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Fluorosilicone O-ring Study

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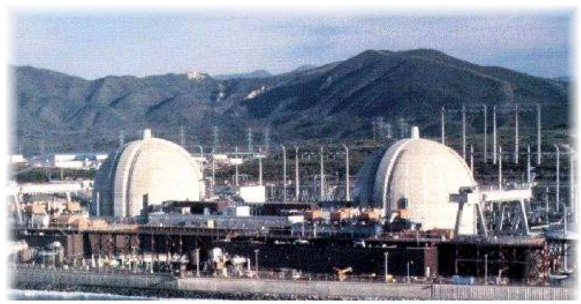
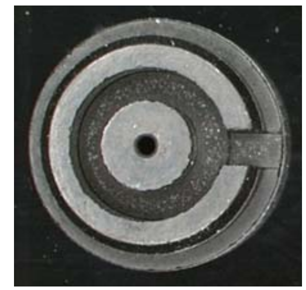
National Nuclear Security Administration

Organic Materials Problems; Organic Materials Aging and Degradation

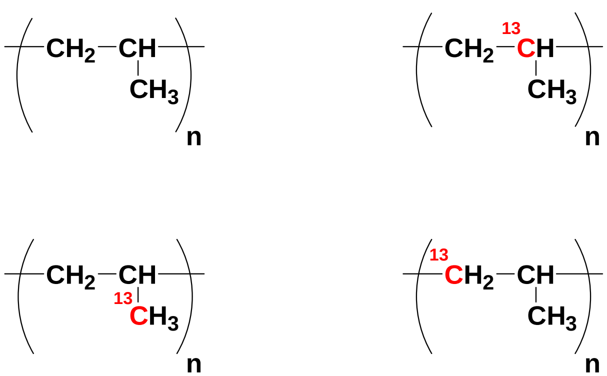
O-rings



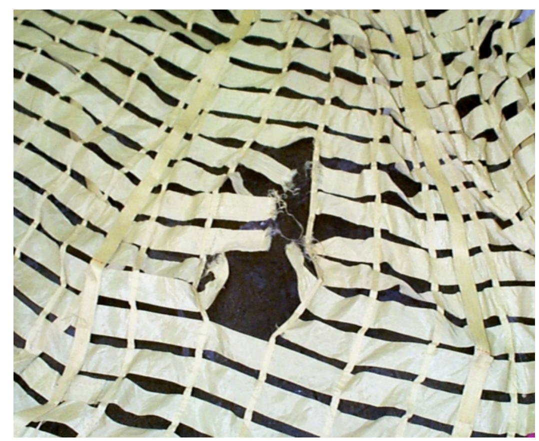
Nuclear Power Plant Cable Insulation



Shorting Plugs



Labeled Polymers



Textiles

Fluorosilicone O-rings

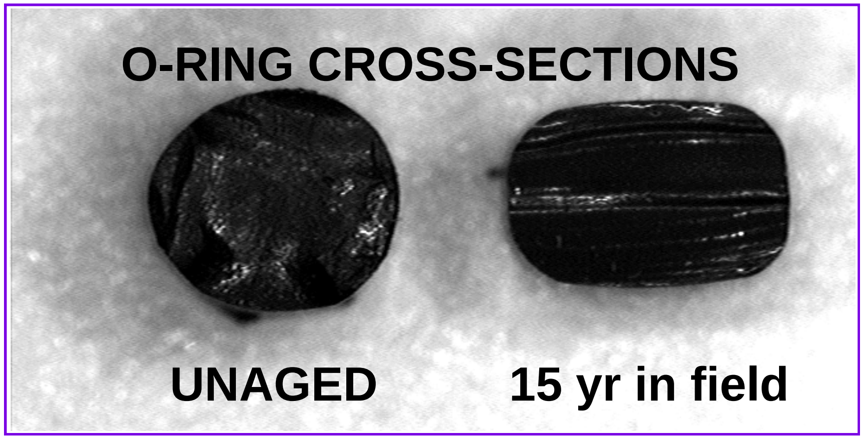
Bernstein, R.; Gillen, K. T. Polymer Degradation and Stability, Predicting the Lifetime of Fluorosilicone O-rings 2009, 94, 2107-2133.

Bernstein, R.; Gillen, K. T. "Fluorosilicone and Silicone O-Ring Aging Study," SAND2007-6781, Sandia National Laboratories, 2007.

O-rings Background

Used as environmental seals or other seals

Many systems filled with inert gas to protect interior components from oxidation & hydrolysis



***Previously*:**

No technique to measure equilibrium sealing force
No technique to rapidly achieve equilibrium compression set
No correlation

CSR Jigs

Gap of jig can be adjusted to any desired size

O-ring pieces cut to allow air circulation

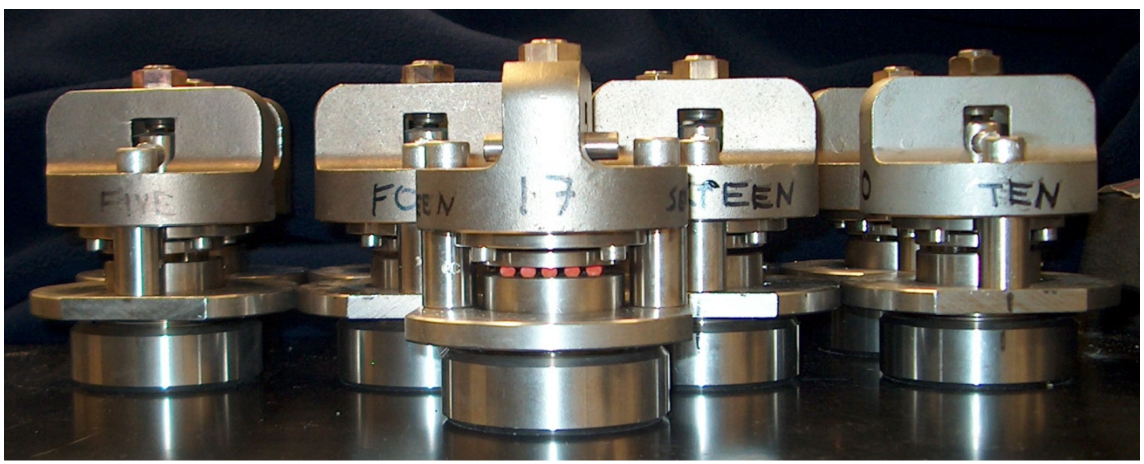
Measurement of force involves very slow and slight compression until electrical contact is broken between the top and bottom plates

Jigs can be placed in ovens, thus providing isothermal measurements

Compression Stress Relaxation (CSR)

Shawbury-Wallace Compression Stress Relaxometer (CSR) MK II

Commercial Instrument
Measure of Force
-O-ring sealing force
Can Adjust Gap Size to Approximate Actual Compression in System



(Wallace Test Equipment, Cryodon, England)

Accelerated aging

1) Physical force decay

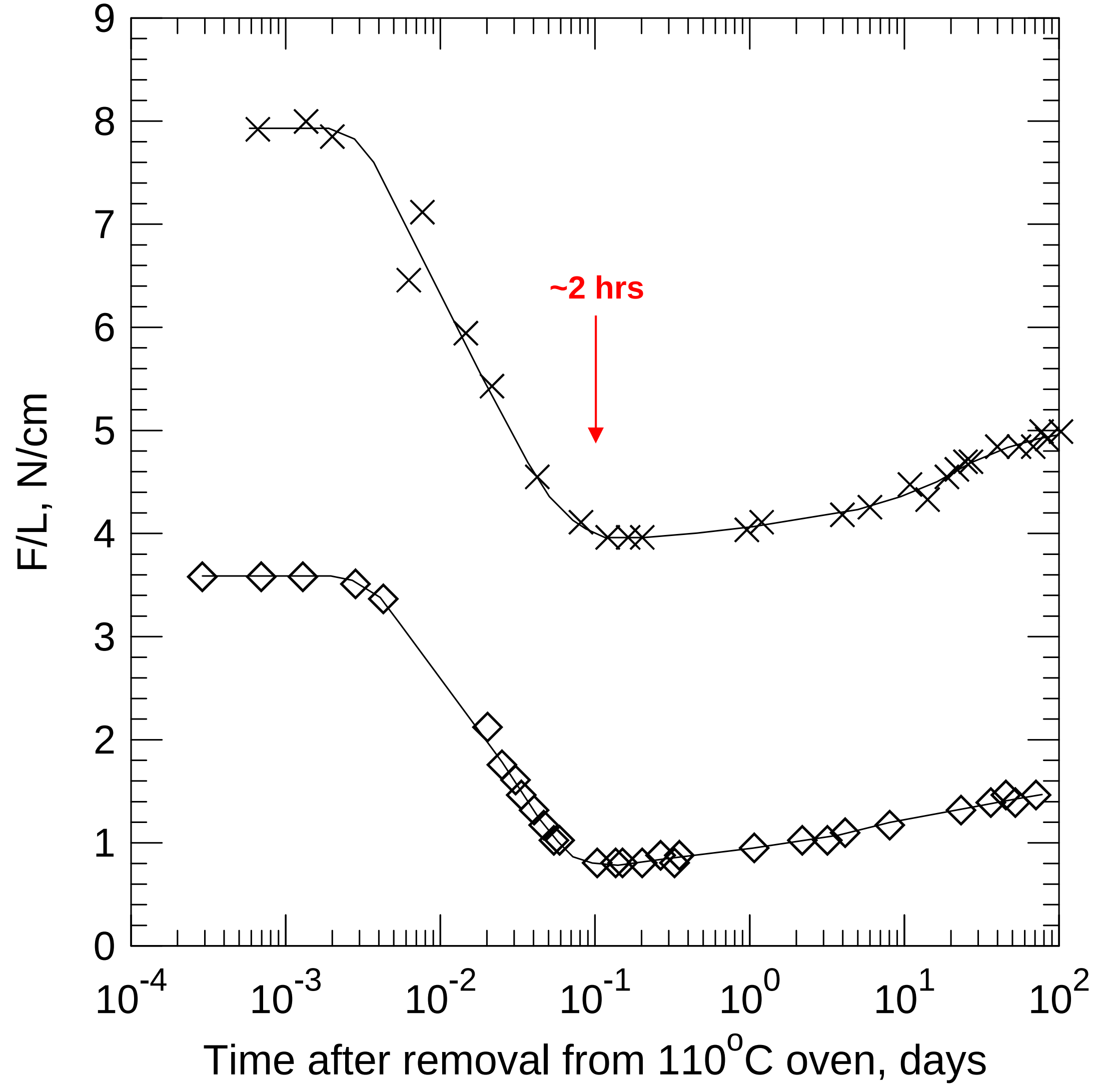
-Equilibrium values achieved –starting point

