

Sampling, Shipping and Analysis of Sludges for Drying Applications

Design of indirect heating/drying equipment requires a reasonably accurate determination of composition and thermal properties of the treated feed. Sludges from industrial operations such as tank cleaning and well-drilling are similar in that they contain a mixture of aqueous and organic fluids along with organic or inorganic solids. These phases will however behave differently in processing equipment and should be characterized to the degree possible to insure effective design and operation.

The following analytical test will determine % Water, % Oil, % Diesel in the sludge. What temperature the different constituents evaporate at and how much energy is required to evaporate those constituents.

Analytical Methods Used:

Step 1: TGA, ASTM e1131 or equivalent

Step 2: DSC or TGA-DSC

Step 3: A proximate analysis (ASTM D6349)

As sludges contain a wide array of constituents with different thermal properties, speciation and determination of these properties is determined using Thermogravimetric Analysis (TGA, ASTM E1131 or equivalent) with simultaneous Differential Scanning Calorimetry (DSC, no specific ASTM method) or TGA-DSC^{1,2}. TGA provides the relationship of mass loss versus temperature and DSC provides a measure of both the incremental and cumulative heat required. The combination of these analytical procedures provides sufficient information to determine the relationship of residual mass versus temperature as well as incremental latent heat of vaporization.

In addition to the TGA and DSC procedures, analyses of sludges should include a proximate analysis (ASTM D6349) indicating moisture and ash content, volatiles and fixed carbon.

Testing Procedure:

As this material typically contains flammable volatiles, it is important that the TGA, DSC testing be performed under an inert gas padded atmosphere such as nitrogen or carbon dioxide. The test samples should be heated from 70 °F to 900 °F. To match our equipment's heating rate as closely as possible, it is recommended that the heating ramp rate be performed accordingly:

70 to 212 @ no more than 10 °F min

Hold for 5 mins

212 to 480 @ no more than 5 °F min

Hold for 5 mins

480 to 900 @ no more than 15 °F min

Therma-Flite does not recommend any particular analytical laboratory; however those offering TGA-DSC analyses include, but are not limited to:

Hazen Research, Golden, CO; (303) 279-4501

Tyler Salibury Project Engineer

4601 Indiana St

Golden, CO 80403

(303)-279-4501 x297

Sampling and Shipping:

To insure representative results, no less than 3 individual samples collected over an appropriate period of time and range of locations should be composited into 1 quart metal can (similar to a paint can) which should be available at your local hardware store. **It is important that the samples not be skimmed off the top.** For the most accurate results it is recommended that the sample contain material from the top, middle and bottom of representative percentages of each. This can be dumped into a larger container, blended then a volume of homogenized material placed in the sample container for shipment.

¹ http://www.perkinelmer.com/CMSResources/Images/44-74556GDE_TGABeginnersGuide.pdf

² <http://www.netzsch-thermal-analysis.com/us/products/simultaneous-thermal-analysis/>



Cans should be packed as follows:

1. In a box large enough to have 2 inches clearance on all sides of can
2. Line the box with a black plastic trash bag
3. Partially fill the box with absorbent (vermiculite or oil sorb)
4. Ensure can lid is sealed, and placed can in the box
5. Fill around and over can with absorbent
6. Close and seal bag
7. Enclose 1 copy of Chain of Custom Form
8. Seal box lid.
9. Affix shipping label and Haz Mat label
10. **Include a copy of this Sampling & Shipping Guide – Email Tyler Notification of Sample being Shipped**

Guidance on Shipping by Ground with UPS

1. **Visit the UPS Guide for Shipping Ground and Air Hazardous Materials** at <http://www.ups.com/content/us/en/resources/ship/hazardous/index.html>. Many of your questions can be answered. All hazardous materials shippers must process their shipments using a UPS compliant shipping solution in conjunction with hazardous materials shipment preparation software. You may utilize [UPS-provided WorldShip®](#), purchase approved third-party software, or use a comparable system that will provide UPS with the necessary electronic information.

2. **Have the DOT hazardous materials training and certification.** DOT states that no person may offer or accept a hazardous material for transportation in commerce unless that person is trained in conformance with 49 CFR and the hazardous material is properly classed (see # 3 below), described, packaged, marked, labeled, and

in condition for shipment as required or authorized by applicable requirements of 49 CFR. [More Info](#)

3. **Start the process with UPS a few weeks early-** At least the first time, UPS will need to visit your site to go over the paperwork requirement, see if you are capable of taking on the “[shipper responsibility](#)”, that you have the correct labels and placards needed and sign the Dangerous Goods Agreement.

4. **Know what you are shipping-** You will be shipping Petroleum Crude Oil which has a I.D. # of UN1267 and requires a FLAMMABLE LIQUID label.

UPS allows a maximum of three chemically compatible materials in one package and [These materials are prohibited](#).

5. **Sign a UPS Dangerous Goods Agreement-** This is a one page agreement between the shipper and UPS that is signed once a year. – [Fill out the agreement online](#)

6. **Use of UPS Multi-part shipping paper-** This document must be computer generated and the following information must appear as indicated (numbers below correspond to number in Figure 1):

The following information must appear as indicated:

1. Your UPS shipper number
2. The 24-hour emergency telephone number in conjunction with the ER Registrant information (when required) §172.604
3. The consignee name, city and state; or the package tracking number; or a reference number that refers back to the consignee. (This field may be handwritten.)
4. When preparing an air shipment, "X" through the "Cargo Aircraft Only" box to confirm the package conforms to the passenger aircraft provision in 49 CFR. If shipping a cargo quantity of Class 9, then "X" through the "Passenger Aircraft" box. When preparing a ground shipment, "X" through both "Cargo Aircraft Only" and "Passenger Aircraft." (This must be done mechanically by the computer.)

