

# Test Method

Number

# 1

## Matter Extractable from Wipers and Other Materials

# 1

## Background and Theory

Distinct from the questions of the burden of ionic content and the generation of particulate debris, is the issue of the quantity of matter accessible to a given solvent from a wiper (or other item) destined for use in a cleanroom. Since, for a given solvent, the total mass of soluble matter is a unique and unequivocal quantity, we at ITW Texwipe chose to employ a method of testing which is aggressive and thorough in the extraction of that quantity. The method is designed to extract the residue in its entirety to a high degree of certainty. This method is generally more thorough in the extraction of residue than other commonly used methods.



Generally speaking, the substances soluble in a particular solvent will be present on the order of, at most, a few percent of the mass of the specimen being tested. In our procedure, we choose a volume of solvent and a mass of specimen such that a *thermodynamic* excess of solvent over the solute is almost certainly always present. In other words, if a solute fails to dissolve in the solvent under these conditions, it will not dissolve in this solvent under virtually any other conditions. To enhance the *kinetics* of solubility—the rate of solubility—the extraction is performed at the boiling point of the solvent.

Frequently, tests for soluble material become cluttered with unnecessary appendages such as the determination of solvent blanks. We are not presuming that under certain circumstances solvent blanks might be required, but generally the solvents used to do the extractions are of reagent or even spectroscopic quality and the blank associated with their use is negligible compared to the mass of material that is being weighed.

Since we presume a thermodynamic excess of the solvent over the solute, it is at best unnecessary and at worst incorrect to specify the selection of a particular mass of the sample. Instead, that mass should be chosen so that a *minimum*, often milligrams, of residual material will ultimately be weighed on an analytical balance. Since the reproducibility of weighing the final mass of extracted matter will rarely be much better than one-half to one milligram. This will ensure a relative precision of the final answer of at least five to ten percent.

### PURPOSE

*To determine the mass of material extractable from a wiper (or other item) using a given solvent.*

## EQUIPMENT

Analytical Balance	At least, 50 g capacity and sensitivity of 0.1 mg.
Hot Plate	Suitable for heating the solvent(s) to a boil.
Fume hood	Providing sufficient face velocity to keep solvent fumes out of the laboratory area.
Glass Beakers (several)	Pyrex type, of various sizes; 25, 50, 250, 500, and 750-mL.
Glass Filter Funnels	Capable of supporting 150 or 185 mm filter papers
Filter Paper	Whatman # 5 Grade
Glass Stirring Rods	10 – 15 cm in length
Small Aluminum Weighing Pans	25-mL volume
Oven	Capable of maintaining 105° C, forced air is preferable
Desiccator	Capable of hold in a constant and relatively low humidity (<10%)
Ring stand	various
Tongs and heat resistant gloves	For moving hot beakers and pans

**All glassware must be cleaned thoroughly to ensure accurate results.**

## REAGENTS

Reagent Grade Water	Deionized water of greater than 16 mohm/cm purity, TS < 10 mg/L
Solvents	ASC Grade, low residue or better
Cleaning Solution	2%, v/v NP-9 or equivalent

# 2

## Protocols and Requirements

### Gowning Protocol

Proper gowning is required to perform this procedure to ensure the accuracy of the test results. Refer to the laboratory gowning procedure in the \\TEXWIPE-NC\VOL1\APPS\ISO2000\QUALITY\W\gowning procedure.doc. The required gowning is as follows:

- Hair net,
- A face cover is required when handling or cutting laundered or finished products,
- Clean lab coat,
- Gloves are required when handling samples, and
- Proper eye protection.

### Glassware Cleaning Protocol

All labware used in this procedure must be cleaned thoroughly using the following procedure.

1. Using gloved hands, scrub the beakers, filter funnel and stirring rods with a non-ionic surfactant solution (2% v/v, NP-9 solution or equivalent), hot tap water, and a low residue wiper.
2. Rinse the glassware with hot tap water until all foaming stops.
3. Rinse the item with acetone, inside and out.
4. Rinse with hot tap water thoroughly.