-Ability to get field returned o-ring force –ending point

2) Chemical force decay

Prediction of force changes as a function of aging

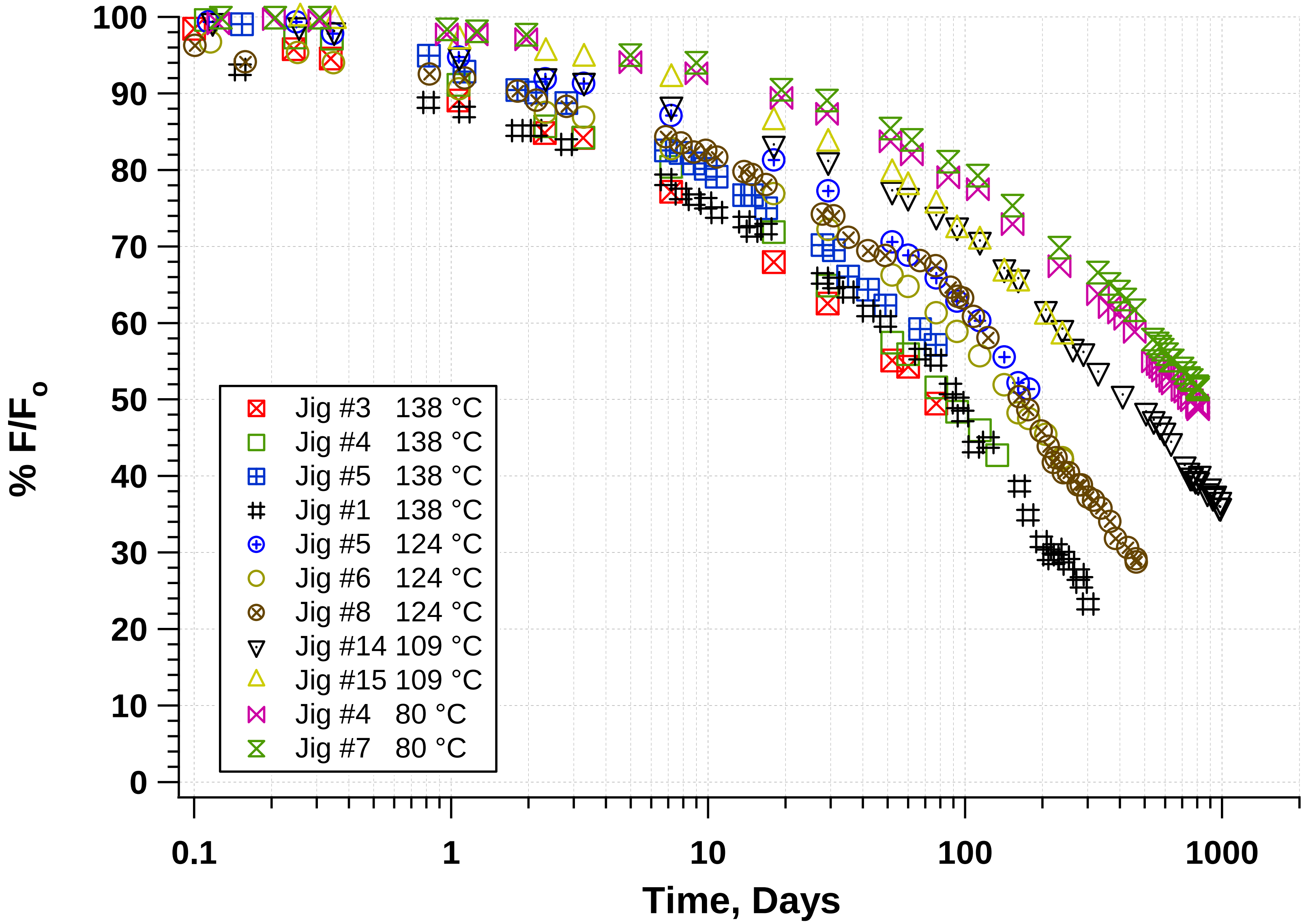
Why we do isothermal measurements…



Sealing force per unit length versus time out of a 110 °C oven for two CSR jigs containing Butyl-A o-ring segments that had aged under 25% compression until the force degraded by ~42% (top curve) and ~72% (bottom curve), respectively.

Gillen, K. T.; Celina, M.; Bernstein, R. In *Polymer Degradation and Stability* Validation of Improved Methods for Predicting Long-Term Elastomeric Seal Lifetimes from Compression Stress-Relaxation and Oxygen Consumption Techniques, 2003; Vol. 82, pp 25-35.

All Jigs at Temperatures -Fluorosilicone



Bernstein, R.; Gillen, K. T. "*Fluorosilicone and Silicone O-Ring Aging Study*," SAND2007-6781, Sandia National Laboratories, 2007.

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Time-Temperature Superposition

Does mechanism change as a function of temperature?

If same mechanism:

- **same shape (log graph)**
- **should be constant acceleration (multiple)**

1. **Pick a reference temperature**
2. **Multiply the time at each temperature by the constant that gives the best overlap with the reference temperature data**
3. **Define that multiple as 'a_T' ($a_T = 1$ for ref. temp.)**
4. **Find a_T for each temperature**

Plot $\log(a_T)$ vs 1/T linear if Arrhenius

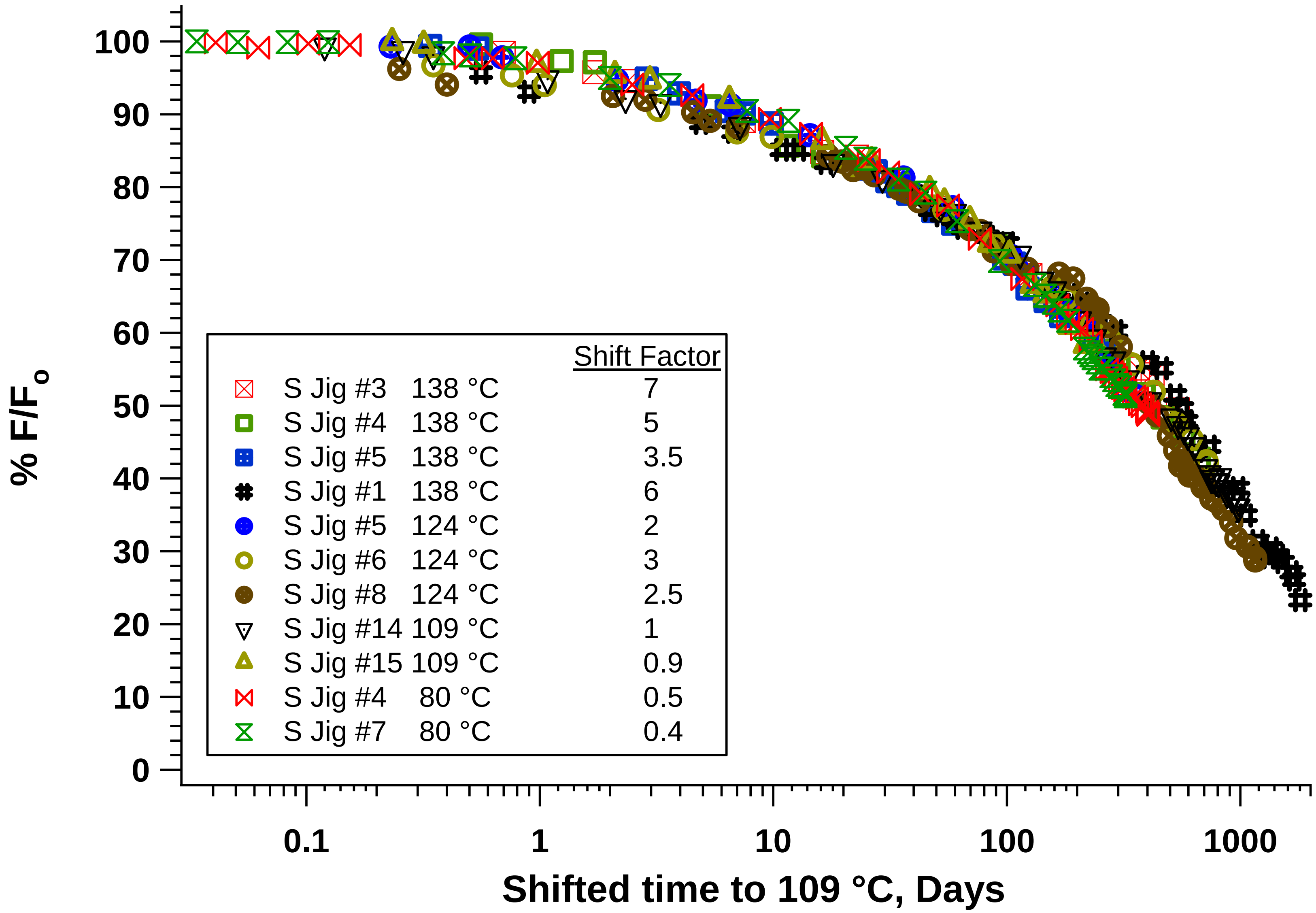
Gillen, K. T.; Celina, M.; Clough, R. L.; Wise, J. ***Trends in Polymer Science,*** **Extrapolation of Accelerated Aging Data -Arrhenius or Erroneous? 1997**, *5*, 250-257.

