



# Container Closure Integrity: Regulations, Test Methods, Application

## Seal Quality Test Methods

### Instructors

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*Vienna Austria, 9-10 November 2017*

## Seal Quality Tests

- Tests used to **characterize and monitor** the **quality and consistency** of a **seal parameter** providing some assurance of the package's ability to remain integral
- **Parameters monitored** may include
  - Seal quality or characteristic
  - Package materials
  - Package components
  - Sealing process
- **SQT are not leak tests**

## Seal Quality Tests

- **Passing SQT  $\neq$  leak-free package**
- Examples
  - **Heat seal strength**
    - A pouch with a strong heat seal peel force may have a pinhole in the pouch face
  - **Closure application force**
    - A well-closed capped bottle may leak due to a scratch on the bottle finish

## Seal Quality Tests

- **Failing SQT = package integrity risk**
- Examples
  - **Heat seal strength**
    - A pouch with a weak heat seal peel force is more likely to leak during product life cycle
  - **Closure application force**
    - A loosely capped bottle may leak during shipping
- **SQT and package leak tests work together to ensure package quality**

## Seal Quality Tests

- Tests included
  - Closure application and removal force
  - Package burst
  - Package seal strength
  - Residual seal force
  - Airborne ultrasound
  - Vision inspection (including x-ray)

## Seal quality tests included

- Described in relevant peer-reviewed publications
- ASTM test with supportive precision and bias data
- Significant variation w/in technologies may be seen among vendors
- Other methods not included may be acceptable

## Closure application and removal force

- **Description**

- **Application force:** The torque required to apply a screw-cap onto a threaded closure
- **Removal force:** The torque required to initiate screw-cap removal

- **Correlation to package integrity**

- Well-closed caps are less likely to back-off during product life-cycle and leak
- Over-closed caps may distort components causing damage and leakage

**Reference :** Numerous ASTM methods for various closure types

## Closure application and removal force

- **Application**

- Test results are impacted by
  - Instrument automation (automatic, not manual, recommended)
  - Tooling to lock bottle/cap in position (use recommended)
  - Testing speed (controlled, constant speed recommended)

- **Examples of reported usage**

- Ophthalmic solution dropper tip bottles with screw-caps



## Closure application and removal force



Image from [www.suretorque.eu.com](http://www.suretorque.eu.com), accessed May 7, 2014

## Package burst

- **Description:** The pressure at which a package ruptures during exposure to increasingly greater internal air pressure forces
- **Correlation to package integrity**
  - Low burst strength packages
    - More likely to rupture during product life-cycle
    - Indicative of sealing process failure or material changes
  - The mode of rupture can indicate seal failure risks
    - E.g., seal bond rupture vs. seal edge rupture

**Reference : ASTM F2054, F1140**

## Package burst

- **Application**

- A restraining plate is used to limit test sample expansion and ensure uniform stress is applied to seal areas
- No restraining plate is used to understand sample strength if exposed to typical conditions of sterilization, shipping, etc.
- Test results are impacted by
  - Air pressure rise rate
  - Pressure sensing mechanisms
  - Tooling dimensions (including restraining plate gap height)

- **Examples of reported usage**

- Flexible pouches or bags

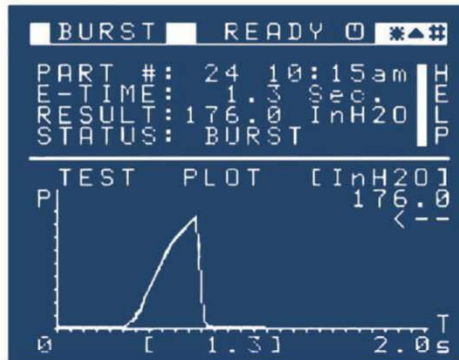


## Burst test

**TM Electronics  
BT-1000  
package tester**



**Optional restraining plate**



The BT-1000 graphically shows the characteristic burst curve for each test.



Accumulating test data are shown in histogram form to give a visual overview of your process.



Ongoing test data are shown on a statistical control chart for tighter process control.

*Graphical and statistical analysis screens from the TM Electronics BT-1000 Package Tester*

