicable.

The Permittees are currently managing all 440 waste containers stored at LANL in a RCRA-compliant manner; however, the containers are not being managed as ignitable (D001) or corrosive (D002) hazardous waste or as waste containing free liquids (unless previously confirmed as such). The Permittees will transfer these waste containers as soon as practicable to a permitted storage unit at TA-54, Area G for storage in a manner that complies with the special Permit requirements for ignitable, corrosive, reactive or incompatible wastes (ICR) (LANL Permit Section 2.8).

In the meantime, the Permittees will promptly implement the following compensatory measures:

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[11] "Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria," Chapter 34, Procedure 0.2 (United Nations, 2009) (Attachment B).

[12] Under NMED regulations, a "corrosive" waste must be: 1) in an aqueous state with a pH less than or equal to 2 or greater than or equal to 12.5; or 2) a liquid and corrode steel a rate greater than 6.35 m (0.250 inch) per year. 40 CFR §261.22(a)(1)(2).

- Administrative controls (e.g., additional signage and work instructions) to ensure that heat or spark producing items/tools are not used in these areas to protect the drums from sources of ignition, and to ensure that drums are not stacked more than two high;
- Enhanced fire suppression/protection measures, as needed;
- Increased daily inspections (from weekly) to visibly inspect container integrity as a measure to address secondary containment and ICR requirements; and
- Inspections to ensure adherence to compensatory measures.

The Permittees will implement the above compensatory measures consistent with the LANL Permit, including the testing, repair and maintenance requirements for enhanced fire control equipment as required by Permit Section 2.10.1. In addition, in the event that compensatory measures are not met, the Permittees will notify NMED as required by Permit Section 1.9.

In the event the additional tests do not support the ignitability (D001) or corrosivity (D002) characteristic, then the Permittees will remove any D001 or D002 labels applied in the interim to the waste containers. Similarly, in the event RTR data and operating records confirm the absence of free liquids, the "free liquids" and "D002" labels will be removed from the specified container(s). In addition, if the D001/D002 labels and/or "free liquid" labels are removed, associated compensatory measures will be phased-out as appropriate. If the testing confirms the applicability of the ignitability (D001) or corrosivity (D002) characteristic, then appropriate treatment options will be assessed.

Off-Site Wastes (LA-CIN01). In addition to the 440 MTRU waste containers stored at LANL, the Permittees have shipped LA-CIN01 waste containers to Waste Control Specialists, Inc. (WCS) and WIPP. Based on review of documentation, the Permittees believe that the number of LA-CIN01 waste containers with free liquids or liquids absorbed with organic kitty litter will not exceed six (6) containers at WCS, and 192 containers at WIPP.[13]

In summary, the Permittee will contact and/or meet with NMED as necessary to provide updated information and discuss further options or contingencies for the management of this waste stream. In addition, the Permittees intend to update NMED in its monthly meetings, regarding these efforts, including the ongoing analytical work discussed above. If these tests do not confirm applicability of the D001 or D002 characteristic, then the Permittees will notify NMED in writing and correct the next annual non-compliance report to reflect the removal of the non-compliances as required by Permit Section 1.9.14.

5. Steps Taken or Planned to Reduce, Eliminate, and Prevent Recurrence

As required by Permit Section 1.9.14, and in response to the Ordered Actions in ACO HWB-14-20, the Permittees are undertaking a thorough investigation and evaluation of steps to reduce, eliminate, and prevent recurrence of these potential non-compliances. Several immediate steps were taken (e.g., the Permittees have halted all processing of TRU/MTRU waste at LANL). As part of

[13] The Permittees review of Batch Data Reports indicate that although these containers met the WIPP-WAC criteria for free liquids, the containers have less than 1% free liquids.

the response to the Ordered Actions in the ACO, the Permittees have engaged in a cooperative and comprehensive effort with NMED to address numerous corrective actions required under the ACO to reduce, eliminate, and prevent recurrence.

As discussed above, this self-disclosure report concerns the same categories of non-compliances identified in ACO HWB-14-20 and addressed the self-disclosures associated with the remediation of the nitrate salt-bearing wastes at WCRRF reported in 2014. No new categories of non-compliances were identified. As with the deficient WCRRF procedures, the non-compliances addressed in this report stem from use of deficient procedures to remediate other legacy MTRU waste streams. The Permittees are confident that the extensive corrective actions required under the ACO to address the deficiencies identified with the nitrate salt-bearing waste stream will fully cover and encompass the non-compliances addressed in this self-disclosure applicable to the other legacy MTRU waste streams managed at LANL.

CERTIFICATION

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

8/31/15

Michael T. Brandt, DrPH, CIH
Associate Director
Environment, Safety, and Health
Los Alamos National Security, LLC
Los Alamos National Laboratory
Operator

Date Signed

8/31/15

Kimberly Davis Lebak, Manager
Los Alamos Field Office
National Nuclear Security Administration
U.S. Department of Energy
Owner/Operator

Date Signed

Attachments to Enclosure 1

Self-Disclosure of Non-Compliances Resulting From the Extent of Condition Review (FY 2006-2014)

A. Los Alamos National Laboratory Report, *Characterization of Potential Hazards Associated with Potential RCRA Treatment Non-Compliances*, LA-UR-15-26595, Drs. D. Funk and D. Clark (Aug. 2015)).

B. Excerpts for Procedure 0.2, Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria," Chapter 34, Procedure 0.2 (United Nations, 2009).

C. Preliminary Analytical Sampling Results (DOT/UN Test 0.2 (Div. 5.1) (Chamberlain Surrogate No. 1 and No.2) (August 20, 2015).

Attachment A

Los Alamos National Laboratory Report

Characterization of the Potential Hazards Associated with Potential RCRA Treatment Non-Compliances

LA-UR-15-26595

Los Alamos
NATIONAL LABORATORY
EST. 1943

Characterization of the Potential Hazards Associated with Potential RCRA Treatment Non-compliances Report

Prepared by:

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Associate Director, Environmental Programs

Subject: Characterization of the Potential Hazards Associated with Potential RCRA Treatment Noncompliances

David J. Funk and David L. Clark

In response to the WIPP incident, Los Alamos National Laboratory conducted an Extent of Condition (EOC) review of waste processes to identify whether additional potential noncompliances (beyond those previously identified with remediated nitrate salts) under the LANL Resource Conservation and Recovery Act (RCRA) Permit were made. This comprehensive review determines the extent of treatment noncompliances and serves as a lesson for future remediation work and workers going forward, so as to meet our commitments with the State of New Mexico in a safe and compliant fashion.

The purpose of this document is to provide a hazard evaluation of the noncompliances and whether any new actions are required to mitigate potential risk to the worker or the public. ***In short, we have reviewed the noncompliances and have concluded that the possibility of exothermic reactions leading to radioactive release is not credible, and in one case, inconceivable, stemming from the fact that the majority fraction of the waste is compatible with organic absorbents and neutralizers. It is not expected that the noncompliances would generate or produce uncontrolled flammable fumes, gases, extreme heat, pressure, fire, explosions, or violent reactions.*** Regarding nuclear safety basis, where consequence should also be considered, we expect there to be a low consequence to the worker or the public: a container breach is not expected due to the small quantities of potentially incompatible materials identified.

We provide a brief summary of the EOC review below, and then discuss the waste streams and the potential for hazards that each waste stream could present.

Overview of the Extent Of Condition (EOC) Review

The instances of noncompliance involve the processing of four mixed transuranic (mixed TRU or MTRU) waste streams (other than nitrate salt-bearing waste) at LANL: oversized box debris waste (LA-MHDxx), drummed debris waste (LA-MHDxx), wastewater treatment sludges (LA-MINxx), and cemented waste (LA-CINxx)[1]. The purpose of the EOC review was to determine whether LANL's prior remediation activities associated with TRU/MTRU waste streams other than nitrate salts had compliance issues similar to the nitrate salts remediation activities. LANL wanted to determine whether:

> (1) the same flaws identified in the nitrate salts remediation procedures may have affected TRU/MTRU waste streams other than the unconsolidated nitrate salts; and

[1] The "xx" indicates that each of these CCP-AK waste streams includes containers in several sub-waste-streams.

(2) said flaws may have affected LANL locations that processed waste in addition to the Waste Characterization, Reduction, and Repackaging Facility (WCRRF) facility (i.e., locations at Technical Area (TA)-54, Area G).

"Remediated" MTRU waste is defined as LANL MTRU waste (from "parent" containers) that was processed to remove liquids through the addition of absorbent material (e.g., kitty litter and/or polymer sorbents) – in some cases, with the addition of neutralizers - and repackaged into new drums or waste boxes (called "daughter" containers). The remediation process was necessary to remove free liquids from parent containers, in order to meet the WIPP Permit requirement that TRU waste containers received at WIPP may not contain free liquids greater than 1% volume of the waste (WIPP Permit, §2.3.3.1).

However, as with the nitrate salts, some remediation activities may have constituted impermissible treatment. EPA defines "treatment" to include "any method, technique, or process, including neutralization, designed to change the physical, chemical, or biological character or composition of the waste ... to render the waste non-hazardous, or less hazardous; safer to transport, store, or dispose..." Specifically, the EOC review focused on identifying whether:

(a) any absorption processes employed to remediate TRU or MTRU wastes may have constituted instances of impermissible treatment of hazardous waste;

(b) any neutralization processes employed to remediate TRU or MTRU wastes may have constituted instances of impermissible treatment of hazardous waste; and

(c) any instances occurred where potentially incompatible absorbents or neutralizers may have been mixed with the waste (potentially resulting in creation of a problematic secondary waste stream).

In review of these records, we identified three categories of noncompliances:

1. **Unpermitted treatment by absorption.** Principally, adding absorbent to the parent container, not to a daughter at the time of generation. In some cases absorbent was added after Real Time Radiography RTR showed liquids were still present, a violation of RCRA, which requires addition of absorbent at the time of generation.
2. **Unpermitted treatment by neutralization.** RCRA allows neutralization if, and only if, the D002 characterstic is applicable. If any other hazardous waste codes apply, then a permit is required to neutralize.
3. **Mixing of Potentially Incompatible Absorbers and/or Neutralizers with the Waste.** RCRA forbids the mixing of incompatible materials to prevent potentially hazardous reactions.

In support of this review, LANL reviewed records for a targeted subset of a population of 9778 MTRU waste containers remediated at LANL between FY2006[2] and 2014. This effort resulted in identifying 566 potential noncompliances (in a total of 508 containers, some with multiple noncompliances) as follows:

- 154 containers were remediated using absorption methods that potentially did not meet the RCRA permit exemption
- 58 containers were remediated using potentially impermissible neutralization methods
- 354 containers were considered potential noncompliances based on mixing potentially incompatible absorbents or neutralizers with the waste.

This document summarizes the potential incompatibilities and the possible hazards resulting from the mixing of potentially incompatible materials. We note that the EOC took a very conservative approach as to whether the addition was incompatible. As an example, we have included the analysis as to whether the addition of Swheat® would be considered incompatible, in Appendix A. Essentially, the absorbent/neutralizer used was compared with the known source of oxidizers identified in the Central Characterization Project (CCP) Acceptable Knowledge (AK) documents (CCP-AK-006, rev. 13 and CCP-AK-004, rev. 7). We note that WIPP has a no migration variance, and as a result, it can accept a very broad spectrum of waste, i.e. heavy metals, absorbed solvents, etc. An evaluation of such a broad waste stream (from organics to metals to oxidizers) will almost inevitably result in incompatibilities[3].

Evaluation of the Potential Hazards for Each Waste Stream

Inorganic Sludges (LA-MIN03-NC.001)

Waste stream LA-MIN03-NC.001 consists of homogeneous dewatered sludge generated during the treatment of caustic and acid waste streams through the addition of base. The main treatment process removed particulate and heavy metals as a sludge that settled out and/or was cleaned out of the clariflocculation tanks. The first step of the main treatment process was the addition of flocculants to produce a precipitate and involved addition of (primarily) calcium carbonate and ferric hydroxide. The precipitate or sludge was then dewatered and contained approximately 25 to 40 percent inorganic solids with a "wet clay" consistency. Perlite or diatomaceous earth was used after dewatering to further filter and absorb liquids present in the

[2] That year was chosen as the demarcation point at which LANS began absorption and neutralization activities on TRU waste.

[3] The AK describes a waste stream that is inherently incompatible with itself. Cf., the LA-MINHD01.001 debris waste stream table summarized on pages 96-105 in CCP-AK-LANL-006, Rev. 13.

sludge. Finally, Portland cement was added to the bottom and top of the sludge during packaging. Prior to remediation, this waste may also contain debris, including but not limited to containers (e.g., unpunctured aerosol cans, vials), metal, personal protective equipment (PPE), plastic, and secondary waste from repackaging.