Arrhenius equation: **Empirical equation**

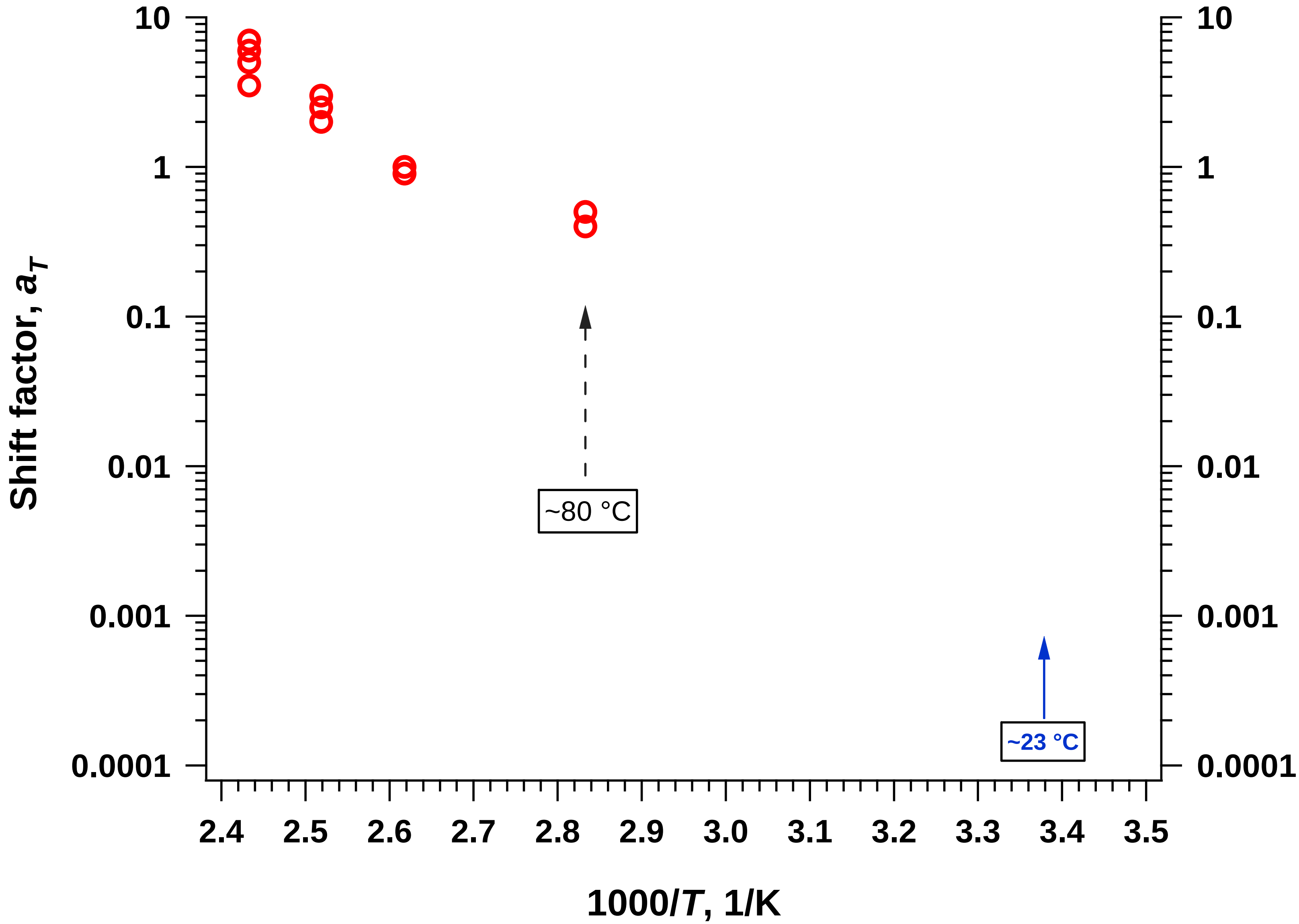
$$\boxed{k = Ae^{-Ea/RT}}$$

$$\boxed{\ln(k) = \ln(A) - Ea/RT}$$

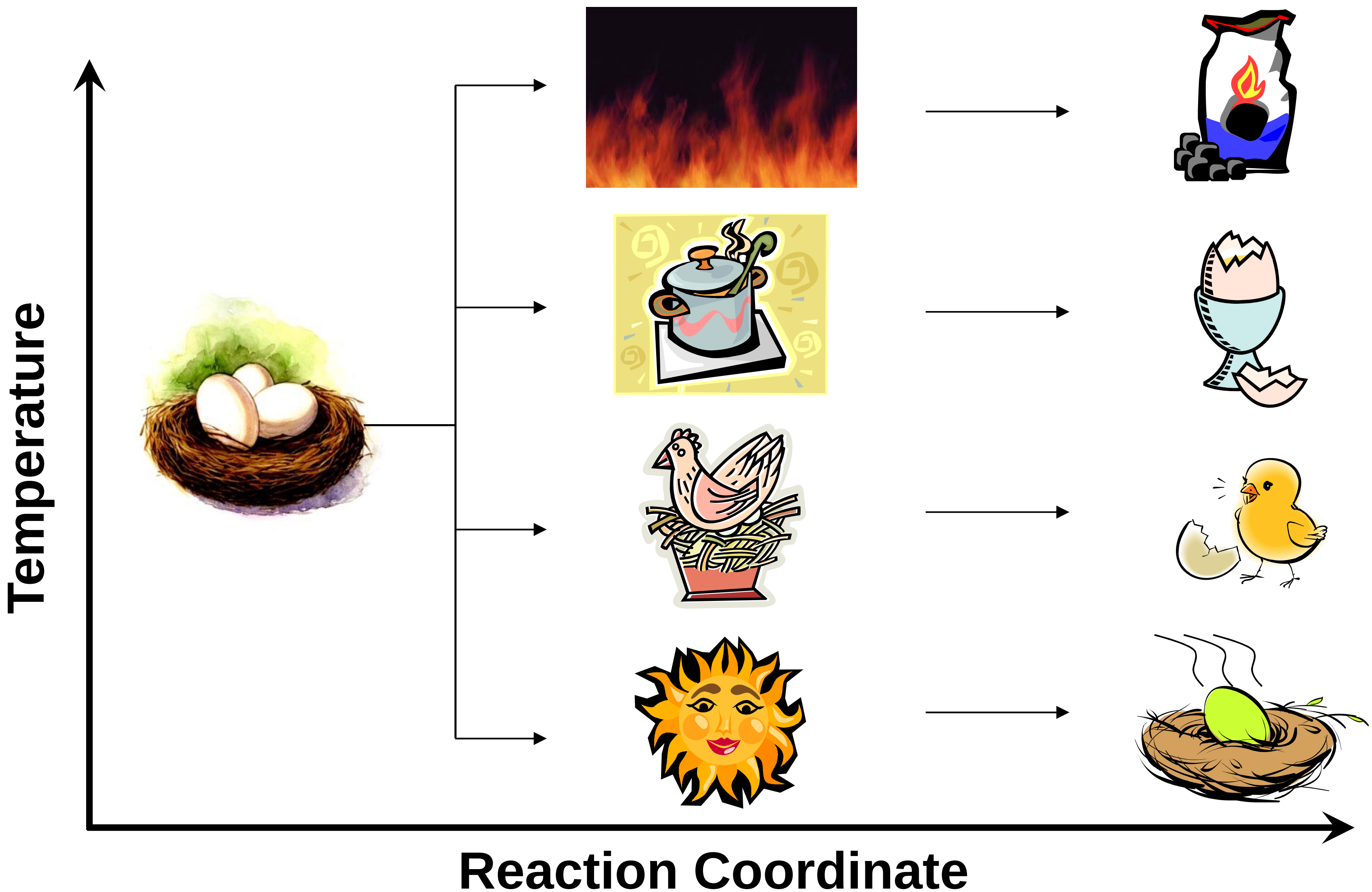
Time-Temperature Superposition



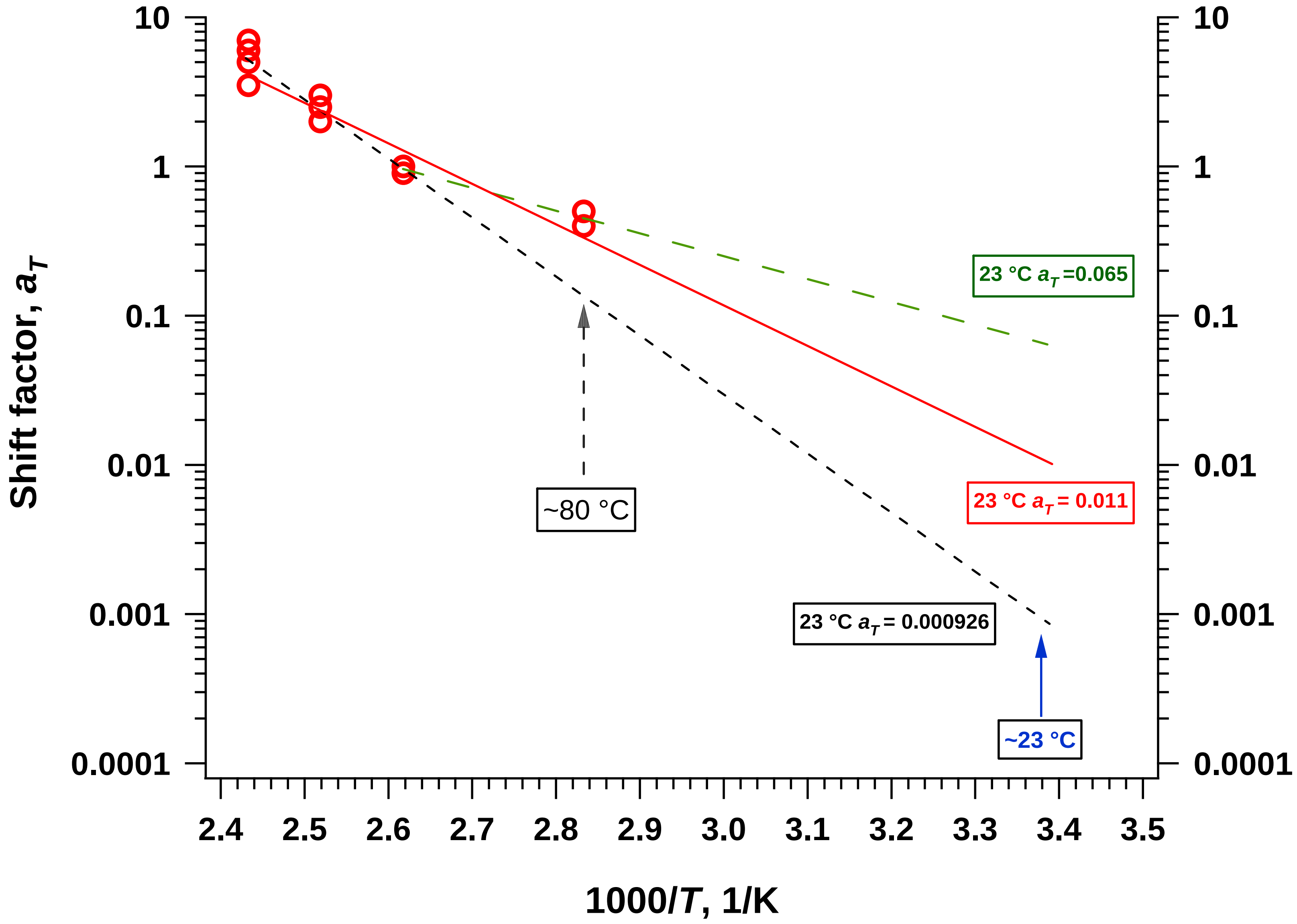
