



# Test Method for Indirect Measurement of Elastomeric Closure Compression Using an Automated Residual Seal Force Tester

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Definitions

Test Method Concept

Method Justification

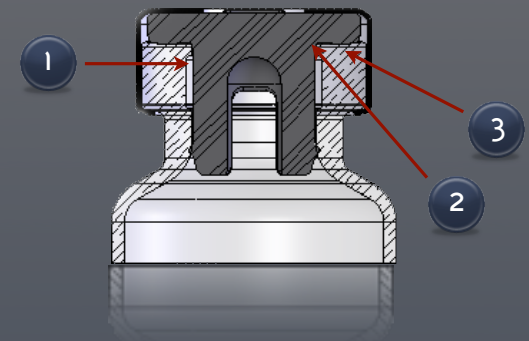
Method



Outline

# The Land Seal

Provides the Primary Seal Post Capping



1



Plug Seal

2



Transition Seal

3

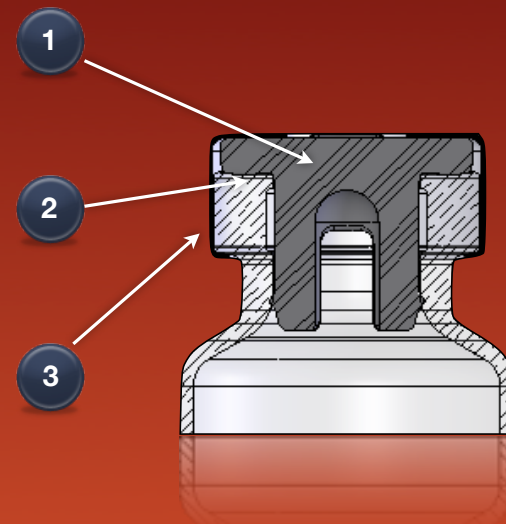


Land Seal

# Definitions

## *Closure Compression (n)*

The extent to which the elastomeric closure flange is vertically deformed against the vial land seal surface by the applied aluminum seal. (viscoelastic deformation)

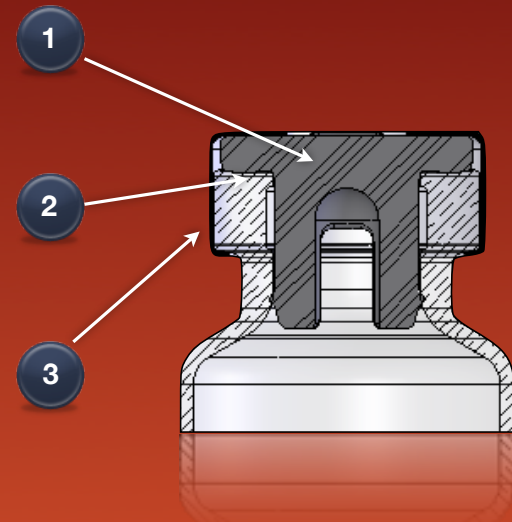


1. Closure Flange
2. Vial Land Seal
3. Aluminum Seal

# Definitions

*Residual Seal Force (RSF), (n)*

The stress a compressed elastomeric closure flange exerts on a vial land seal surface after application of an aluminum seal (crimping).



1. Closure Flange
2. Vial Land Seal
3. Aluminum Seal

# Test Method Concept

- There is an OPTIMUM WINDOW of Closure Compression
- Too MUCH Compression on Capping
  - Glass Breakage
  - Closure Dimpling (i.e., closure is forced into the vial mouth)
  - Capillary Leaks From Wrinkled Closure Coating
- Too LITTLE Compression on Capping
  - Failure to Seal
  - Loss of Integrity
  - Loose Cap
- Poor Compression Cannot be Visually Detected
  - RSF is an Indirect Measure of Compression

# RSF Testing Justification

- Long History of Use
  - 19 Years RSF Testing Using Universal Tester
  - 7 Years of Testing Using Genesis Automated Tester
- Published Method
  - J PDA 6 Peer Reviewed Articles (1988, 1992, 1995, 1996)
- Recognized Technology
  - PDA Technical Report No. 27 (1997) *Pharmaceutical Package Integrity*
  - USP <1207> *Sterile Product Packaging - Integrity Evaluation*
  - FDA Guideline (2008) *Package Integrity Testing in Lieu of Sterility as Function of Stability Testing*

# Scope

Package: Vials

Plastic or Glass

Serum Finish  
(ISO 8362 or GPI 27103)