## Package seal strength

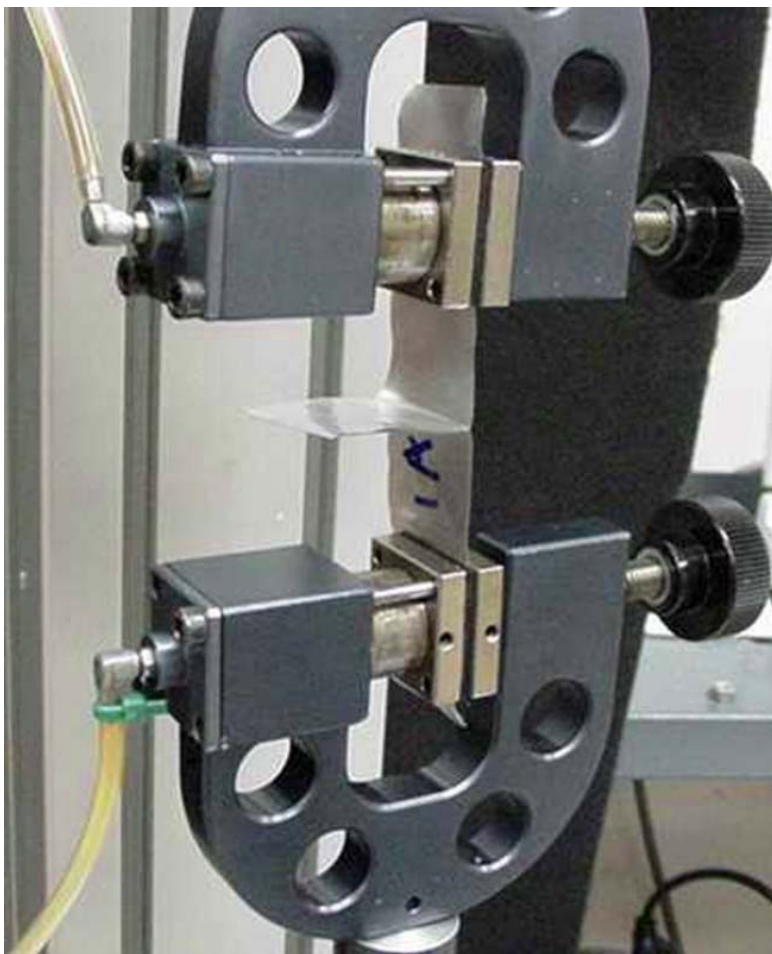
aka Peel test

- **Description:** The forces required to peel apart two bonded surfaces, including maximum and average force
- **Correlation to package integrity**
  - Low seal strength packages
    - More likely to open during product life-cycle
    - Indicative of sealing process failure or material changes
  - The mode of rupture can indicate seal failure risks
    - E.g., seal bond rupture vs. seal edge rupture

**Reference : ASTM F88**

## Package seal strength aka Peel test

- **Application**
  - Performed using a universal stress-strain instrument in the extension force mode
  - Special tooling required to position/hold test sample.
  - Test results are impacted by
    - Tooling design
    - Peel angle
    - Pull direction
    - Pull speed
- **Examples of reported usage**
  - Flexible bags or pouches
  - Lidded trays

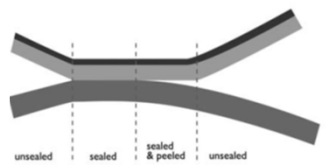


**ASTM F88/F88M – 09  
Standard Test Method  
for Seal Strength of  
Flexible Barrier  
Materials**

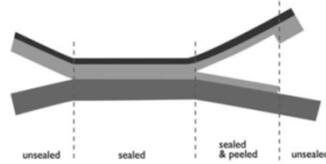
**Image from  
[www.testresources.net](http://www.testresources.net)  
Accessed May 7, 2014**



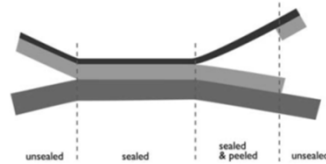
## SEAL SEPARATION MODES



ADHESIVE PEEL



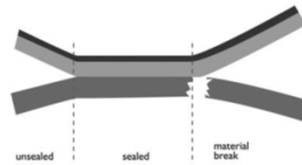
COHESIVE PEEL



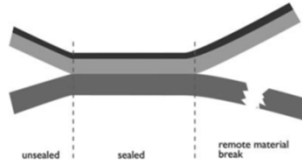
DELAMINATION

## INTERFERENCES

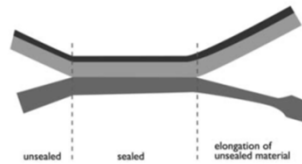
Though the diagrams show only one web being affected, it is possible for either or both webs to partially or fully exhibit interferences. Delamination, when not a designed seal separation mode, is an interference.



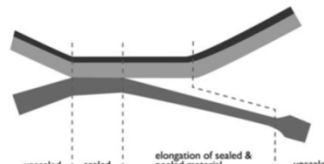
MATERIAL BREAK



MATERIAL BREAK (REMOTE)



MATERIAL ELONGATION



PEEL WITH ELONGATION

## ASTM F88/F88M – 09 Standard Test Method for Seal Strength of Flexible Barrier Materials

Examples of seal  
separation modes and  
interferences

## Residual seal force

- **Description:** An indirect measure of the compressive force (lb-f or newton) exerted by an elastomeric closure onto a parenteral vial finish post package assembly (capping)
- **Correlation to package integrity**
  - Low RSF
    - Loosely capped vial more likely to leak during product life-cycle
  - High RSF
    - Overly compressed vial more likely to leak due to vial scoring or closure/seal distortion
  - Low or High RSF
    - Indicative of possible sealing process failure
    - Indicative of possible component changes

## Residual seal force

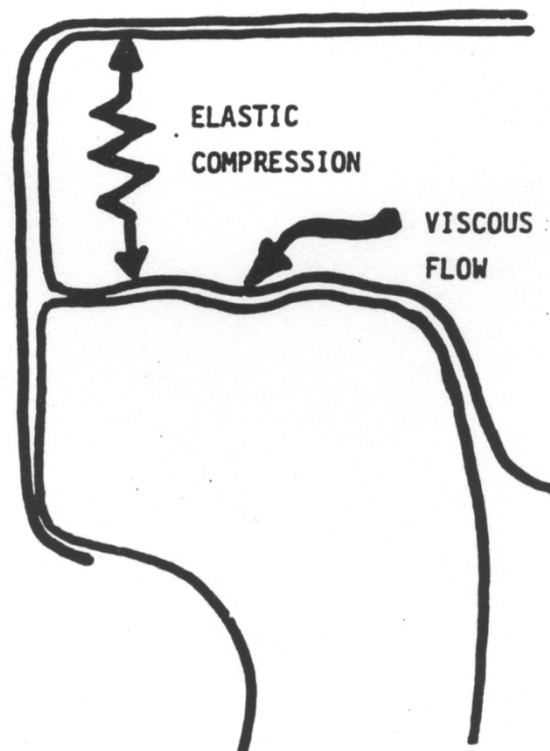
- **Application**

- Performed using a universal stress-strain instrument in the compression force mode. Test sample top is fitted with a cap anvil tool
- RSF is calculated by the stress-strain response curve 2<sup>nd</sup> derivative
- Supplier: Genesis Packaging Technologies
- Test results are impacted by
  - Test sample flip cap (no cap recommended)
  - Cap anvil tooling design
  - Test speed
  - Mode of calculating RSF