Shift Factor Plot



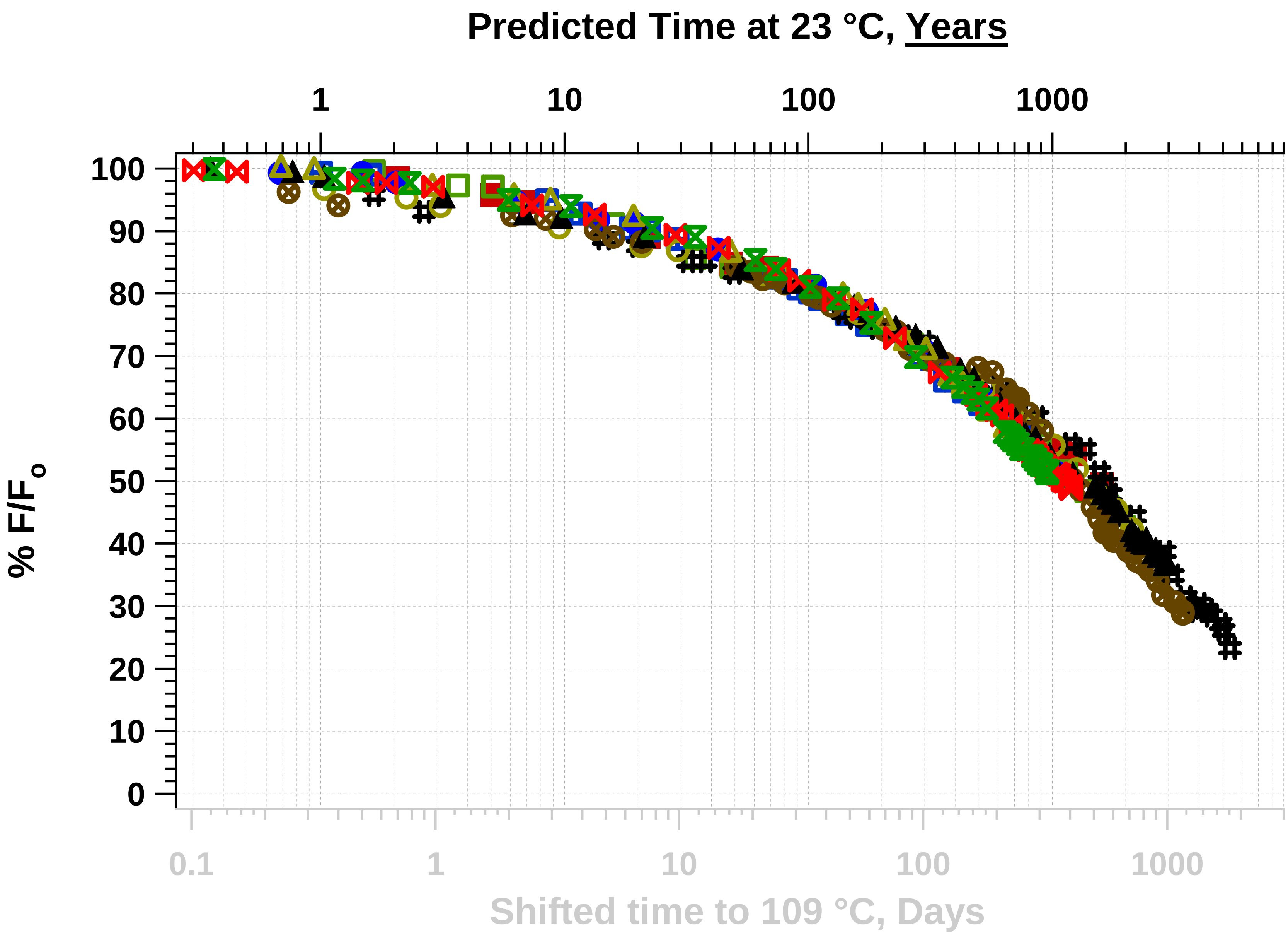
‘Accelerated Aging’



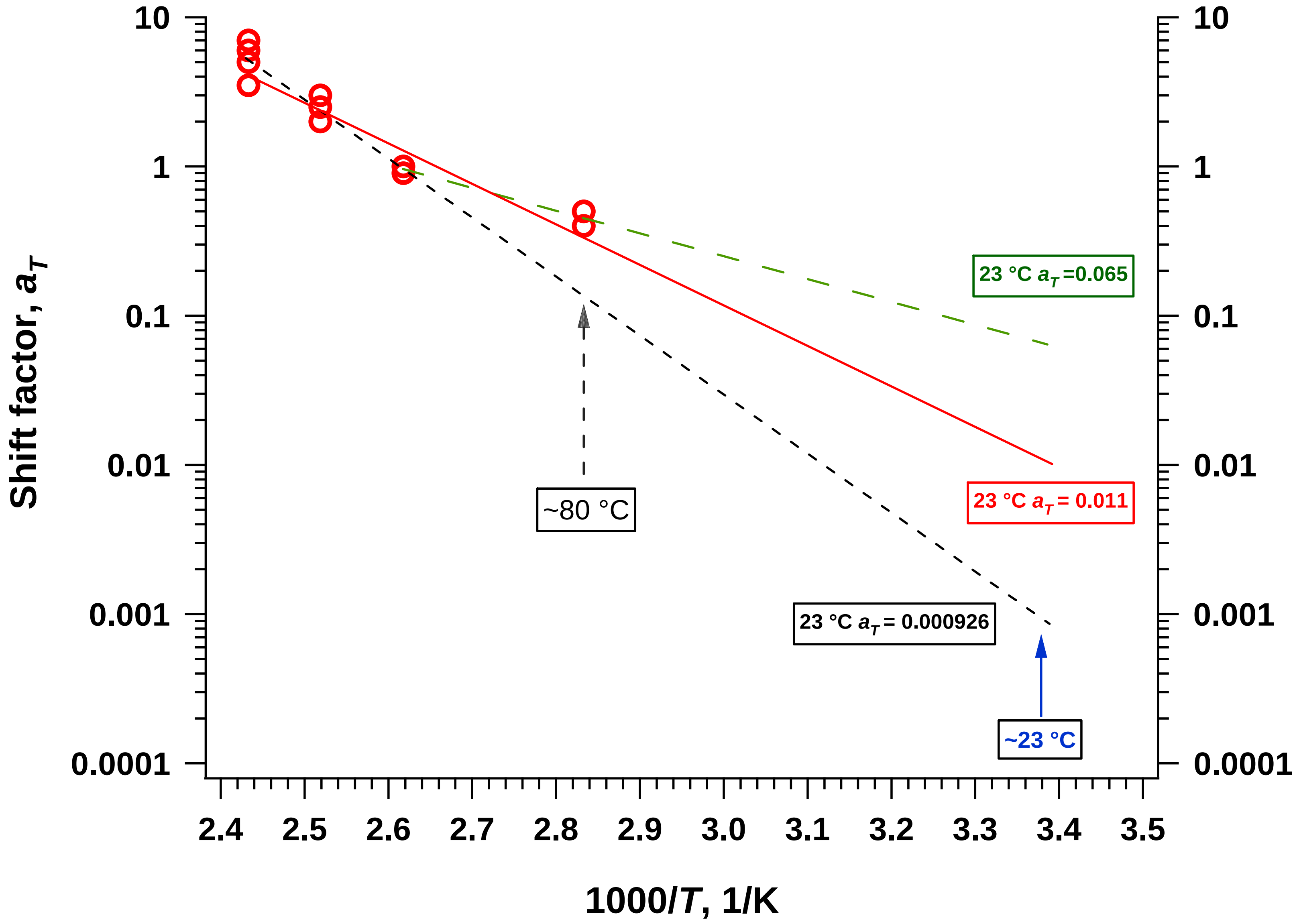
Shift Factor Plot



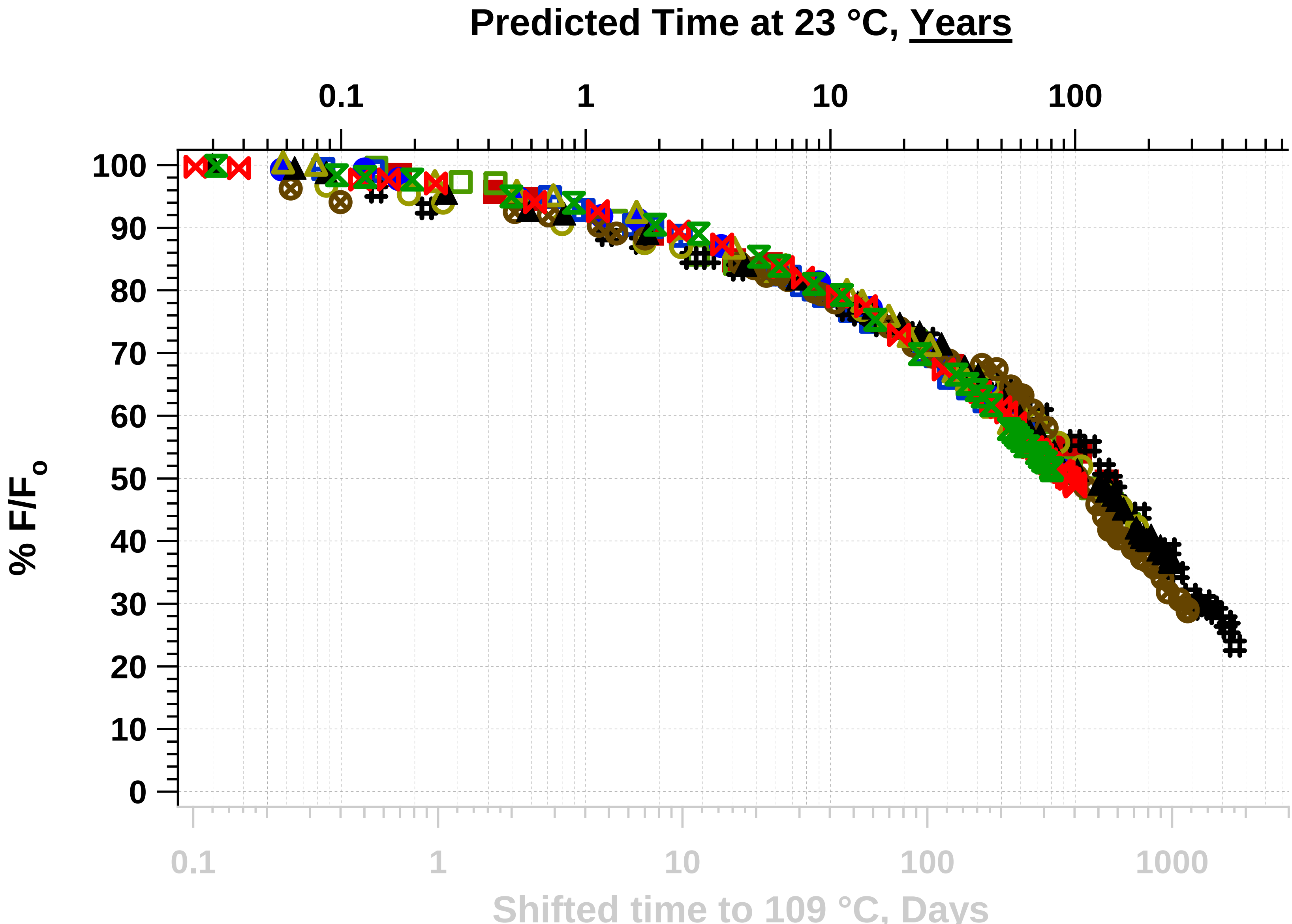
Shifted Data with RT 'Prediction' w/o 80 C data