# Scope

Package: Closures

Elastomeric Material

Uncoated or Coated  
(*barrier or lubricant*)

Serum, Lyophilization,  
Disc or other

13mm to 28mm  
in Flange Diameter

# Scope

Package: Aluminum Seals

Crimp, Single Piece Design

Multiple Pieces

*(better results when plastic flip top is removed pre-testing)*

# Scope



## Purpose of Test

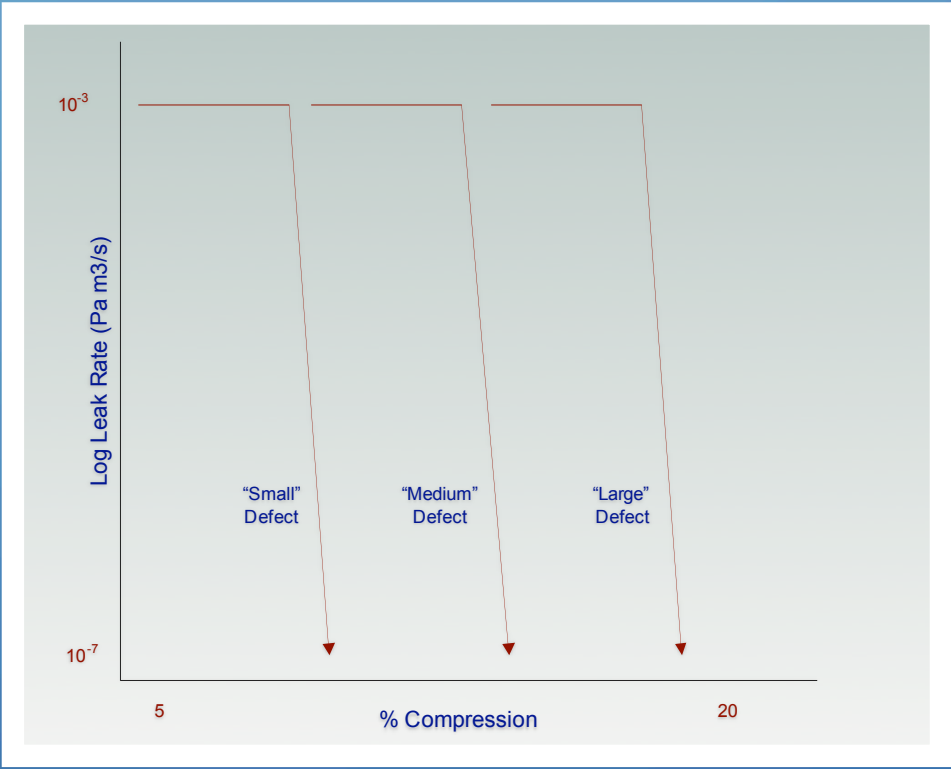
Indirect Measure of Capping Uniformity and Effectiveness

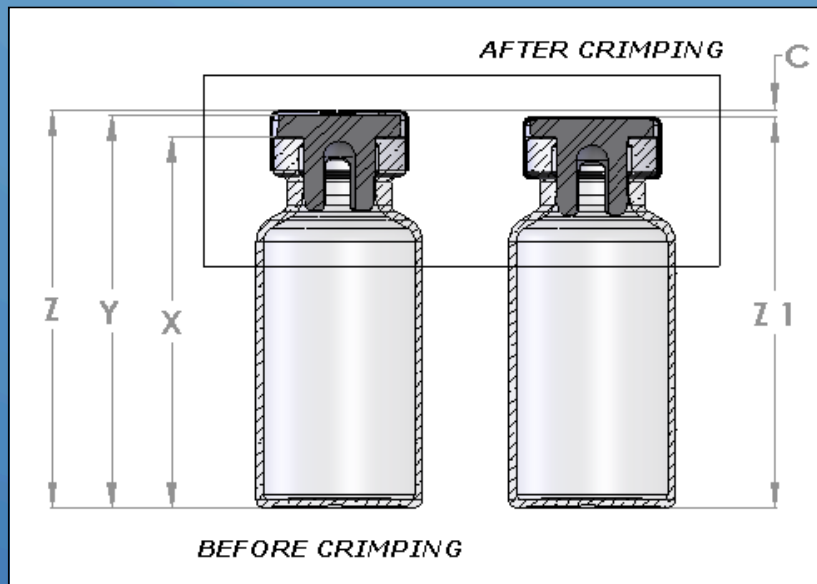
Predictor of Final Seal Integrity

## Test Results

- Quantitative *Newtons (or English equivalent)*
- Range 20 to 245 N
- Acceptance Criteria
  - Unique for Each Package System
  - Established by Correlation to Other Package Integrity Criteria
    - Leak Test
    - Closure Flange Compression