- **Examples of reported usage**

- Vials, bottles, cartridges with stoppers having a land-seal flange, closed with aluminum caps

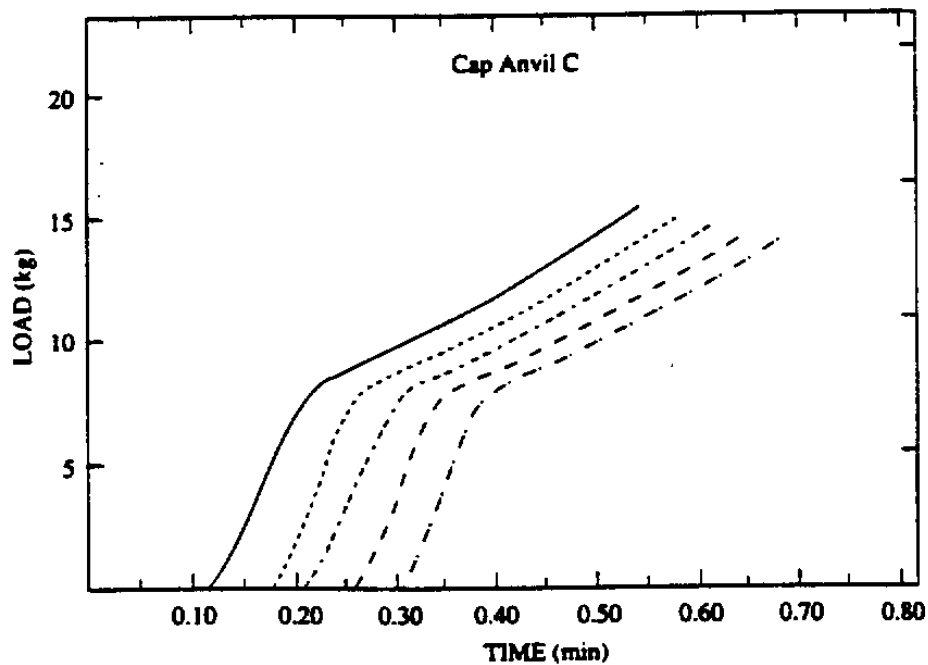
## Elastomeric Compression Seal



Closures are both...

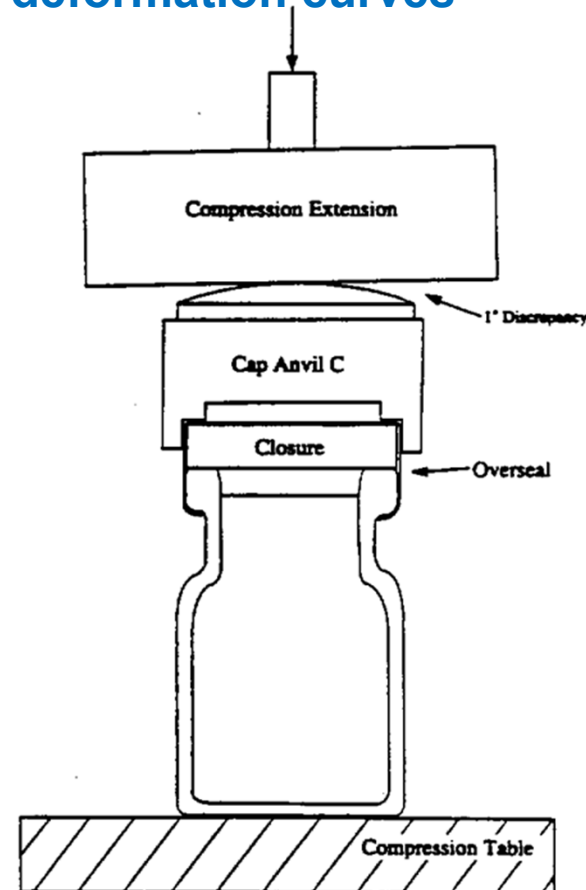
- **VISCOUS** for flow into the vial finish
- **ELASTIC** for continuous pressure against the finish

## Universal stress-strain tester



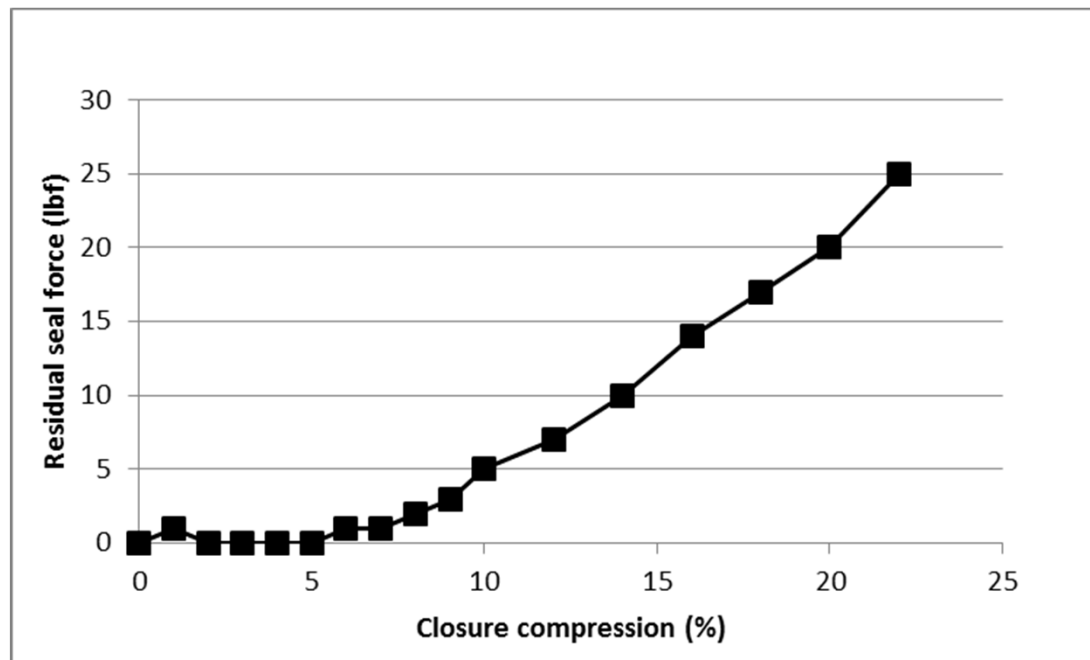
**RSF:** Stress at which further strain results in a shift in stress response curve and increased elastomer viscous flow.