5. Rinse with deionized water thoroughly.

6. Inspect the item for visible residue and markings. If present, repeat the cleaning process until acceptable.

7. Place the glassware on a drying board until dry.

8. Move the item to storage as soon as dry, to reduce the possibility for recontamination.

### Quality Assurance Requirements

#### Selection of Material Weight for Extraction

The quantity of residue extracted is dependent upon the mass of material tested and the quantity of solvent used. Select a mass of material that is likely to provide at least 0.0020g of extracted residue. If the analysis yields less than 0.0020g of residue, repeat the analysis with a higher mass of material. Obviously, there is a limit to the weight of material that can reasonably be processed utilizing this procedure. In general, no more than 50 grams of material need be extracted in an effort to meet the minimum mass of extracted residue. If, due to the nature of the material, sufficient material cannot be extracted to meet the minimum requirement, test the largest sample mass that can reasonably be processed. Note in the final report that the extracted residue did not meet acceptance criteria, the reason why, and indicate the result is an approximate value.

# 2

## Protocols and Requirements (con't.)

### Balance

The analytical balance must have a resolution of 0.0001 g. The analytical balance must be checked for proper leveling and accuracy before use each day. Check the leveling bubble and make adjustments if necessary. The weight of a certified one-gram and fifty-gram weight is checked to determine if the scale is in acceptable working order. The initial weight checks must meet the following acceptance criteria before work can proceed.

Weight, g	Acceptance Range, g
One-gram	0.9990 – 1.0010
Fifty-gram	49.9980 – 50.0020

In addition to the initial weight checks, the balance should be checked using a one-gram certified weight before each set of weighings. The weight check must meet the same acceptance criteria as required for the initial one-gram weight check. If weighings are performed within two hours of the initial weight checks, the additional one-gram check is not required.

If the balance does not meet acceptance criteria for any checks, discontinue testing and notify the Laboratory Manager or the equipment calibration analyst. The balance must be zeroed before each weighing.

### Solvent Blanks

A solvent blank must be performed on each lot of solvent before use for testing. The percent non-volatile residue of the solvent should be <0.005% by weight. If the solvent does not meet this acceptance criteria, select another more suitable grade of solvent. If a suitable substitute is not available, consult the Laboratory Manager on the suitability of the solvent for use.

### Method Blanks

A method blank is generally not necessary when using high purity solvent. However, analysis of a method blank can yield information about the cleanliness of the equipment and the analyst's technique. Method blanks must be performed at a rate of one per twenty sample analyses per analyst. The resulting value must meet the acceptance criteria given below. If the method blank is outside the acceptance criteria, another method blank must be analyzed immediately. If the method blank is still outside the acceptance criteria, all NVR testing must be halted and corrective action taken before testing can resume.

Analysis	Acceptance Criteria
NVR Method Blank	<0.005 %, w/v

### Duplicate Analyses

A duplicate analysis of a sample must be performed at a rate of one per twenty sample analyses. The resulting relative percent difference (RPD) must meet the acceptance criteria listed below. If the RPD is outside the acceptance criteria, the sample must be

reanalyzed. If the RPD is still outside the acceptance criteria, another sample must be selected and analyzed in duplicate. If that set of analyses fails to produce an acceptable RPD, all NVR testing must be halted and corrective action taken before testing can resume. If the RPD for the new set of samples is acceptable, the failure is attributable to inconsistent dispersion of NVR within the original sample.

NVR Concentration Range, %	RPD Acceptance Criteria, %
<0.01 %	Do Not Use for RPD Calculation
0.01 – 0.10 %	<30 %
>0.10 %	<20 %

### Safety

The test procedure is to be carried out using good laboratory practices and in a safe manner. The protocols established in the Chemical Hygiene Plan (\\TEXWIPE-NC\VOL1\APPS\ISO2000\Lab\SOP\Chemical Hygiene Plan.doc) must be followed. Proper Personal Protection Equipment (PPE) must be used. This includes the use of eye protection and protective clothing.

Solvent use and handling should be held to a minimum and conducted in a fume hood or other suitably well ventilated area. Flammable or toxic solvents must be used in a fume hood and the required personal safety equipment must be utilized. Refer to the Material Safety Data Sheets for all solvents and chemicals before use. Solvent disposal must comply with the Chemical Hygiene Plan. Isopropyl alcohol (2-propanol) is one of the solvents commonly used in the procedure. This solvent is highly flammable and slightly toxic. Use proper care when handling alcohols. Other solvents may be utilized in this procedure.

This procedure routinely utilizes boiling water in the extraction. Boiling water can cause serious burns. Use tongs, heat resistant gloves, or other personal protective equipment when handling hot water.

