

STANDARD OPERATING PROCEDURE TDSC-01 SAMPLE COLLECTION PROCEDURE FOR TITANIUM DIOXIDE SAMPLE LABELLING, PACKAGING AND SHIPPING

1.0 PURPOSE

This standard operating procedure (SOP) provides the guidance necessary for sample collection personnel to collect titanium dioxide (TiO₂) product samples. Samples should be collected by personnel familiar with the facility operations and this SOP. Additionally, this SOP addresses field documentation and sample labelling, packaging and shipping. This SOP was prepared by Environmental Standards which has expertise in data collection procedures so that uniform approaches are undertaken by the Titanium Dioxide Stewardship Council (TDSC) member companies' sample collection personnel.

2.0 GENERAL CONSIDERATIONS

This SOP cannot predict all situations the sample collection personnel may encounter. If situations are encountered that dictate modifying these procedures for successful sample collection, then the modifications must be documented.

Safety first. Sample collection personnel must comply with applicable company specific health, safety and management requirements. Sample collection personnel should wear nitrile surgical gloves during sample collection activities to minimize contamination of the samples and for personal protection.

3.0 COMPANION DOCUMENT

The following document provides information relative to this sampling event:

- Quality Assurance Project Plan Evaluation of the Presence of Polychlorinated Biphenyls in Titanium Dioxide (QAPP, Environmental Standards, February 2020)

4.0 PROCEDURES

Sampling kits containing the necessary equipment to collect TiO₂ samples will be shipped to the sample collection personnel in advance of the sample collection date(s). Arrangements for the sample kits will be made by Environmental Standards personnel. The kits will be prepared by the project laboratory, SGS North America, located in Wilmington, North Carolina.

The sample kits will include:

- Stainless steel spoons or scoops (precleaned)
- 4-ounce amber sample containers
- Sample labels
- Chain-of-Custody record forms
- Sealable plastic bags for samples and COC forms

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- Sealable 1-liter plastic bags for containing ice
- Shipping cooler
- Custody seals
- Return shipping label
- Temperature bottle blank
- Field blank material – clean sodium sulfate in a labelled 4-ounce amber sample container
- Packing materials such as bubble wrap, and large plastic bag to line the sample cooler

NOTE: The sample kit will not contain wet ice necessary to provide proper temperature preservation when shipping the samples to the laboratory. Ice should be obtained the day of sample collection and placed in the 1-liter sealable bags.

The sampling personnel should open the sample kits several days prior to the sample collection event and ensure they are familiar with the items delivered and inventory the items. A packing list will accompany the sample kits.

4.1 Field Blank Collection

A field blank will be collected at the sample collection location immediately prior to collecting the TiO₂ sample using the following stepwise process:

1. Locate the 4-ounce container marked “field blank material.” Inert sodium sulfate should fill about $\frac{3}{4}$ of the container.
2. Complete a sample label and affix the label to an empty 4-ounce container. Use a field blank identification of “FB Month/Day/Year” using the date of collection.
3. Remove the tops of both the empty container and the container that has the field blank material (inert sodium sulfate).
4. Transfer the sodium sulfate from the full container using a stainless-steel spoon to the empty pre-labelled field blank container. When no more material can be moved with the stainless-steel spoon, use a combination of tilting the container and scraping the sodium sulfate with the spoon from the one jar to the other. Again, this should occur near where the TiO₂ sample will be collected.
5. It is not necessary that all the sodium sulfate be transferred into the field blank container as some small amount is expected to remain. Once the transfer of material is complete, cap the container of the now complete field blank and place the container in a sealable plastic bag and then in the sample cooler on ice.
6. Complete the COC (see SOP-001).

4.2 TiO₂ Sample Collection

The TiO₂ sample is to be collected at the point where the product is being bagged. Collect the sample as soon as possible after the finished product is bagged.

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1. Complete a sample label and affix the label to an empty 4-ounce container. Use the field designation identification that corresponds to the TiO₂ sample listed on QAPP Worksheet #18.
2. Remove the cap of the sample container.
3. Using the same stainless-steel spoon that was used to collect the field blank, insert the spoon into the TiO₂ contained in the product bag three times to “rinse” the spoon with product prior to actual sample collection. The stainless-steel spoon should only contact TiO₂, so ensure it does not come in contact with the bag.
4. Once the sampling spoon is rinsed, obtain the sample of the TiO₂ from the product bag using the stainless-steel spoon, and place the sample carefully into the container until the container is approximately ³/₄ full. Again, ensure the sampling spoon only contacts TiO₂.
5. Cap the container of the now complete sample and place the container in a sealable plastic bag and then in the sample cooler on ice.
6. Complete the COC (see section 4.3.2).

4.3 Field Documentation

Proper documentation of sample collection is an important part of the process. Each sample collection team or individual should maintain a field record (or equivalent documentation per existing company product quality sample collection procedures) to document the activities performed each day that sample collection is conducted. The following information should be recorded:

- Name and location of the site.
- Date(s) of sample collection.
- Names of sample collection personnel.
- Pertinent field observations. Ensure key information about the TiO₂ product is recorded such as lot number or other unique identifying information. Record any physical observations that might be important to understanding and interpreting the sample data. **Note:** Only objective field observations shall be recorded. Subjective observations such as opinions and speculations shall not be recorded.
- Time of collection of each sample.
- Numbers and types of samples collected and sample identification numbers.