## Stress deformation curves



J. Ludwig, et al, J Parenteral Sci & Technol, 47, 5, 1993, p. 211, and 49, 5, 1995, p. 253

## Typical RSF vs. % Stopper compression correlation Glass vial with elastomeric stopper



Fictional data for illustration only



## Residual seal force test



Test sample with cap anvil

Genesis Packaging Technologies  
Residual Seal Force Tester

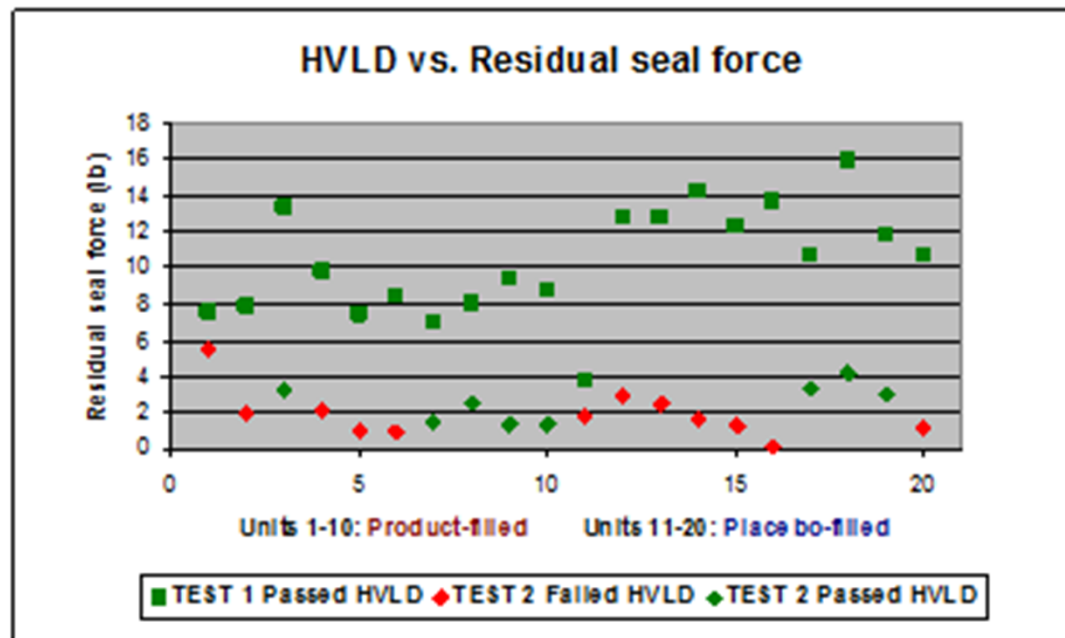
PDA PSIG meeting, Mar 2010, Orlando, FL

33

S. Orosz, D. Guazzo, Glass vial finish defects - Leak detection and product risk assessment, PSIG session of the PDA Annual Meeting, Orlando, FL, March 16, 2010



## Leak detection vs. Residual seal force



PDA PSIG meeting, Mar 2010, Orlando, FL

47

S. Orosz, D. Guazzo, Glass vial finish defects - Leak detection and product risk assessment, PSIG session of the PDA Annual Meeting, Orlando, FL, March 16, 2010





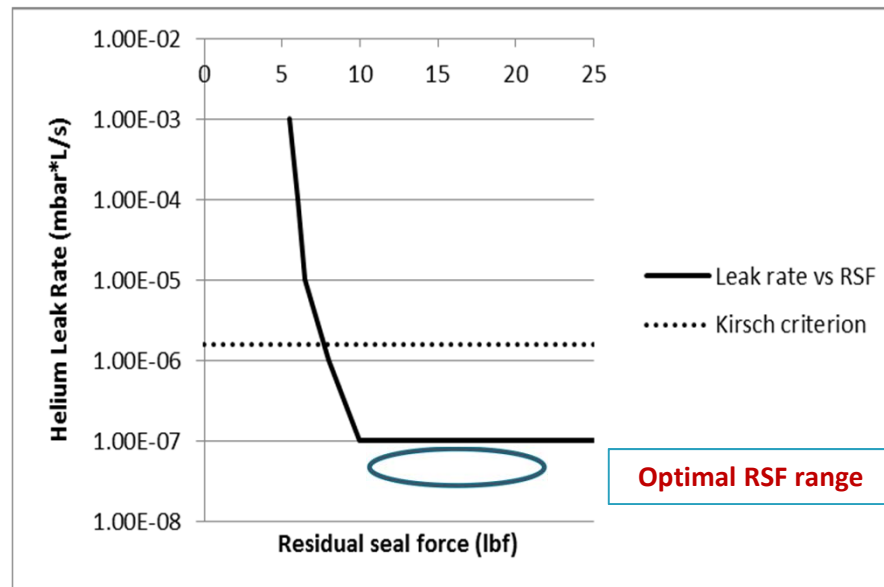
## Leak detection vs. Residual seal force