# Leak Rate Cut Off





# Compression

$$(Z - Z_1) / (Y - X)$$



## ASTM Listings

Leak Test Methods  
Packaging & Distribution Environments  
Thickness Measurements  
P& B Protocol

## Other

PDA Technical Report No. 27  
J PDA Articles by Morton Guazzo, et al (4)  
J PDA Articles by Ludwig, et al (2)

# Referenced Documents

I.Vial With Cap Anvil is Positioned in Tester

Summary of Test Method

2. At START, Tester Compresses Cap Anvil top  
(therefore closure) at Slow, Constant Strain Rate

Summary of Test Method



### 3. Load Cell Measures Stress Resistance

Summary of Test Method

#### 4. RSF is Automatically Identified Via Algorithm

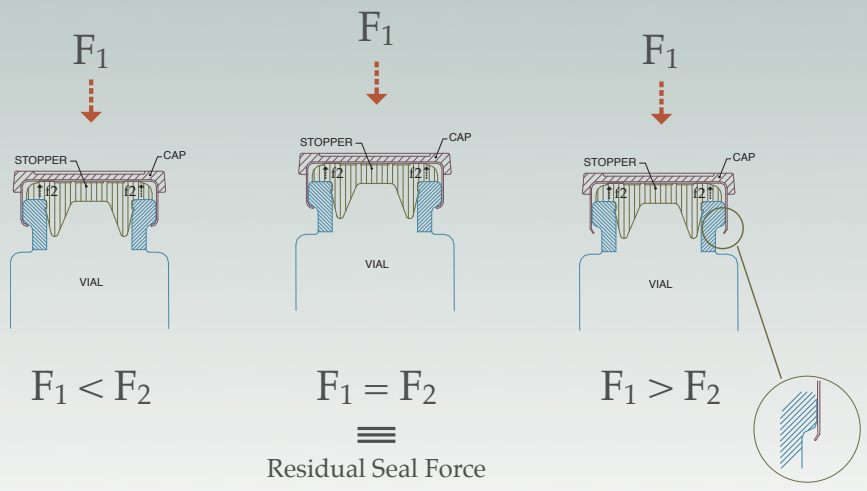
- *Minimum of the 2nd Derivative of the Stress-Strain Curve*
- *Confidence Factors Used for Reliability*
- *RSF Result = Mean of 3 Readings Per Test*

Summary of Test Method

- Theory

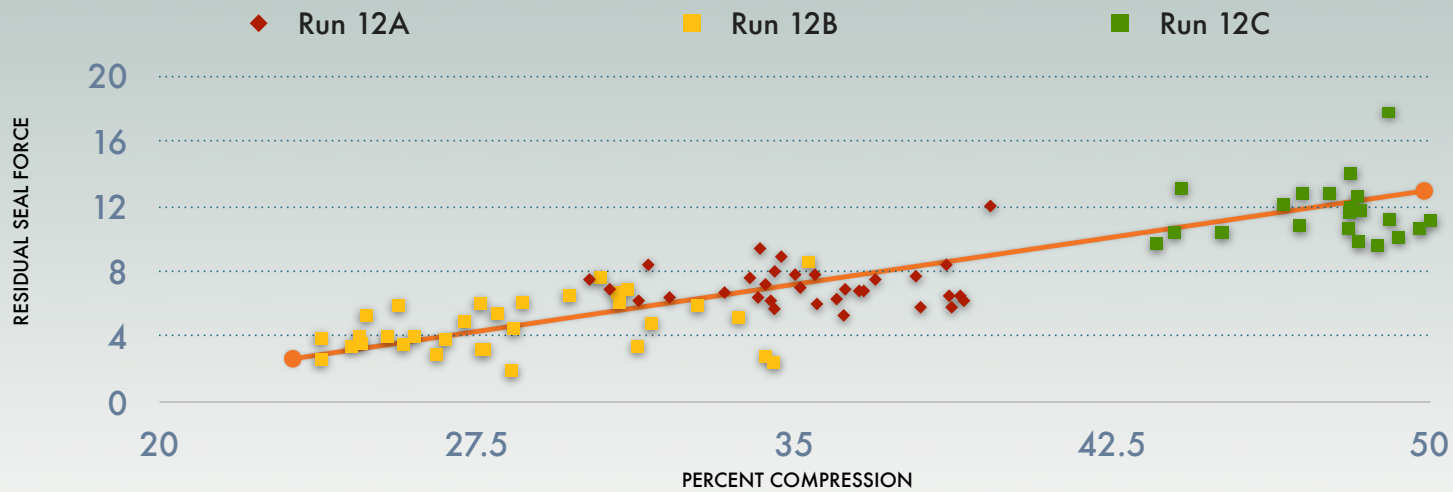
- Upon Capping, Closure Flange is Compressed Onto Vial Land Seal Surface
  - Closure Acts Like a “Compressed Spring”
- Tester exerts Force on Cap/Stopper
  - When Tester Force > Closure Compression Force, the Stress-Strain Slope Drops
  - This “knee” in Curve = RSF
- > Compression by Capping... > Closure Compression Sealing Force on Vial... >RSF