Because the nitrate ion is stable and highly soluble it is not amenable to removal by conventional water treatment processes such as coagulation and precipitation. As a result, the sludge would not contain any significant concentration of nitrates (oxidizers). ***While the sludge is technically incompatible with organic neutralizers/sorbents, the potential for thermal runaway from these mixtures is not credible due to the fact that these low concentrations cannot lead to significant chemical reaction.***

TA-35 Heterogenous Waste

This waste was generated from a TA-35-2-Wing C glove box in which the plastic glove box extension was bagged out into a 55 gallon drum and it has not been assigned to a CCP-LANL AK at this time, though it appears to fit in the MHDXX waste stream. Operators at WCRRF noted that the drum contained the following: gloves, kitty litter, plastic, fiberglass, wipe balls, cardboard. None of these components exhibit incompatibilities with organic neutralizers/sorbents. ***The possibility of any reaction from this drum would be inconceivable due to the compatibility of organic absorbents and neutralizers with this waste.***

Waste Stream LA-MIN02-V.001

Waste stream LA-MIN02-V.001 consists primarily of inorganic particulate waste generated in TA-55. The waste is largely comprised of TRU waste such as liquids and solids absorbed or mixed with absorbent (e.g., Ascarite II, [sodium hydroxide coated silicate], diatomaceous earth [silica and quartz], kitty litter [clay], vermiculite [hydrated magnesium-aluminum-iron silicate], and/or zeolite [aluminosilicate mineral]), though a wide range of chemicals and other materials are included in the complete AK. For example, this is the waste stream that included the nitrate salts, i.e. those responsible for the WIPP incident. Since there were only six records associated with these noncompliances, we reviewed each record against the generator and operator comments listed below, and we find no incompatibilities:

Generator Record	Operator Comments
Solidified Organic	Metal cans, vermiculite, plastic, liquid
PD-241; kerosene (30% DBEP) vermiculite (70%) - Organics: Benzene, toluene, styrene, polystyrene fragments (decomposition from heating Polystyrene, contents is inhomogeneous)	Plastic, vermiculite, 5 gallon container

Organic liquids absorbed in vermiculite	Plastic, (2) 5 gallon buckets, solidified inorganic and organic solids
Pyrochemical salts in 5 gal pails; paint chips/stripper, cheesecloth in 5 gal buckets	Plastic, liquids, (2) 5 gallon buckets, solidified inorganic and organic solids.
Room trash assigned Code A60 (other combustible)	Poly liner lid, banding, but most of drum is like brown sand clay gravel mixed together. Kitty litter Hex armor gloves on last daughter.
Room trash assigned Code A60 (other combustible)	Poly liner lid, banding, but most of drum is like brown sand clay gravel mixed together. Kitty litter Hex armor gloves on last daughter.

LANL believes that MIN02 was assigned to this waste stream on the basis of the vermiculite or inorganic material (sand, clay, gravel) that was identified in the waste. We note that pyrochemical salts consist of sodium and potassium chloride and are compatible with organic sorbents/neutralizers. ***We find that the possibility of thermal runaway for these drums is not credible, since organic absorbents and neutralizers are compatible with the majority components of the waste.***

Boxline and Drum Debris Waste Stream MHD0X.0xx

The typical AK for debris waste stream included a statement such as: the waste "consists primarily of mixed heterogeneous combustible and non combustible debris. Example of combustible materials include: paper, rags, plastic, rubber, wood-based high-efficiency particulate air (HEPA) filters...". In addition, the AK identified all of the potential chemicals that might be expected to be associated with this stream, and as noted previously, if taken literally, would be incompatible with itself.[3] The majority of these chemicals are minor or trace constituents and while technically incompatible, provide little or no means to support chemical reactions that could lead to any kind of runaway within a debris drum. Thus, the addition of other organics (e.g. Swheat) to absorb any liquid would not lead to an incompatibility of any significance.

A question arises as to where the liquids that were absorbed came from. Oversized TRU legacy waste was from cold war operations and was brought to Area G up to 30-40 years ago. This waste, which consisted of piping, filters, gloveboxes, tanks, equipment and pencil tanks, were packaged at the generator sites primarily in Fiber-Re-enforced Plywood (FRP) crates that were buried underground for many years, and then unearthed and placed into storage domes. Both the underground storage and placement in above ground sites that sometimes experienced leaks, allowing water to be captured within the debris waste form. In addition, residual liquid in bottles that were used to decontaminate during initial packaging were a second source of liquid. Third, many of the drums were power-washed when brought above grade and their condition would have potentially allowed

water into the waste. In any event, given the nature of the waste stream (mixed combustible and noncombustible material) the Swheat or other organic absorbers/neutralizers would be technically incompatible but not present a source of chemical reactions. ***We conclude that the possibility of thermal runaway for these drums is not credible, since organic absorbents and neutralizers are compatible with the majority components of the waste.***

Cemented Inorganic Waste, LA-CIN01.001, Treated with Swheat Kitty Litter (Absorption of Liquids with Organic Absorbers)

Waste stream LA-CIN01.001 consists primarily of inorganic homogeneous solid waste (cemented TRU waste) generated within TA-55. Many waste products were encased within cement in this fashion. For the purposes of this discussion, we are referring to the subset of LA-CIN01.001 that was generated with evaporator liquids from TA-55 nitric acid plutonium recovery operations. These liquids were expected to be near saturation with nitrate salts (the salts were precipitated and the remaining liquid was used in the cementation process). This aqueous waste was immobilized in Portland or gypsum cement using processes that were not as robust as those of today. This waste stream was generated prior to 1991, and radiographic analysis of the waste stream often indicated liquids (dewatering) failing the WIPP WAC, and as a result, this waste stream required remediation prior to acceptance by WIPP.

Remediation was conducted within the Glove Box at WCRRF, and during the timeframe of interest, Swheat Kitty Litter was the absorbent used, both to remediate liquids, and based on operator interviews, often as a precaution against dewatering (even when liquid was ***not*** reported to be present).

The use of Swheat in this manner, when combined with nitrate salt solution, triggers a measure of concern: nitrates are incompatible with organics, and solutions of concentrated nitrates can be characterized as oxidizers (see below). However, the risks associated with the use of the organic absorbent in this manner are expected to be very different than the risks associated with the remediated nitrate salts (RNS):

- Quantities of absorbent used were only that required to absorb the small quantity of liquid present.
- The chemical environment of the liquid is expected to be alkaline (use of grout) versus strongly acidic for the nitrate salts, which would not support solution phase nitration of the Swheat.
- The concentration of nitrates in the free liquids is much lower than those of the neat nitrate salts, though they could be at oxidizer levels (see below).
- NO_2/N_2O_4 will not be generated (salts are in solution), and the Swheat

will not become nitrated through gas-phase reactions, in contrast to the Remediated Nitrate Salt (RNS) waste stream.

- The large volume of cement comprising the vast majority of the contents of the drum is inherently inert.
- No organic neutralizers were used with the free liquids.

Ultimately, the degree of hazard will depend on the concentration of the nitrate salts in solution, as well as their chemical makeup, resulting in their classification as Div. 5.1 Oxidizer in some instances (e.g., 45 wt.% sodium nitrate solution, Packing Group III) and ***not*** Div. 5.1 in other cases (e.g., saturated solution of calcium nitrate).[4] Liquid nitrates are tested using UN DOT O.2 testing, to determine their oxidizing potential and appropriate Packing Group for transportation.

To evaluate the potential hazard created by absorbing with Swheat, an understanding of the chemical environment of the liquid is required. To achieve that understanding, a campaign to sample free liquids from eight CIN01 drums was initiated. Chemical analysis from the first free liquid sample was recently completed, indicating nitrate salt concentrations that may be at "oxidizer levels" (approximately 34% by wt., cf. Appendix B). As a result, we will conduct UN DOT O.2 testing of non-radioactive surrogated to evaluate whether the liquid exhibits the D001 characteristic (45% sodium nitrate is a Packing Group III oxidizer, the least "oxidizing" of liquid oxidizers).

We do expect that the degree of hazard presented by this waste form to worker (and public) is very different from, and does not approach, the hazard presented by the RNS waste form. As indicated previously, the quantities, pH, salt concentration, lack of nitration, lack of organic neutralizer, and environment (large mass of inert cement), create an environment very different from the remediated nitrate salt drums. ***As a result of these differences, the possibility for thermal runaway of the Swheat used in the absorption process is not credible.***

To support this analysis, we developed the following strategy to understand the nature of the hazard as well as the degree of reactivity taking place within the CIN01 drums remediated with Swheat:

1. Differential Scanning Calorimetry (DSC) is a useful screen to evaluate thermal stability/sensitivity. We conducted tests on surrogates that included a known oxidizer, 45 wt.% sodium nitrate;

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[4] Recommendations on the Transport of Dangerous Goods Manual of Tests and Critera, United Nations Publishing (New York and Geneva), Fifth Edition (2009).

and a solution prepared from our most thermally active salt surrogate WB8 as follows:

a. 45 wt.% solution of $NaNO_3$ mixed with Swheat in 1:1 and 3:1 ratios (by volume)

b. A saturated solution of WB8 also mixed with Swheat in 1:1 and 3:1 ratios (by volume)

c. Tests were conducted with both wet and dry surrogates. In all cases, reactions did not initiate until the mixture was over 100 °C, with an estimate of the most reactive occurring at 125 °C (cf. Appendix C).

2. We will conduct headspace gas analysis on the CIN01 drums. As noted in Clark and Funk (2015),[5] CO_2:H_2 ratios greater than 6.5 are indicative of processes other than radiolysis such as oxidation. Thus, ratios of less than 6.5 would likely be indicative of only radiolytic processes, leading to the conclusion that little chemistry is taking place within the CIN01 drum.

Furthermore, in our testing of nitrate salt/Swheat surrogates, we note that the material (pure salt mixed with Swheat), failed the UN DOT O.1 testing (solid oxidizer), but was on the edge of "passing" (would not have been considered an oxidizer). Our expectation is, that given the low concentration of salt relative to Swheat, that the material will likely test out as a D001 characteristic waste, and we will propose conducting these tests as part of our strategy to characterize these materials. *This testing will be conducted to support a regulatory determination of the characteristic of the waste and should not be construed as additional evaluation of the hazards of this waste form.* Thus, we will:

1. Conduct UN DOT O.1, Method 1030, and Method 1050 for the Swheat/absorbed liquid and O.2 testing for the neat liquid using surrogates, to properly evaluate this waste form for RCRA characteristic D001 (using external laboratories such as Southwest Research Institute; SWRI).

The tests and sampling are expected to take several weeks to complete. In addition, we expect to continue to sample the other seven CIN01 drums to ensure we have "representative" samples. Based on testing to date, we do not anticipate the need to include the CIN01 drums remediated with Swheat (though we have examined this as an option) in the container isolation plan. ***Based on the discussion above and the testing completed***

[5] Clark, D. L. and D. J. Funk, 2015. Chemical Reactivity and Recommended Remediation Strategy for Los Alamos Remediated Nitrate Salt (RNS) Wastes. LANL Report LA-UR-15-22393

to date, thermal runaway is not credible for the LA-CIN01.001 waste stream.

Spontaneous combustion from Swheat self-heating

As noted in the Clark and Funk[5] report, the possibility of self-heating of Swheat has been considered as the potential initiator for the spontaneous combustion of Drum 68660, the drum responsible for the radiation release within Room 7 of Panel 7 at WIPP. In the cases identified above, the Swheat has been determined to be compatible with the majority fraction of the waste, and were any self-heating to occur, the reaction could not be sustained due to the lack of oxidizer that would be available. This is in contrast with the nitrate salt waste, where self-heating would be fed by the oxygen that would be released from the incompatible oxidizer, the nitrate salts themselves. ***The risk of spontaneous combustion leading to drum breach is not credible, a result of the fact that organic absorbents and neutralizers are compatible with the majority faction of the waste form.***

Conclusions

Utilizing a conservative approach to the noncompliances by comparing the use of organics (neutralizers/sorbents) with the CCP AK data for the waste streams, chemicals identified within the AK could potentially be present in the waste that, in pure form and/or sufficient concentrations, could be incompatible with organic neutralizers/sorbents (such as Swheat® or Kolorsafe®). ***However, a careful review of the waste stream, and understanding the majority components, we find that the possibility of thermal runaway for containers associated with these noncompliances is not credible, and in once case, inconceivable, stemming from the fact that the majority fraction of the waste is compatible with organic absorbents and neutralizers. It is not expected that the noncompliances would generate or produce uncontrolled flammable fumes, gases, extreme heat, pressure, fire, explosions, or violent reactions.*** Regarding nuclear safety basis, where consequence should also be considered, we expect there to be a low consequence to worker or public: a container breach is not expected due to the small quantities of potentially incompatible materials identified. As a an added measure of conservatism, LANL will conduct surrogate testing and Headspace Gas Sampling of the CIN01 waste stream, to support our conclusion that the levels of oxidizers contained within the CIN01 liquids to not support assigning the RCRA D001 characterstic to this waste stream.

APPENDIX A.

ANALYSIS OF CCP-AK-004 AND CCP-AK 006 WASTE STREAMS FOR POTENTIAL INCOMPATIBILITY WITH Swheat™ SORBENT

Four mixed transuranic (MTRU) waste streams were remediated by ADEP/LTP using organic sorbents and/or neutralizers between 2006 and 2014. The purposes of this analysis were as follows:

> (1) to identify any chemicals that may have been present in four remediated waste streams that were potentially incompatible with organic sorbents (*the organic sorbent Swheat™ is the specific focus of this analysis*); and
>
> (2) if so, to address the following question: were the chemicals likely to be present in forms or quantities sufficient to constitute an incompatibility of wastes and sorbents in the same container?