RSF 13.7 lb  
**Passed** HVLD

RSF 1.5 lb  
**Failed** HVLD

## Typical helium leak rate as a function of residual seal force Glass vials with elastomeric stoppers



Leak rate declines as RSF increases,  
reaching a steady state at optimal RSF range

**RSF too low risks leakage**

**RSF too high risks vial breakage, closure distortion**

## Airborne ultrasound

- **Description:** An ultrasound signal is passed through the sealed area of a test sample. The magnitude of the transmitted signal through the seal is compared to that through so-called 'good' seals.
- **Correlation to package integrity**
  - Low signal
    - Loosely bonded seal more likely to leak during product life-cycle
    - May be indicative of product inclusion in seal
  - High signal
    - May be indicative of damage to bonded surfaces, materials
  - Low or High signal
    - Indicative of possible sealing process failure
    - Indicative of possible component changes

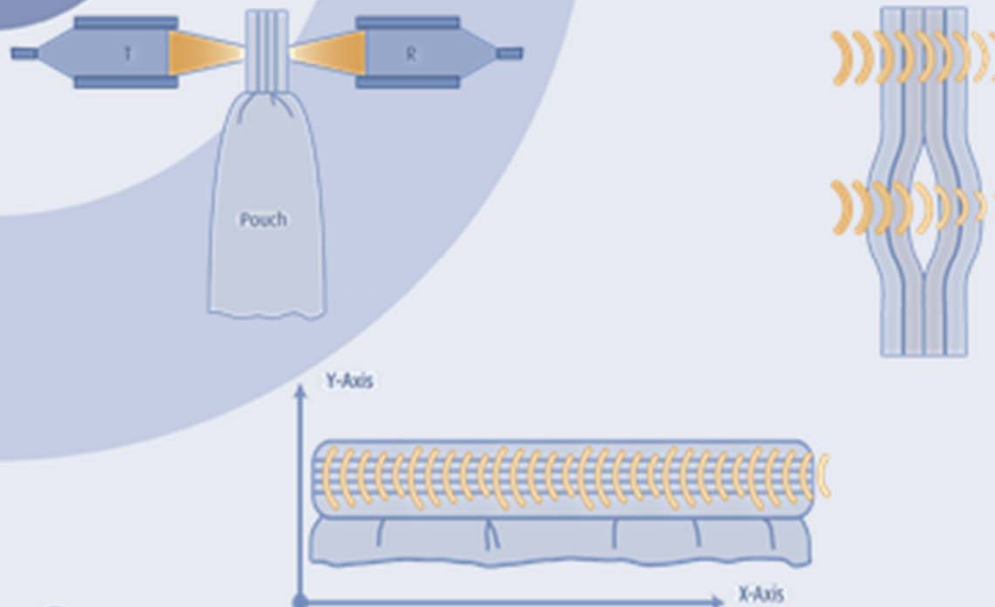
**Reference : ASTM F3004**

## Airborne ultrasound

- **Application**
  - This non-contact test is performed using an airborne ultrasound instrument equipped with
    - A transducer to provide US signal
    - Fixturing to hold and move the test sample
    - A detection transducer to capture intensity of transmitted US signal
    - Test sample is scanned in either a single x-y linear mode or a full surface x-y-z scanning mode
  - Supplier: Packaging Technologies & Inspection
  - Test results are impacted by
    - Air gap between sample and transducers
    - US signal strength
    - Scanning speed
    - Test sample material of construction and surface topography
- **Examples of reported usage**
  - Pouches, bags
  - Blister packaging