## Summary of Test Method

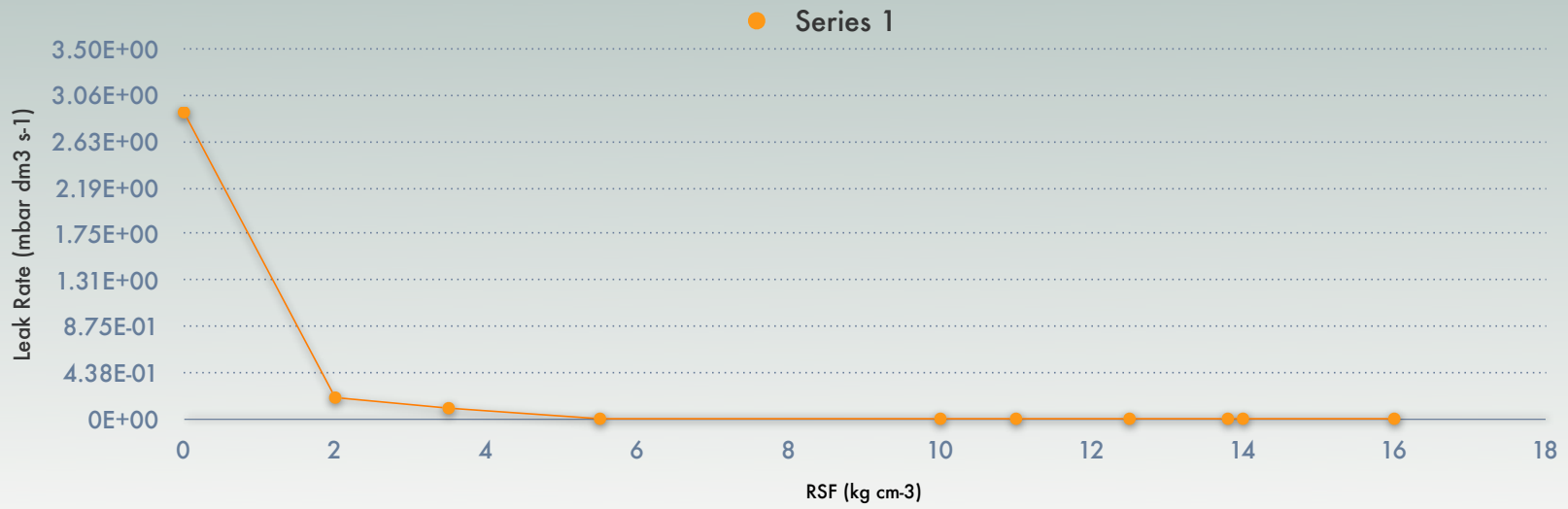


Residual Seal Force

# Measuring Residual Seal Force

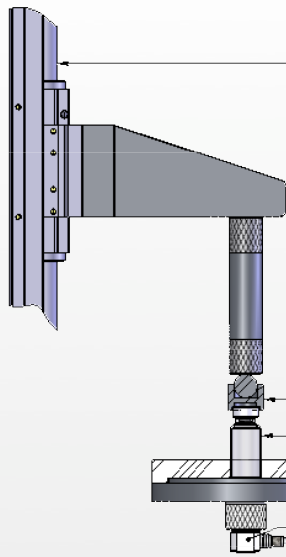


RSF versus Closure Compression



RSF versus Leak Rate

**Distance (MM) to Processor**



MOTORIZED LINEAR ACTUATOR

ANVIL

SEALED/CRIMPED VIAL

CENTERING PUCK

VIAL/BOTTLE REST

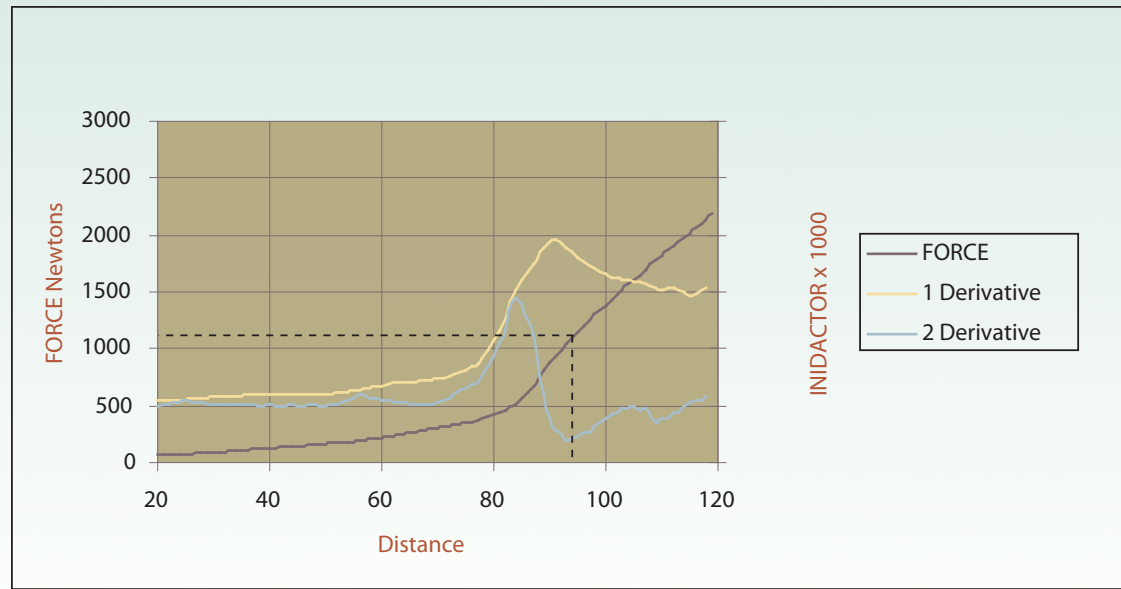
LOAD CELL

**Force (Newtons) to Processor**

# Automated RSF Tester



# Stress Strain Analysis





# Summary of Test Method

- RSF Major Variables
  - Closure Size (flange thickness)
  - Exposure to Heat Post Capping
  - Time Lapse Post Capping (Maxwell Degenerative Curve)
- RSF Minor Variables
  - Closure Formulation
    - Durometer
    - Compression Set Characteristics
  - Closure Lot to Lot Variation
  - Closure Degree of Cure
- RSF Controls to Minimize Test Variables
  - Cap Anvil Design and Dimensions
  - Vial Puck to Center and Hold Package

# Significance and Use

- Poor Compression Cannot be Visually Detected
  - RSF is an Indirect Measure of Compression
- Too MUCH force on Capping
  - Glass Breakage
  - Closure Dimpling
  - Capillary Leaks with Coated Closures
- Too LITTLE force on Capping
  - Failure to Seal
    - Loss of Integrity
    - Loose Cap

# Significance and Use

- Method is Semi-Automatic
  - Proven Algorithms
  - PLC Controlled
  - Simple to Use
  - R&D
    - Establish Optimum Capping Conditions
    - Determine Effects of Package Variables
      - E.g., Dimensions, Component Processing
      - E.g., Assembled Package Processing, Distribution, Storage
- Production
  - Verify Capping Equipment Set-Up

# Significance and Use

- Results
  - Quantitative RSF Value (N)
  - No Pass/Fail Criterion
    - WHY?
      - Acceptance Criterion Must be Established Using Another Package Parameter
        - % Closure Flange Compression
        - Leak Rate
          - Helium
          - Vacuum Decay
          - Dye Ingress

# Automated Residual Seal Force Tester

# Appendix

- A1. Cited Technical Literature
  - PDA Technical Report No. 27
  - D. Morton Guazzo, J PDA Publications (4)
  - J. Ludwig, J PDA Publications (2)



Thank You!