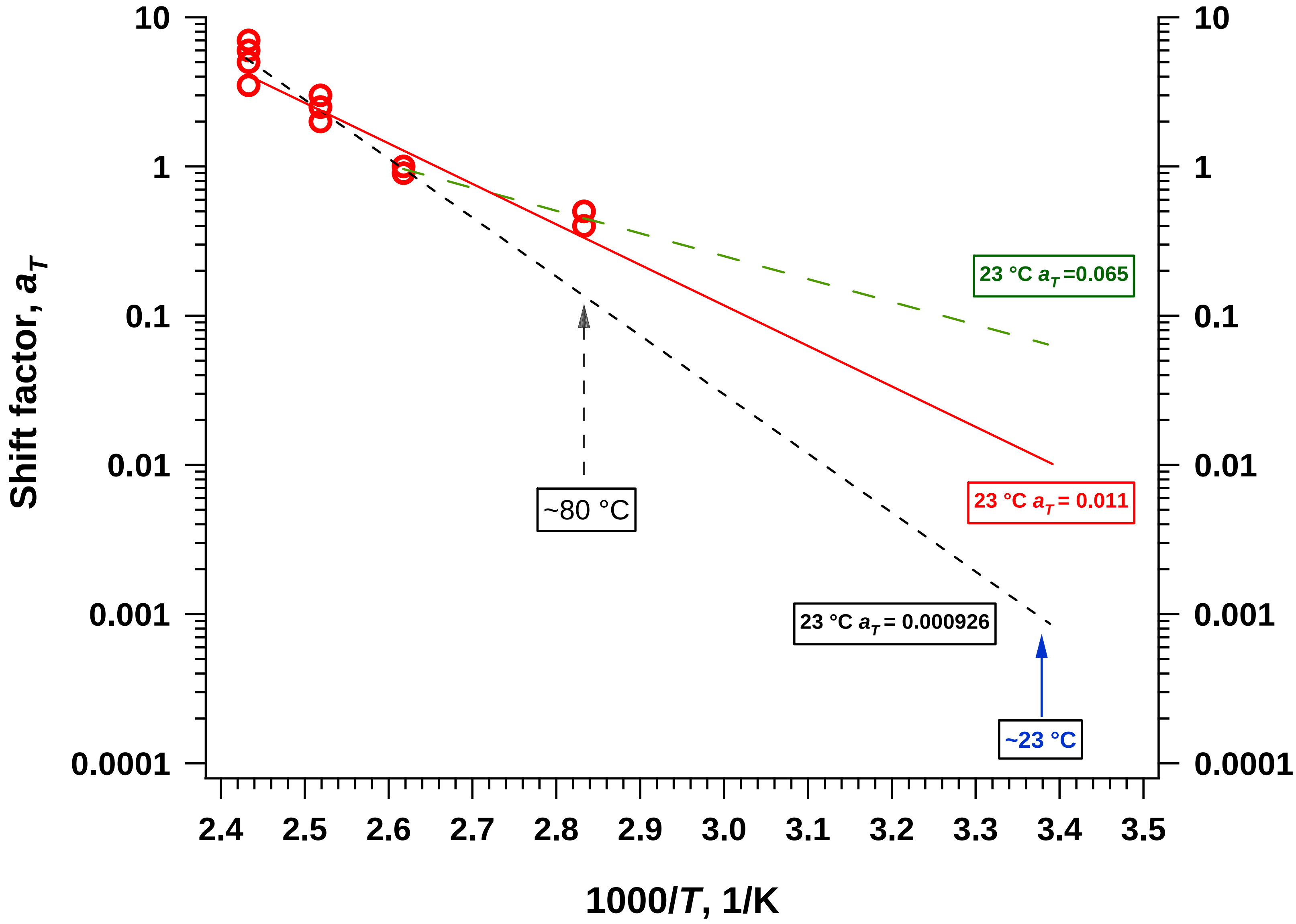
Shift Factor Plot



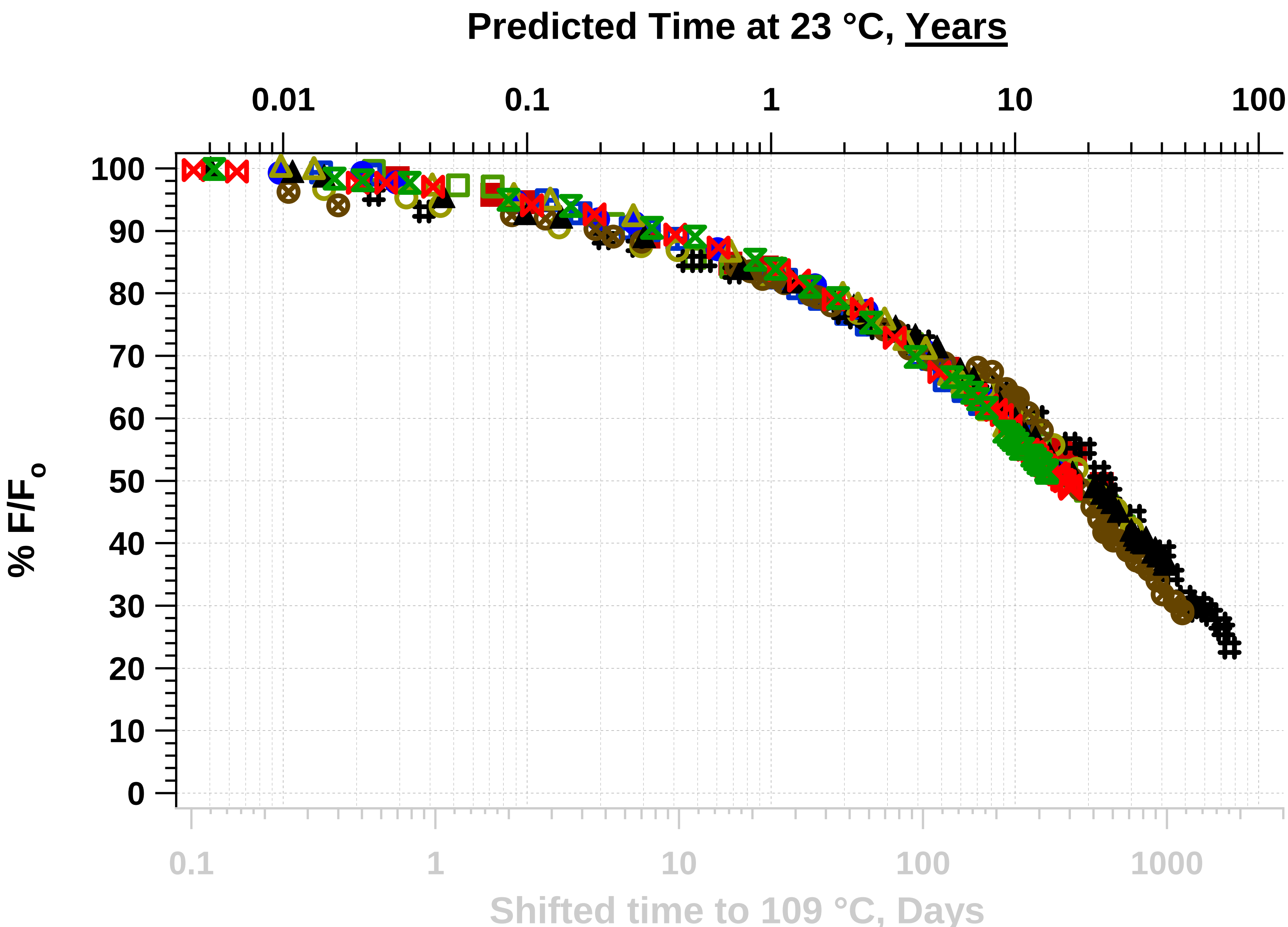
Shifted Data with RT 'Prediction' All data



