

<b>BASF Corporation</b>	<b>Title:</b> <i>Shake Test.</i>		
<b>Individual Unit</b>	<b>Function:</b> <i>For use to check cleanliness of totes and/or certification of other equipment, and cleanliness of critical raw materials such as filter bags, gloves, rags.</i>		
<b>Procedure</b>	<b>SAP-QM:</b> MBP22A23	<b>Page:</b> 1 of 12	
<b>N-ECN</b>	<b>Reviewed:</b> 03/20/12	<b>Effective:</b>	<b>Supersedes:</b> 11/01/06
<b>Legacy TM TC083</b>	<b>Technical Owner:</b> Jim Laugal	<b>Approver: Technical Managers</b> Jim Laugal, Jessica Rangel	

**PROCEDURE CHANGES:**

**08/30/2005:** Corrected error in 9.1.4. "If the solvent sample is transparent, has no color, and/or particulate materials, proceed to 9.1.6 (rather than 9.4 as written in the previous version)".

**03/09/2005:** Modified test method to include its use for certification of equipment and certification of critical raw materials, including filter bags, gloves, rags, etc.

**02/16/2006:** Clarified use of test method to include testing Solvent and Solvent Mixtures. Re-approval of method is not required.

**11/01/2006:** Modified 9.1.1 to change the time of the sample collection at the tote cleaner.

**03/20/2012:** Modified to use an 8-oz glass jar as well as a vial for evaluation of totes

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**1. PURPOSE:**

1.1. This procedure defines a qualitative method for detection of contamination in 2-butoxy-ethanol (butyl cellosolve) final rinse for totes, solvents and solvent mixtures, and for detection of contamination present in other solvents used to clean equipment and/or used in extractions of non-raw materials such as filter bags, gloves, gaskets, rags, etc.

**2. SCOPE:**

2.1. This procedure applies to testing of totes cleaned by Tote Cleaning suppliers of BASF, NAFTA region.

2.2. This method can detect the presence of silicone oil contamination down to a concentration of 0.3 ppm and the presence of residual coating materials such as clearcoats, melamines, basecoats, primers and electrocoat present in concentrations down to 0.2%.

2.3. This method can be used to certify/recertify equipment for use in paint manufacturing.

2.4. This method can be used to detect the presence of silicone oil in critical non-raw materials, including filter bags, gloves, rags, etc.

**3. DEFINITIONS AND TERMS:**

3.1. Control sample: vial or 8-oz glass jar containing clean 2-butoxy-ethanol (butyl cellosolve) or vial or 8-oz glass jar containing clean solvent as used for the test sample.

3.2. Test sample: vial or 8-oz glass jar containing 2-butoxy-ethanol (butyl cellosolve) to be tested (collected as described in 9.2) or vial or 8-oz glass jar containing the solvent to be tested.

3.3. Solvent from extraction of non-raw materials: solvent removed from a clean container in which about one inch square of a filter bag, glove, etc. was soaked for 3-5 minutes.

**4. REFERENCED DOCUMENTS:**

4.1. NA

**5. SUMMARY OF METHOD:**

**5.1. For tote use:**

5.1.1. A vial containing 10 - 15 ml of 2-butoxy-ethanol (butyl cellosolve) or an 8-oz glass jar containing 4-5 oz is shaken along with a vial or 8-oz glass jar containing the same amount of a known good 2-butoxy-ethanol (butyl cellosolve) solvent (control sample). The rate of disappearance of bubbles in the two containers is observed and compared.

**5.2. For all other uses:**

5.2.1. A vial containing 10 - 15 ml of the solvent of choice is shaken along with a vial containing about the same amounts of a known good solvent (control sample). The rate of disappearance of bubbles in the two vials is observed and compared.

