

DRAFT

LC/MS/MS Analysis of Articles and Products: Samples and Suggested Sampling Procedures

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I. Preliminary Study – Samples for Method Development and Extraction Efficiency

During the course of this preliminary study, the contract facility will develop extraction efficiency data on three matrices – carpet, textile and paper – at three different concentrations: 1 ppb, 10 ppb and 100 ppb for articles only. In order to do that work, we have determined that the contract lab needs individually prepared controls in the following quantity:

Product	Matrix	Number Requested	Approx. wt of each sample
Untreated Shaved Carpet	Nylon	54*	10 grams
Untreated Textile	Polyester	54*	2 grams
Untreated Paper	Cellulose	54*	2 grams

Total pieces: 162

*18 of each of these 54 untreated samples are required/requested for method development.

II. Treated and Untreated Articles

Number of Samples Required for Analysis

FOR CARPET

Product	Matrix	Number Requested	Approx. wt of each sample
Each Treated Shaved Carpet		6	10 grams
Each Untreated Shaved Carpet*		4	10 grams

* Used to verify extraction efficiency at 1 ppb, 10 ppb and 100 ppb, as well as control

We plan on having three different carpet samples.

	Shaved Carpet I	Shaved Carpet II	Shaved Carpet III
Treated Samples	6	6	6
Untreated Samples	4	4	4

18 samples analyzed

30 total treated and untreated articles

FOR TEXTILES

Product	Matrix	Number Requested	Approx. wt of each sample
Treated Textile		6	2 grams
Untreated Textile*		4	2 grams

* Used to verify extraction efficiency at 1 ppb, 10 ppb and 100 ppb, as well as control

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We plan on having seven textile samples.

	Tex I	Tex II	Tex III	Tex IV	Tex V	Tex VI	Tex VII
Treated Textile	6	6	6	6	6	6	6
Untreated Textile	4	4	4	4	4	4	4

42 samples analyzed

70 total treated and untreated articles

FOR PAPER

Product	Matrix	Number Requested	Approx. wt of each sample
Treated Paper		6	2 grams
Untreated Paper*		4	2 grams

* Used to verify extraction efficiency at 1 ppb, 10 ppb and 100 ppb, as well as control

We plan on having two treated paper products analyzed.

	Paper I	Paper II
Treated Paper	6	6
Untreated Paper	4	4

12 samples analyzed

20 total treated and untreated articles

III. Product Formulations Samples

Number of Samples Required for Analysis

Product	Number Requested	Approx. vol. of Each sample
Formulation I	1	40 mL
Formulation II	1	40 mL
Formulation III	1	40 mL
Formulation IV	1	40 mL
Formulation V	1	40 mL
Formulation VI	1	40 mL
Formulation VII	1	40 mL
Formulation VIII	1	40 mL
Formulation IX	1	40 mL
Formulation X	1	40 mL
Formulation XI	1	40 mL
Formulation XII	1	40 mL

IV. Procedure for Preparing Samples

This study should be viewed as a screening study. Currently, a validated methodology does not exist for the analysis of the test substance in carpet (shavings), textiles and paper. The purpose of the preliminary study is to demonstrate the suitability of the existing LC/MS/MS method to analyze the test substance in your matrices. A report detailing the methodology used to obtain 70-110% extraction efficiencies for 1 ppb, 10 ppb and 100 ppb test substance in each of these three matrices will be available to all TRP members. The accuracy and precision of the methodology will be determined by extracting 6 untreated articles (each of carpet, paper and textile) each at 1 ppb, 10 ppb and 100 ppb on two successive days.

Once validated and verified for accuracy and precision, this method will be used to analyze our now 12 treated articles. In order to minimize the variability in sampling, it is key that steps be taken to generate a representative sample for each treated article. It is important that the sampling procedures used to generate these samples be documented and retained in your study records.

A. Textile Sampling Procedure

1. Determine the size of a 2-gram sample of paper.
2. Suggested sampling approach:

Treated Samples from Roll of Fabric

-If the treated sample is taken from a roll, first discard a minimum of 1 meter of fabric from the very outside of the roll – equivalent to at least one wrap - then take a full-width swatch 0.5 meter long from the end of the roll of fabric.