Permit Requirements

Permit Section 2.8 requires the Los Alamos National Laboratory (LANL) Permittees to "*…take precautions during the treatment or storage of ignitable or reactive waste, the mixing of incompatible waste, or the mixing of incompatible wastes and other materials to prevent reactions...*"

Permit Sections 2.4.1(3) and 2.4.1(4) require the LANL Permittees to "*…obtain and document all of the information that must be known to treat, store, or otherwise manage a hazardous waste stream in accordance with 40 CFR Parts 264 and 268 including… waste characterization necessary to prevent the mixing or placing of incompatible wastes in the same container*" and "*…waste characterization necessary to prevent accidental or spontaneous ignition or reaction of ignitable or reactive wastes, including, but not limited to, ignition or reaction in containers.*"

Review Approach

Identification of Oxidizing Chemicals

Compliance with these permit conditions requires knowledge of the chemicals present in a container and a determination of their compatibility with other materials in the container such as sorbents (i.e. Swheat™). Because Swheat™ is a natural, organic material, its incompatibility would be with oxidizing chemicals. Because the waste streams involve legacy waste, chemical analytical data from the waste generators for the four waste streams in the LTP Extent of Condition (EOC) review is minimal to nonexistent. Therefore, ENV-CP's review approach was as follows. The four waste streams in the LTP Extent of Condition review are addressed in two CCP Acceptable Knowledge (AK) documents (CCP-AK-006, rev. 13 and CCP-AK-004, rev. 7). These two CCP AK documents were selected because, upon review, ENV-CP determined that they effectively summarize all

the LANL AK documentation that is currently available regarding the historical chemical composition of the four waste streams. Therefore, the chemical summaries in the two CCP AK reports were reviewed to identify oxidizers in the four waste streams that might be potentially incompatible with Swheat™ or other organic sorbents.

Each CCP AK document contains a Chemical Identification and Use Summary Table that lists chemicals found in the MTRU waste streams addressed. Each Summary Table was checked to identify whether any chemicals listed would be classified as oxidizers.

These oxidizing materials were then checked, using EPA and DOT methodologies and data, to determine if a potential incompatibility exists.

EPA 40 CFR 264 Appendix V Compatibility Analysis

The first compatibility determination was completed using the EPA method and data in 40 CFR 264 Appendix V. LANL is conservatively considering Swheat™ to be a Reactivity Group 101 "Miscellaneous Combustible or Flammable Material" (i.e., a 40 CFR 264 Appendix V material in Group 6B) for purposes of this analysis. If a Group 6A (oxidizing) chemical is present in the CCP Chemical Identification and Use Summary Table, it would theoretically be incompatible with Swheat™ for purposes of this analysis. Oxidizing materials from the CCP AK documentation were compared to the chemical groups and classes listed in Compatibility Group 6A.

DOT 49 CFR 177 Compatibility Analysis

Two segregation requirements under DOT were used to assess potential incompatibility based on references to DOT requirements in the LANL Permit. DOT segregation requirements codified at 49 CFR 177.848 apply to the segregation of containers with differing hazard classes and divisions on trucks and transport tanks and not to the mixing of incompatible materials in a container. If used as a guide for incompatibility determination for mixing within a container, the oxidizing materials from the AK documents could not be mixed with materials in incompatible classes as shown on the Segregation Table For Hazardous Materials in 48CFR 177.848.

DOT 49 CFR 173 Compatibility Analysis

Actual compatibility requirements for mixtures in containers under DOT are provided in 49 CFR 173.24(e)(4), and do not provide a method for determining incompatibility. However, they do prohibit mixing of materials that would cause fires, create heat or toxic gases, etc. To determine potential incompatibility under 49 CFR 173.24(e)(4), the MSDSs for oxidizing materials were used to determine if mixing the chemical with Swheat™ could result in the hazardous conditions listed.

WIPP Appendix C-1 Compatibility Analysis

Finally the method outlined in Appendix C1 to the WIPP Part B Permit Application entitled "Chemical Compatibility Analysis of Waste Forms and Container Materials" was applied to the waste materials when mixed with Swheat™ for potential incompatibility. This method involves entering the materials into an unspecified database that checks for incompatibility based on the method outlined in the U.S. EPA document, *A Method for Determining the Compatibility of Hazardous Waste* (Hatayama et al.,1980). Although the database was unavailable for LANL use the materials were checked using the compatibility chart provided in the reference document.

Results

Identification of Oxidizing Chemicals

1. LA-MHD01, LA-CIN01.001[6] (debris waste stream, cemented cans waste stream)

Table 9 (Chemical Identification and Use Summary) in CCP-AK-006, rev. 13 lists chemicals found in the LA-MHD01 and LA-CIN01.001 waste streams. The following Group 6A chemicals (theoretically incompatible with Swheat™ per 40 CFR 264 Appendix V) were listed in CCP-AK-006 Table 9,

Table 1 Potentially Incompatible Chemicals - CCP-AK-006

Aluminum Nitrate	Mercuric Nitrate
Antimony Pentafluoride	Perchloric acid (*probably present as perchlorate; 'perchlorate' is listed in Appendix V*)
Bromine	Potassium Chromate
Cerium Nitrate	Potassium Dichromate
Cobalt Nitrate	Potassium Permanganate
Ferric Nitrate	Silver Nitrate
Hydrogen Peroxide	Sodium Chlorite
Magnesium Perchlorate	Sodium Chromate
Lanthanum Nitrate	Sodium Nitrate
Lead Nitrate	

[6] *NOTE: CCP-AK-006 also covers LA-MIN02-V.001 (mixed absorbent waste stream). However, LA-MIN02-V.001 containers were not a subject of this analysis. Some LA-MIN02-V.001 containers are believed to have been remediated using WasteLock 770™; however, WasteLock™ remediation was not included in this analysis.*

The text of CCP-AK-006 also describes RCRA chemicals in the LA-MHD01 and LA-CIN01.001 waste streams. No Group 6A chemicals were listed in the text in addition to those in Table 9 mentioned above.

2. LA-MIN03-NC.001(cemented sludges from TA-50 RLWTF)

Table 8 (Chemical Identification and Use Summary) describes RCRA chemicals in the LA-MIN03-NC.001 waste stream. The following chemicals or chemical categories (potentially incompatible with Swheat™) were confirmed to occur in CCP-AK-004 Table 8:

Table 2 Potentially Incompatible Chemicals - CCP-AK-004

Aluminum Nitrate	Lanthanum Nitrate
Bromine	Mercuric Nitrate
Calcium Nitrate	Perchloric Acid (*probably present as perchlorate)*
Cerium Nitrate	Silver Nitrate
Chromic Acid	Sodium Chromate
Chromium Trioxide	Sodium Hypochlorite
Cobalt Nitrate	Sodium Nitrate
Ferric Nitrate	Sodium Perchlorate
Hydrogen Peroxide	

Twenty (20) EPA Compatibility Group 6A chemicals were confirmed to occur in the LA-MHD01 and LA-CIN01.001 waste stream documentation. Eighteen (18) EPA Compatibility Group 6A chemicals were confirmed to occur in the LA-MIN03-NC.001 waste stream documentation.

40 CFR 264 Appendix V Compatibility Analysis

Because LANL is conservatively considering Swheat™ to be a Reactivity Group 101 "Miscellaneous Combustible or Flammable Material" (i.e., a 40 CFR 264 Appendix V material in Group 6B) for purposes of this analysis, all of the oxidizing materials listed in the Tables 1 and 2 above have a potential incompatibility with Swheat™ used as a sorbent.

DOT 49 CFR 177 Compatibility Analysis

All of the materials in the Tables 1 and 2 above would be considered Class 5.1 Oxidizers and as such would require segregation, under this section, from explosives, flammable liquids, poisonous liquids and corrosive liquids. Because Swheat™ is not a DOT hazardous material and has no assigned hazard class, a direct segregation determination cannot be made. This method would not be appropriate to make determinations for mixing substances within a drum or container.

DOT 49 CFR 173 Compatibility Analysis

Since the materials listed in Tables 1 and 2 are all classified as oxidizers, their MSDSs list a potential incompatibility with organic and/or flammable or combustible materials with the result being fire, heat generation, and toxic or asphyxiant gasses, all of which are prohibited for mixtures under 49 CFR 173.24(e)(4).

WIPP Appendix C-1 Compatibility Analysis

With the exception of hydrogen peroxide with a number 30, all of the chemicals listed in Tables 1 and 2 are strong oxidizers and are assigned a number 104 on the compatibility chart used for the Appendix C-1 analysis. Swheat™, as a combustible material, would be assigned number 101. In accordance with the compatibility chart, mixing Swheat™ (101) with strong oxidizers (104) could generate H (heat), F (fire), and/or G (gasses). Mixing Swheat™ with hydrogen peroxide (30) could generate H, F, and GT (toxic gasses). Therefore, Swheat™ as a sorbent would potentially be incompatible with any of the chemicals listed in the tables above.

Chemical Forms and Quantities

All chemicals listed in the Chemical Identification and Use Summary Tables in both CCP AK documents were assigned based on their reference as feed or process chemicals in procedures, material ordering documentation, and/or interviews with personnel performing operations. The chemicals would not be expected in the waste streams in pure form since they would have been involved in chemical reactions, decontamination activities, TRU recovery operations, and/or waste treatment processes prior to placement in their current waste containers. The quantities of chemical wastes will vary widely depending upon the type of waste and from one container to the next depending upon how much and what kind of cementation or sorption was applied and how many remediation activities were performed on each container.

The debris (LA-MDH01) waste stream, by volume, contains mostly materials and equipment that was contaminated through contact or use that has residual contamination such as gloves or other PPE, process containers or hoses, etc. Additionally the debris stream would include other combustible materials such as rags, plastic, and cellulose that would be in the same combustible class as Swheat™ but have not caused incompatibility issues during the 30+ years they have been in storage.

The cemented TRU waste stream (LA-CIN01) consists of waste materials that have been encased in Portland or gypsum cement. Again, these waste materials would have been subjected to reaction, decontamination, TRU recovery, and/or waste treatment or volume reduction prior to the cementation processes. The cementation process would reduce the oxidizing potential of the waste materials decreasing the likelihood of reactions with combustibles such as Swheat™.

The sludge waste stream (LA-MIN03) consists of dewatered waste materials from the treatment of wastewaters. These waste materials would also have been subjected to reaction, decontamination, TRU recovery, as well as the liquid waste treatment processes and dewatering prior to placement in the container with cement added to further solidify them. The addition of cement would reduce the oxidizing potential of the waste materials decreasing the likelihood of reactions with combustibles such as Swheat™.

Conclusions

Based on the conservative approach of using the CCP AK data for the waste streams, there are chemicals that could potentially be present in the waste that, in pure form and/or sufficient concentrations, could be incompatible with Swheat™ sorbent. Because determining precisely the incompatibility of wastes in specific containers with the sorbent added would be technically impossible at this point, LANL must report, conservatively at this time, that certain chemicals known to be incompatible with the sorbents used may be present in some of the waste streams analyzed. However, based on the fact that the incompatible chemicals would not be in their pure form and have been subject to process reactions and various dewatering, solidification, and other remediation processes which would reduce their oxidizing potential, and the lack of historical incompatibility reactions in containers of the subject wastes, the wastes should not be considered to be in danger of imminent reaction.

References

Hatayama et al. 1980. *A Method for Determining the Compatibility of Hazardous Waste*. EPA-600/2-80-076, USEPA, Washington D.C.

Appendix B.

Los Alamos NATIONAL LABORATORY EST.1943

memorandum

Actinide Analytical Chemistry

To/MS: Bruce Robinson, ADEP, MS K491
David Funk, ADEP, MS J910
From/MS: Rebecca Chamberlin, C-AAC
Phone/Fax: 7-1841/Fax 5-4737
Symbol: C-AAC-15-0024
Date: June 24, 2015

SUBJECT: Analytical Results for Drum S8111785 Free Liquid Sample

Sample Summary	
Drum #	**S8111785**
Sample collection date	05/13/2015
Analysis start date	05/28/2015
Sample description	Heterogeneous: Brown liquid with lighter-colored sludge that was difficult to keep in suspension. After settling 10 min, the solids were ~25% of volume of the sample.
Sample mass	55.2 g
Sample volume	41 mL
Density (calculated)	1.35 g/mL
pH (potentiometric)	10.9
Radionuclides (NDA, SNAP)	**Ci/mL** (+/- 30% modeling uncertainty)
Pu-239	Not detected
Am-241	1.91E-10
Cs-137	7.71E-12
Anions (Ion Chromatography)	**g/100 mL** (+/- 10%)
Nitrate (NO_3^-)	32.2
Nitrite (NO_2^-)	2.7
Chloride (Cl^-)	0.4
Fluoride (F^-)	Not detected
Sulfate (SO_4^-)	1.1
Oxalate ($C_2O_4^-$)	Trace
RCRA Metals (ICP-MS/AES)	**µg/mL** (+/- 20%)
Silver (Ag)	0.035
Arsenic (As)	<0.9
Barium (Ba)	1.7
Cadmium (Cd)	0.14
Chromium (Cr)	34
Mercury (Hg)	0.30
Lead (Pb)	590
Selenium (Se)	<1.4

Major Elements (ICP-MS/AES)	µg/mL, except as noted (+/- 20%)
Sodium (Na, %)	12.4 g/100 mL
Potassium (K, %)	1.2 g/100 mL
Aluminum (Al)	200
Calcium (Ca)	72
Iron (Fe)	50
Magnesium (Mg)	21
Silica (SiO_2, crystobalite)	Detected in undigested residue

Sample photos

As-received sample, after mixing

As-received sample, after standing 10 min

Analytical procedures and work instructions used:

1) ANC 212, Ion Chromatography
2) ANC 102, Inductively Coupled Plasma—Mass Spectrometry Using the VG Elemental Plasma Quad
3) ANC 221, Operating the Jobin-Yvon (JY) Inductively Coupled Plasma – Atomic Emission Spectrometer
4) WI-5, Analytical Sample Receipt, Subsampling, and Distribution within Analytical Chemistry
5) WI-30, Chemical Analysis, Characterization and Research
6) WI-42, Radiochemical Research and Development at CMR
7) WI-57, X-ray Diffraction
8) ANC 214, Spectrophotometric Determination of Silicon in Plutonium Materials (potentiometric pH method)

Cy: C-AAC File

Appendix C

UNCLASSIFIED

Los Alamos
NATIONAL LABORATORY

memorandum

M-DO: Explosive Science & Shock Physics
M-7: High Explosives Science & Technology

To/MS: David J. Funk, ADEP, djf@lanl.gov
From/MS: Geoffrey Brown, M-7, geoffb@lanl.gov
Phone/Fax: 7-6718 / 7-0500
Symbol: M7-15-6008
Date: July 10, 2015

GWB 7/10/15

SUBJECT: (U) Decomposition Onsets for Dried Swheat Mixtures in Analytical Lab 52143

M-7 recently carried out differential scanning calorimetry (DSC) testing of samples related to Waste Isolation Pilot Plant (WIPP) waste forms. The samples were

1. 1:1 ratio of saturated solution of WB8 mixed with Swheat kitty litter
2. 3:1 ratio of saturated solution of WB8 mixed with Swheat kitty litter
3. 1:1 ratio of 45 wt.% $NaNO_3$ mixed with Swheat kitty litter
4. 3:1 ratio of 45 wt. % $NaNO_3$ mixed with Swheat kitty litter

After the initial DSC runs, the samples were then dried for one week at room temperature in a hood. The DSC testing was then repeated.