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## 6. REQUIRED APPARATUS:

- 6.1. Wheaton 25 ml disposable scintillation vials with foil lined caps (VWR #66021-453) or equivalent.
- 6.2. Control vial (same type of vial as above) containing clean 2-butoxy-ethanol (butyl cellosolve) or
- 6.3. For totes, can use an 8-oz glass jar that is approved on the Critical Non-Raw Material Database (All-Pak/Qor-Pak GLC01732/GLC016 series)

## 7. Equipment Specifications

- 7.1. NA

## 8. REAGENTS AND MATERIALS:

- 8.1. For tote cleaning operations: 2-butoxy-ethanol (butyl cellosolve), practical grade.
- 8.2. For other than tote cleaning operations: solvent of choice, practical grade.

## 9. PROCEDURE:

### 9.1. For tote use, solvent, or solvent mixture to be tested:

- 9.1.1. Solvent Collection
  - A. For Totes: Collect 2-butoxy-ethanol (butyl cellosolve) by fully opening the bottom valve of the tote after a minimum of 10 seconds after the final rinse step was stopped.
  - B. For Solvent and Solvent mixtures: Collect the appropriate amount of the material to be tested
- 9.1.2. Add approximately 10-15 ml of this solvent/solvent mixture in the test vial or add 4-5 oz of solvent/solvent mixture to an 8-oz glass jar.
- 9.1.3. Visually inspect the solvent/solvent mixture for color, clarity and/or particulate materials.
- 9.1.4. If the solvent/solvent mixture sample is transparent, has no color, and no particulate materials, proceed to 9.1.6.
- 9.1.5. If the solvent/solvent mixture is not clear, has a color, and/or particulate materials, the tote shall be sent back for re-cleaning or the solvent/solvent mixture is rejected.
- 9.1.6. Hold both the test container and the control container in one hand and shake them vigorously (5-10 shakes).
- 9.1.7. Immediately after shaking, monitor and compare the bubble dissipation in the two vials.

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9.1.8. Repeat steps 9.1.6 to 9.1.7 two times.

**9.2. For all other uses:**

- 9.2.1. Cut approximately 1 inch square of the critical raw material to be tested (choose a stitched area where applicable).
- 9.2.2. Add approximately 10-15 ml of a solvent in the test vial and place the piece to be tested.
- 9.2.3. Soak for 3-5 minutes.
- 9.2.4. By using tweezers, take testing piece out of the vial.
- 9.2.5. Hold both the test vial and the control vial in one hand and shake them vigorously (5-10 shakes).
- 9.2.6. Immediately after shaking, monitor and compare the bubble dissipation in the two vials.

*Repeat steps 9.2.5 to 9.2.6 two times.*

**10. EVALUATION AND CALCULATIONS:**

10.1. If the bubble dissipation rate in the test sample is the same as in the control sample, the tote, equipment and/or raw material passed the inspection (Pass).

Note: Test vials, which contained a sample that passed this test, may be recycled. Empty the contents of the vial into the waste and cap the vial immediately to avoid contamination. Visually evaluate each vial prior to re-use to assure that the aluminum backing is not damaged. Vials with damaged aluminum backing should be disposed off.

10.2. If the bubble dissipation rate in the test sample is slower than in the control sample the tote, equipment and/or raw material failed the inspection (Failed) and shall be sent back for re-cleaning, recertification and/or rejected.

Note: Bubbles of all sizes and shapes should be considered when evaluating the sample.

Follow and compare the bubbles formed on top of the two vials, which are the most visible when compared to bubbles which may be formed throughout the entire solvent content.

**11. RESULTS:**

**11.1. For tote use:**

11.1.1. Pass – clear, no color, no particles and equal bubble dissipation rate in the test sample and control sample.

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11.1.2. Fail – color, opaque, particles, and/or slower bubble dissipation in the test sample versus the control sample.

**11.2. For all other uses:**

11.2.1. Pass –equal bubble dissipation rate in the test sample and control sample.

11.2.2. Fail –slower bubble dissipation in the test sample versus the control sample.

12. **MSA:** Results attached.

**13. RESPONSIBILITY:**

13.1. Associates performing the Test Method are responsible for following the procedure as well as obtaining documented training for any necessary equipment included in the Test Method.

