Dense Phase CO2 Outgassing and Cleaning of Critical Components: Principles, Methods, and Equipment

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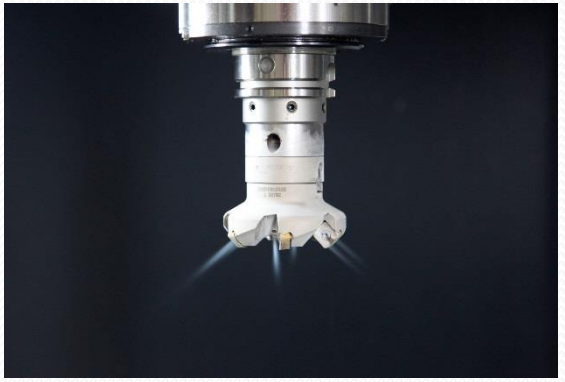
Who Is Cool Clean Technologies?

- A company that uses CO_2 in all phases:
  - for cleaning and surface preparation of precision surfaces;
  - machine tool cooling and lubrication for precision machining applications;
  - selective extraction.
- Strong Proprietary Products - Patents and Know-How
- All our processes for all these applications have these important attributes:
  - Processes are effectively dry;
  - Processes generate Zero to trace byproducts;
  - Applications are environmentally friendly and use environmentally safe products and energy efficient processes;
  - User - not a producer of Carbon Dioxide (CO2).

Core Competencies

- Spray Cleaning:
  - Particle Removal;
  - Residue Removal;
  - Surface Preparation / Functionalization.
- Machine Tool Cooling and Lubrication:
  - Dry machining (hard turning and hard milling)
  - Plastic machining
  - Opening machines, VTLs, Gang lathes
  - Drilling thru spindle
- Dense Phase Extraction/Cleaning:
  - Precision Degreasing;
  - Silicone Extraction;
  - Aerospace Outgassing;
  - Porous Metal Cleaning;
  - Garment Cleaning
  - Botanical Oil Extraction.

Spray Cleaning – Particle/Residue Removal



Machine Tool Cooling and Lubrication

Dense Phase Extraction / Cleaning

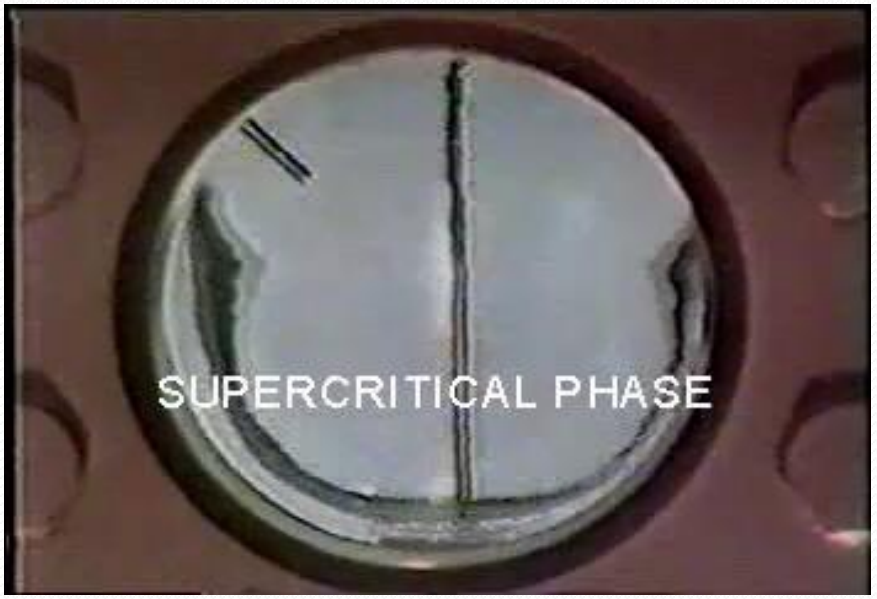
Outline

- CO2 Technology Background
- Process and Systems Overview
- Liquid CO2 Extraction of Silicones
- Aerospace Component Cleaning

Presentation contains information previously presented by authors and by former colleagues – J. Elders & W. Elias, Raytheon, “LCO2 Cleaning of Space Hardware Assemblies”, Presented at CCMPP meeting July 2015.

