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

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The Land Seal
Provides the Primary Seal Post Capping
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
- Recognized Technology
  - USP <1207> Sterile Product Packaging - Integrity Evaluation
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)
**Purpose of Test**

Indirect Measure of Capping Uniformity and Effectiveness

Predictor of Final Seal Integrity

**Scope**

- 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

**Test Results**

- Leak Test
- Closure Flange Compression
Leak Rate Cut Off

Log Leak Rate (Pa m³/s)

% Compression

10⁻³

10⁻⁷

5  10  20

“Small” Defect
“Medium” Defect
“Large” Defect

"Small" Defect "Medium" Defect "Large" Defect
Compression

\[
\frac{Z-Z_1}{Y-X}
\]
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
3. Load Cell Measures Stress Resistance
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
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
Measuring Residual Seal Force

\[ F_1 < F_2 \]
\[ F_1 = F_2 \]
\[ F_1 > F_2 \]

Residual Seal Force
RSF versus Leak Rate

Leak Rate (mbar dm³ s⁻¹) vs. RSF (kg cm⁻³)
Automated RSF Tester

Distance (MM) to Processor

MOTORIZED LINEAR ACTUATOR

- Anvil
- Sealed/Crimped ViAL
- Centering Puck
- ViAL/Bottle Rest
- Load Cell

**Force** (Newtons) to Processor
Stress Strain Analysis

![Graph showing force and derivative versus distance.](image-url)
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
  - Quantitive 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

  - PDA Technical Report No. 27
  - D. Morton Guazzo, J PDA Publications (4)
  - J. Ludwig, J PDA Publications (2)
Thank You!