Drying samples 1 and 2 changed them only minimally. Their exothermic activity before and after drying occurred at high temperatures with onsets of decomposition between 250 °C and 325 °C.

Samples 3 and 4 also changed minimally upon drying. Both showed a residual broad endotherm centered near 100 °C and a broad exotherm ranging from 150 °C to 400 °C. The 1:1 mixture showed some sharper features within the endotherm and exotherm regions but no feature on either dried sample exceeded 2 W/g heat flow. Given that the nominal baseline of the instrument can be +/- 1 W/g heat flow, the signals from the dried samples indicate minimal exothermic activity.

The onset of the exothermic activity of samples 3 and 4 cannot be explicitly determined because of the residual endotherm. Based on our experience with energetic materials however, it is very unlikely that both features became stronger after drying and are masking each other. As a result we expect that the exothermic onsets of samples 3 and 4 are in the 125 °C range.

GB:mq

Cy: M-DO DCM File, P942, wxdct@lanl.gov

Reviewed and determined to be UNCLASSIFIED.
This review does not constitute clearance for public release.
Derivative Classifier: Geoffrey Brown
Date: July 09, 2015

M-7 High Explosives Science and Technology

52143

Analytical Laboratory Report

Analytical Lab # 52143

Requester: BROWN **Group:** M-7

Sample ID: 45wt% NaNO3 Swheat (1:1), 45wt% NaNO3 Swheat (1:3), Saturated WB8 Swheat (1:1), Saturated WB8 Swheat (1:3) wet and dry

Tests Completed	Date Completed	Analyst
150611001		
DSC (wet)	06/11/15	SANDSTROM
DSC (dry)	06/24/15	SANDSTROM
150611002		
DSC (wet)	06/11/15	SANDSTROM
DSC (dry)	06/24/15	SANDSTROM
150611003		
DSC (wet)	06/11/15	SANDSTROM
DSC (dry)	06/24/15	SANDSTROM
150611004		
DSC (wet)	06/11/15	SANDSTROM
DSC (dry)	06/24/15	SANDSTROM

This document deemed Unclassified by
Mary M Sandstrom 7/9/15
(DC)

Validated: Geoffrey W. Brown 7/9/15

Approved: Mary M. Sandstrom 7/9/15

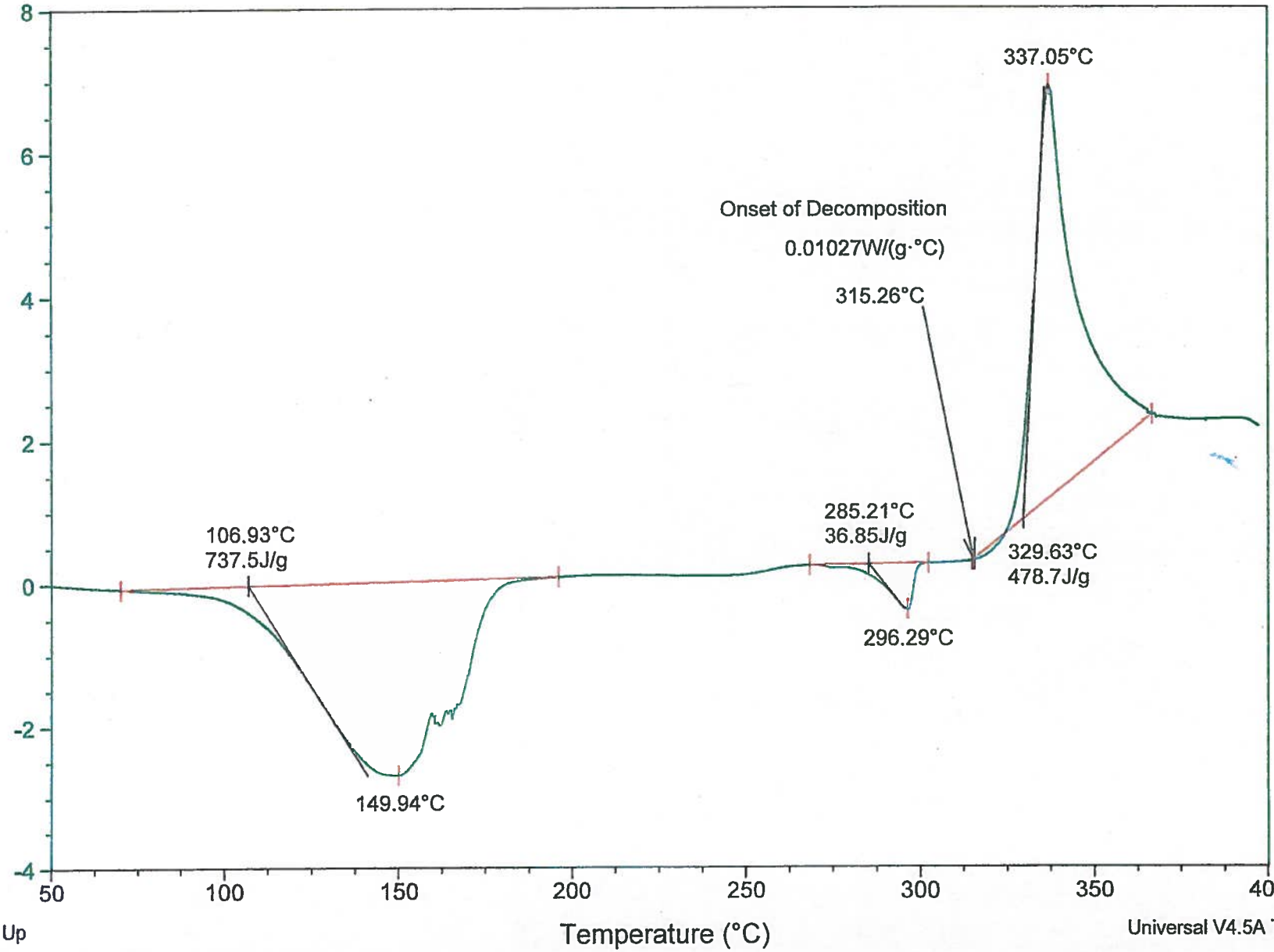
Date Submitted: 06/11/2015
Date Competed: 06/24/2015

Los Alamos

ıple: 45wt% NaNO3 Swheat (1-1)
: 2.7720 mg
ıod: 10C Ramp

DSC

File: C:...\52143\45wt% NaNO3 Swheat (1-1).001
Operator: MMS
Run Date: 11-Jun-2015 21:55
Instrument: DSC Q2000 V24.11 Build 124



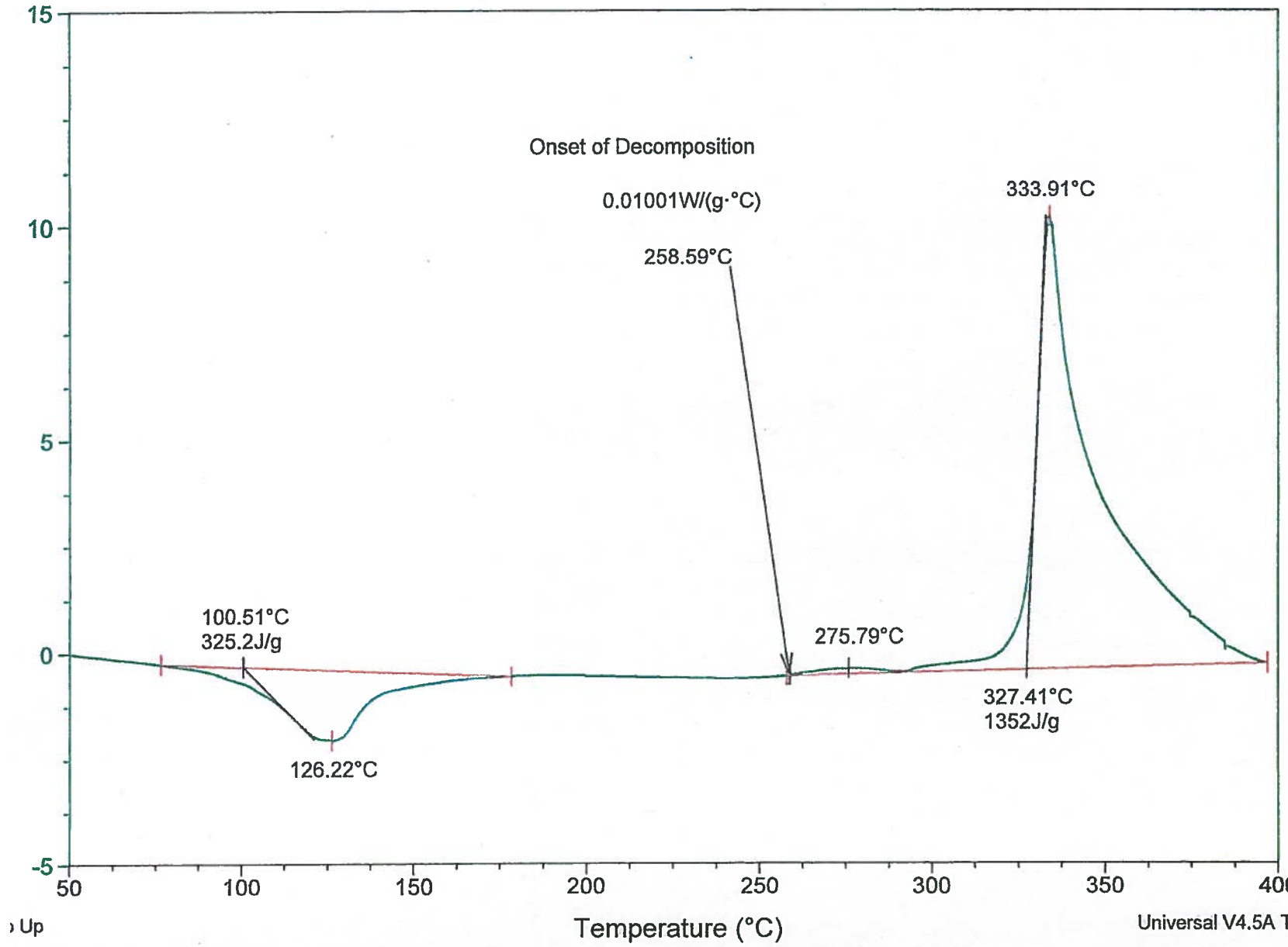
› Up

Universal V4.5A TA Instruments

ıple: 45wt% NaNO3 Swheat (3-1)
: 1.0430 mg
hod: 10C Ramp

DSC

File: C:...\52143\45wt% NaNO3 Swheat (3-1).001
Operator: MMS
Run Date: 11-Jun-2015 22:45
Instrument: DSC Q2000 V24.11 Build 124



