Non-Volatile Residue (NVR)
Contamination from Dry Handling and Solvent Cleaning

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Presentation Overview

- Background
- Items Tested
- Work Instructions
- Gravimetric Determination Method
- Contact Transfer Method
- NVR Calculation Equations
- Consumables Database
- Recommendations
- Questions
Background

- Non-volatile Residue (NVR) consists of organic molecules/compounds
  - Transferred to surfaces via dry handling or solvent contact

- NVR may adversely affect
  - Bonding strength
  - Cleanliness of optics
  - Cross Contamination via Outgassing or Contact Transfer

- Sources of NVR generally expected to be “clean”
  - Gloves
  - Wipes
  - Swabs
  - Garments
  - Bagging material & film
  - Tape

- Situations in which NVR may be an issue
  - Surface preparations
  - Clean room operations
  - Assembly of components
  - Cleaning
  - Project requirements/contamination allocations
  - Sensitive hardware near contaminated surfaces
Items Tested

- **Gloves**
  - Polyethylene
  - Latex
  - Nitrile
  - Vinyl
  - Polyurethane

- **Wipes**
  - Polyester
  - Cotton

- **Swabs**
  - Cotton
  - Poly

- **Miscellaneous**
  - Cleanroom Paper

- **Garments**
  - Coveralls
  - Beard Covers
  - Bouffant Caps
  - Shoe Covers
  - Frocks

- **Bagging Material**
  - Static Shield Bags
  - Poly Bags
    - Clear
    - Pink

- **Film**
  - LDPE Film
  - Anti-static Film
  - Silver Film
  - Pink Poly anti-static film
  - Packaging Film
Work Instructions

Work Instructions provide a standard method for analysis of consumables

- **541-WI-5330.1.20**
  - Gravimetric Determination of Non-Volatile Residue in Clean Room Wipes and Swabs

- **541-WI-5330.1.21**
  - Gravimetric Determination and Contact Transfer of Non-volatile Residue (NVR) in Cleanroom Glove Samples

- **541-WI-5330.1.27**
  - Determination of Extractable Nonvolatile residue and Contact Transfer Residue from Cleanroom Garments

- **541-WI-8072.1.7**
  - Determination of Extractable Nonvolatile Residue (NVR) and Contact Transfer NVR from Bagging and Polymeric Films
Gravimetric Determination Method

**Gloves & Wipes**
- Extracted in 300ml solvent in an ultrasonic bath at 35°C
  - Solvent is typically isopropyl alcohol, acetone, or hexane
  - Gloves are extracted for 15 minutes
  - Wipes are extracted for 30 minutes
- Three trials
  - Gloves - Each trial shall consist of two pieces of glove no less than 5cmx5cm and shall not contain any material from the fingers or cuff
  - Wipes - Total area of tested wipes in each trial shall be no less than 1ft²
- Solvent is evaporated down to ~30ml in a RapidVap Evaporator using a nitrogen purge
- Remaining solvent is evaporated to dryness in clean, preweighed aluminum pans
- Residue is analyzed with Fourier Transform Infrared (FTIR) Spectroscopy

**Swabs**
- Extracted in 40ml solvent on a hot plate for 30 minutes
  - Solvent is typically isopropyl alcohol, acetone, or hexane
- Three trials
  - Each trial includes the equivalent of 5 cotton swabs
- Solvent is evaporated to dryness in clean, preweighed aluminum pans
- Residue is analyzed with Fourier Transform Infrared (FTIR) Spectroscopy
Gravimetric Determination Method

- Garments & Miscellaneous Items
  - Extracted in ~30-40ml isopropyl alcohol for 30 minutes
  - Three trials, each consisting of an 8.7cm diameter piece of garment
  - Solvent is evaporated to dryness in clean, preweighed aluminum pans
  - Residue is analyzed with Fourier Transform Infrared (FTIR) Spectroscopy

- Bagging Material & Film
  - Interior of bag or surface of film is rinsed with ~50-70ml of isopropyl alcohol
  - Three trials
  - Solvent is evaporated to dryness in clean, preweighed aluminum pans
  - Residue is analyzed with Fourier Transform Infrared (FTIR) Spectroscopy
Contact Transfer Method