## ASTM Airborne Ultrasound Integrity Test

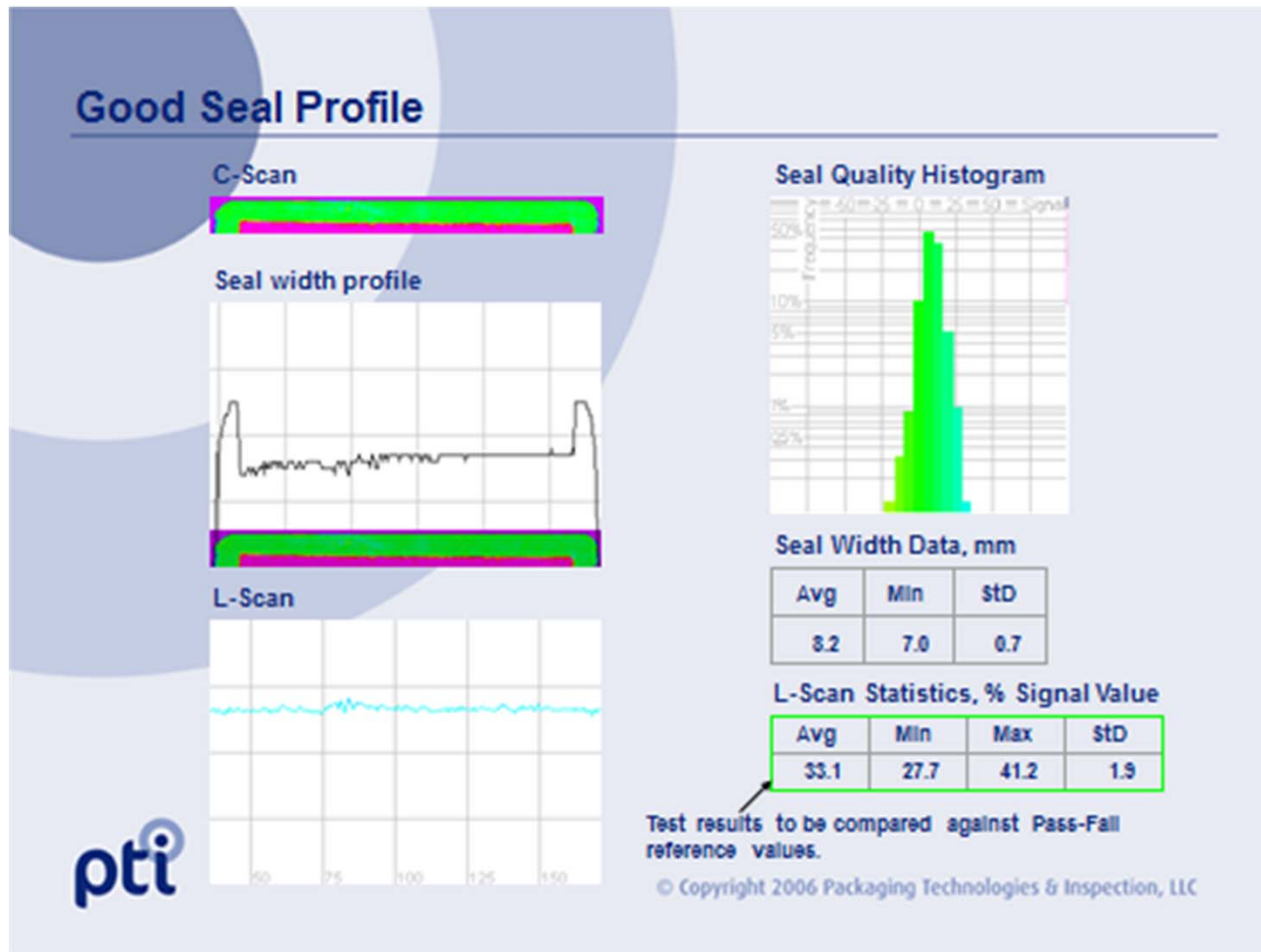
Ultrasonic signal is transmitted along the X-axis through seal and signal is recorded.



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© Copyright 2006 Packaging Technologies & Inspection, LLC

H. Wolf, D. Guazzo, ASTM Standard Test Method for Nondestructive Airborne Ultrasound Seal Integrity Test for Flexible Packages, ASTM F02 Committee meeting, April 18, 2007



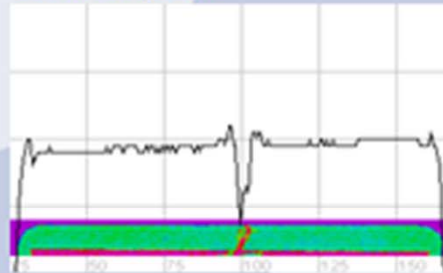
H. Wolf, D. Guazzo, ASTM Standard Test Method for Nondestructive Airborne Ultrasound Seal Integrity Test for Flexible Packages, ASTM F02 Committee meeting, April 18, 2007

## Defective Seal Profile (Channel Leak)

C-Scan



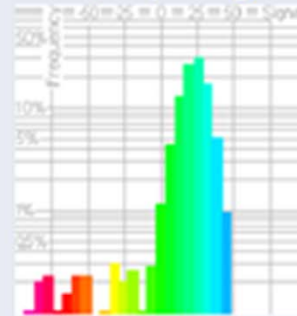
Seal width profile



L-Scan



Seal Quality Histogram



Seal Width Data, mm

Avg	Min	stD
9.4	3.5	1.0

L-Scan Statistics, % Signal Value

Avg	Min	Max	stD
1.7	-80.5	10.0	8.0

Low average and minimum, with high standard deviation.

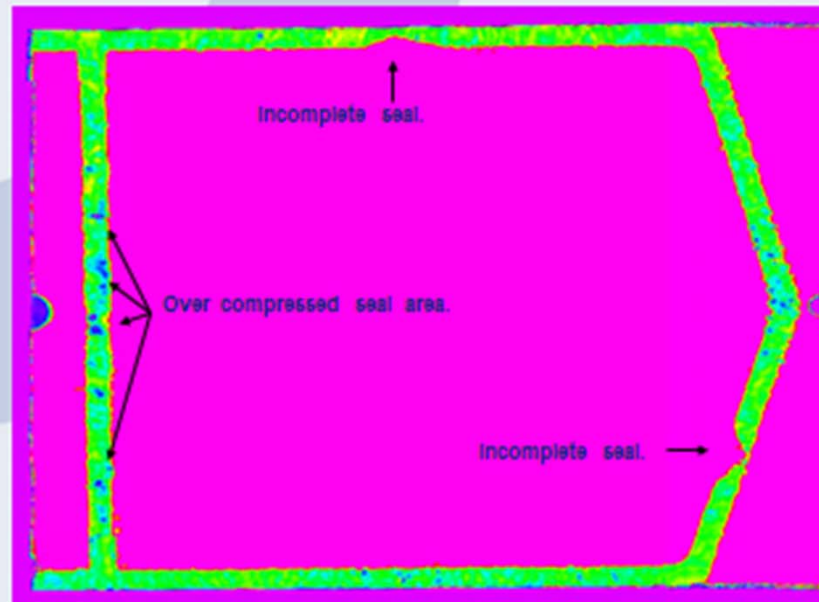
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H. Wolf, D. Guazzo, ASTM Standard Test Method for Nondestructive Airborne Ultrasound Seal Integrity Test for Flexible Packages, ASTM F02 Committee meeting, April 18, 2007