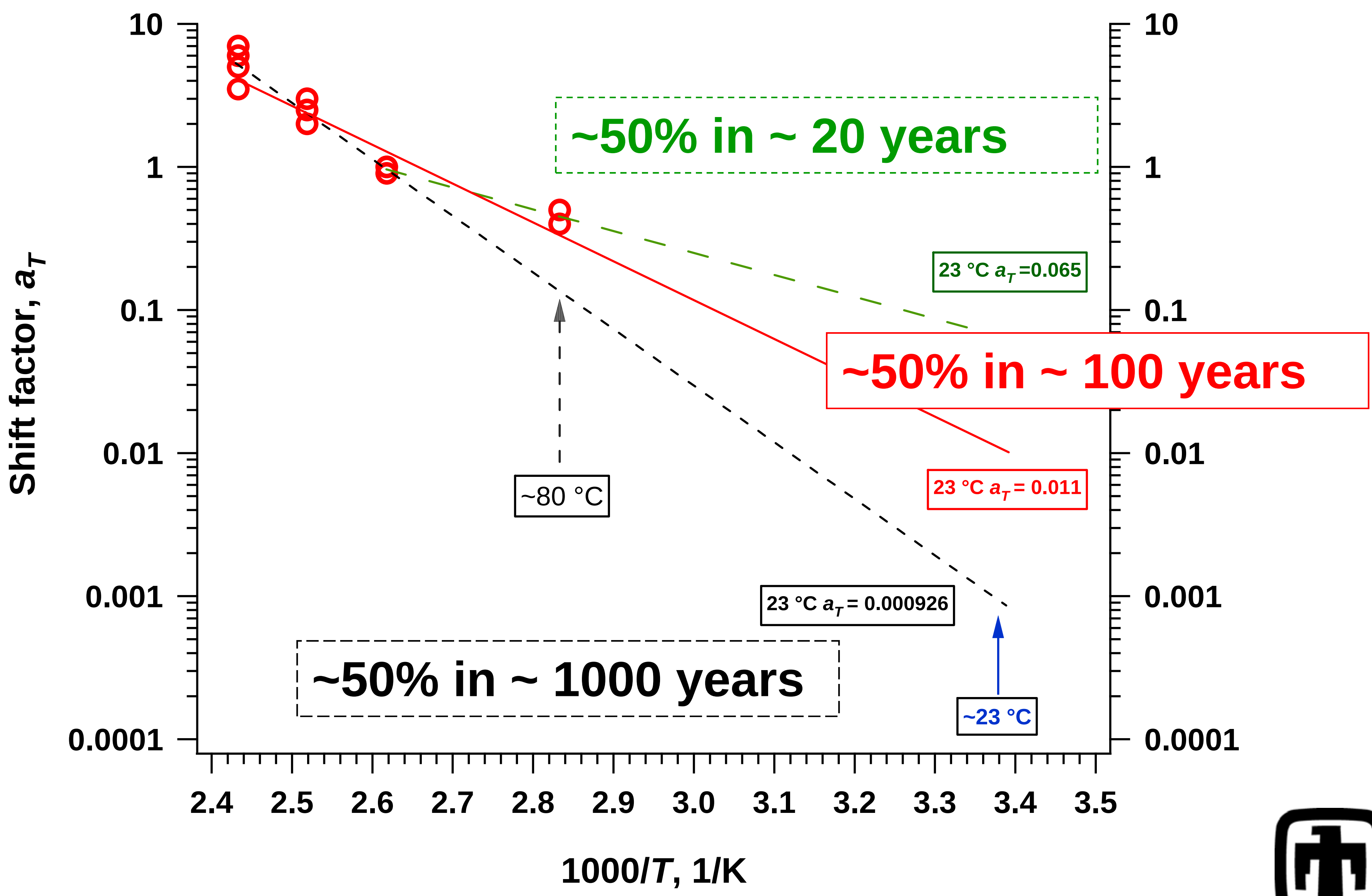
Shift Factor Plot



Shifted Data with RT ‘Prediction’ 109 and 80 only



Shift Factor Plot



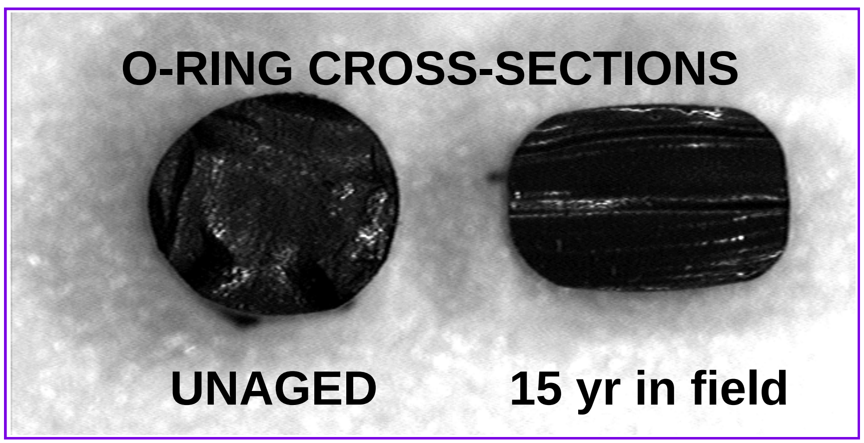
O-ring

Sealing force arguably most important parameter

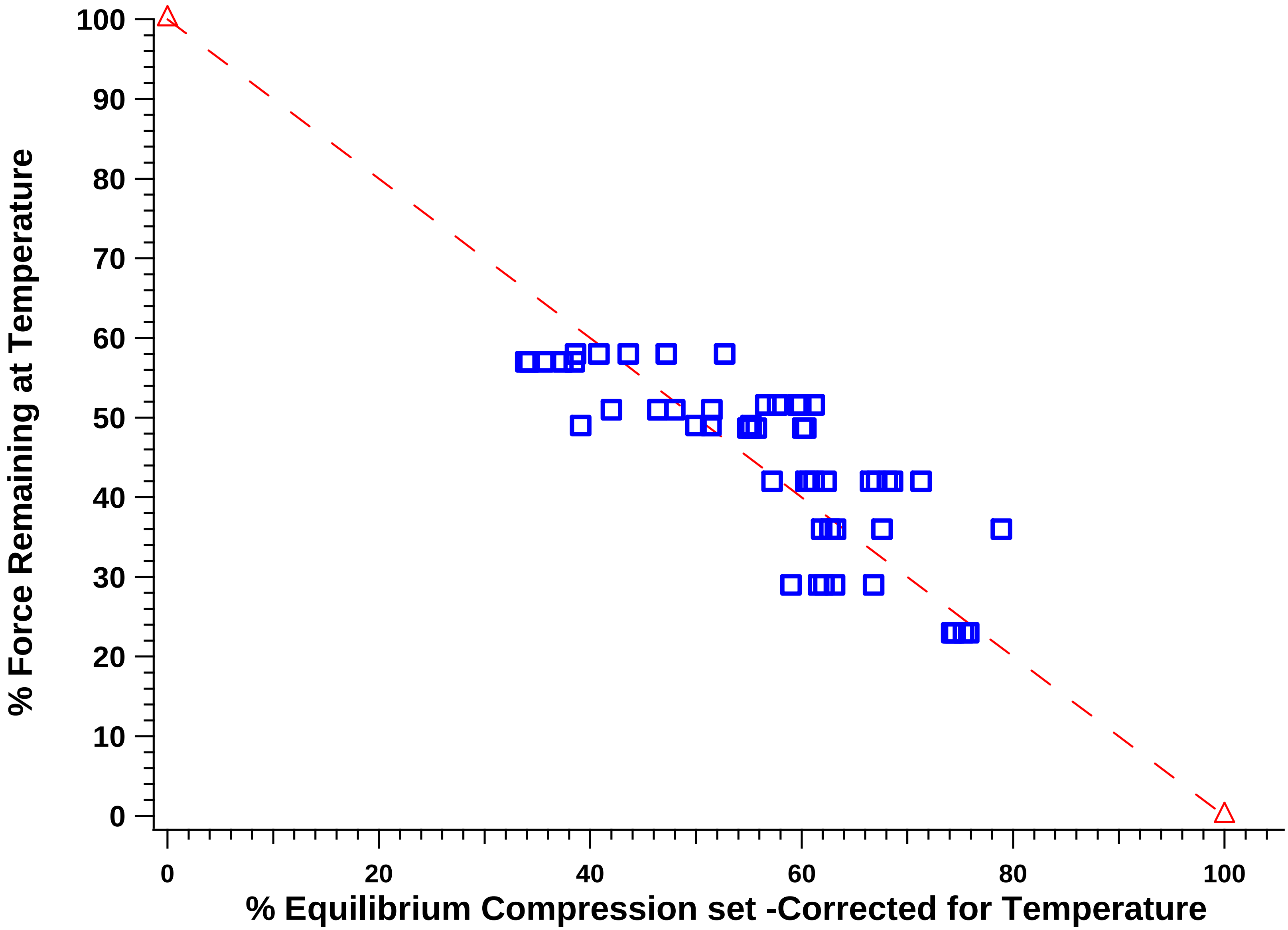
Correlation between equilibrium values

sealing force ⟷ **compression set**

sealing force	compression set
Difficult to measure slow and laborious	Easy to measure quick and simple

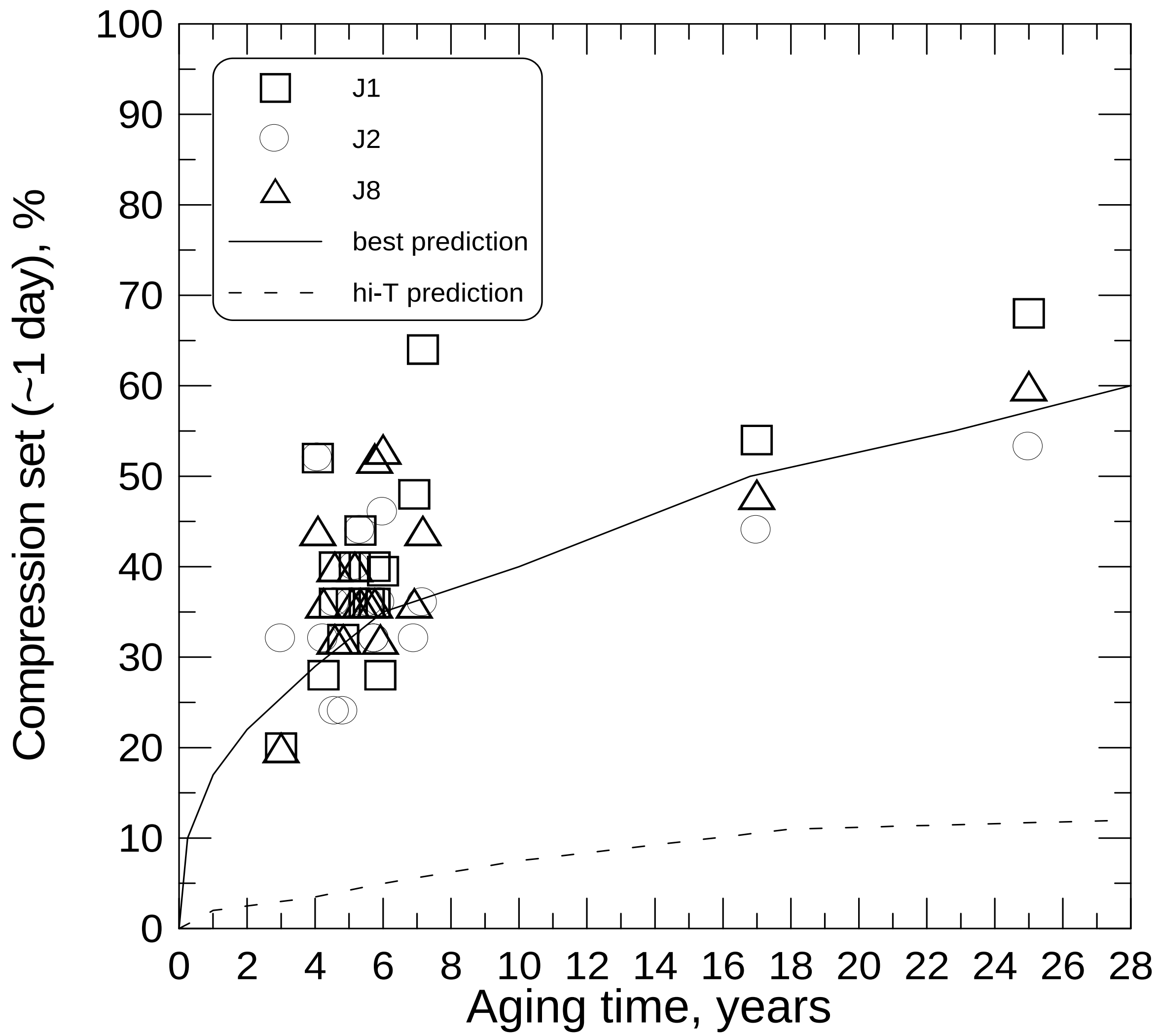


Force versus Compression Set Data -Fluorosilicone



Field Data*

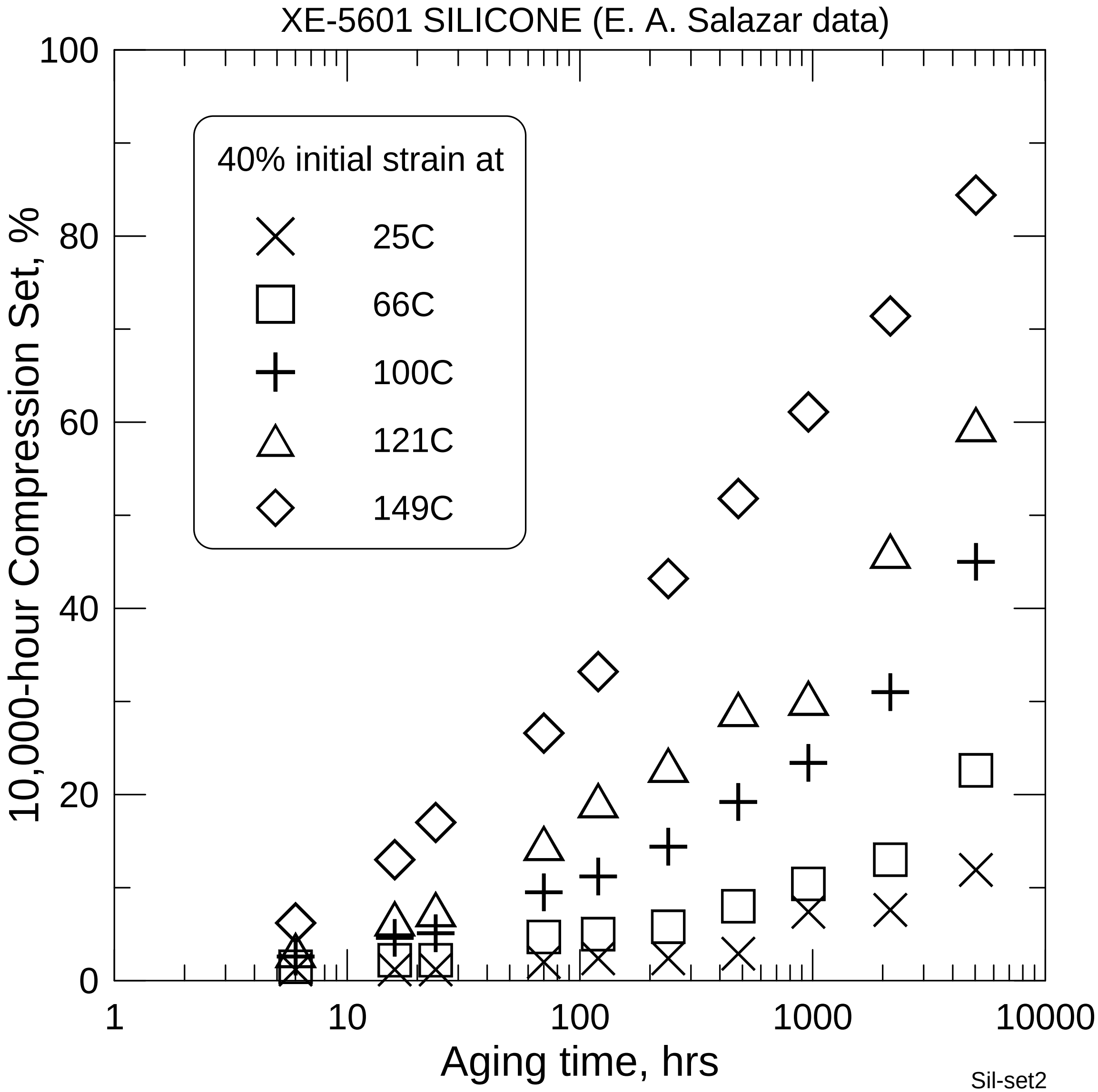
*** not quite the whole story, but good enough for this conversation!**



Compression set measurements of three fluorosilicone o-rings taken on field returned units approximately 1 day after removal from the unit. The solid curve and the dashed curve assume a linear relationship between set and force decay.

