

Eichrom Resin Shelf-life

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Radiobioassay & Radiochemical Measurements Conference



eichrom®

 **RRMC**
Radiobioassay & Radiochemical Measurements Conference

RRMC 2014
Knoxville, Tennessee USA
October 27 - October 31, 2014

Eichrom Quality System

- ISO 9001:2008
 - quality management systems standards
 - demonstrate the ability to consistently provide product(s) that meet customer requirements.
 - aim to enhance customer satisfaction
 - effective application of the system
 - continual improvement of the system
- Produce and conform to procedures governing:
 - sourcing or producing quality raw materials
 - converting raw materials into finished products
 - testing finished products for initial quality
 - re-testing products to evaluate quality after a time period

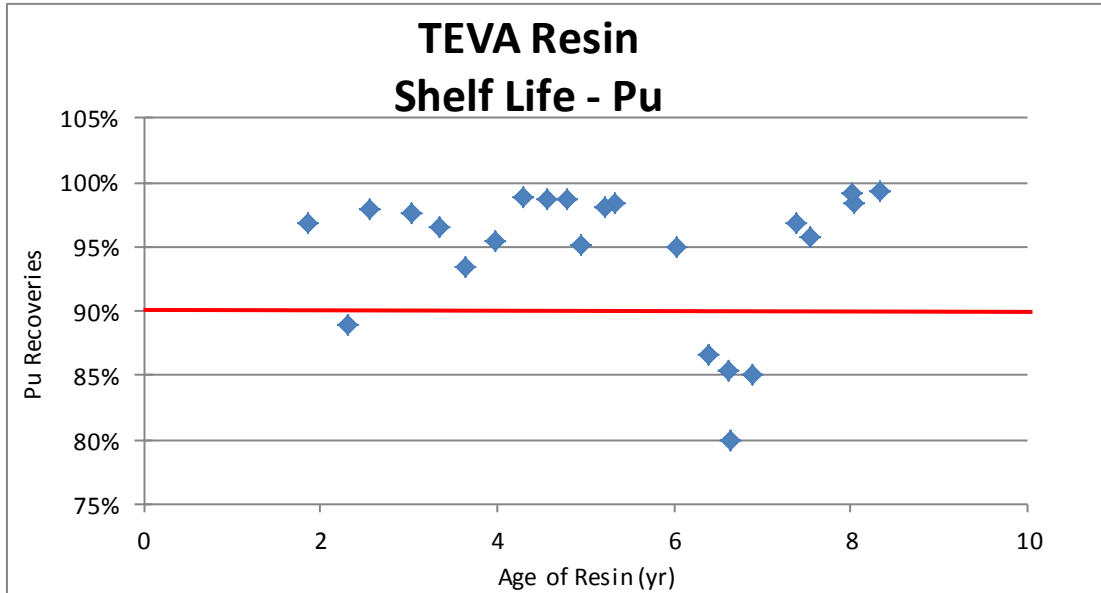
Resin Shelf-life testing

- Started with Sr Resin in 2008 due to customer comments
- Extended to TEVA, TRU, UTEVA, DGA in 2009-2010 to support agencies desiring to stockpile resins for emergency preparedness.
- Has resulted in numerous improvements:
 - Raw material purification
 - Production techniques
 - Quality control testing procedures
- Prior to 2005, QC
 - Batch Uptake Measurements
 - Limited column separation testing
- In 2005-2007, QC
 - Cartridge/Column Separations
 - At least 2 analytes

Good News!!!