- **Gloves**
  - A pre-cleaned, verified aluminum plate, 10cmx10cm minimum is inserted into a glove that has been turned inside out
  - Three trials
  - The inside out gloves with inserted aluminum plates are wrapped in foil and placed in a hydraulic press with 1000kg of weight for 90 minutes
  - The plates are removed and rinsed with ~30ml isopropyl alcohol on both the front & back
    - Solvent is collected in clean, preweighed aluminum pans and evaporated to dryness
  - Residue is analyzed with Fourier Transform Infrared (FTIR) Spectroscopy

- **Garments & Miscellaneous Items**
  - 6”x6” section of garment is sandwiched between two pre-cleaned pieces of aluminum foil (foil should be a minimum of 7”x7”)
  - A 6”x6”x1/4” stainless steel plate weighing approximately 5lb is placed on the foil/garment sample for 24 hours
  - Three trials
  - Rinse foil pieces which were in contact with garment sample with ~50ml of a 50/50 mixture of isopropyl alcohol and chloroform
    - Solvent for each trial shall be collected in a clean, preweighed aluminum pan
  - Solvent is evaporated to dryness and residue is analyzed with Fourier Transform Infrared (FTIR) Spectroscopy

- **Bagging Material & Film**
  - A piece of aluminum foil large enough to fit inside the bag or to be sandwiched between the film (preferred area is (1ft²) shall be rinsed with spectroscopic grade chloroform
  - Once dry, foil is placed inside bag/sandwiched between film and the bag/film is sealed
  - The sample sits at room temperature for 10 days
  - After 10 days, the foil is removed and rinsed with ~50-70ml isopropyl alcohol into a clean, preweighed aluminum pans and evaporated to dryness
  - Residue is analyzed with Fourier Transform Infrared (FTIR) Spectroscopy
  - A 24 hour test may be performed if enough bagging material is provided
NVR Calculation Equations

\[ S_f - S_i = S_t \]
Calculation of Total Sample Weight (g)

\[ S_t - S_b = S_a \]
Calculation of Adjusted Sample Weight (g)

\[ \frac{S_a}{A} \times \frac{10^3 \mu g}{1 mg} = NVR \]
Calculation of NVR for gloves, wipes, bags, film (µg/cm²)

\[ \frac{S_a}{\# \text{Swabs}} = NVR \]
Calculation of NVR for Swabs (mg/swab)

\[ \frac{S_a}{\pi \left( \frac{d}{2} \right)^2} \times \frac{10^3 \mu g}{1 mg} = NVR \]
Calculation of NVR for garments (µg/cm²)

<table>
<thead>
<tr>
<th>A = Area (cm²)</th>
<th>S_b = Blank Sample Weight (mg)</th>
<th>S_t = Total Sample Weight (mg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>d = Diameter (cm)</td>
<td>S_i = Sample Post Weight (mg)</td>
<td></td>
</tr>
<tr>
<td>S_a = Adjusted Sample Weight (mg)</td>
<td>S_i = Sample Initial Weight (mg)</td>
<td></td>
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</tbody>
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Consumables Database

- Provides a way to track lot to lot variation

- Allows a comparison of the performance of different types of consumables using the same procedure

- Provides a useful resource for projects

- A ‘living’ document, continuously updated with new data
Testing for Consumables Database

- **Consumable Item**
  - New Item
  - Old Item (different lot)

- **Test for Acceptability**
  - Criteria determined by Codes 541 and 546 for general usage
  - Add results to consumables database
  - If material is purchased for general use on Center, Code 541 works to develop a fast QA inspection test that can be used as an incoming screening tool by the 549 contractor lab

- **Periodic Retest**
  - Periodic retest of items is necessary to catch any process changes
Recommendations

- Gloves
  - Polyethylene gloves are the first choice for use when solvent exposure is a possibility
  - Some nitrile gloves are a suitable replacement for latex gloves, and often have lower NVR levels than latex gloves
    - Nitrile gloves intended to be used as a replacement for latex gloves should be tested prior to lab use
    - Nitrile gloves with a powder coating on the interior are not acceptable

- Wipes
  - Wipes should be extracted prior to use to reduce the amount of NVR
Questions