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Title: Gas Analysis for LANL Weapons Surveillance

Author(s): Chris Leibman, Crystal Densmore, David Dogruel, and David Martinez

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Gas Analysis for LANL Weapons Surveillance

Abstract: Gas analysis is performed at LANL as one part of weapons surveillance conducted to identify stockpile aging concerns and to predict the lifetime of weapon components and materials. These analyses include gas dissolved in and evolved from polymeric materials. Radiolysis and thermal decomposition of polymers are examples of processes which lead to gas production. These are accessed using canister aging studies with gas analysis typically performed using gas chromatography and gas chromatography/mass spectrometry. More recently we have developed methods to measure dissolved gases in polymeric materials suspect in the failure of potting materials. This talk provides a brief overview of recent work.

Gas Analysis for LANL Weapons Surveillance

DAMM

November 16, 2011

**Chris Leibman, Crystal Densmore, David
Dogruel, David Martinez**

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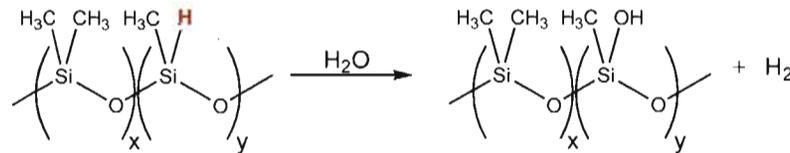
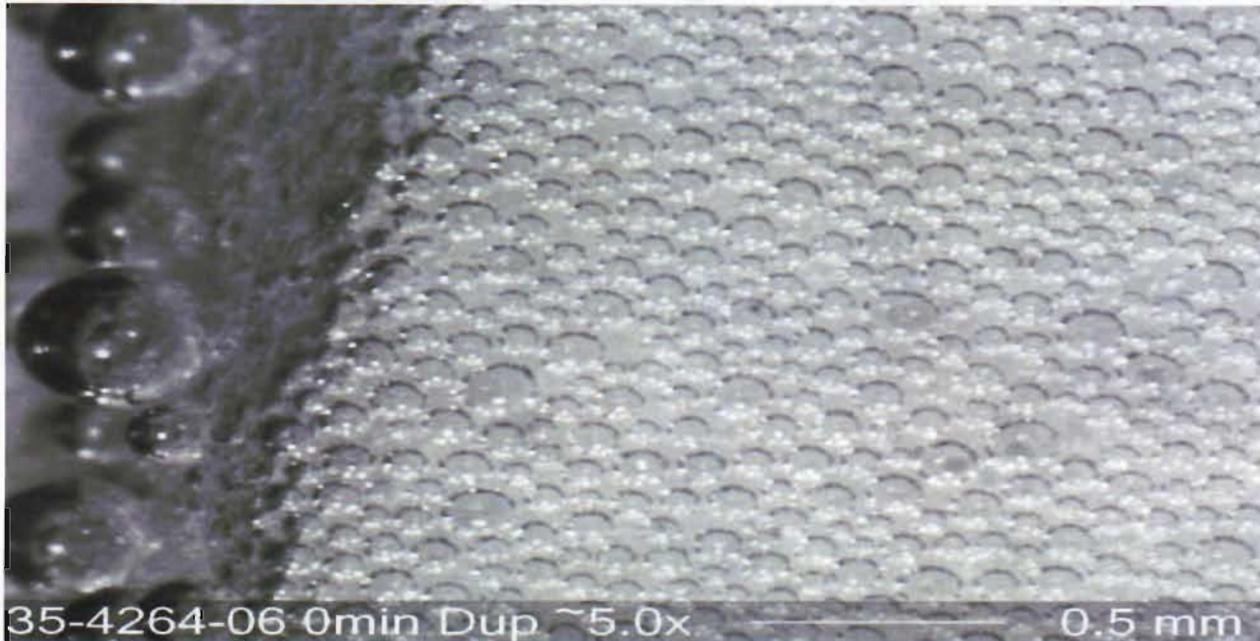
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Gas Analysis Overview

- Dissolved Gases in Polymeric Materials
 - Karl Fisher
 - Headspace Gas GC/MS
 - Purge & Trap GC/TCD/HePDD
- Polymer Aging Studies – Off-gas from Thermal Degradation and Radiolysis
- Emergency Response
 - Canister Based Air Sampling
 - Solid Phase Microextraction (SPME)