Confirm the operational status of the fume hood before beginning testing. Observe the air flow in the unit to ensure adequate venting in the hood. The fume hood sash must be in the down position when not directly working in the hood. At all other times during the testing, the sash should not be raised higher than the maximum recommended sash height indicator.

# 3

## Selection of Extraction Solvents

The selection of extraction solvent is based on the application in which the material is to be used and the affinity of the extractable materials for the solvent. While one extraction solvent may be capable of extracting all residues for various materials, deionized water and isopropyl alcohol (IPA) have been chosen by ITW Texwipe as the solvents routinely used in evaluating the performance of wipers and other materials with regard to extractable residue. All testing for extractable residue will be performed using laboratory grade deionized water and low residue isopropyl alcohol, unless fully evaluated and authorized by the Laboratory Manager.

Other solvents can be substituted in this procedure when deemed necessary to better evaluate product performance. Caution should be exercised when selecting solvents and solvent blends. The safety of personnel is the main consideration in solvent selection. Toxicity of the solvent, safe handling requirements, solubility of the base material in the solvent, and waste disposal are other items to be addressed before selecting an extraction solution. No solvent should be used in this procedure until it has been fully evaluated and has been approved for use by the Laboratory Manager and Chemical Hygiene Officer.

# 4

## Procedure

1. Place a sufficient number of evaporation dishes/pans into the oven at 80°C and heat for at least one hour. After drying, place the dishes into a desiccator and allow them to come to room temperature. Determine the mass of the dishes to four significant digits. Record the values on the worksheet. The type of evaporation dishes to be used depends on the solvent used in the extraction. Aluminum weighing pans are satisfactory for solvents such as alcohols and acetone and most water extractions. Because of the potential for oxidation of the aluminum pans, glass may be needed for water extractions of low residue materials.
2. Procure a representative sample of the material to be tested. Because a small portion of material is analyzed, collection of a representative sample is paramount to obtaining useful test data. It may be necessary to perform several tests on portions taken at various spacing or intervals throughout the roll, lot or other unit to obtain representative test results. When only one test is to be performed, take multiple small sample portions from throughout the material roll or unit and composite these into one test sample. If this is not practical, take samples from the beginning and end of a roll of material and composite these before testing. If the testing of only one head end is necessary, cut a swath of material running from one side of the material to the other, relative to the machine direction. Further, subsample this swath by taking evenly spaced portions from along the length until sufficient material is obtained.
3. Cut a representative test specimen from the material sample and weigh to four significant figures. The weight of sample selected for extraction is dependent upon the moisture content and the amount of extractables in the material. Select a sample weight that is expected to yield at least 10 mg of extractable materials.
4. Dry the test sample at 80°C until a constant weight is obtained. Usually 30 minutes in the oven is sufficient. Place the dried test sample in a desiccator and allow it to cool to room temperature. As an alternative, perform a percent moisture analysis on a separate but representative portion of material. Adjust the sample weight used in the NVR calculation to compensate for the moisture content of the material.
5. Set up a ring stand to hold a glass funnel so that the funnel stem will be just above the top of a 1000 mL beaker. Place a fluted filter paper into the funnel and rinse it three times with a small quantity of the same solvent as used in the extraction. Discard the washings. Place a clean 1000-mL beaker beneath the funnel.
6. Determine the mass of the test specimen to four significant figures. Determine the basis weight (see Texwipe Test Method No. 20) on the specimen or on another sample portion from the same lot of material. Record the information on the worksheet.
7. Place the specimen in the 500-mL<sup>2</sup> beaker; it is necessary to cut the sample beforehand into smaller sized pieces.
8. Add sufficient volume of the chosen solvent<sup>1</sup> to cover the material in the beaker completely. Usually 250 mL is sufficient<sup>1</sup>. Bring the liquid to a boil on a hot plate inside the fume hood. The material must be completely covered by the solvent, with sufficient volume to allow for mixing.
9. Boil the contents of the beaker for five minutes, prodding and stirring with a glass rod to ensure complete mixing and to prevent “bumping” of the solvent. With the more volatile solvents, it is usually necessary to add small amounts of solvent to the beaker to ensure the material remains covered throughout the boiling period.
10. After the five-minute boiling period, remove the beaker from the hot plate and pour the readily decantable portion of the solvent into the washed filter paper in the glass funnel. Collect the filtrate in the suitable size beaker.
11. Add a second portion<sup>1</sup> of solvent to the beaker containing the test specimen. Place the beaker back onto the hot plate and bring the mixture to a boil. Decant the free solvent through the same filter, combining the filtrate with the first portion. Repeat the extraction with third portion<sup>1</sup> of solvent, adding the filtrate to the filter and combining all three filtrates. Using a glass stirring rod press the free solvent out of the material and add it to the funnel.