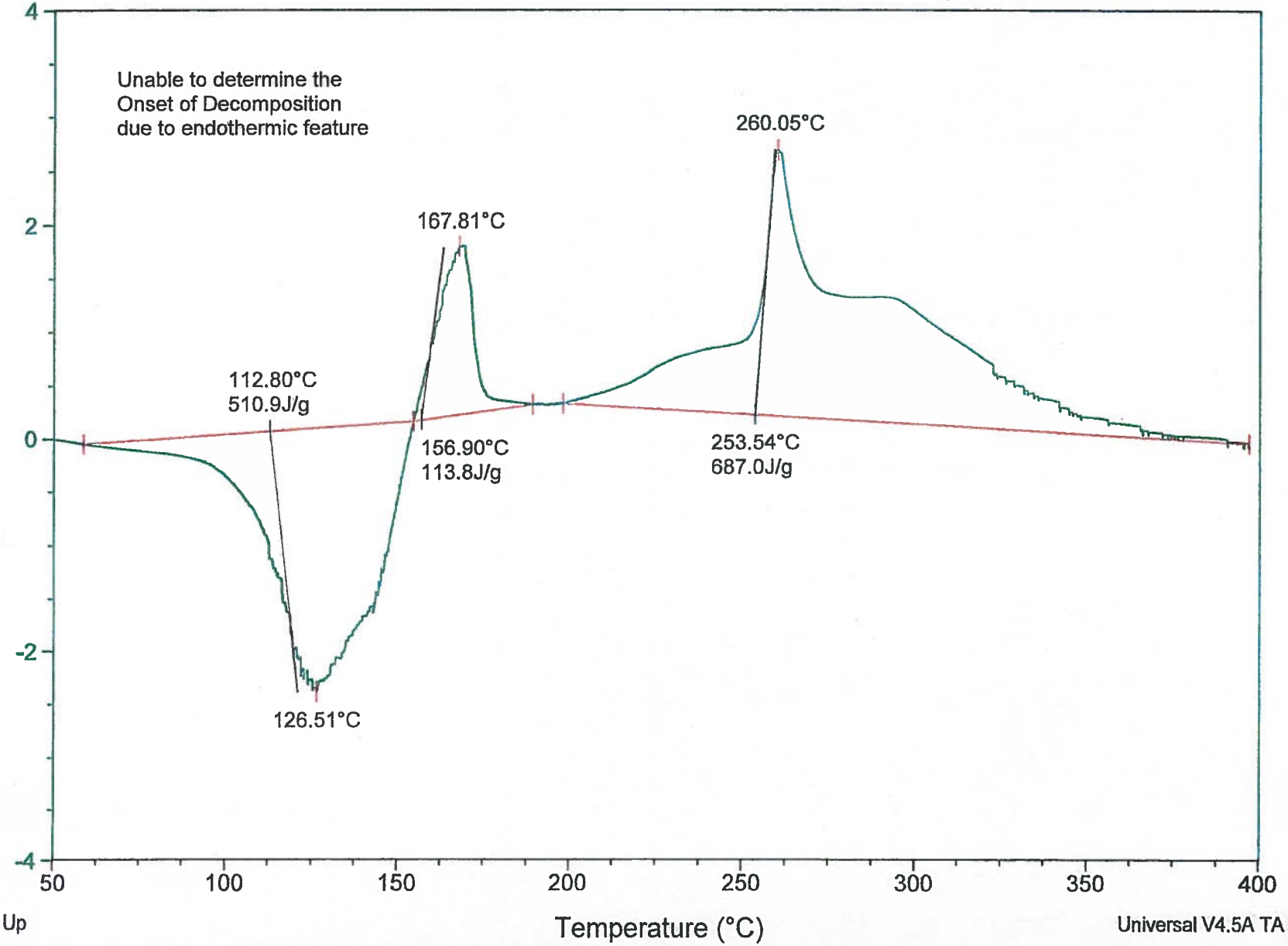
› Up

Universal V4.5A TA Instruments

mple: Saturated NaNO3 Swheat (1-1)
ze: 1.9080 mg
ethod: 10C Ramp

DSC

File: C:...\Saturated NaNO3 Swheat (1-1).001
Operator: MMS
Run Date: 11-Jun-2015 23:35
Instrument: DSC Q2000 V24.11 Build 124



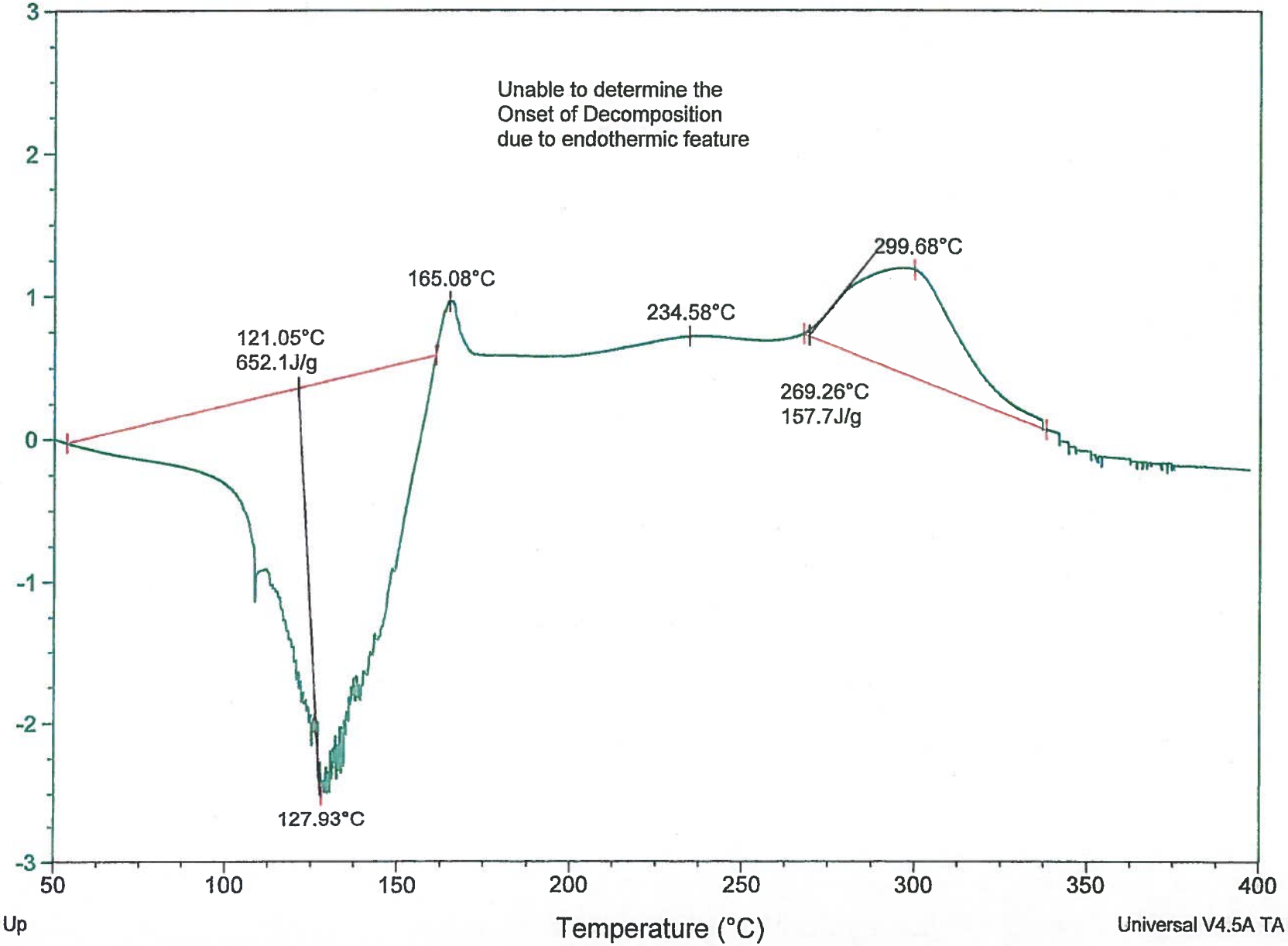
to Up

Universal V4.5A TA Instruments

ıple: Saturated NaNO3 Swheat (3-1)
: 1.5030 mg
hod: 10C Ramp

DSC

File: C:...\Saturated NaNO3 Swheat (3-1).001
Operator: MMS
Run Date: 12-Jun-2015 00:25
Instrument: DSC Q2000 V24.11 Build 124

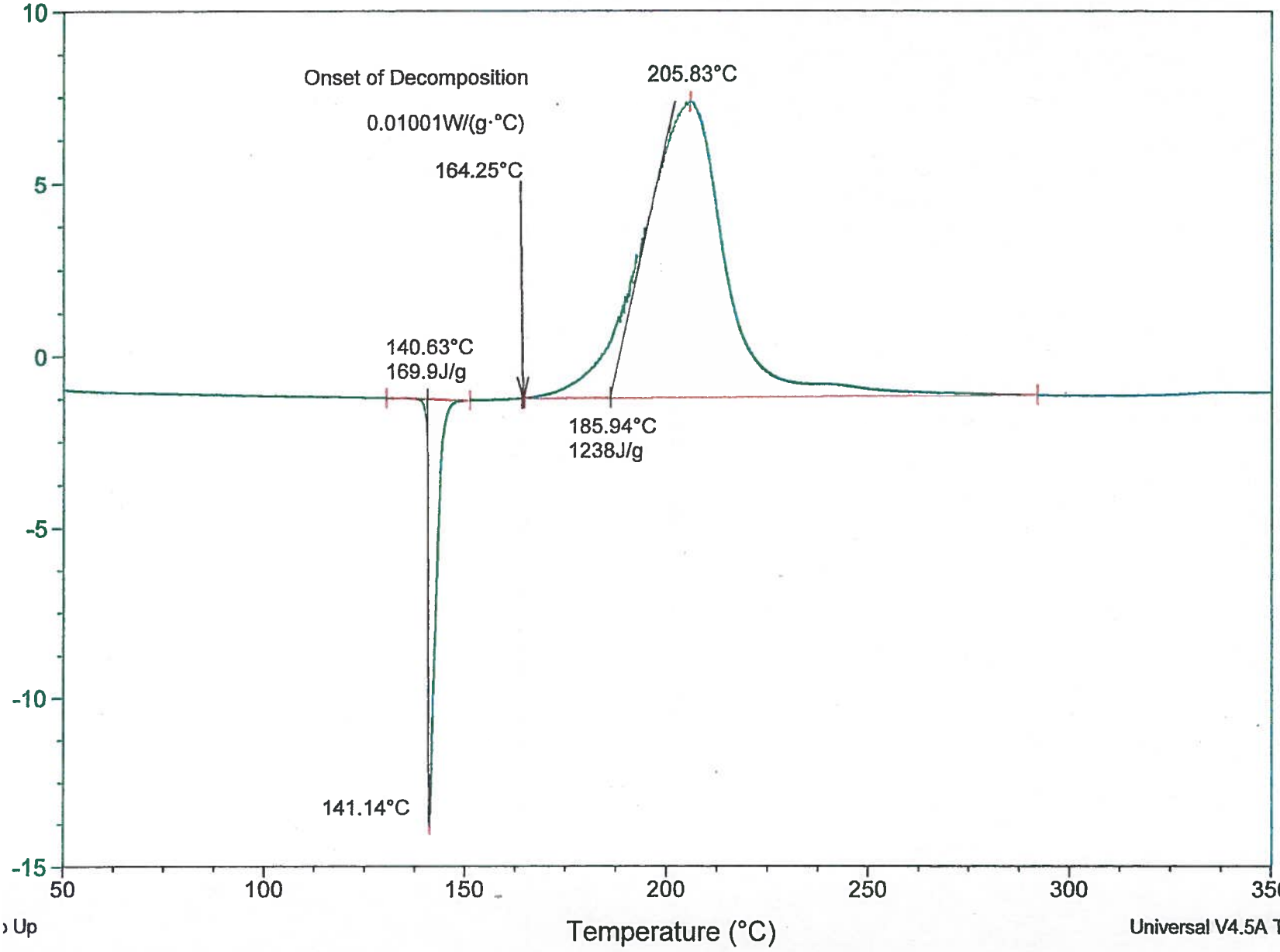


Universal V4.5A TA Instruments

nple: PETN 9401
e: 0.8960 mg
hod: 10C Ramp

DSC

File: C:...\061115 Instrument Control.001
Operator: MMS
Run Date: 11-Jun-2015 16:42
Instrument: DSC Q2000 V24.11 Build 124