## Pouch Analysis Examples

### Tyvek®/Poly Sample



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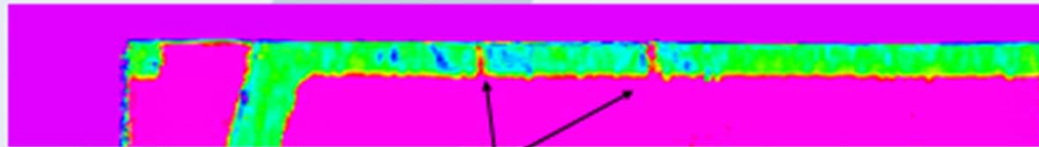
H. Wolf, D. Guazzo, ASTM Standard Test Method for Nondestructive Airborne Ultrasound Seal Integrity Test for Flexible Packages, ASTM F02 Committee meeting, April 18, 2007



## Pouch Analysis Examples

### Sample of Folded Tyvek®

#### Tyvek®/Poly Sample

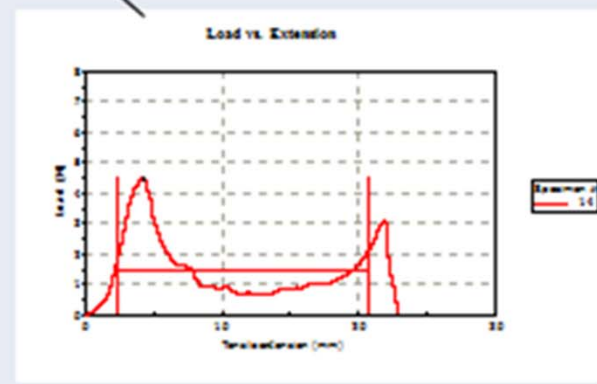
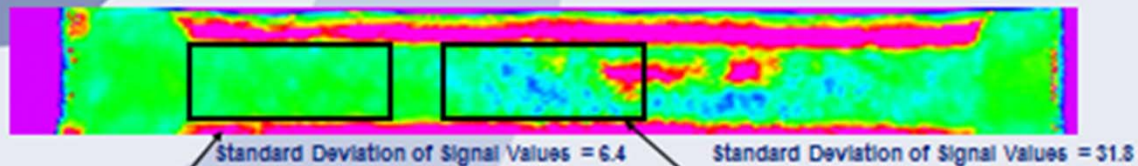


Two folds in Tyvek® seal

Folding a Tyvek® seal can lead to the separation of Tyvek® fibers. Seal-Scan™ technology is sensitive to the separation of Tyvek® fibers and is capable of detecting the folded areas. These areas could be potential defects, putting the product contents at risk.

## Seal-Scan™ vs. Peel Strength Test

Tyvek®/Poly Seal



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H. Wolf, D. Guazzo, ASTM Standard Test Method for Nondestructive Airborne Ultrasound Seal Integrity Test for Flexible Packages, ASTM F02 Committee meeting, April 18, 2007



PTI Seal Scan

## Vision and X-ray inspection

- **Description:**

- **Vision systems** are used to inspect test samples for visibly evident dimensional nonconformities, assembly mishaps, and package or component defects
- **X-ray** is used to detect those nonconformities hidden from view

- **Correlation to package integrity**

- May or may not be indicative of package leakage
  - Depends on the type and extent of nonconformity

## Vision and X-ray inspection

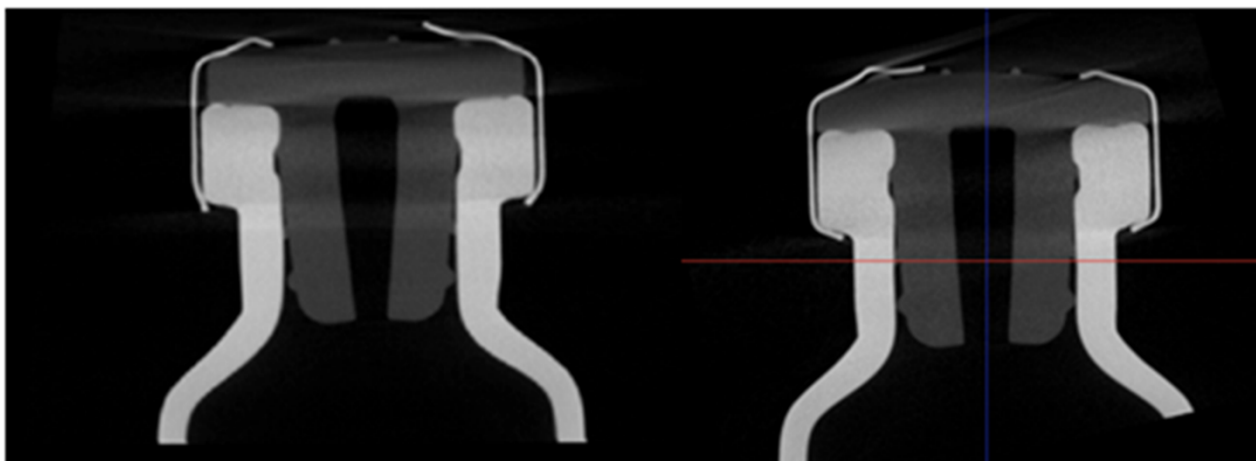
- **Application**

- A wide variety of inspection systems are available
- Test results are impacted by
  - Instrumentation capabilities
  - Test sample inspection angle, design, size, material of construction, contents
  - Inspection speed