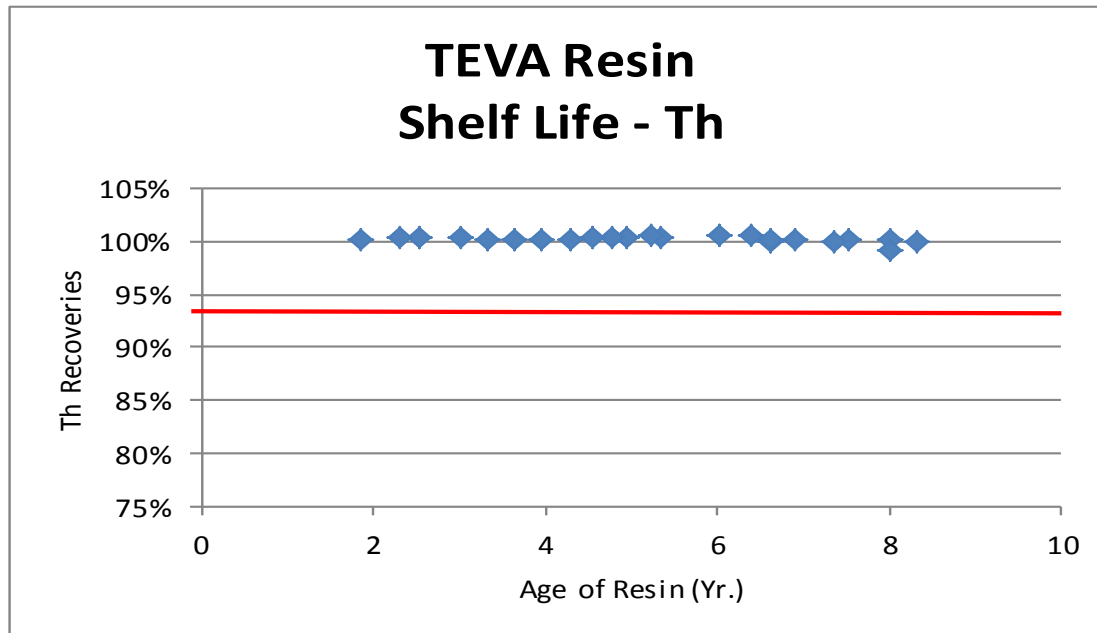


TEVA



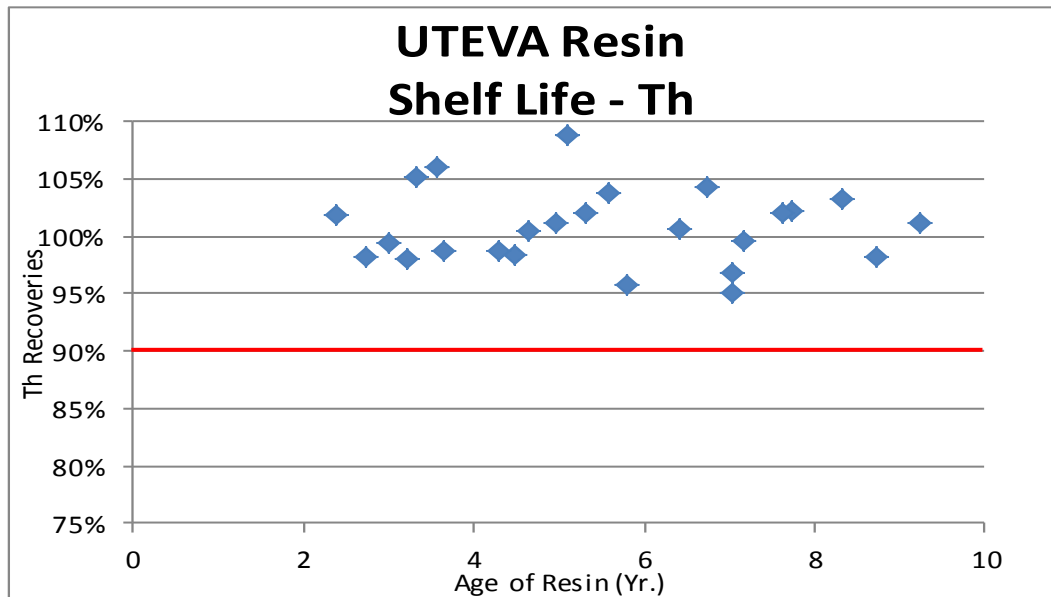
Recovery in
0.02M HCl-
0.02M HF

Performed in
2010 on lots
from 2001-
2008.