-Create a grid with pencil on the treated fabric, eliminating 3-5 cm from the fabric edges. The grid should be composed of six equivalent blocks. These six blocks represent the six areas where you will take one sample. Divide each of the large blocks into smaller blocks, so that each sub-block is equivalent to a 2-gram sample. Systematically number all the smaller blocks in each larger block. Use a random number generator (in Excel) to generate a list of random numbers between 1 and the number of smaller blocks in each larger block. Use the first six numbers to select the six locations of the treated article sample in each larger block.

Treated Samples from Discrete Squares of Fabric

-If each discrete square of treated fabric is small enough to generate one to six samples, take one random sample from each discrete square of treated fabric using the block design concept described above. Therefore, from six discrete squares of treated fabric, take one random sample.

Untreated Fabric Samples

Untreated fabric samples can be taken randomly from untreated fabric – avoid edges – avoid contamination; supply four untreated samples.

3. Document the sampling approach.

B. Paper Sampling Procedure

1. Determine the size of a 2-gram sample of paper.

2. Suggested sampling approach:

Treated Samples from Roll of Paper

-If treated sample is taken from a roll, first discard a minimum of 1 meter of paper from the very outside of the roll – equivalent to at least one wrap - then take a full-width swatch 0.5 meter long from the end of the roll of paper in the lot sample.

-Create a grid with pencil on the paper, eliminating 3-5 cm from the paper edges. The grid should be composed of six equivalent blocks. These six blocks represent the six areas where you will take one sample. Divide each of the large blocks into smaller blocks, so that each sub-block is equivalent to a 2-gram sample. Systematically number all the smaller blocks in each larger block. Use a random number generator (in excel) to generate a list of random numbers between 1 and the number of smaller blocks in each larger block. Use the first six numbers to select the six locations of the treated article sample in each larger block.

Treated Samples from Discrete Squares of Paper

-If each square of discrete treated paper is small enough to generate one to six samples, take one random sample from each square of treated paper using the block design concept described above. Therefore, from six discrete squares of treated paper, take one random sample. Collect six samples.

Untreated Paper Samples

-Untreated samples can be taken randomly from untreated paper – avoid edges – avoid contamination; supply four untreated samples.

3. Document your sampling approach.

C. Carpet Sampling Procedure

1. Determine the size of carpet that will produce approximately 10-grams of carpet shavings.

2. Suggested sampling approach:

Samples from Discrete Sections of Carpet

-If each discrete section/square of treated carpet can generate one to six samples, take one random sample from each section of treated carpet using the block design concept described above for textile and paper. Therefore, from six discrete squares of treated carpet take one random sample.

-When sample location is identified, use hair shears to remove carpet shavings (fibers) from the carpet backing. Shear fibers as close to carpet backing as possible. It is recommended to clear the hair shears with cheesecloth, as cheesecloth should not be contaminated with the test substance.

Untreated Samples of Carpet

Untreated samples of carpet can be taken randomly from untreated carpet –avoid edges – avoid contamination; supply four untreated samples of carpet shavings.

3. Document your sampling approach.

V. Sample Packing Procedure

A. Articles

-Triple wrap each sample with aluminum foil. For a 10-cm square sample, cut a piece of foil that is at least 25-cm long. Fold foil at the mid-point of the length. Place article in the pocket. Fold-up about 1 cm on each edge. Fold up another cm of each edge. Then crimp tight. Repeat with two more pieces of aluminum foil. Place in zip-lock bag – remove air from bag and seal.

-Place in a second zip-lock bag. Place provided labels with sample identification numbers on outside of second plastic bag. Sample should be labeled with Project Number, Sample Identification Number and sample type. Sample label should also clearly state if sample is treated or untreated. Complete Sample Identification Sheet for the study records.

-Sample identification numbers have been provided in excel spreadsheet. Labels will be shipped on February 13, 2003.

-Pack untreated sample separately from treated samples.

-Include a chain-of-custody form in each package.

-Follow shipping procedure.

B. Products

- Place 40 mL of product formulation used to treat article in the appropriate containers for shipment. Double-bag each sample, using zip-lock plastic bags. Place label with sample identification number, project number and sample lot number on outside of second plastic bag. Include chain-of-custody form and modified MSDS in box containing formulated product samples. Ship products separately from articles. Follow shipment procedures. Complete Sample Identification Sheet for the study records.