Bernstein, R.; Gillen, K. T. *Polymer Degradation and Stability, Predicting the Lifetime of Fluorosilicone O-rings 2009, 94, 2107-2133*

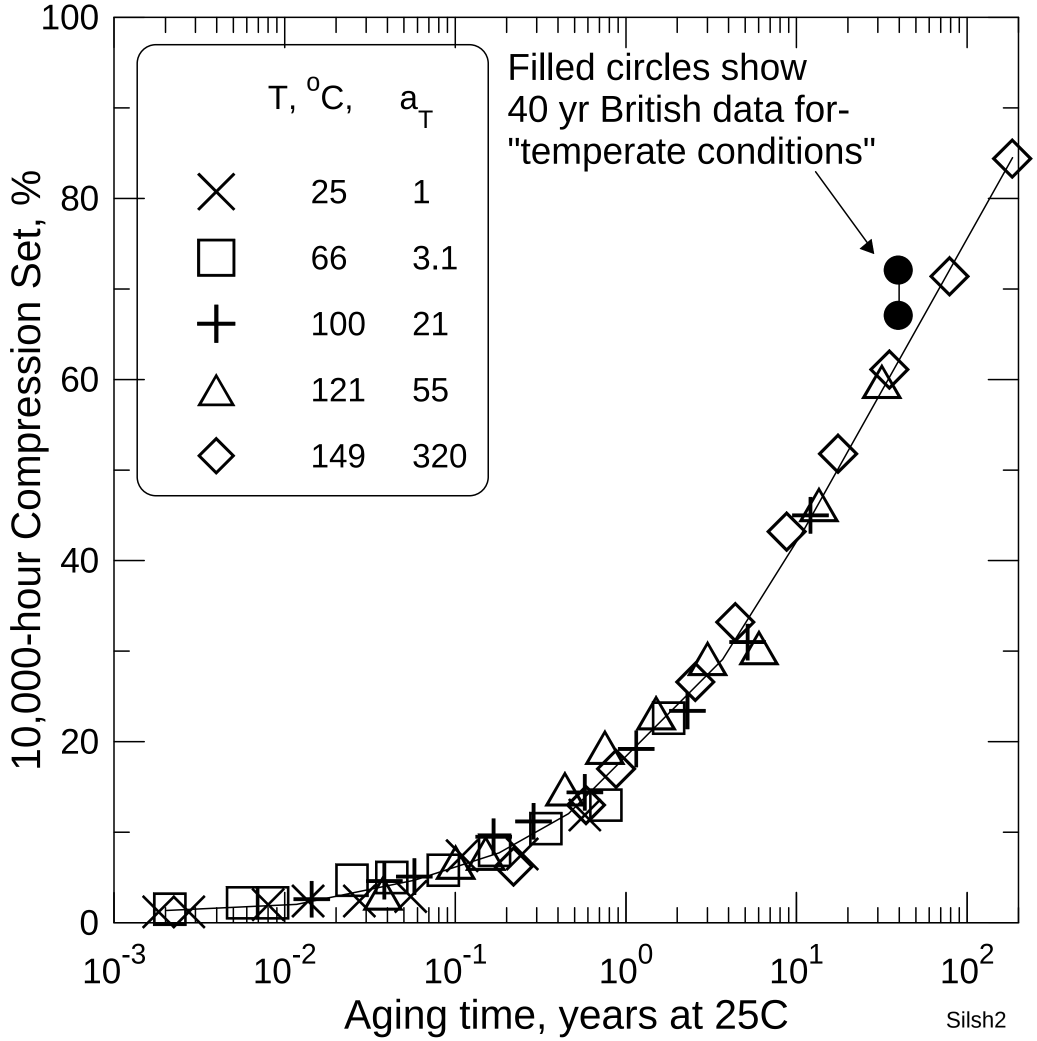
Other Compression Set Data



Different Silicone
Different Program
Different PI

Gillen, K. T. "Silicone seal analysis," Internal Memo, SNL, 2001.

Compression Set

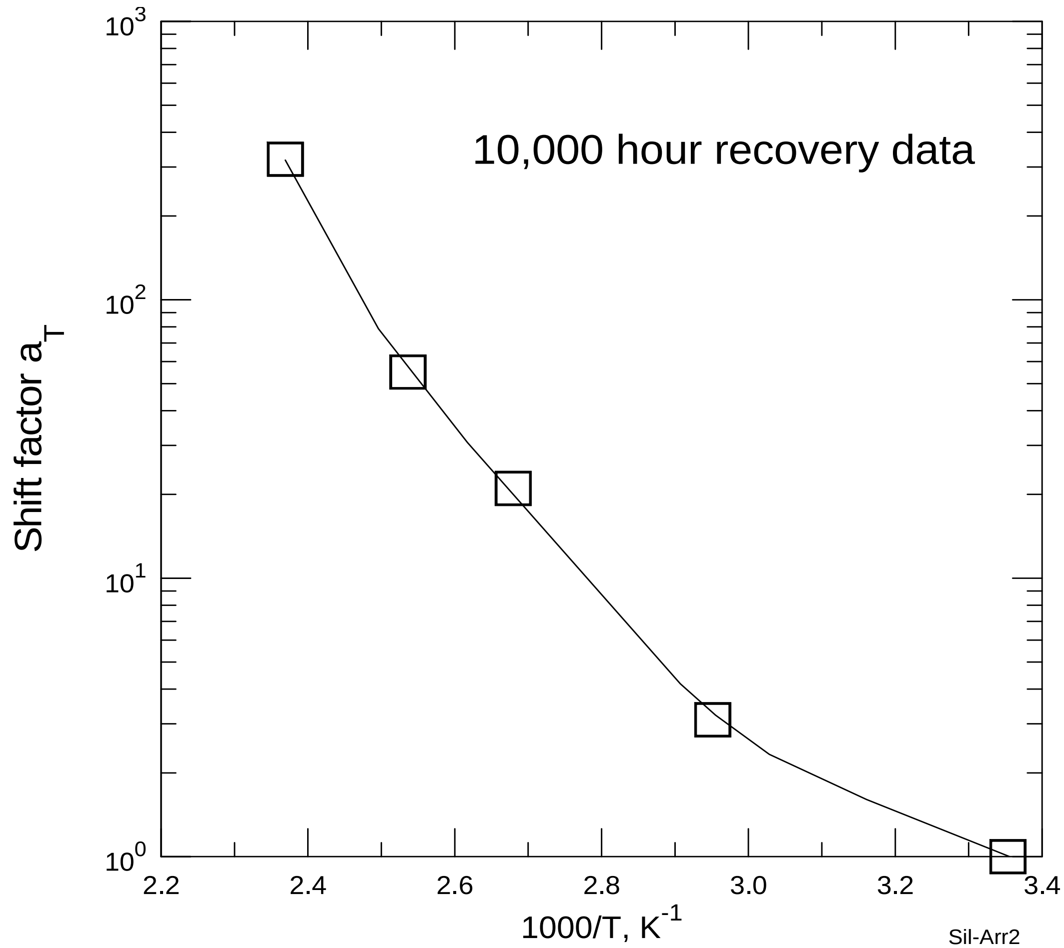


Data Analyzed by Gillen

Added in 40 year RT data from another source

Gillen, K. T. "Silicone seal analysis," Internal Memo, SNL, 2001.