5. The basic description including the following information in sequence separated by commas, semicolons, or multiple spaces:

- Identification number (when assigned) §172.202(a)(1)
- Proper shipping name §172.202(a)(2)
- Technical name(s) for N.O.S. or generic materials in parenthesis (if required)
- Hazard Class or division number §172.202(a)(3)
- Subsidiary hazard class or division number in parentheses (if assigned) §172.202(a)(3)
- Packing group number (when assigned) §172.202(a)(4)
- Additional description(s) required §172.203
- Other regulatory information, such as "Limited Quantity" or "LTD QTY," DOT exemption/special permit number, and EX numbers
- Number and type of packages (mandatory October 1, 2007) §172.202(a)(7)
- Quantity of material §172.202(a)(5)

Note: In accordance with DOT Docket HM-215I, UPS will allow the use of basic description sequence which starts with the proper shipping name until January 1, 2013.

6. Sign and date the shipping paper. (This field can be handwritten or computer generated.)



Figure 1

Hazardous materials shipments authorized for non-specification packaging that require a shipping paper must be in outer packages at the following minimum levels (overpacks are excluded from this requirement):

- All packages must meet the requirements of [International Safe Transit Association \(ISTA\) Procedure 3A testing](#).
- Packages must be in a minimum 200 lbs. Burst Strength or 32 Edge Crush Test (ECT) certified box. The box must also be of sufficient strength to protect the contents while in transit.
- Any non-specification package that displays the ISTA seal indicating the package has been tested and certified to ISTA Procedure 3A is not subject to the minimum ECT or burst strength requirements.

With the exception of shipments containing dry ice, hazardous materials packages must not exceed 70 lbs. gross weight. Lower limits apply to certain materials.

Guidance on Shipping by Ground with FedEx

1. **View the [FedEx Ground Hazardous Materials Shipping Guide](#)**
2. **Have the training and certification**
Hazardous material training is required for all employees who perform a hazardous material function. It is the duty of each hazmat employer to comply with the training requirements listed in 49 CFR 172.704.
3. **Complete the Qualification Process**
In order to ship hazardous materials via FedEx Ground, shippers must complete a qualification process before they can ship hazardous materials. To begin this process, contact your FedEx Account Executive who can assist you or call FedEx Customer Service at 1.800.GoFedEx (1.800.463.3339).

Qualification process:

Complete a hazardous materials qualification form (OP-910).

Provide information on the hazardous materials you'll be tendering to FedEx Ground.

You can complete a sample hazardous materials certification form (OP-950) or prepare a spreadsheet listing the hazardous materials you ship.

Provide proof that you have been trained in shipping hazardous materials in accordance with federal requirements (49 CFR 172.704).

Acceptable forms of proof are:

1. A certificate from a training company stating that you have completed its program. This certificate must contain the date in which the course was completed.
2. A letter on company letterhead indicating who is responsible for hazardous materials training at your company and confirming that your employees have received the required training.
3. Completing the ShipSafeShipSmart online hazardous materials training program
4. **Know what you are shipping** You will be shipping Petroleum Crude Oil which has a I.D. # of UN1267 and requires a FLAMMABLE LIQUID label.
5. **Complete Hazardous Materials Shipping Papers**
A hazardous materials shipping paper (OP-900, OP-900B, OP-900LG or OP- 900LL) and hazardous material certification form (OP-950) are documents used to identify the hazardous materials package(s) being offered for transportation. It is the shipper's responsibility to ensure when offering hazardous material packages, that each package is accompanied with the appropriate documentation and that all forms are accurate and complete. To meet DOT shipping paper and certification requirements, FedEx Ground uses the following forms:

Shipper's Hazardous Materials Certification, OP-950:

Note: All forms must have the appropriate DOT and FedEx Ground shipping information

6. Hazardous Materials Label, OP-900: Note:

The OP-900 must be preprinted or can be computer generated. NO Handwritten documentation is accepted.

To view completed sample completed forms visit <http://www.fedex.com/us/service-guide/our-services/dangerous-goods-hazmat/index.html?qgroup=toggle-c2 - Shipping Forms>

7. Marking & Labeling

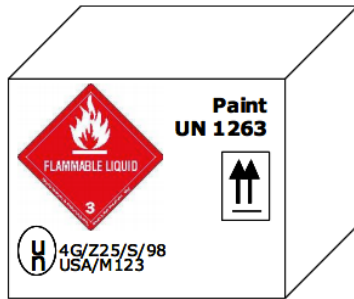
All packages must be properly marked in accordance with 49 CFR requirements. FedEx Ground requires all packages to have a minimum of four package markings.

1. Shippers Address
2. Consignee (Recipient) Address
3. The DOT Proper Shipping Name
4. The UN/NA Identification Number

All liquid hazardous materials and Class 7 Radioactive must be legibly marked, with package orientation arrows on two opposite vertical sides of the package. They may be hand drawn or affixed by a label and they must conform pictorially to ISO Standard 780-1985: two arrows up with a line underneath and enclosed within a rectangle. Depicting a rectangular border around the arrows is

optional.

Completed package example:



Additional examples can be found at <http://www.fedex.com/us/service-guide/our-services/dangerous-goods-hazmat/index.html?qgroup=toggle-c2#Examples>

Results:

Copies of analytical results should be provided in searchable electronic (Adobe PDF) format to Therma-Flite, attention: Preston Whitney (pwhitney@therma-flite.com).