Trapped Gas in Cured Sylgard 184 Results in Parts Rejection



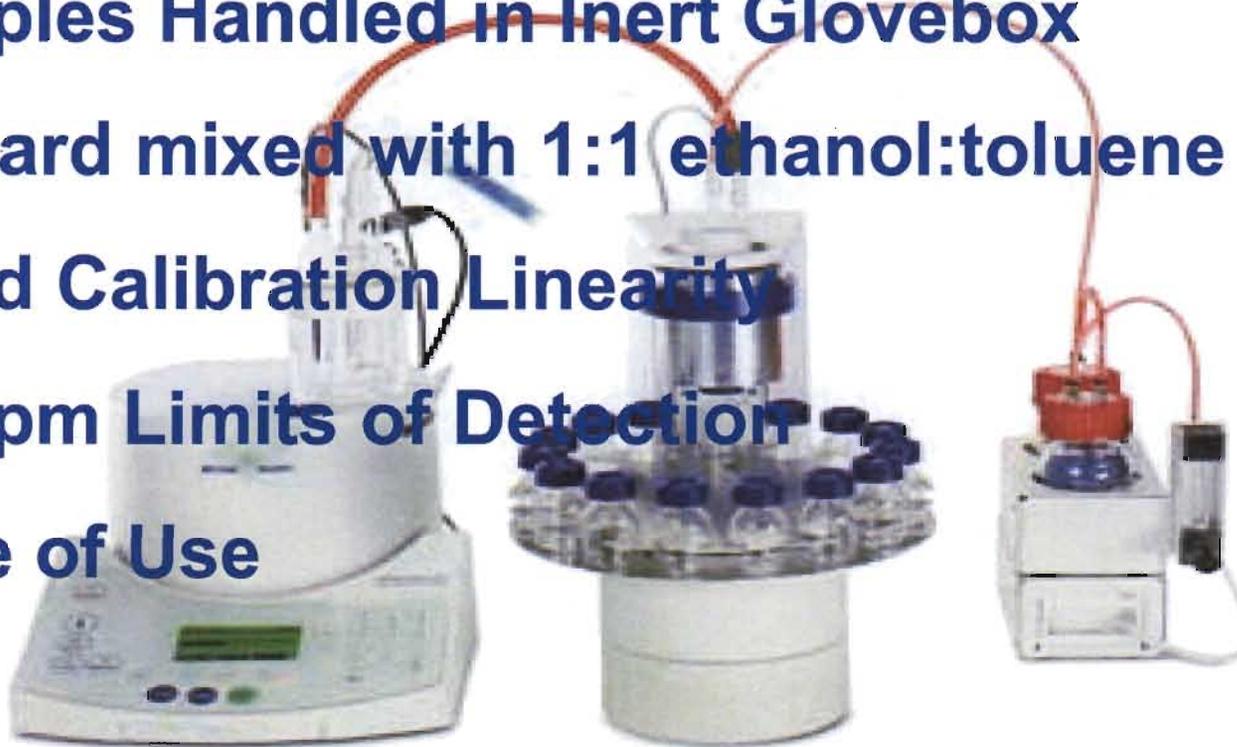
LANL Method Development for Determining Water Content in Uncured Sylgard 184

Potential Basis for Sylgard 184 Shelf Life Extension and Acceptance

- Water is a contributor to champagne bubble formation in Sylgard 184.
- Water difficult to measure in Sylgard 184.
- Principal Methods Developed & Evaluated by LANL
 - Headspace Gas/GC/MS
 - Purge and Trap/GC
 - Karl Fisher Coulometry
- Analyses were performed on 2 Pantex Lots received 6/15/2011 – 12 Samples, Sealed in Al foil barrier bags prepared at Pantex ~ one month earlier.
 - 6 samples then stored in Drybox (< 0.2 ppmv water vapor).
 - 6 samples Stored in Environmental Chamber under “Extreme Conditions” of 85% RH/85 °F.

Karl Fisher Coulometry Developed as Preferred Method

- **Samples Handled in Inert Glovebox**
- **Sylgard mixed with 1:1 ethanol:toluene**
- **Good Calibration Linearity**
- **50 ppm Limits of Detection**
- **Ease of Use**



Data Suggest Sylgard 184 Shelf Life can be Extended

Water Content Same - Independent of Storage Conditions

Pantex Sample Number	Expiration Date	(mg water/g Sylgard)	Sylgard Kit 3 month Storage Condition
078-1_B	6/18/2011	0.253	Dry
078-5_B	6/18/2011	0.262	Dry
079-1_B	6/19/2011	0.184	Dry
079-3_B	6/19/2011	0.283	Dry
078-2_B	6/18/2011	0.202	Wet
078-3_B	6/18/2011	0.283	Wet
079-4_B	6/19/2011	0.192	Wet
079-6_B	6/19/2011	0.219	Wet

Data Suggest Sylgard 184 Shelf Life can be Extended

- Moisture content of drybox stored samples and samples stored under “extreme” conditions equivalent 4 months after kit preparation.
 - Water measured at ~200ppmv in all samples.
 - Aluminum foil barrier bags with desiccant seem to prevent uptake of additional water in Sylgard 184.
- Extension of Sylgard 184 shelf life maybe warranted **provided**
 - Aluminum foil barrier bag integrity verified using bag compression test prior to use
 - Desiccant use in storage bags is continued.
- Karl Fisher Coulometry – Simple turnkey method for water analysis
 - Analyze Reserve Samples at LANL for further shelf life extensions.
 - Develop calibration standards for use at Pantex.

Gas Analysis using GC/MS - Surveillance and Emergency Response Support

- Polymer Aging Studies – Off-gas from Thermal Degradation and Radiolysis
- Emergency Response
 - Prevented needless parking lot excavation
 - False Positive from Handheld Instrument
 - GC and GC/MS confirmed absence of Natural Gas
- Air Sampling
 - Passivated Canisters
 - Solid Phase Microextraction (SPME)
 - Analyze within Hour/Report

JEOL GCMate - High Resolution GC/MS

■ Benchtop GC/double-focusing mass spectrometer

- high sensitivity, high resolution (up to 5,000 at 10% valley) and accurate mass measurement for determining elemental compositions.
- 44 - propane, C_3H_8 , and ethanal, CH_3CHO .
- C_3H_8 44.0624 CH_3CHO 44.0261



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