
Question 43: What are your best practices when shipping ecat, fines, feed, and slurry to suppliers for testing? Please also comment on some best practices for sampling equilibrium catalyst.

TODD HOCHHEISER (Johnson Matthey)

When shipping ecat or fines, an appropriate sample container should be used. Catalyst suppliers will typically provide refiners with sample containers if needed. Catalyst shipping containers should be made of plastic or metal. Glass containers are not recommended due to potential breakage but can be used with appropriate packaging. A screw top lid is preferred over a snap on lid sometimes found on metal containers. Prying opening a snap on lid can result in personnel dust exposure. Catalyst samples should not be shipped in plastic sandwich bags or other containers not designed for catalyst service.

JM has found that metal sample containers with a screw top lid are best when shipping low vapor pressure hydrocarbon samples. A best practice is to place the sample container in a plastic bag containing adsorbent pads. These pads should minimize the chance of hydrocarbons leaking out of the box if the sample container leaks.

For hazardous catalyst and hydrocarbon samples, a GHS complaint label must be placed on the sample container. The safety data sheet must also be included with the shipment. Most catalyst suppliers prefer for a safety data sheet to be included even if the sample isn't considered hazardous. Other regulations and requirements may apply especially for sample shipments between countries.

Common sense precautions are also recommended. Some examples are shipping only the quantity of sample that is required, packaging in strong boxes, and using labels with high quality adhesive. Our lab has received sample boxes containing multiple ecat samples and multiple labels that are no longer attached to the sample containers. Clearly identifying the date of the ecat samples is critical for unit monitoring.

For any sample that is shipped, it is recommended that a company representative certified under DOT or applicable regulations be involved in the packaging and shipping process. Carriers also have specific requirements for shipping hazardous material.

KEN BRYDEN AND LUIS BOUGRAT (W. R. Grace & Co.)

For all samples, it is important to provide a safety data sheet (SDS) when shipping the sample and to follow appropriate Department of Transportation (DOT) and International Air Transport Association (IATA) rules when packaging and sending the sample. Samples should not be sent by U.S. Mail or any service that transfers to U.S. Mail. Based on our experience receiving and testing thousands of

customers Ecat and hydrocarbon samples each year, Grace has the following suggestions on best shipping practices.

Ecat and Fines Samples

For Equilibrium catalyst (Ecat) and fines samples, we have found that for routine testing a 500 mL screw top plastic container is an ideal size. Grace provides complimentary Ecat Express containers for this purpose. Screw-top metal containers are another packaging option for Ecat. Glass containers are unsuitable for Ecat since they tend to break in shipment. Containers with paint can lids are unsuitable since the lids tend to come off during shipment and spill catalyst. Bags are also unsuitable containers since they tend to leak. For any container, do not put any tag, string or wire between the cap and the container lid since they will compromise the seal and cause leaking. For large quantities of Ecat, we have found that five-gallon (or 20 liter) plastic screw top buckets are good containers.

For Ecat and fines samples, proper labeling is important in making sure the desired tests are done and reported. At a minimum, samples should be labeled with the following information:

- Refinery or company name.
- Refinery location. For example, city and state.
- Unit Name: Especially important if there is more than one FCC unit at the refinery location.
- Sample Date: The date that the sample was collected.
- Sample ID: (Optional) A sample number or name, for your reference.
- Sample Type: for example, Ecat, fines, purchased Ecat, non-routine, etc.

As part of the complimentary Ecat Express kits, Grace provides container labels that already have the refinery name and unit written and barcoded on the label.

Feed and Oil Slurry Samples

For routine analytical testing to measure the properties of feed or oil slurry samples, a 16-ounce (or 500 mL) sample size is preferred. In shipping hazardous materials, proper packaging and labeling is essential to ensure compliance with the appropriate regulations. This will prevent fines from the carrier

and delays in your shipment. In addition, poorly packaged samples can leak, which results in the sample being compromised and thus unsuitable for analysis. There are many good packaging systems available from suppliers that may be chosen to meet the packaging requirements of IATA and CFR49. Which system to use has to be determined by each individual shipper for their samples. The most common system that we see customers use is a 4GV shipper where the hydrocarbon sample is packaged in a metal can, which is then placed inside a plastic bag with an absorbent sleeve. The entire assembly is then placed in a certified cardboard box. It is important to make sure that the lid is screwed on securely. We occasionally receive leaking samples where the container lid vibrated loose in shipment. In preparing containers, make sure tags and wires from labels are not in the thread area of a cap. A string or wire from a label tag put into the sample container, with the cap sealed over it, will act as a wick. This will always cause leaking. Container types that we have noted problems within the past are a) paint cans- the lids often pop off during shipment, and b) glass bottles- they have a tendency to break during shipment.