**14. SAFETY:**

14.1. All site safety practices should be adhered to when performing this Test Method.

**15. RECORD RETENTION**

15.1. Records are to be maintained for 6 months at the tote cleaning facilities.

**16. APPENDICES**

16.1. NA

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**MODIFICACIONES EN EL PROCEDIMIENTO:**

**08/30/2005:** Error corregido en el punto 9.1.4. "Si la muestra de solvente es transparente, incolora, y/o no presenta partículas de materia extraña, proceda al 9.1.6" (en lugar del 9.4 como se especificaba en la versión anterior).

**03/09/2005:** Modificación del método de evaluación para incluir la certificación de equipo y/o materiales no productivos incluyendo bolsas para filtrado, guantes, trapos, etc.

**02/16/2006:** Aclaración del método de evaluación para incluir pruebas de solvente y mezclas de solvente. La re-aprobación del método no es requerida.

**11/01/2006:** Modificación del punto 9.1.1 para cambiar el tiempo de retención de muestras del lavado de totes.

**03/20/2012:** Modificación para el uso de un frasco de vidrio de 8 oz o un vial para la evaluación de totes.

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## 1. PROPOSITO:

**16.2.** Este procedimiento describe el método cualitativo para la detección de contaminación en el enjuague final de totes con 2-butoxi-etanol, otro solvente y/o mezclas de solventes, y la detección de contaminación presente en los solventes empleados en el lavado de equipo y/o de la extracción de materiales no productivos como bolsas para filtrar, guantes, juntas, trapos, etc.

## 2. ALCANCE:

- a. Este procedimiento aplica para la evaluación del lavado de totes por proveedores de BASF y región NAFTA.
- b. Este método detecta la presencia de contaminación por derivados de silicón menor a 0.3 ppm y la presencia de residuos de pintura como clearcoats, melaninas, bases, primers y electro recubrimientos presentes en concentraciones menores a 0.2 %.
- c. Este método puede ser utilizado para la certificación/re-certificación de equipo empleado para la manufactura de pintura.
- d. Este método puede ser utilizado para detectar la presencia de derivados de silicón en materiales no productivos incluyendo bolsas para filtrado, guantes, trapos, etc.

## 3. TERMINOS Y DEFINICIONES:

- a. Muestra de control: vial o frasco de vidrio de 8 oz con 2-butoxi-etanol limpio o en su defecto el solvente utilizado en la muestra de evaluación.
- b. Muestra de evaluación: vial o frasco de vidrio de 8 oz con 2-butoxi-etanol para evaluación (recolectado como se describe en el punto 9.2) o con el solvente a evaluar.
- c. Solvente de la extracción de materiales no productivos: solvente removido de un contenedor limpio, en el cual aprox. 1 pulg<sup>2</sup> de una bolsa para filtrar, un guante, etc. se remoja durante 3-5 minutos.

## 4. DOCUMENTOS DE REFERENCIA:

- a. NA

## 5. RESUMÉN DEL MÉTODO:

- a. **Para uso en totes:**
  - i. Un vial con 10-15 ml de 2-butoxi-etanol o un frasco de vidrio de 8 oz con 4-5 oz se agita junto con un vial o un frasco de vidrio de 8 oz con la misma cantidad de 2-butoxi-etanol estándar o limpio (muestra de control). El grado de disipación de las burbujas en los dos recipientes se observa y se compara.
- b. **Para otros usos:**

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- i. Un vial con 10-15 ml del solvente seleccionado se agita junto con un vial con aprox. la misma cantidad del solvente en condiciones estándar (muestra de control). El grado de disipación de burbujas en los dos recipientes se observa y se compara.

## 6. EQUIPO REQUERIDO:

- a. Viales desechables Wheaton de 25 ml con tapas forradas de papel aluminio (VWR #66021-453) o sus equivalentes.
- b. Viales de control (mismo tipo que las anteriores) con 2-butoxi-etanol limpio, ó
- c. Para totes, puede utilizar un frasco de vidrio de 8 oz, aprobado como material no productivo (All- Pak/Qor-Pak GLC01732/GLC016 series).