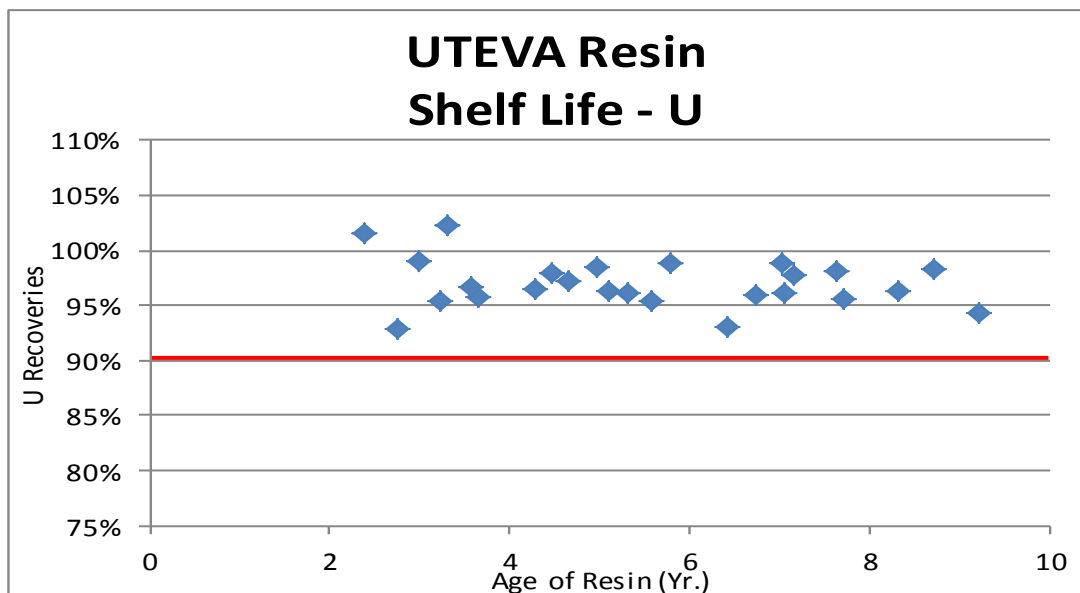
Recovery in
6M HCl

UTEVA



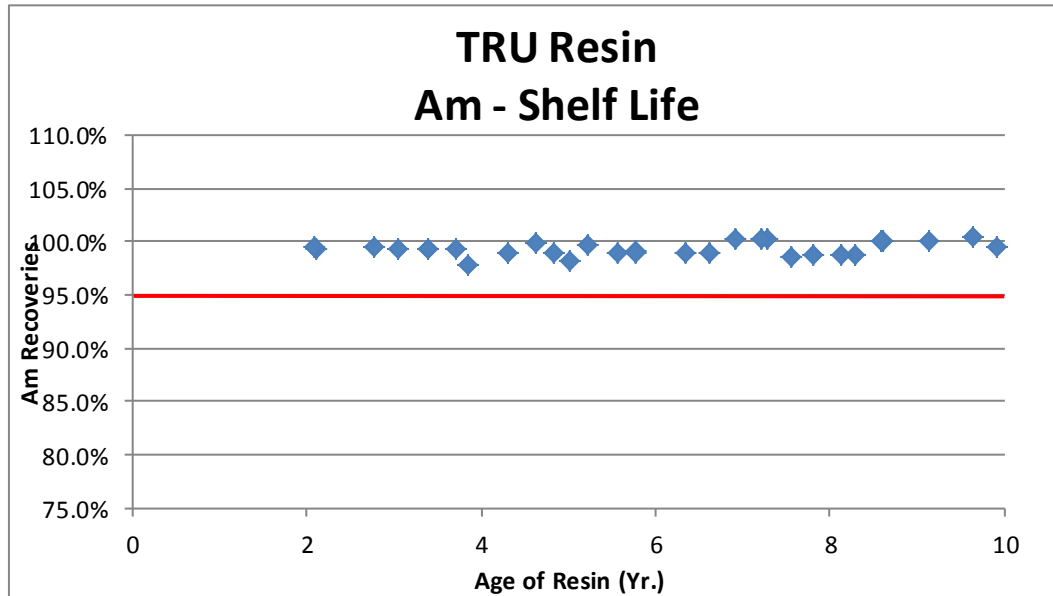
Recovery in
5M HCl-
0.05M Oxalic
Acid.

Performed in
2010 on lots
from 2001-
2008.