# 4

## Procedure (con't.)

12. Place the beaker containing the combined filtrates onto the hot plate and evaporate the extract to a volume of approximately 10 mL. If, due to size of the beaker used, the solvent begins to char before the volume is reduced to 20 mL, quantitatively transfer the solvent to a 50 or 25-mL beaker and continue the evaporation. Rinse the large beaker with three small portions of fresh solvent and add these rinses to the smaller beaker. The extractable material in the beaker must never be allowed to go dry during the evaporation process. If this occurs, discard the test and repeat the extraction with a new portion of material.

13. When the extract is approximately 10 mL in volume, quantitatively transfer the fluid into a weighed evaporating dish. Rinse the beaker with three small portions of fresh solvent and add these rinses to the evaporation dish.

14. Transfer the evaporation dish/pan to an oven at 80°C. Allow the extract to evaporate completely in the oven, and then continue the drying for an additional hour.

15. After the drying period, transfer the evaporation dish to a desiccator and allow it to come to room temperature. Determine the weight of the evaporating dish and residue to four significant figures. Record the value on the worksheet

# 5

## Calculations

1. Test results can be adjusted to account for moisture content if a dried sample is not used. This is accomplished by performing a percent moisture determination on a portion of the material. The sample weight used in the calculations is adjusted for moisture content using the following equation:

$$W_d = W_w * (1 - M_p)$$

where:

$W_d$  is the dry weight of the material [grams]

$W_w$  is the weight of the material as received [grams]

$M_p$  is the decimal percent of the moisture content of the sample as received [decimal %]

Note: This conversion of the sample weight is not necessary and should not be performed, if the material is dried before analysis or is to be reported on an as received basis.

2. The extractable material is reported on a dry weight basis as a percentage and/or as a weight per unit area of the material, according to the following equations:

$$EM\% = 100 * (m_{ds} - m_d) / m_w$$

$$EM_A = [(m_{ds} - m_d) / m_w] * bw$$

where:

EM% is the percent extractable matter [%]

EMA is the extracted matter per unit area (grams/m<sup>2</sup>)

$m_{ds}$  is the mass of the evaporation dish plus the extracted matter [grams]

$m_d$  is the mass of the evaporation dish [gram]

$m_w$  is mass of the extracted matter [gram]

bw is the basis weight of the material [grams/meter<sup>2</sup>]

# 6

## Footnotes and References

1. The volume of solvent to be used in the extraction is dependent on the amount of material required to obtain a satisfactory amount (>10 mg) of extractable material. Materials that are low in extractable matter will require sample weights of 50 grams or more to produce acceptable test results. Other materials such as unlaundered cottons may require as little as two to five grams. In all cases, the volume of extraction fluid must be in excess. The material must be free floating in the solvent and easily mixed during extraction.

2. The size of the beaker used is dependent on the weight of fabric and volume of solvent required. Select the beaker size as necessary to ensure adequate mixing of the material during the extraction. The volume used in the extraction will dictate the size of the beaker to be used to collect the decanted solvent after extraction. Select a size that will provide adequate capacity.

## ITW Texwipe®

**North America**  
300B Route 17 South  
Mahwah, NJ 07430  
Tel (800) TEXWIPE ext 120  
(201) 684-1800 ext 120  
Fax (201) 684-1801  
[www.texwipe.com](http://www.texwipe.com)  
[info@texwipe.com](mailto:info@texwipe.com)

**Europe/Middle East**  
Skejby Nordlandsvej 307  
DK-8200 Aarhus N  
Denmark  
Tel +45 87 400 220  
Fax +45 87 400 222

**Asia/Pacific**  
50 Tagore Lane  
#02-01 Markono  
Distri Centre  
Singapore 787494  
Tel +65 6468 9433  
Fax +65 6468 6772