## 7. ESPECIFICACIONES DEL EQUIPO:

- a. NA

## 8. REACTIVOS Y MATERIALES:

- a. Para lavado de totes: 2-butoxi-etanol (butil celusolvente), grado práctico.
- b. Para otras funciones: solvente de su elección, grado practico.

## 9. PROCEDIMIENTO:

### a. Evaluación del solvente o mezcla de solventes para uso en totes:

- i. Recolección de solvente
  1. Para totes: recolecte 2-butoxi-etanol abriendo completamente la válvula en el fondo del tote mínimo 10 segundos después de terminada la última etapa de enjuague.
  2. Para solvente o mezcla de solventes: recolecte la cantidad apropiada del material a ser evaluado.
- ii. Agregue de 10-15 ml del solvente/mezcla de solventes en el vial de evaluación o agregue de 4-5 oz del solvente/mezcla de solventes a un frasco de vidrio de 8 oz.
- iii. Realice una inspección visual del solvente/mezcla de solventes para detectar color, claridad y/o partículas extrañas.
- iv. Si el solvente/mezcla de solventes es transparente, incolora, y no presenta partículas de materia extraña proceda al punto 9.1.6.



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- v. Si el solvente/mezcla de solventes no es clara, presenta color, y/o partículas de materia extraña, el tote deberá ser enviado de regreso para su re-lavado o el solvente/mezcla debe ser rechazada.
- vi. Sostenga ambos recipientes, el de control y el de evaluación, en una mano y agítelos vigorosamente (5-10 agitaciones).
- vii. Inmediatamente después de agitar, monitoree y compare la disipación de las burbujas en los dos viales.
- viii. Repita los pasos 9.1.6 y 9.1.7 dos veces más.

**b. Para otros usos:**

- i. Corte aprox. 1 pulg<sup>2</sup> del material no productivo a ser evaluado (elija un área representativa).
- ii. Agregue aprox. de 10-15 ml del solvente en el vial de evaluación y coloque la pieza del material.
- iii. Remoje la pieza de 3-5 minutos.
- iv. Utilizando pinzas, saque la pieza del vial.
- v. Sostenga ambos recipientes, el de control y el de evaluación, en una mano y agítelos vigorosamente (5-10 agitaciones).
- vi. Inmediatamente después de agitar, monitoree y compare la disipación de las burbujas en los dos viales.
- vii. Repita los pasos 9.2.5 y 9.2.6. dos veces más.

**10. EVALUACION Y CALCULOS:**

- a. Si el grado de disipación de burbujas en la muestra evaluada es el mismo que en la muestra de control, el tote, equipo y/o el material aprueban la inspección.  
Nota: Los viales de evaluación que contenían las muestras aprobadas pueden ser reciclados. Vacíe el contenido al desperdicio y tapé inmediatamente el vial para evitar su contaminación. Evalúe visualmente cada vial previo a su reuso para asegurar que el vial no este dañado. Los viales con dañados deberán ser desechados.
- b. Si el grado de disipación de burbujas en la muestra evaluada es más lento que en la muestra de control, el tote, equipo, y/o material no aprueban la inspección y deben ser enviados de regreso para su re-lavado, re-certificado y/o su rechazo.

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Nota: Las burbujas de todos los tamaños y formas deben ser consideradas cuando se evalúa la muestra.

Observe y compare las burbujas formadas en la superficie, las cuales son más visibles en comparación a las burbujas formadas en el interior del solvente.

## 11. RESULTADOS:

### a. Para uso en totes:

- i. Aprobado – claro, incoloro, sin partículas extrañas y/o grado de disipación de burbujas igual en ambas muestras, de control y de evaluación.
- ii. Rechazo – color, turbio, partículas y/o lenta disipación de burbujas en la muestra de evaluación en comparación con la muestra control.

### b. Para otros usos:

- i. Aprobado – grado de disipación de burbujas igual en ambas muestras, de control y de evaluación.
- ii. Rechazo – lenta disipación de burbujas en la muestra de evaluación en comparación con la muestra de control.