- **Examples of reported usage**

- Vision: All product-package types
- X-ray: vials, syringes

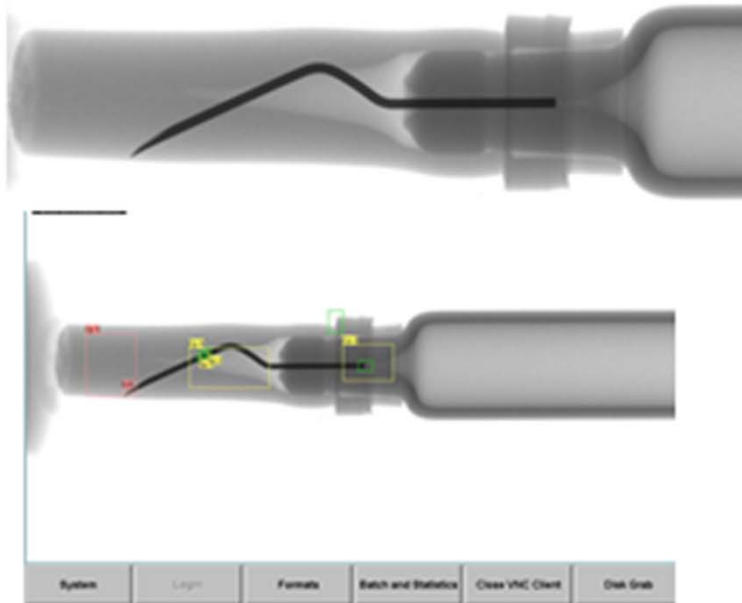
## X-Ray Tomography Visualization Techniques



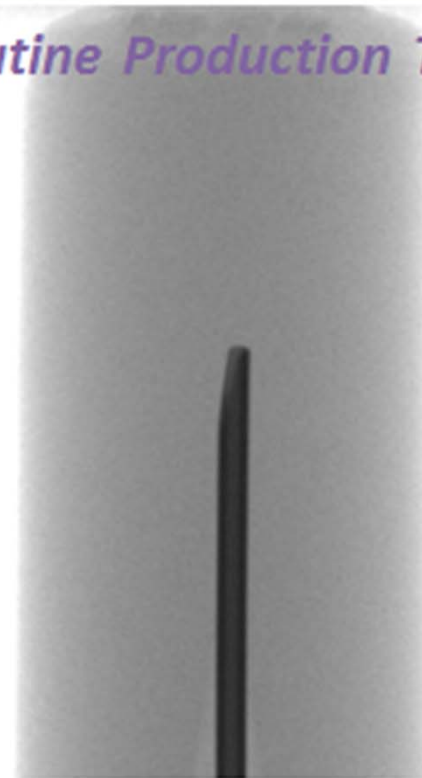
SkyScan Image Courtesy of Micro Photonics, Inc.



## *X-Ray Inspection - Routine Production Test*



Images property of Wilco AG



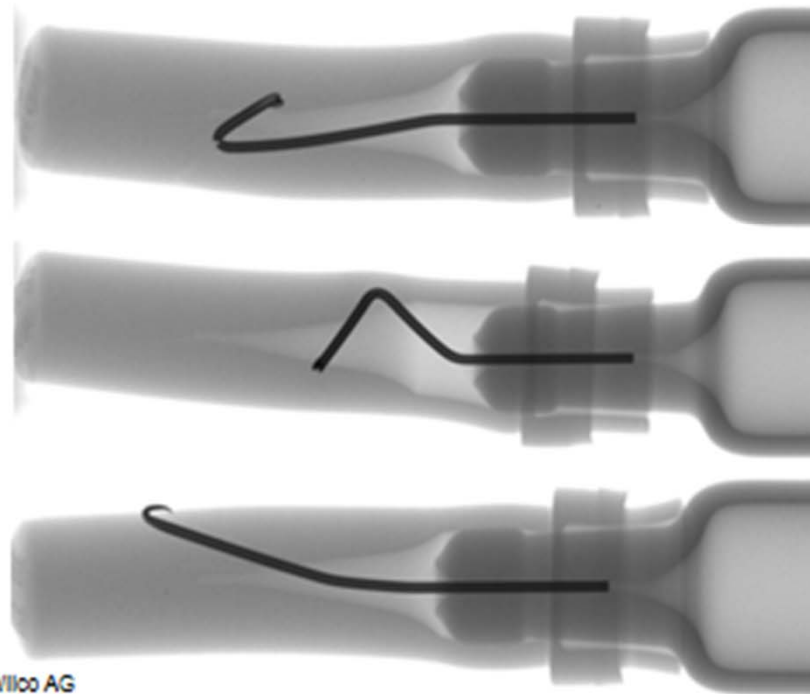
RxPax, LLC 152

D. Guazzo, Container closure integrity challenges unique to prefilled syringes and cartridges, PDA 7<sup>th</sup>

Annual Global Conference on Microbiology, Bethesda, MD, Oct 22-24, 2012



## *X-Ray Inspection - Routine Production Test*



Images property of Wiloo AG

RxPax, LLC 152

D. Guazzo, Container closure integrity challenges unique to prefilled syringes and cartridges, PDA 7<sup>th</sup>

Annual Global Conference on Microbiology, Bethesda, MD, Oct 22-24, 2012



## Seal quality test methods

- Significant variation w/in technologies may be seen among vendors
- Outcome varies based on test variables chosen
- Other methods not included may be acceptable
- **SQT ≠ leak tests**
  - Passing SQT ≠ leak-free package
  - Failing SQT = package integrity risk
- **SQT and package leak tests work together** to ensure package quality

## Leak test methods

### • **Deterministic and Probabilistic methods**

- Significant variation w/in technologies is seen among instrument vendors
- Outcome varies based on test variables chosen
- Other methods not included may be acceptable
- **No method** is appropriate for all product-packages
- **All methods** are valuable when used appropriately