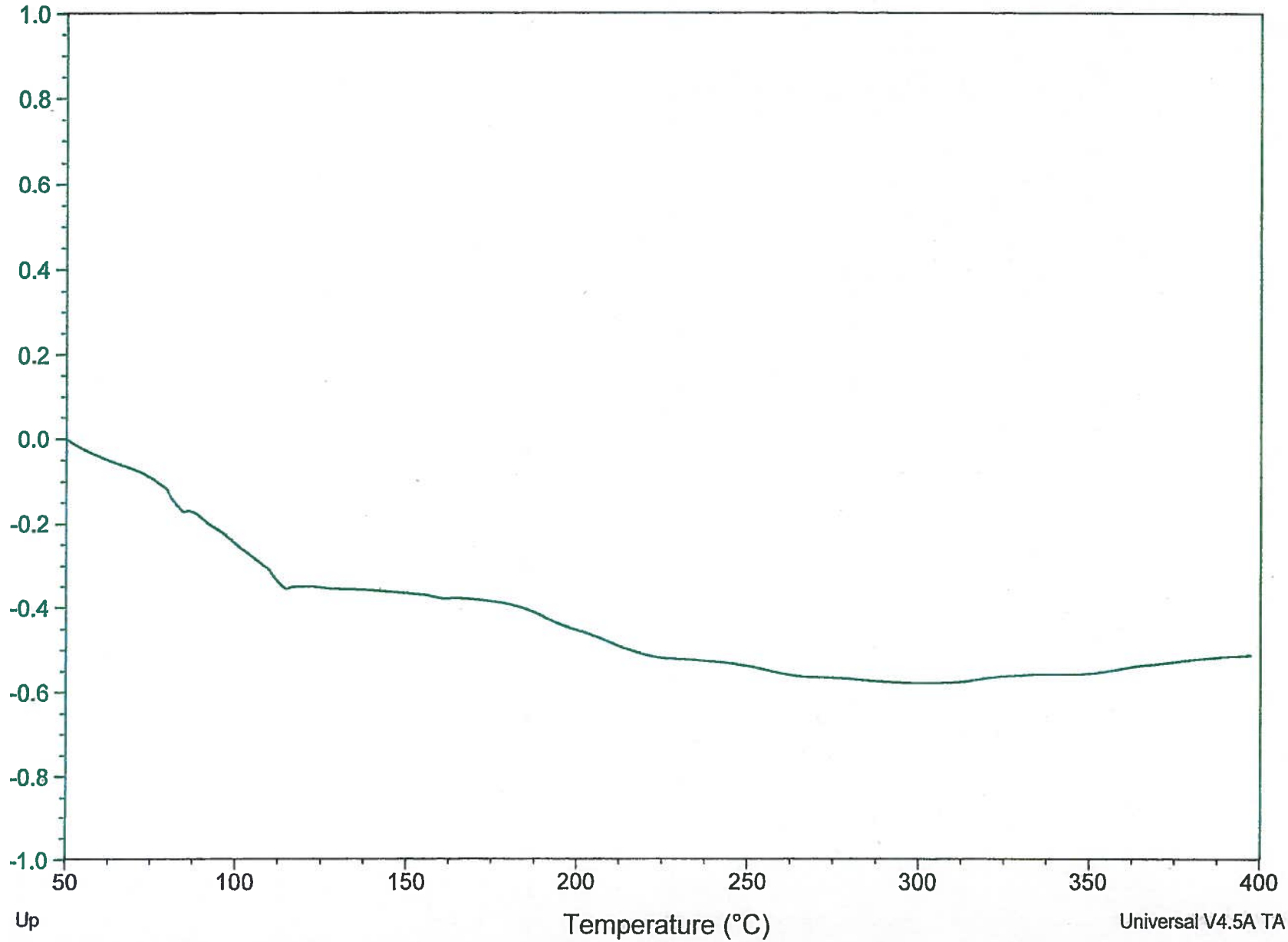
› Up

Universal V4.5A TA Instruments

ıple: Pan Blank
: 0.0000 mg
hod: 10C Ramp

DSC

File: C:...\2015\061115 Pan Blank.002
Operator: MMS
Run Date: 11-Jun-2015 15:52
Instrument: DSC Q2000 V24.11 Build 124