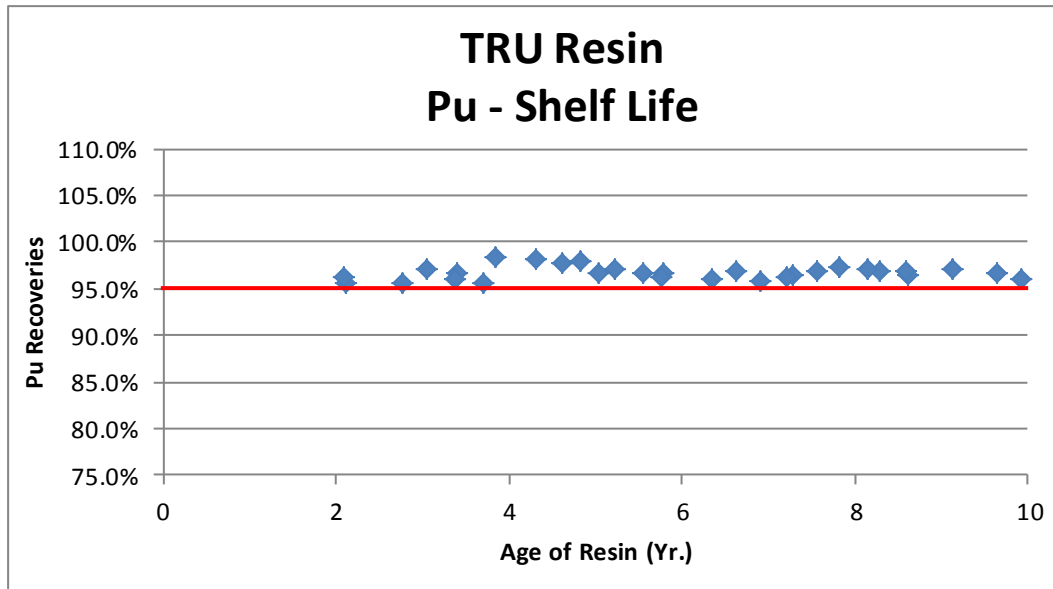
Recovery in
1M HCl

TRU Resin



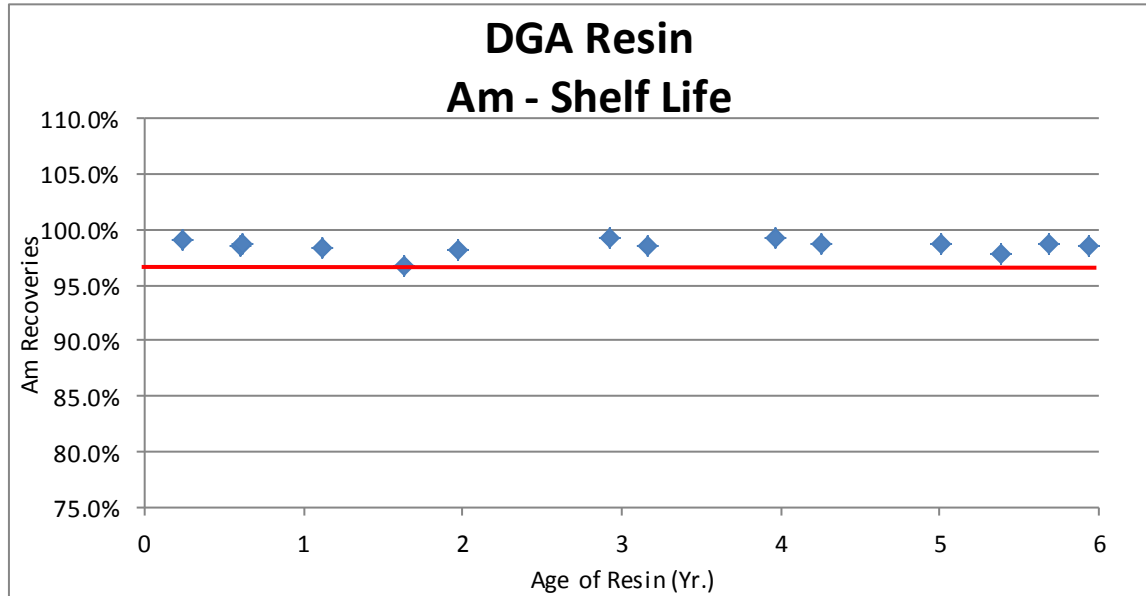
Recovery in
4M HCl

Performed in
2009 on lots
from 2000-
2008.



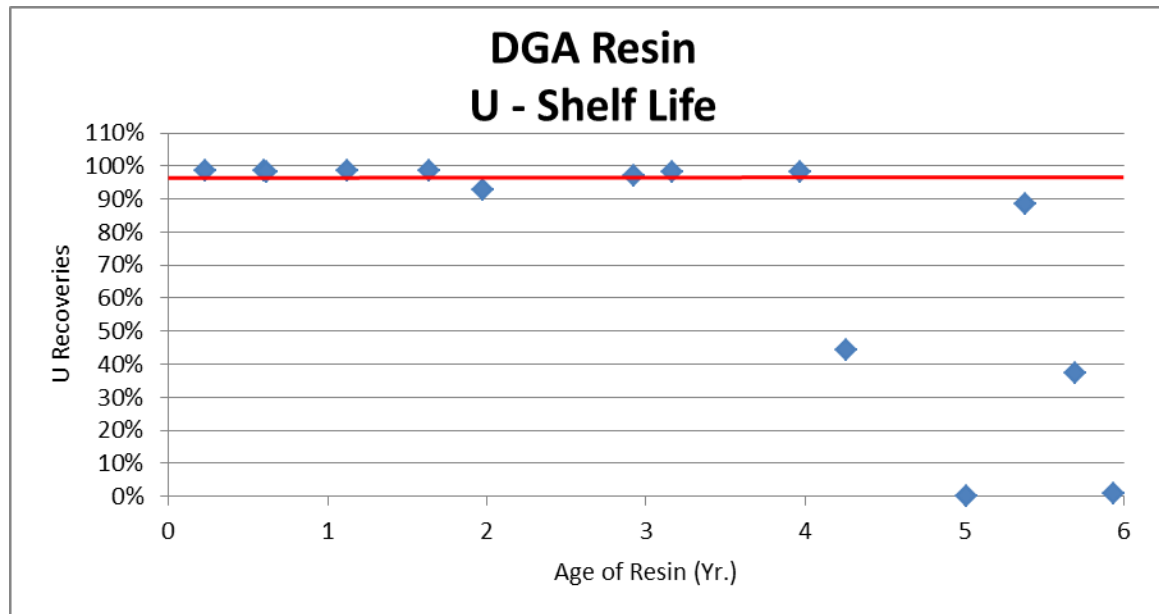
Recovery in
0.1M
ammonium
biocalate

DGA Resin



Recovery in
0.1M HCl

Performed in
2010 on lots
from 2004-
2010.