## 12. MSA: Resultados adjuntos.

## 13. RESPONSABILIDAD:

- a. Los colaboradores que realicen el método de evaluación son responsables de seguir el procedimiento así como de tener capacitación documentada para cualquier equipo utilizado en el método de evaluación.

## 14. SEGURIDAD:

- a. Todas las prácticas de seguridad del sitio deben ser consideradas cuando se lleve a cabo el método de evaluación.

## 15. REGISTRO DE RETENCION:

- a. Los registros se deben mantener por 6 meses en las instalaciones de lavado de totes.

## 16. APENDICES

- a. NA

# Measurement System Analysis

TC-083

Crater Shake Test Method

Method Date: 6/04/2003

MSA Date: 05/14/03

## Recommendations

**Spec Width:** N/A

**Repeatability:** When a qualified associate performs this test repeatedly on the same sample, the result will agree 96% of the time.

**Reproducibility:** When two qualified associates perform this test on the same sample, the result will agree 96% of the time.

**Improvement Opportunities:** None Noted.

## Basis

The spec width, repeatability and reproducibility statements were based on a MSA format that utilized three operators and fifty samples with three replicate measurements. Associates participated from Windsor, Cygnet and Dana. Results were analyzed using the attribute method.

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# Measurement System Analysis

TC-083

Crater Shake Test Method

Method Date: 6/04/2003

MSA Date: 05/14/03

## Summary of Results

### Within Appraiser - Repeatability Assessment

#### Assessment Agreement

Appraiser #	Inspected #	Matched #	Percent (%)	95.0% CI
1	50	50	100.0	( 94.2, 100.0)
2	50	50	100.0	( 94.2, 100.0)
3	50	48	96.0	( 86.3, 99.5)

# Matched: Appraiser agrees with him/herself across trials.

### Between Appraisers – Reproducibility Assessment

#### Assessment Agreement

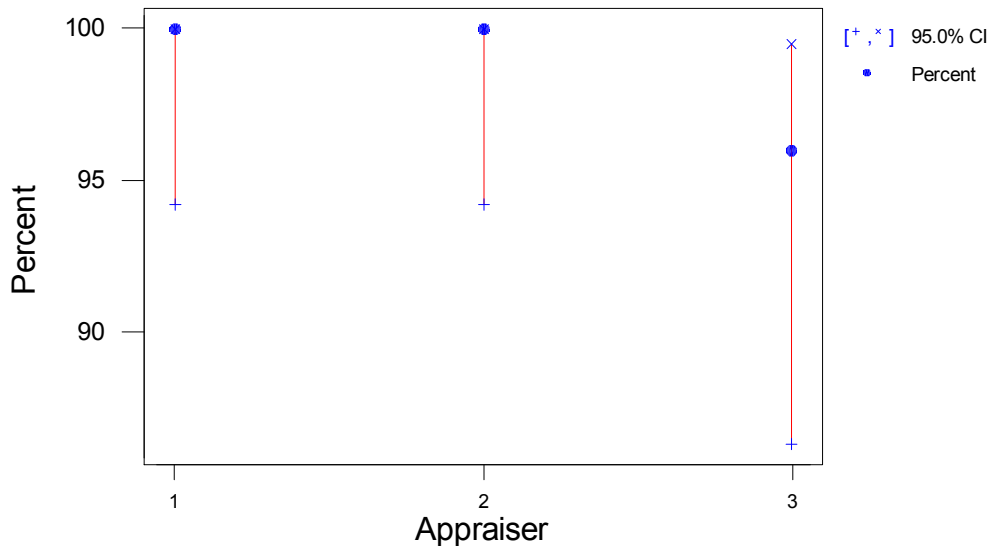
# Inspected	# Matched	Percent (%)	95.0% CI
50	48	96.0	( 86.3, 99.5)

# Matched: All appraisers' assessments agree with each other.

### Assessment Agreement

Date of study: 05/14/03  
 Reported by: D. Kessel  
 Name of product: Crater Shake Test

#### Within Appraiser



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