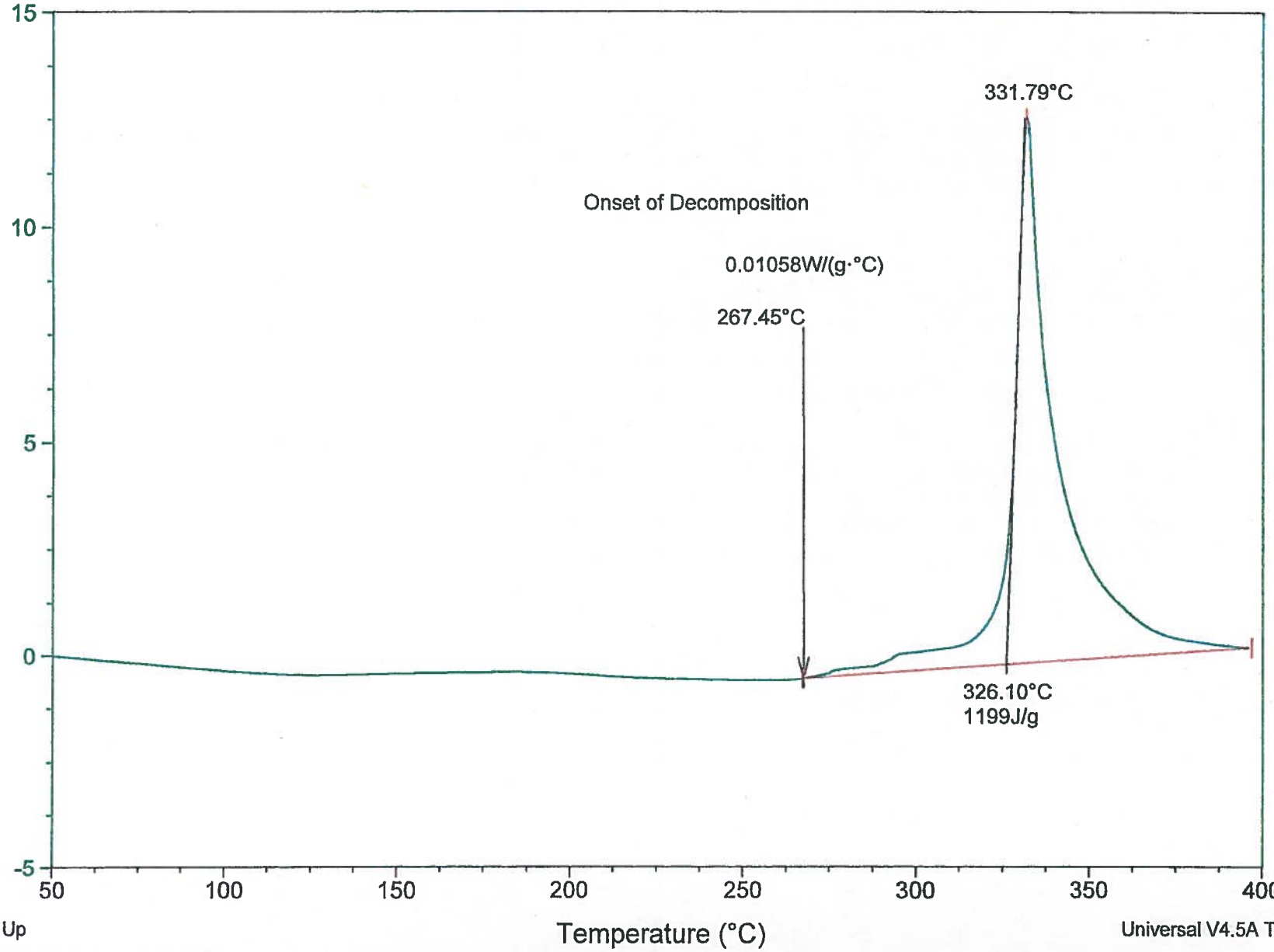


nple: 45wt% NaNO3 Swheat (3-1)- Dry
e: 1.2460 mg
:hod: 10C Ramp

DSC

File: C:...\45wt% NaNO3 Swheat (3-1)- Dry.001

Run Date: 24-Jun-2015 04:32
Instrument: DSC Q2000 V24.11 Build 124



› Up

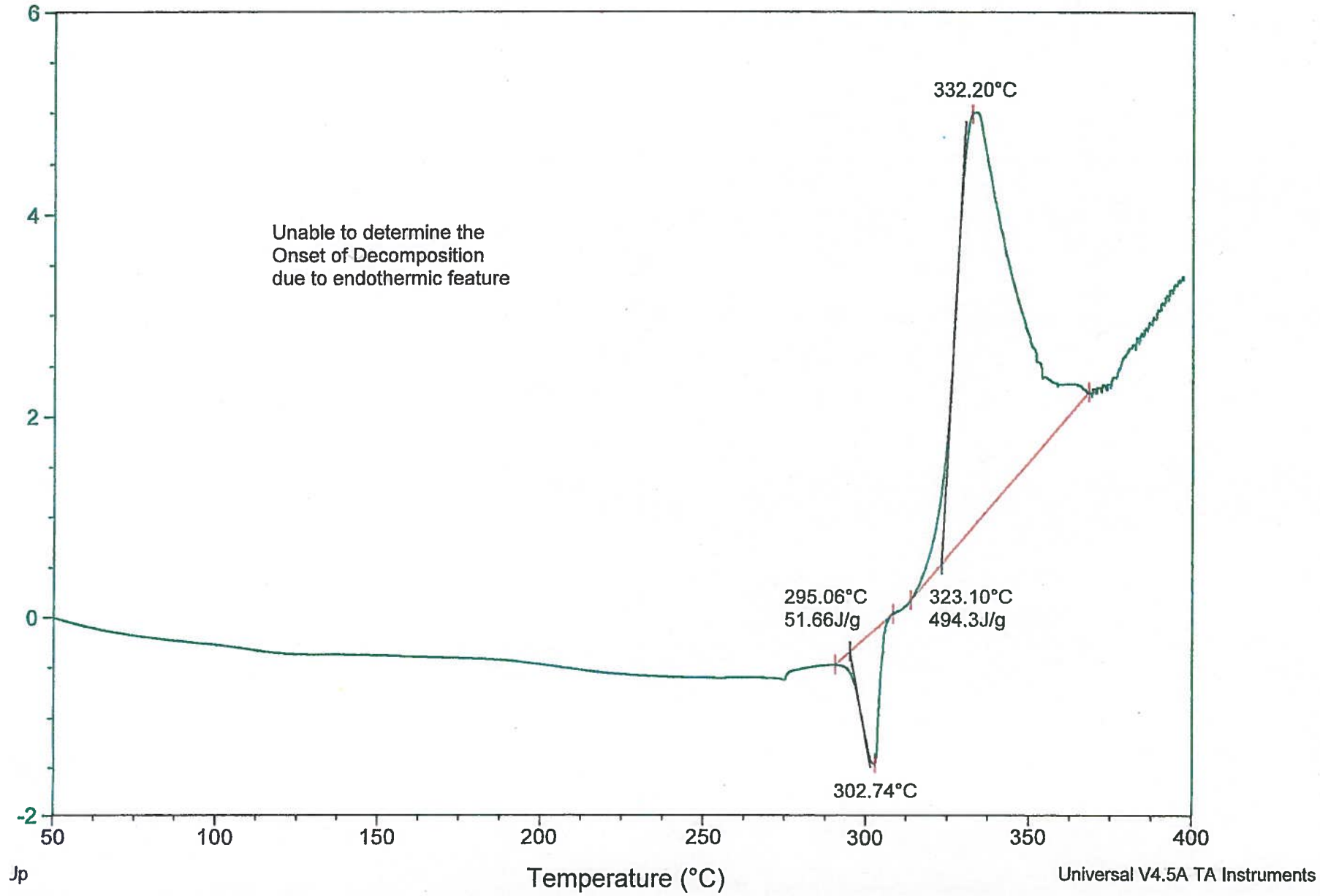
Universal V4.5A TA Instruments

ple: 45wt% NaNO3 Swheat (1-1)- Dry
0.9840 mg
od: 10C Ramp

DSC

File: C:...\45wt% NaNO3 Swheat (1-1)- Dry.001

Run Date: 24-Jun-2015 05:22
Instrument: DSC Q2000 V24.11 Build 124

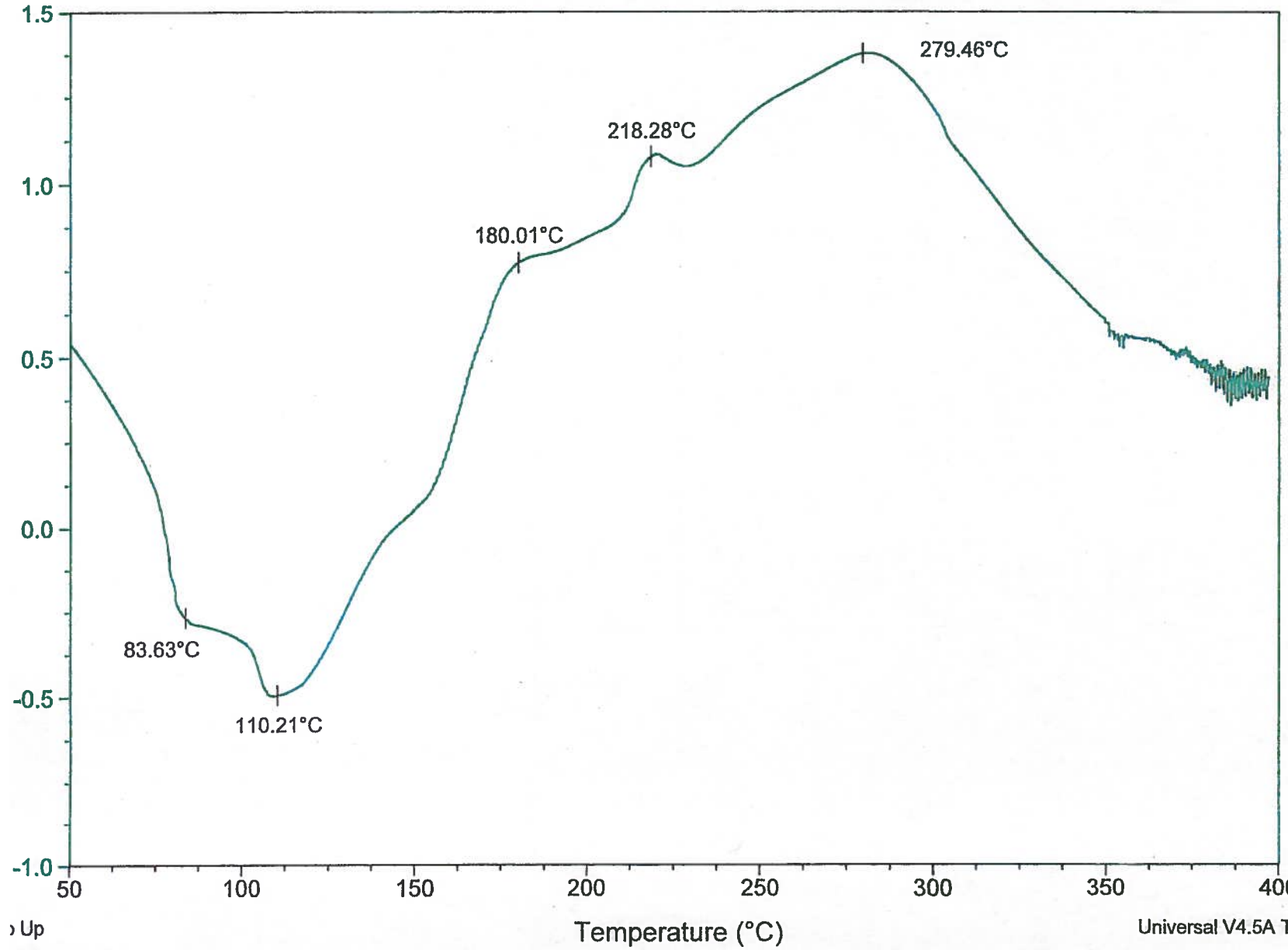


ıple: Saturated NaNO3 Swheat (3-1) Dry
ɛ: 0.8650 mg
hod: 10C Ramp

DSC

File: ...\Saturated NaNO3 Swheat (3-1) Dry.001

Run Date: 24-Jun-2015 07:02
Instrument: DSC Q2000 V24.11 Build 124



› Up

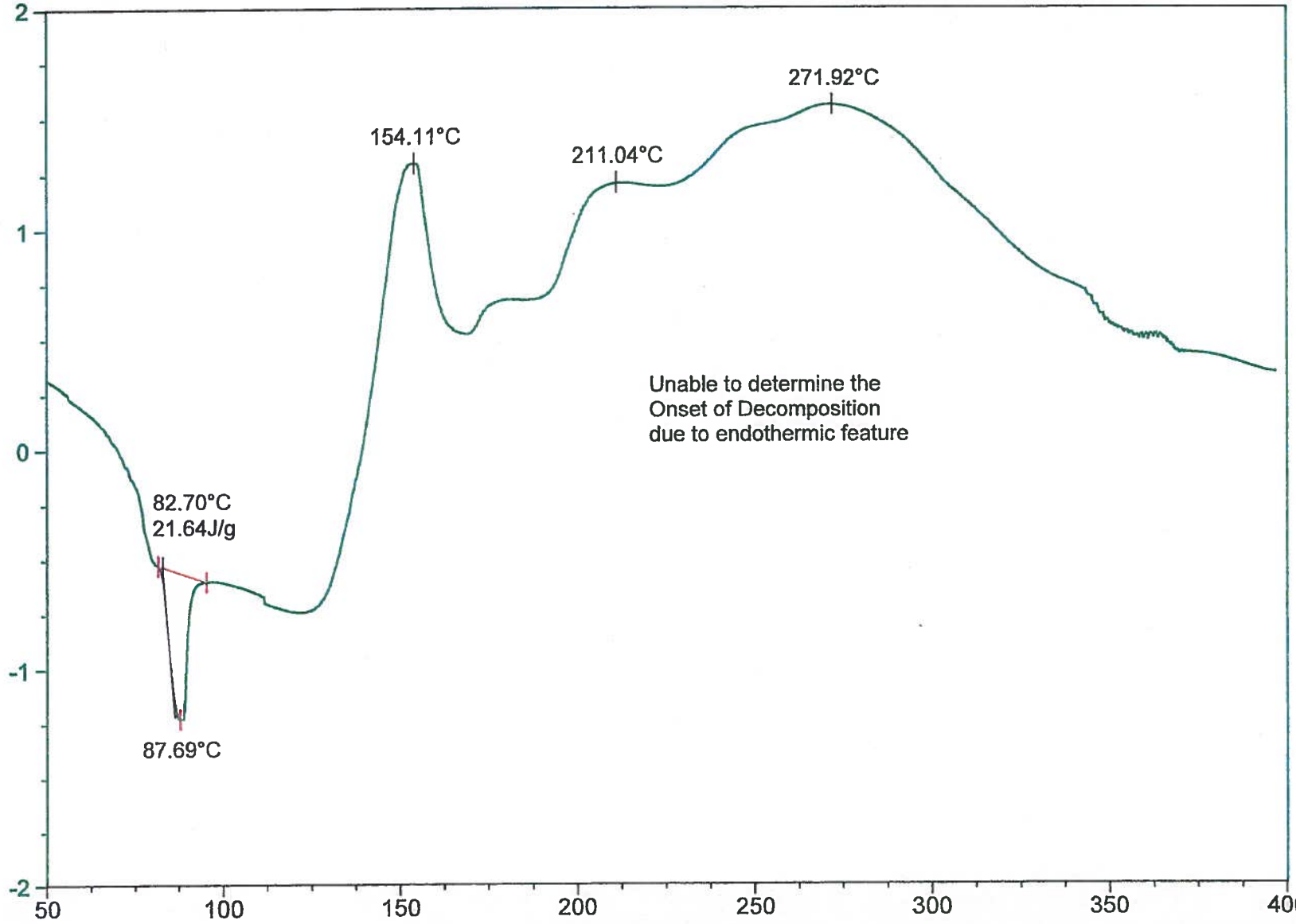
Universal V4.5A TA Instruments

ıple: Saturated NaNO3 Swheat (1-1) Dry
: 1.2040 mg
ıod: 10C Ramp

DSC

File: ...\Saturated NaNO3 Swheat (1-1) Dry.001

Run Date: 24-Jun-2015 06:12
Instrument: DSC Q2000 V24.11 Build 124



ı Up

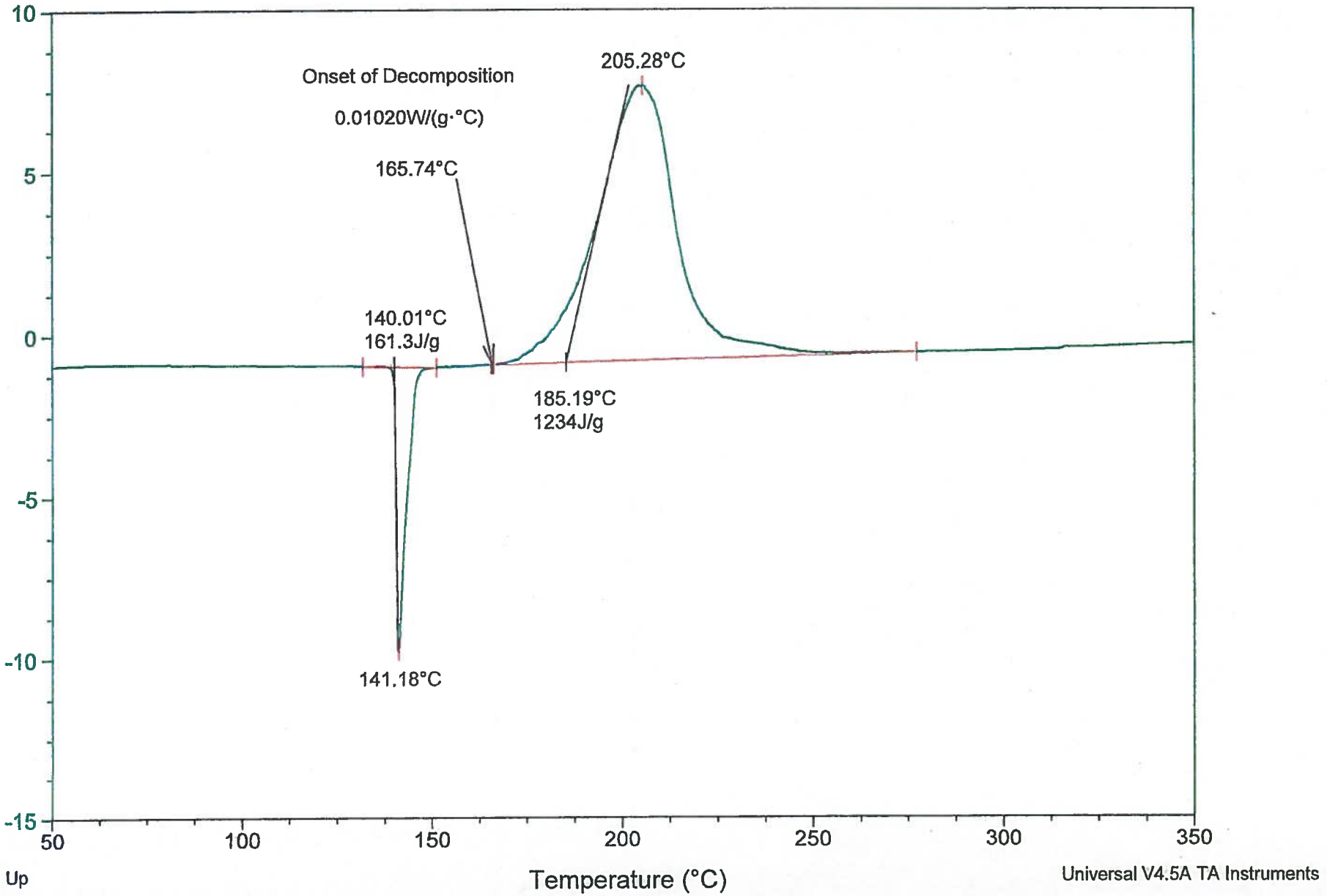
Universal V4.5A TA Instruments

ple: PETN 9401B
: 0.9320 mg
ıod: 10C Ramp

DSC

File: C:...\062315 Instrument Control.001

Run Date: 23-Jun-2015 18:10
Instrument: DSC Q2000 V24.11 Build 124

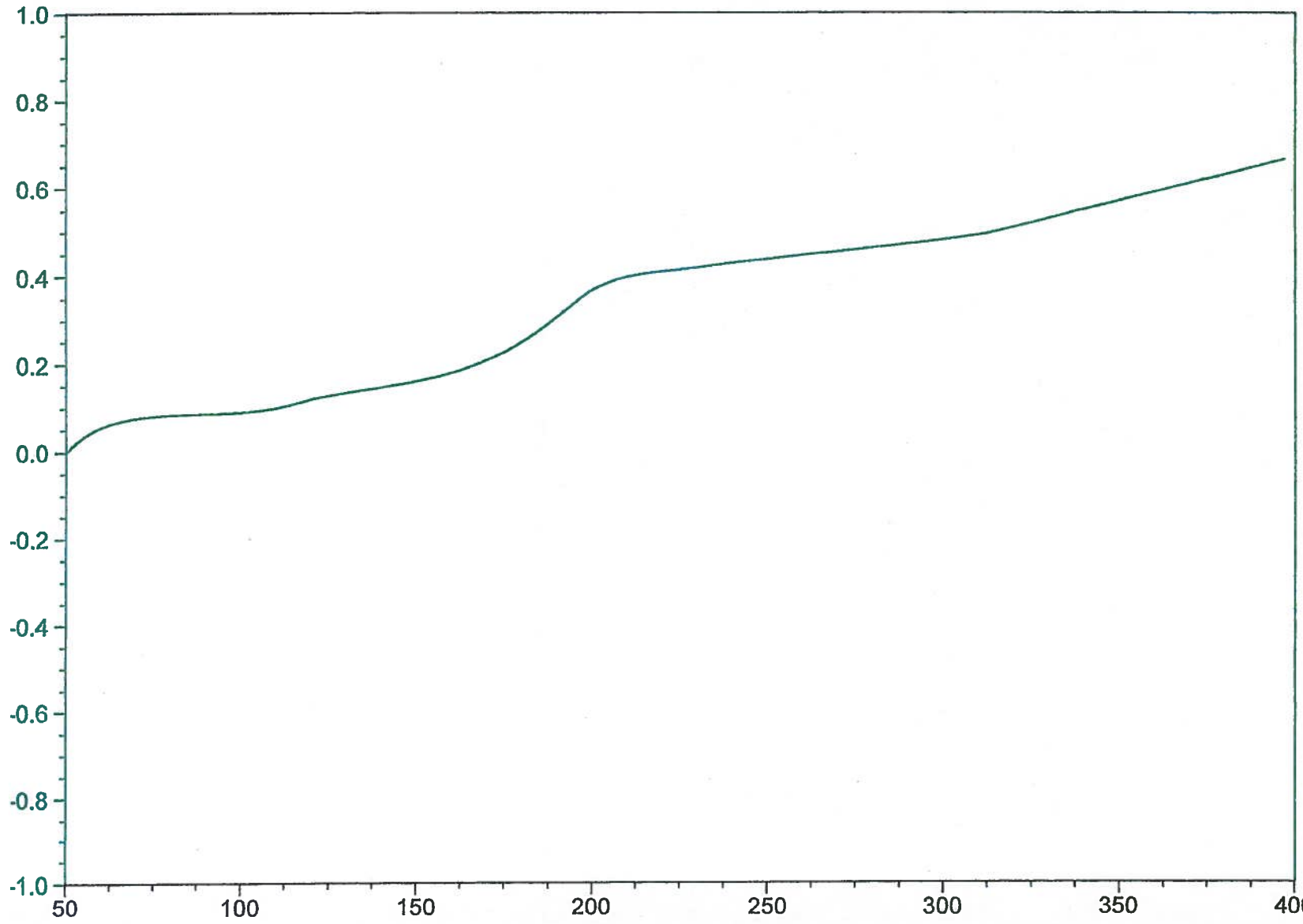


ple: Pan Blank
: 0.0000 mg
iod: 10C Ramp

DSC

File: C:...\2015\062315 Pan Blank.001

Run Date: 23-Jun-2015 17:20
Instrument: DSC Q2000 V24.11 Build 124



Universal V4.5A TA Instruments

Attachment B

Excerpts for Procedure 0.2,
"Recommendations on the Transport of Dangerous Goods, Manual of Test and Criteria," Chapter 34, Procedure 0.2 (United Nations, 2009).