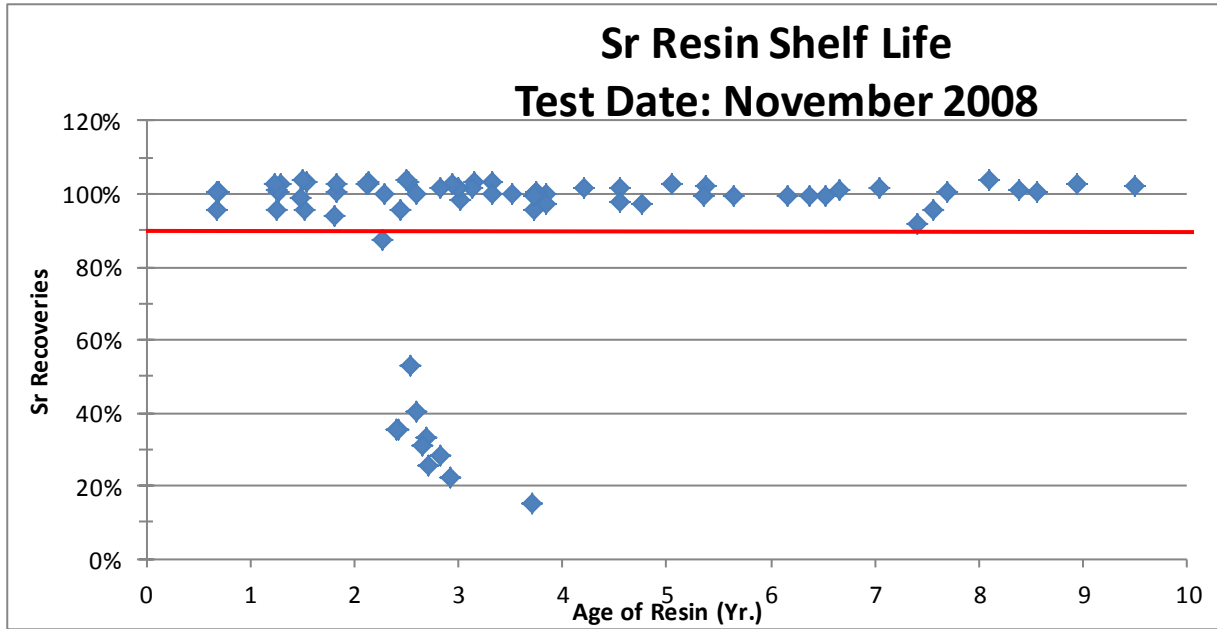


Recovery in
0.1M HNO₃

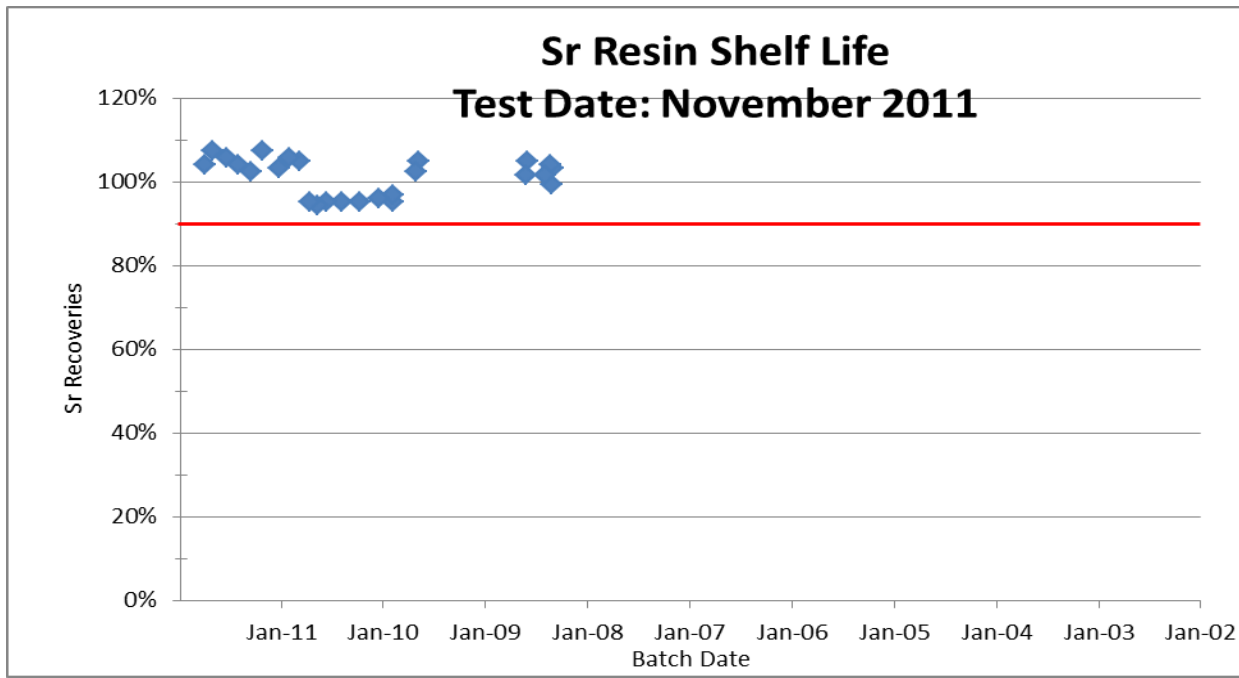
Sr Resin



Sr Resin



Recovery in
0.05M HNO₃
1998-2007
lots



Recovery in
0.05M HNO₃
2008-2011
lots

Parameters Correlating with failures

- 1) Bulk Resin (not columns or cartridges)
- 2) Failure at >1.5 years from production date
- 3) Exposure to high (>30°C) or low temperature (<4°C).

Testing points to physical issue, not chemical degradation

- loss of 1-octanol diluent (smell)
- waxy/solid crown ether
- re-formulating resin with recovered crown

Frequencies of Failures

Original Testing (2008)