A summary of the key elements of field documentation procedures is provided below.

- Recorded notes or entries on the field record shall be made in indelible ink.
- Data must be recorded directly and legibly in the field record.
- Corrections in notes or field records shall be lined out (a single line strike-through), initialed, and dated.
- Avoid leaving any blank line(s) between recorded entries. Cross out any lines intentionally left blank with a single line and initial and date the cross-out.
- Cross out any blank areas that exist with a single line and initial and date the cross-out.
- Multiple blank lines may be crossed out using a single diagonal line that passes from the top left of the first blank line to the bottom right of the last blank line.

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- The field team member keeping the records or notes must sign and date each page of the field record.

The following materials will be necessary:

- Chain-of-custody (COC) forms (will be provided by the laboratory).
- Custody tape or seals (will be provided by the laboratory).
- Field record.
- Ballpoint black ink pens or permanent markers.
- Sample labels (will be provided by the laboratory).
- Waterproof bags for COCs (will be provided by the laboratory).
- Clear-plastic sealing tape.
- Camera (optional)

4.3.1 Sample Labels

When feasible, sample containers should be pre-labeled prior to sample collection to minimize the potential for misidentifying the location. Sample labels shall include the following information:

- Unique sample identification (see QAPP Worksheet #18).
- Parameter to be analyzed; PCBs by Method 1668C.
- Date and time of collection.
- Sampler's initials.
- Preservative(s), if any. This will be NONE.

4.3.2 Chain-of-Custody Documentation

The chain-of-custody (COC) form is intended to be a legal record of possession of samples from the time of collection to laboratory analysis. COC forms once completed will not be shared beyond Environmental Standards and the laboratory. Each TDSC company will receive copies of the COC forms for the samples it submitted. This will help to ensure the anonymity of the sample origin and to protect confidential business information. The COC shall be initiated in the field by the sampling personnel at the time of sample collection and should bear the name of the person responsible for the secure and appropriate handling of the samples.

The sample collection personnel responsible for the collection event shall maintain the COC during sample collection activities. The minimum information required for COC documentation includes the following:

- Name and location of the site. This will be a generic reference to protect anonymity of company.
- Name and affiliation of samplers.
- Sample identification number.
- Date and time of sample collection.
- Matrix and type of sample collected (e.g., TiO₂ and grab).

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- Number of containers per sample.
- Preservatives and fixatives. This will be NONE.
- Parameters to be analyzed. PCBs by Method 1668C.
- Identification of couriers.
- Identification of laboratory. SGS North America Wilmington, NC.
- Data deliverable requirements and turn-around-time (TAT). Level 4 and standard TAT.

Environmental Standards will coordinate with the laboratory as part of the sample kit delivery to have much of the COC pre-populated with the correct information. When completing COCs, field personnel must employ the applicable field documentation techniques described previously for field records.

The person identified as the Sampler shall sign and date the COC when the samples are relinquished. The designated laboratory representative shall accept custody of the samples by signing with affiliation, date, and time in the “accepted by” section. **Note:** Commercial couriers (e.g., UPS, Federal Express) shall NOT sign the COC; however, tracking numbers and shipping documentation should be retained as part of the project files.

4.4 Sample Packing and Shipping

After the samples are collected, they will be placed in a cooler on ice for temperature preservation and then be prepared to ship to the laboratory.

4.4.1 Temperature Preservation

In order to facilitate preservation of the samples, they will be chilled to an appropriate temperature (< 6°C but not frozen) and maintained at this temperature from the point of collection through transport and receipt at the analytical laboratory. Wet ice contained in sealable plastic bags will be used for temperature control. Blue Ice® or similar media shall not be used to chill samples.

4.4.2 Sample Packing

The following is a summary of steps required for packing the samples for shipment.

- For packaging the samples using wet ice for temperature preservation, place a garbage bag or liner in the cooler.
- Place a temperature bottle blank in the center of the cooler. The temperature blank will be supplied by the laboratory in the sample kit.
- Place individual sample containers into the cooler.
- Place ample amounts of wet ice contained in doubled resalable bags inside the garbage bag/liner in cooler. As needed, place bubble wrap or other inert packing material around the garbage bag/liner in the cooler.
- Seal the garbage bag/liner with duct tape. This is to ensure that if the contents were to spill that the garbage bag/liner would contain the spill.

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- One COC form should be completed for each cooler, placed in a large, resealable plastic bag, and taped to the inside lid of the cooler. The shipper's tracking number (e.g., courier and courier air bill number, if applicable) should also be written on the COC form.
- The cooler should be taped closed with strapping tape and sealed with custody tape on two sides such that opening the cooler will break the custody tape.

4.4.3 Sample Shipping

The samples shall be shipped as environmental samples to the laboratory using the non-hazardous shipping forms provided by the laboratory in the sample kits. When preparing the cooler for shipment, any old labels from the outside of the cooler should be removed. Samples will be shipped for overnight, next day delivery.

END OF SOP