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34.4.2 ***Test O.2: Test for oxidizing liquids***

34.4.2.1 *Introduction*

This test method is designed to measure the potential for a liquid substance to increase the burning rate or burning intensity of a combustible substance when the two are thoroughly mixed or to form a mixture which spontaneously ignites. The liquid is mixed in a 1 to 1 ratio, by mass, with fibrous cellulose, the mixture heated in a pressure vessel and the rate of pressure rise determined[2].

34.4.2.2 *Apparatus and materials*

34.4.2.2.1 A pressure vessel, as used in the time/pressure test (see Part I, test 1 (c) (i)), is required. It consists of a cylindrical steel pressure vessel 89 mm in length and 60 mm in external diameter (see Figure 34.4.2.1). Two flats are machined on opposite sides (reducing the cross-section of the vessel to 50 mm) to facilitate holding whilst fitting up the firing plug and vent plug. The vessel, which has a bore of 20 mm diameter is internally rebated at either end to a depth of 19 mm and threaded to accept 1" British Standard Pipe (BSP). A pressure take-off, in the form of a side arm, is screwed into the curved face of the pressure vessel 35 mm from one end and at 90° to the machined flats. The socket for this is bored to a depth of 12 mm and threaded to accept the 1/2 " BSP thread on the end of the side-arm. If necessary, an inert seal is fitted to ensure a gastight seal. The side-arm extends 55 mm beyond the pressure vessel body and has a bore of 6 mm. The end of the side-arm is rebated and threaded to accept a diaphragm type pressure transducer. Any pressure-measuring device may be used provided that it is not affected by the hot gases or the decomposition products and is capable of responding to rates of pressure rise of 690-2 070 kPa in not more than 5 ms.

34.4.2.2.2 The end of the pressure vessel furthest from the side-arm is closed with a firing plug which is fitted with two electrodes, one insulated from, and the other earthed to, the plug body. The other end of the pressure vessel is closed by a bursting disc (bursting pressure approximately 2 200 kPa (320 psi)) held in place with a retaining plug which has a 20 mm bore. If necessary, an inert seal is used with the firing plug to ensure a gas-tight fit. A support stand (Figure 34.4.2.2) holds the assembly in the correct attitude during use. This comprises a mild steel base plate measuring 235 mm × 184 mm × 6 mm and a 185 mm length of square hollow section (S.-H.S.) 70 × 70 × 4 mm.

34.4.2.2.3 A section is cut from each of two opposite sides at one end of the length of S.H.S. so that a structure having two flat sided legs surmounted by 86 mm length of intact box section results. The ends of these flat sides are cut to an angle of 60° to the horizontal and welded to the base plate. A slot measuring 22 mm wide × 46 mm deep is machined in one side of the upper end of the base section such that when the pressure vessel assembly is lowered, firing plug end first, into the box section support, the side-arm is accommodated in the slot. A packing piece of steel 30 mm wide and 6 mm thick is welded to the lower internal face of the box section to act as a spacer. Two 7 mm thumb screws, tapped into the opposite face, serve to hold the pressure vessel firmly in place. Two 12 mm wide strips of 6 mm thick steel, welded to the side pieces abutting the base of the box section, support the pressure vessel from beneath.

---

[2] *In some cases, substances may generate a pressure rise (too high or too low), caused by chemical reactions not characterising the oxidizing properties of the substance. In these cases, it may be necessary to repeat the test with an inert substance, e.g. diatomite (kieselguhr), in place of the cellulose in order to clarify the nature of the reaction.*

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34.4.2.2.4 The ignition system consists of a 25 cm long Ni/Cr wire with a diameter 0.6 mm and a resistance of 3.85 ohm/m. The wire is wound, using a 5 mm diameter rod, in the shape of a coil and is attached to the firing plug electrodes. The coil should have one of the configurations shown in Figure 34.4.2.3. The distance between the bottom of the vessel and the underside of the ignition coil should be 20 mm. If the electrodes are not adjustable, the ends of the ignition wire between the coil and the bottom of the vessel should be insulated by a ceramic sheath. The wire is heated by a constant current power supply able to deliver at least 10 A.

34.4.2.25 Dried, fibrous cellulose[3] with a fibre length between 50 and 250 µm and a mean diameter of 25 µm, is used as the combustible material. It is dried in a layer not more than 25 mm thick at 105 °C for 4 hours and kept in a desiccator, with desiccant, until cool and required for use. The water content of the dried cellulose should be less than 0.5% by dry mass. If necessary, the drying time should be prolonged to achieve this.

34.4.2.2.6 50% perchloric acid, 40% aqueous sodium chlorate solution and 65% aqueous nitric acid are required as reference substances.

34.4.2.2.7 The concentration of the substance tested should be specified in the report. If saturated solutions are tested, they should be prepared at 20 °C.

34.4.2.3 *Procedure*

34.4.2.3.1 The apparatus, assembled complete with pressure transducer and heating system but without the bursting disc in position, is supported firing plug end down. 2.5 g of the liquid to be tested is mixed with 2.5 g of dried cellulose in a glass beaker using a glass stirring rod. ***For safety, the mixing should be performed with a safety shield between the operator and mixture.*** (If the mixture ignites during mixing or filling, no further testing is necessary.) The mixture is added, in small portions with tapping, to the pressure vessel making sure that the mixture is packed around the ignition coil and is in good contact with it. It is important that the coil is not distorted during the packing process. The bursting disc is placed in position and the retaining plug is screwed in tightly. The charged vessel is transferred to the firing support stand, bursting disc uppermost, which should be located in a suitable, armoured fume cupboard or firing cell. The power supply is connected to the external terminals of the firing plug and 10 A applied. The time between the start of mixing and switching the power on should be about 10 minutes.

34.4.2.3.2 The signal produced by the pressure transducer is recorded on a suitable system which allows both evaluation and the generation of a permanent record of the time pressure profile obtained (e.g. a transient recorder coupled to a chart recorder). The mixture is heated until the bursting disc ruptures or until at least 60 s has elapsed. ***If the bursting disc does not rupture, the mixture should be allowed to cool before carefully dismantling the apparatus and precautions taken to allow for any pressurisation.*** Five trials are performed with the mixture and each of the reference substances. The time taken for the pressure rise from 690 kPa to 2 070 kPa above atmospheric is noted. The mean time interval should be used for classification.

34.4.2.4 *Test criteria and method of assessing results*

34.4.2.4.1 The test results are assessed on the basis of:

(a) Whether the mixture of substance and cellulose spontaneously ignites; and

(b) The comparison of the mean time taken for the pressure to rise from 690 kPa to 2 070 kPa gauge with those of the reference substances.

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[3] *Source reference available from the national contact for test details in Sweden (see Appendix 4).*

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34.4.2.4.2 The test criteria for determining the oxidizing properties of the substance are:

Packing group I:	-	Any substance which, in the 1:1 mixture, by mass, of substance and cellulose tested, spontaneously ignites; or
	-	The mean pressure rise time of a 1:1 mixture, by mass, of substance and cellulose is less than that of a 1:1 mixture, by mass, of 50% perchloric acid and cellulose.
Packing group II:	-	Any substance which, in the 1:1 mixture, by mass, of substance and cellulose tested, exhibits a mean pressure rise time less than or equal to the mean pressure rise time of a 1:1 mixture, by mass, of 40% aqueous sodium chlorate solution and cellulose; and
	-	The criteria for packing group I are not met.
Packing group III:	-	Any substance which, in the 1:1 mixture, by mass, of substance and cellulose tested, exhibits a mean pressure rise time less than or equal to the mean pressure rise time of a 1:1 mixture, by mass, of 65% aqueous nitric acid and cellulose; and
	-	The criteria for packing groups I and II are not met.
Not Division 5.1:	-	Any substance which, in the 1:1 mixture, by mass, of substance and cellulose tested, exhibits a pressure rise of less than 2 070 kPa gauge; or
	-	Exhibits a mean pressure rise time greater than the mean pressure rise time of a 1:1 mixture, by mass, of 65% aqueous nitric acid and cellulose.

For substances having additional risks, e.g. toxicity or corrosivity, the requirements of section 2.0.3 of the Model Regulations should be met.

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34.4.2.5 *Examples of results*

Substance	Mean pressure rise time for a 1:1 mixture with cellulose (ms)	Results
Ammonium dichromate, saturated aqueous solution	20 800	Not Div. 5.1
Calcium nitrate, saturated aqueous solution	6 700	Not Div. 5.1
Ferric nitrate, saturated aqueous solution	4 133	PG III
Lithium perchlorate, saturated aqueous solution	1 686	PG II
Magnesium perchlorate, saturated aqueous solution	777	PG II
Nickel nitrate, saturated aqueous solution	6 250	Not Div. 5.1
Nitric acid, 65%	4 767 [a]	PG III [b]
Perchloric acid, 50%	121 [a]	PG II
Perchloric acid, 55%	59	PG I
Potassium nitrate, 30% aqueous solution	26 690	Not Div. 5.1
Silver nitrate, saturated aqueous solution	[c]	Not Div. 5.1
Sodium chlorate, 40% aqueous solution	2 555 [a]	PG II
Sodium nitrate, 45% aqueous solution	4 133	PG III
Inert substance		
Water:cellulose	[c]	

[a] *Mean value from inter-laboratory comparative trials.*

[b] *Packing group III by the test but Class 8 by the Precedence of Hazards table.*

[c] *Maximum pressure of 2 070 kPa not reached.*

Attachment C

Analytical Sampling Results

Summary of Test Results of Oxidizing Liquid

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SCE P-1356
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August 20, 2015

Table 1

Summary of Test Results for Oxidizing Liquid

Material Tested: Chamberlain Surrogate #1 (8/11/15)
Chamberlain Surrogate #2 (8/11/15)

Material Tested	Trial No.	Did Sample/Cellulose Mixture Ignite Spontaneously?	Maximum Pressure (kPa)	Time for Pressure to Rise from 690 kPa to 2070 kPa (sec)
Chamberlain Surrogate #1 (8/11/15) and Dried Cellulose in 1:1 (by mass) mixture	1	No	1401	--
	2	No	1877	--
	3	No	2249	33.1
	4	No	2394	18.7
	5	No	1898	--
				Mean: 25.9
Chamberlain Surrogate #2 (8/11/15) and Dried Cellulose in 1:1 (by mass) mixture	1	No	1656	--
	2	No	1359	--
	3	No	1001	--
	4	No	1649	--
	5	No	1842	--
				Mean: NA
50% perchloric acid and cellulose in 1:1 Mixture (by mass) (Reference I)	5 Trials	No	>2400	0.29 Mean time
40% aqueous sodium chlorate solution and cellulose in 1:1 Mixture (by mass) (Reference II)	5 Trials	No	>2400	2.99 Mean time
65% Aqueous Nitric Acid and Dried Cellulose in 1:1 (by mass) mixture (Reference III)	5 Trials	No	>2400	4.7 Mean time

N/A = Not applicable

Table 2
Overall Test Result/Classification

Material Tested	Liquid Oxidizer Classification	Results/Remarks
Chamberlain Surrogate #1 (8/11/15)	**Not Division 5.1**	Since the mean pressure rise time (from 690-2070 kPa) for the 1:1 mixture, by mass, of a liquid sample and cellulose tested, was greater than the mean pressure rise time of a 1:1 mixture, by mass, 65% aqueous nitric acid and cellulose.
Chamberlain Surrogate #2 (8/11/15)	**Not Division 5.1**	Since the 1:1 mixture, by mass, of a liquid sample and cellulose tested, did not exhibited a pressure rise of 2070 kPa gauge.

DOT/UN Test O.2 (Division 5.1) ;Test Criteria and Method for Assessing Results

The classification test results are assessed on the basis of:

- Whether the mixture of the substance and cellulose spontaneously ignites; and
- The comparison of the mean time taken for the pressure to rise from 690 kPa gauge to 2070 kPa gauge with those of the reference substances.

Assignment of Packing Groups

Liquid oxidizing substances are assigned to a packing group in accordance with the following criteria:

Packing Group I: any substance which, in the 1:1 mixture, by mass, of a substance and cellulose tested, spontaneously ignites; or the mean pressure rise time of a 1:1 mixture, by mass, of substance and cellulose is less than that of a 1:1 mixture, by mass, of 50% perchloric acid and cellulose.

Packing Group II: any substance which, in the 1:1 mixture, by mass, of a substance and cellulose tested, exhibits a mean pressure rise time less than or equal to the mean pressure rise time of a 1:1 mixture, by mass, of 40% aqueous sodium chlorate solution and cellulose; and the criteria for Packing Group I are not met.

Packing Group III: any substance which, in the 1:1 mixture, by mass, of a substance and cellulose tested, exhibits a mean pressure rise time less than or equal to the mean pressure rise time of a 1:1 mixture, by mass, 65% aqueous nitric acid and cellulose and cellulose test criteria for Packing Groups I and II are not met.

Not Division 5.1: any substance which, in the 1:1 mixture, by mass, of a substance and cellulose tested, exhibits a pressure rise of less than 2070 kPa gauge; or exhibits a mean pressure rise time greater than the mean pressure rise time of a 1:1 mixture, by mass, of 65% aqueous nitric acid and cellulose.

For substances having additional risks, *e.g.* toxicity or corrosivity, the primary and secondary risks should also be determined.