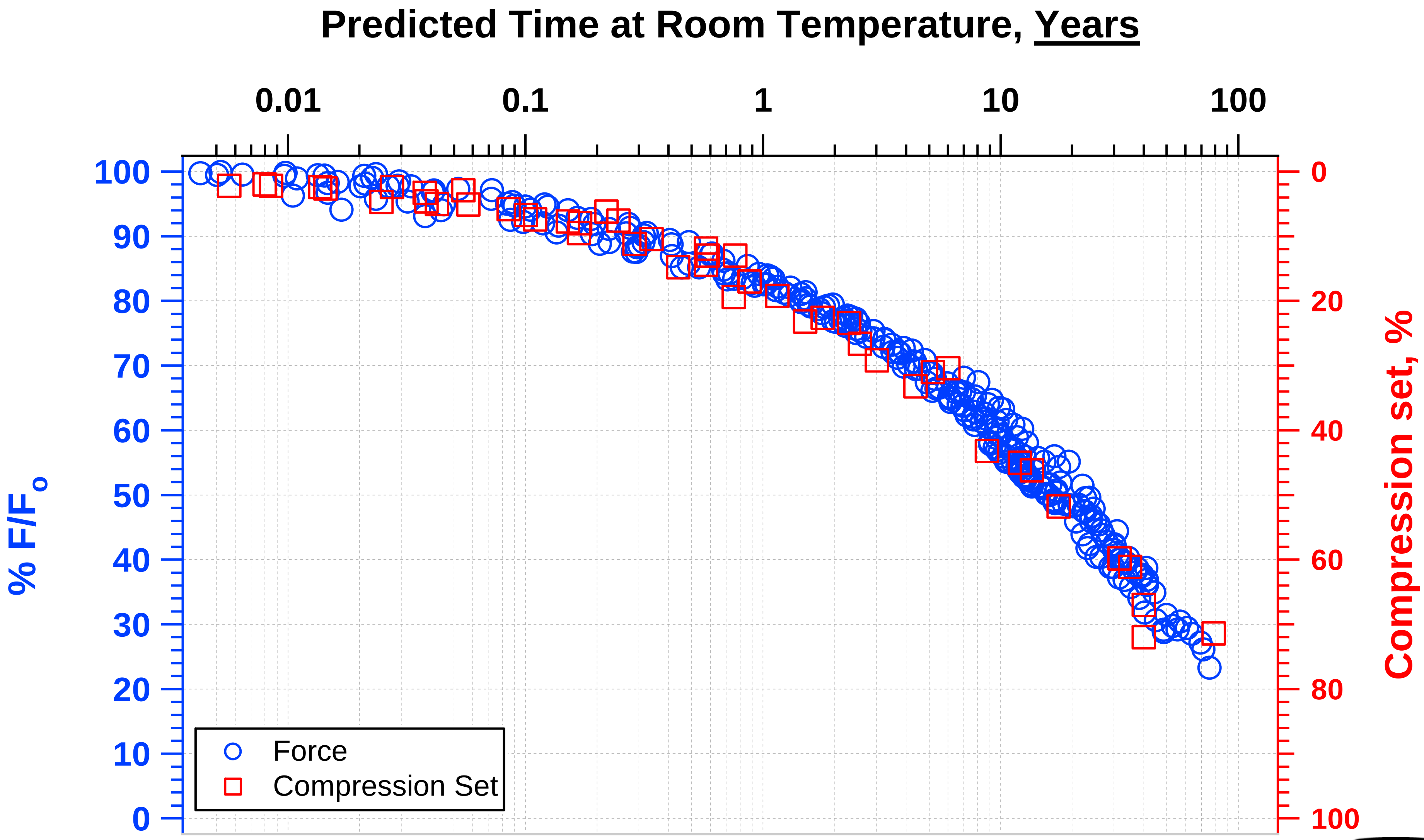
Arrhenius Plot for Compression Set



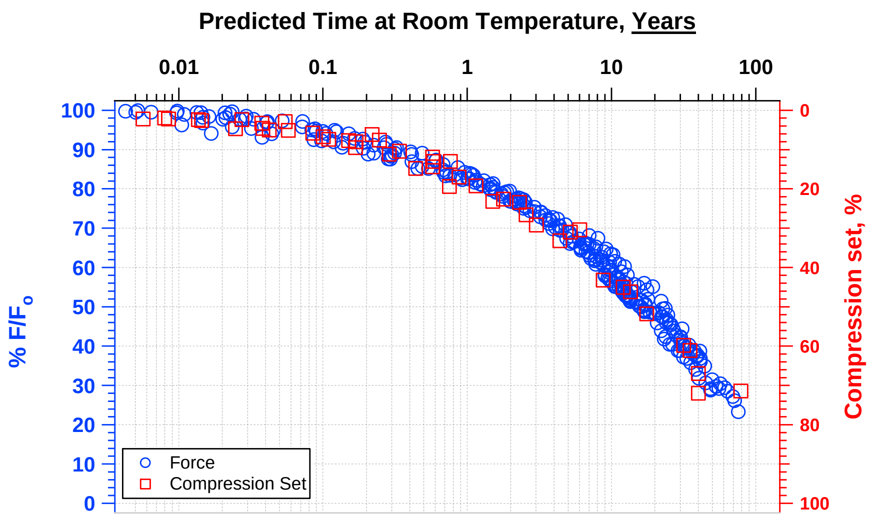
Arrhenius plot of the shift factors for silicone compression set which leads to an aging room temperature prediction for compression set

Gillen, K. T. "Silicone seal analysis," Internal Memo, SNL, 2001.

Force versus Compression Set Data



Force versus Compression Set Data

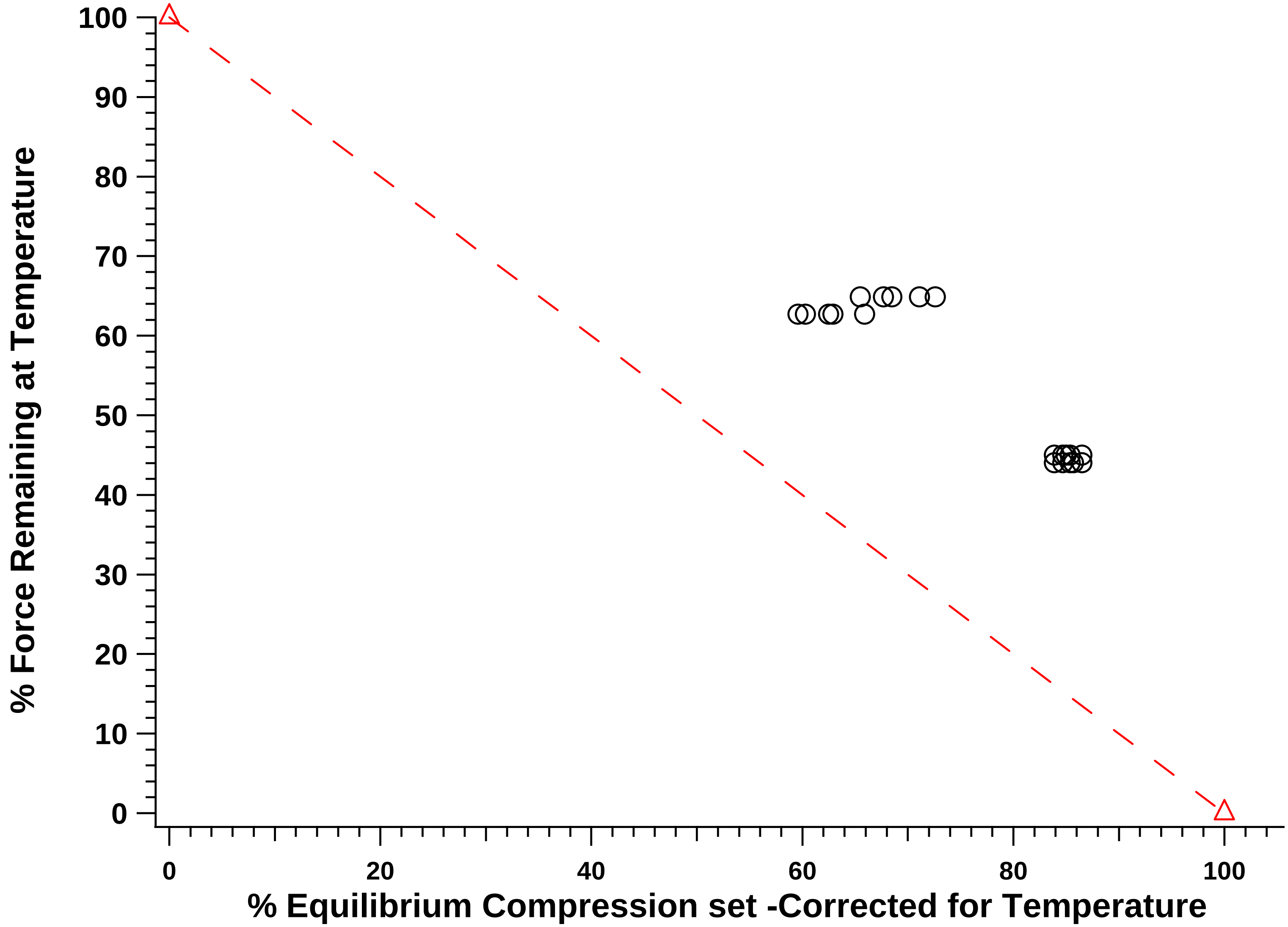


Correlation between current Silicone Force data and Compression set data obtained from *three different sources (and different sizes!)*

Fluorosilicone versus Silicone!!

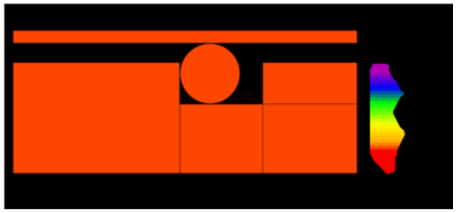
Displays confidence in generalized predictions about silicone o-rings state of health (CS easy to measure) under oxidative environments*

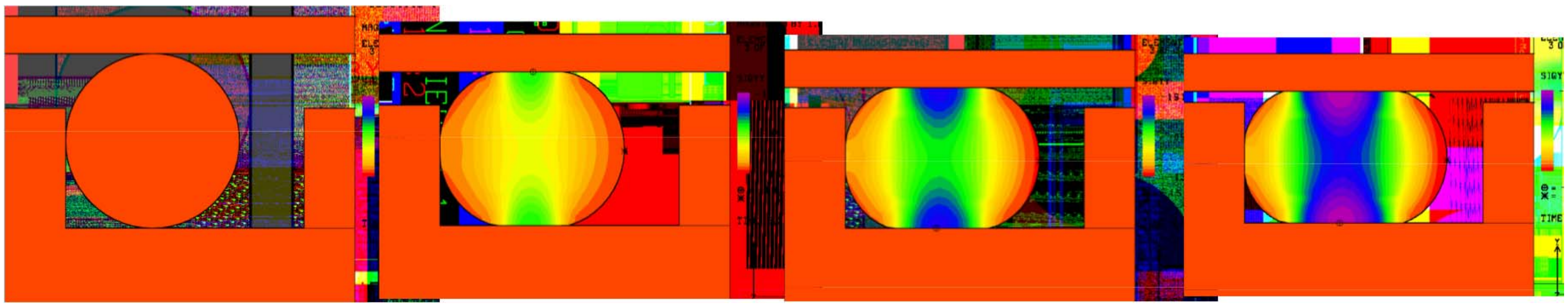
Heavily Filled Silicone



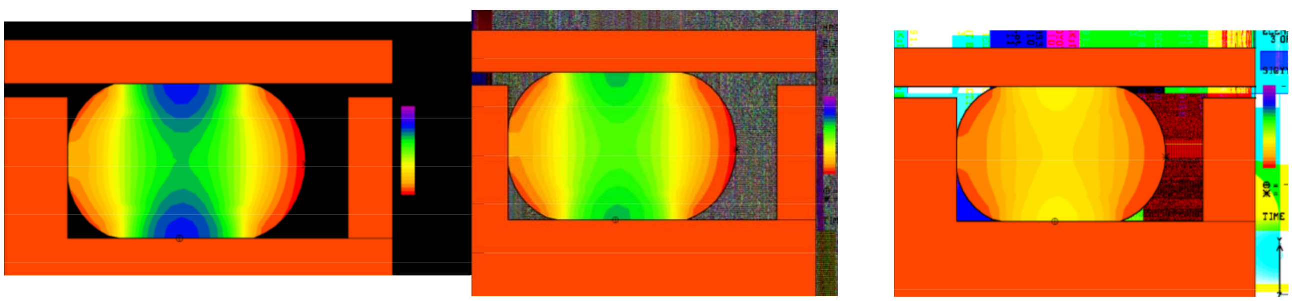
Conducting o-rings compression set versus force remaining

Progression of Stress Relaxation due to Chemical Aging





Time = 0 **Time = .5sec** **Time =.8sec** **Time = 1sec**



Time = 30 years **Time = 56 year** **Time 90 years**

SIGYY

-191 psi=133 N/cm^2

-89 psi= 61 N/cm^2

13 psi= 9 N/cm^2

Slide Courtesy of David Lo

Take home messages…

1) Be aware of mechanism changes
2) Do things for very *very* long time
3) Validate against real world

Lots of help…

Dora Derzon, Brad Hance, Don Bradley, Roger Assink, James Hochrein, Steven Thornberg, David Lo, Kathy Alam, Laura Martin, John Schroeder, Patti Sawyer, Mark Stavig, and Ken Gillen

Questions…