December 2005 – June 2006 MAJOR failures

-8 out of 11 failed after 2 years

-2007 no failures

2011 Testing

No failures for 2008-2011 lots

Subsequent Testing

2008 2 failures

2009 2 failures

2010 1 failure so far

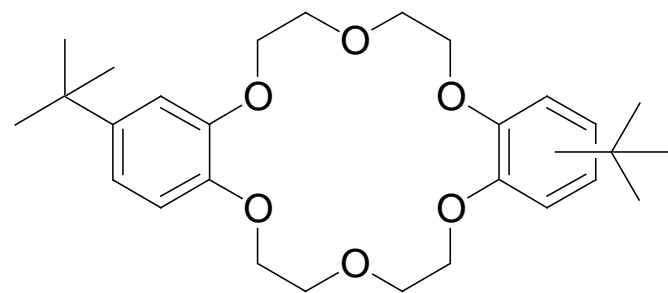
2011 no failures to date

2012 1 failure

2013 no failures to date

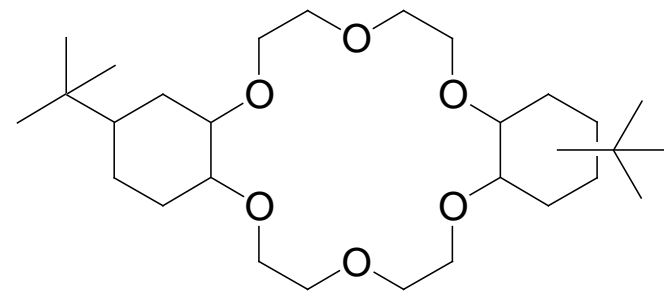
2014 no failures to date

Sr Resin Production



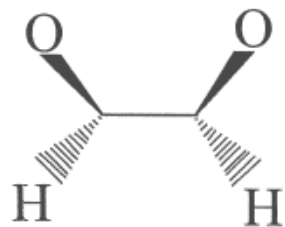
4,4'(5')-di(t-butylbenzo)18-crown-6

H₂, catalyst

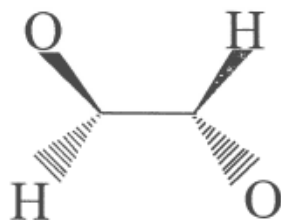


4,4'(5')-di(t-butylcyclohexano)18-crown-6

2 isomers with different connectivity of *t*Bu group.