As with Ecat samples, labeling of feed and slurry oil samples is important. The container should be labeled with the material identity and the appropriate Global Harmonized System (GHS) hazard symbols. Additionally, the sample should be labeled with the following information:

- Refinery or company name
- Refinery location: for example, city and state
- Unit Name: especially important if there is more than one FCC unit at the location
- Sample Date: the date that the sample was collected
- Sample ID: (Optional) a sample number or name, for your reference
- Sample Type: for example, feed, oil slurry, etc.

Process Ecat Sampling

Routine and representative sampling of the circulating Ecat inventory represents a critical part of FCC performance monitoring and optimization. Samples of the circulating inventory should be collected from a fluidized and accessible section of the unit to enable representative sampling of the catalyst system. From a safety standpoint, regenerated catalyst represents an inherently safer sampling source than spent catalyst due to the lack of entrained hydrocarbons and the lower coke concentration along the surface of the catalyst. However, the process temperatures associated with regenerated catalyst are significantly higher than those of spent catalyst and should be mitigated accordingly.

The regenerated catalyst standpipe represents the most common sampling location due to the continuous catalyst flow and accessibility associated with this standpipe. Although the flowing catalyst is well fluidized within this type of standpipe, it is important to properly fluidize the sampling manifold as well when obtaining a catalyst sample. Plant or instrument air are the most common fluidization media for regenerated catalyst sampling stations, which can also be equipped with steam connections to serve as blast points for line plugging troubleshooting. An air or steam purge into the process should be maintained at all times across the standpipe sampling nozzle to prevent catalyst ingress and nozzle plugging. The fluidization medium should correspond to a reliably dry source to prevent potential catalyst agglomeration issues throughout long-term operation. The sampling outlet nozzle should be purged prior to lining up the sampling line to the process to ensure that the manifold is clear of fouling and to confirm that the sample fluidization medium is available and properly dry. The key considerations and best practices for the Ecat sampling process, among others, are as follow:

1. Field personnel should be equipped with all necessary PPE prior to collecting the Ecat sample. Contact your catalyst vendor if any additional feedback or specific PPE guidelines are required.
2. Any potential impacts on instrument readings or safety interlocks by the Ecat sampling process should be thoroughly identified. Ecat sampling activities should be communicated to the board operators prior to starting the sampling process to help ensure that instrument and safety interlock functions are not compromised while sampling.
3. Ensure that the sampling container or recipient is adequately rated for the normal process temperatures associated with the circulating Ecat inventory. The sample containers used for shipping are not typically rated for these elevated temperatures. Metallic containers are typically required to accommodate Ecat sampling.
4. The sampling valve and the sampling outlet nozzle configuration should, ideally, enable sample collection without exposing field personnel to catalyst and entrained flue gas at the high process temperatures. A remote point where the operator can operate a HIC (Hand Indicate Controller) valve to take the sample in line of sight of the sample station but a safe distance away is practiced by several refiners. The sampling recipient can be attached to a long metallic or high-temperature-resistant handle to help mitigate personnel exposure to high temperatures throughout the sampling procedure.
5. Sufficient sample flow should be established to enable collection of a representative Ecat sample. Insufficient purging of the sampling manifold with the flowing Ecat can lead to non-representative or compromised results due to the presence of stagnant Ecat from previous sampling rounds, or other similar contamination sources. Collection of a slip stream during continuous Ecat flow through the sampling line tends to yield a more representative sample than collecting a vial sample from a drum or (large container) of Ecat sample inventory.
6. Excessive superficial velocities through the sampling manifold should be prevented while sampling to help mitigate potential erosion and attrition issues. Excessive catalyst attrition

through the sampling line can lead to false PSD profiles for the circulating catalyst inventory that can prompt unnecessary troubleshooting activities. Adequate velocities through the sampling nozzle also help reduce turbulence and dust as the flowing Ecat reaches the sampling container, thus preserving as much of the fines content present in the circulating inventory as possible.

7. A pint of Ecat sample is usually sufficient volume to accommodate routine lab testing for process monitoring purposes. Excess Ecat sampling volume should be properly handled and discarded via spent catalyst drums or disposal lines routed to the spent catalyst hopper, if available.
8. Ecat samples should be allowed to properly cool before filling the corresponding shipping containers. Windy or wet environments should be avoided for the cooling period to avoid altering the physical properties of the Ecat sample.

The guidelines and best practices previously referenced should be followed when shipping the Ecat sample containers. Board operators, unit engineers and other supporting staff for the FCC complex should visually inspect Ecat samples before the sample is shipped to the catalyst vendor. Visual inspection can help qualitatively gauge the health of the circulating catalyst inventory – especially with respect to coke on regenerated catalyst (CRC), drastic PSD shifts, and/or potential Fe poisoning contamination – well before the corresponding lab results become available.

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2019