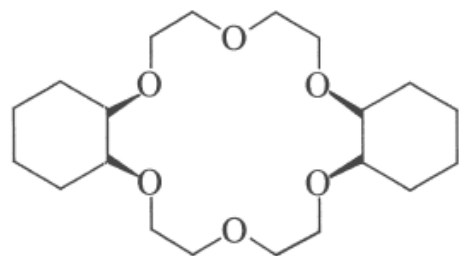


cis

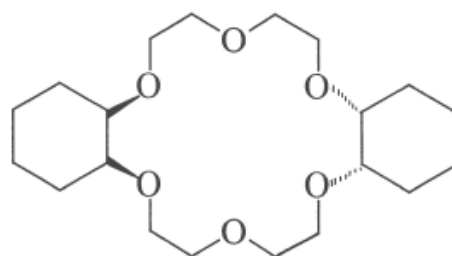


trans

40 possible stereoisomers

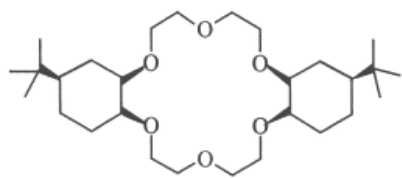


cis-syn-cis-dicyclohexano-18-crown-6

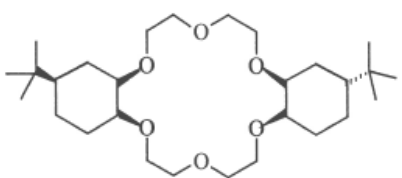


cis-anti-cis-dicyclohexano-18-crown-6

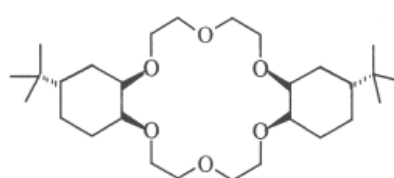
12 cis-cis isomers are predominant.



4(z),4'(z)-cis-syn-cis

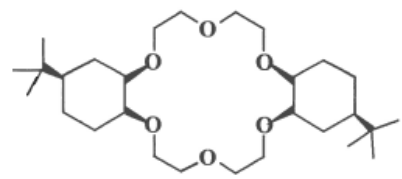


4(z),4'(e)-cis-syn-cis

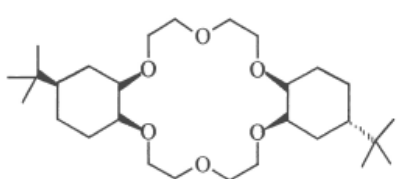


4(e),4'(e)-cis-syn-cis

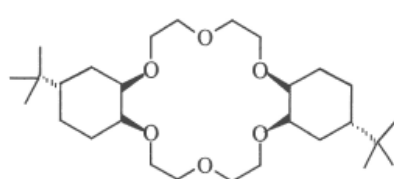
9 cis-cis isomers are responsible for Sr retention



4(z),5'(z)-cis-syn-cis



4(z),5'(e)-cis-syn-cis

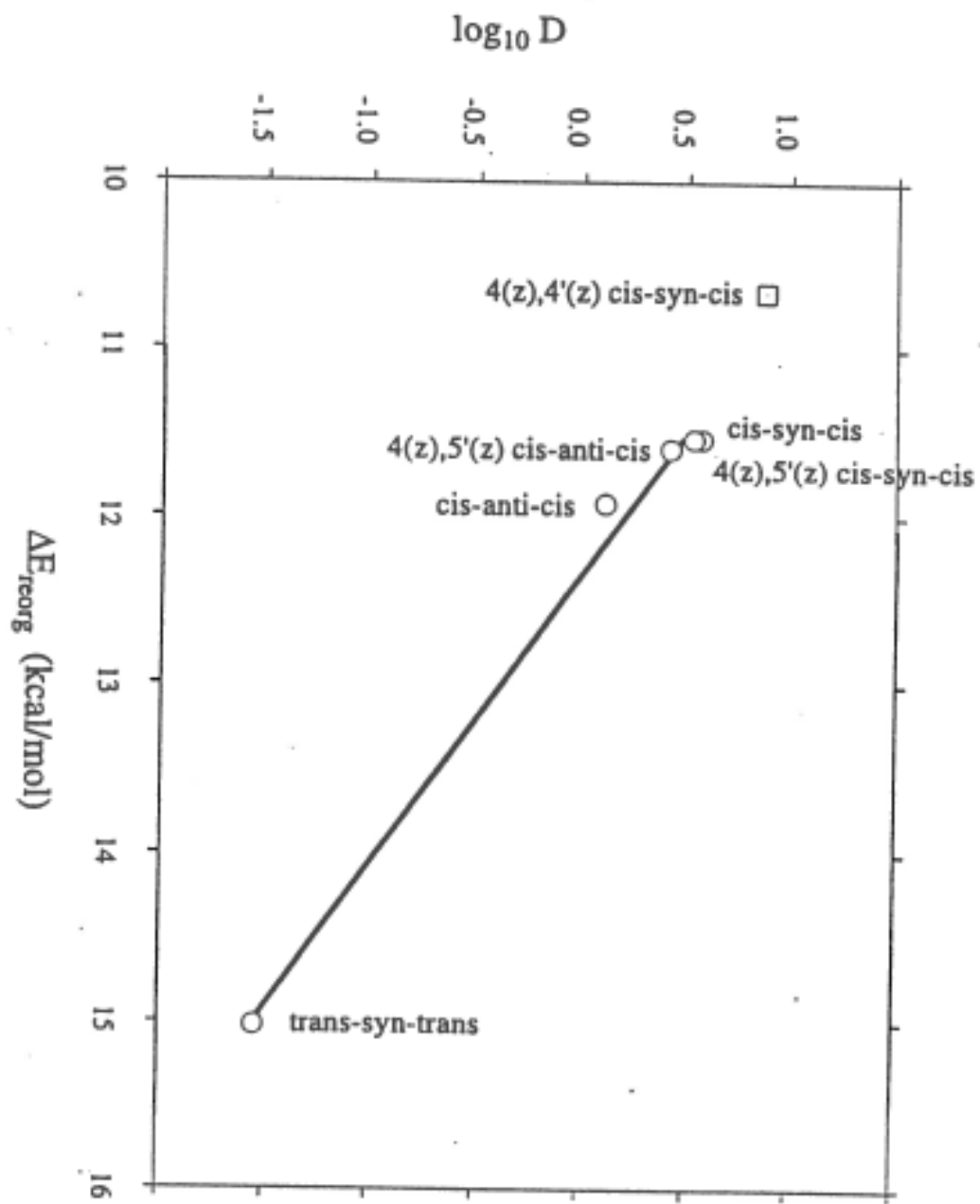


4(e),5'(e)-cis-syn-cis

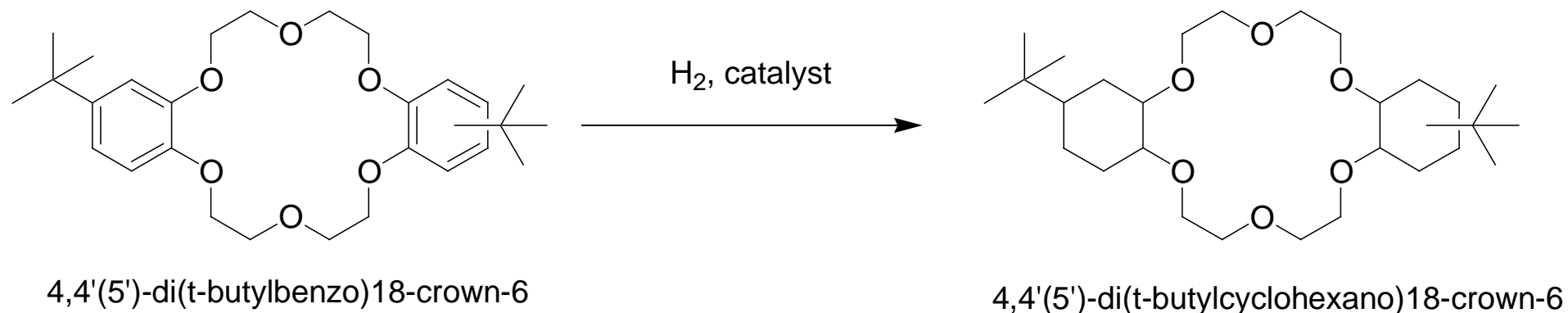
Table 1. Predicted D_{gr} at 0.1 M ligand concentration for the n-octanol:1 M nitric acid system.

4(z),4'(z) <i>cis-syn-cis</i>	9.50±1.6	4(z),5'(z) <i>cis-syn-cis</i>	3.00±0.5
4(z),4'(e) <i>cis-syn-cis</i>	5.79±1.0	4(z),5'(z) <i>cis-anti-cis</i>	2.77±0.5
4(e),4'(e) <i>cis-syn-cis</i>	4.93±0.8	4(e),5'(e) <i>cis-anti-cis</i>	2.52±0.4
4(z),5'(e) <i>cis-syn-cis</i>	4.57±0.8	4(e),4'(e) <i>cis-anti-cis</i>	0.052±0.009
4(e),5'(e) <i>cis-syn-cis</i>	3.67±0.6	4(z),4'(z) <i>cis-anti-cis</i>	0.064±0.010
4(z),4'(e) <i>cis-anti-cis</i>	3.21±0.5	4(z),5'(e) <i>cis-anti-cis</i>	0.022±0.004
<i>cis-syn-cis</i>	3.00±0.5	<i>trans-syn-trans</i>	0.028±0.005
<i>cis-anti-cis</i>	1.79±0.3	<i>trans-anti-trans</i>	0.0004±0.0001
18-crown-6	0.77±0.13		

M. Dietz, et al. "Comparison of Column Chromatographic and Precipitation Methods for the Purification of a Macrocyclic Polyether Extractant, Sep. Sci. Tech., 34(15), 2943-2956 (1999)



Sr Resin Production



- 1) Small test batch – Dw Sr from 3.1M HNO₃
- 2) Blend crown lots to consistent Dw Sr value (120-130)
- 3) Full batch - Dw Sr from 3.1M HNO₃
 - Gravimetric Recovery/Blank Residue
 - Stability in 8M HNO₃
 - Sr Recovery from Ca/Ba/Y matrix

Recommendations

- 1) If stockpiling, consider Sr Resin cartridges or columns
- 2) Exposure to high ($>30^{\circ}\text{C}$) or low temperature ($<4^{\circ}\text{C}$).
- 3) Tightly seal resin bottles and repackage into smaller containers as necessary.