CO_2 Technology Background

Dense Phase CO2

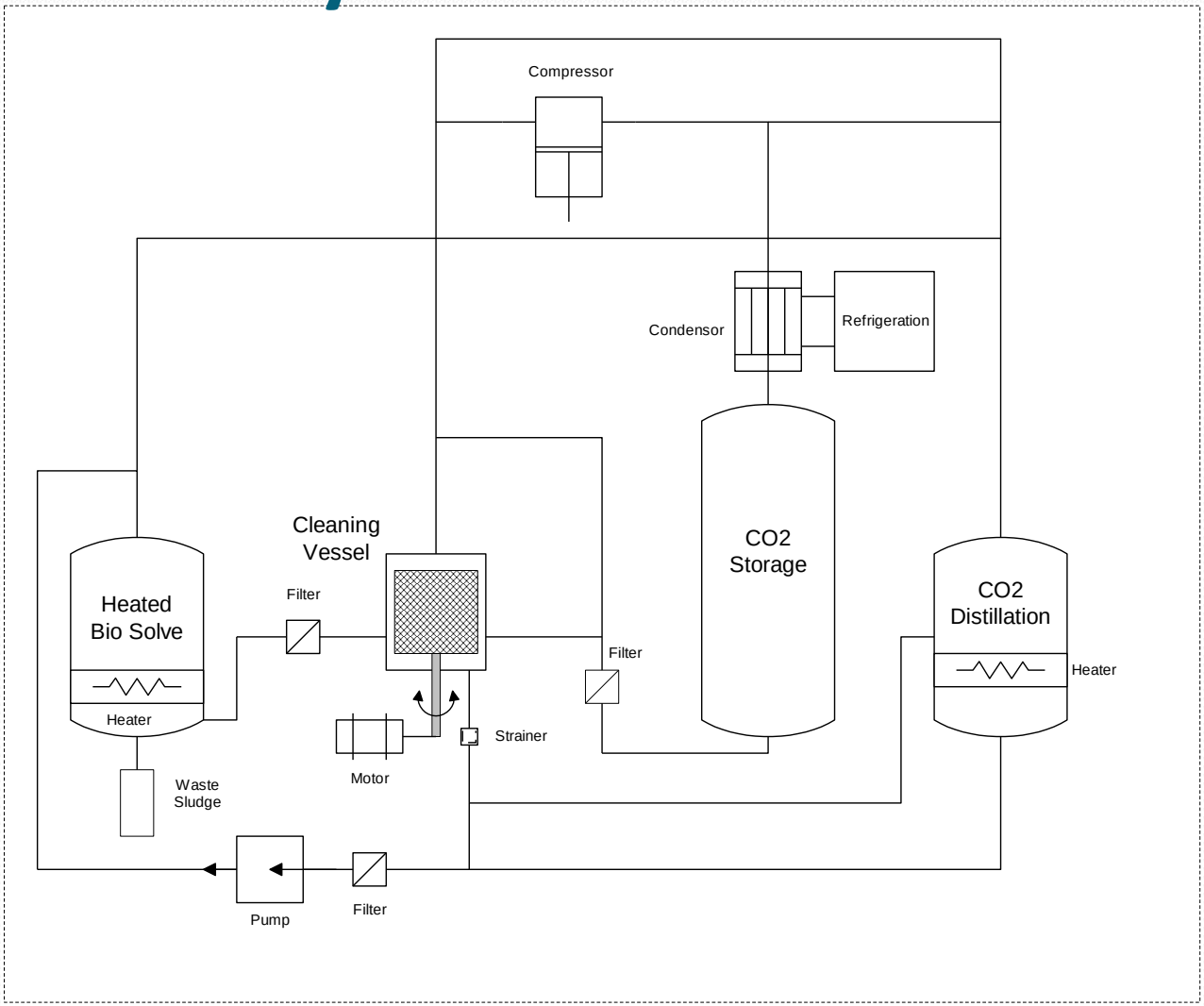


Key Properties Favorable for Dense Phase CO2 Cleaning

- Low Viscosity – Only about 8% of H_2O viscosity
- Low Surface Tension – Only about 7% of the H_2O surface tension
- Variable Solubility – depending on operating pressure and temperature
- Solubilizes many other co-solvents – alcohols, ketones, glycol ethers

How Enertia™ LCO2 System Works

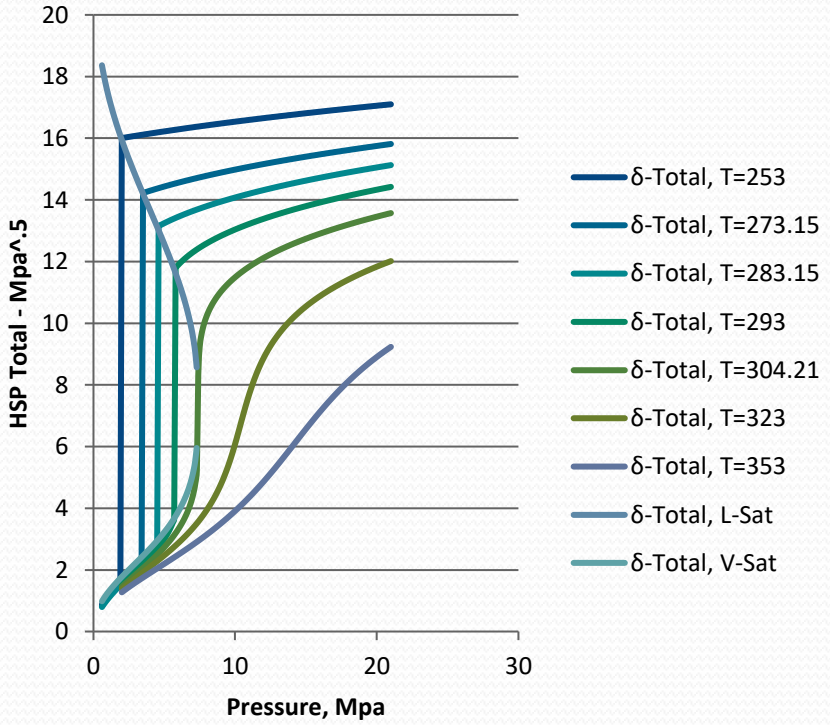
- CO2 Chemistry
  - Alone (neat)
  - In combination with soluble additives
  - In combination with High Boiling Solvents (HBS)
  - Solvency is Tunable
- Operating conditions:
  - Temperature: -50 to 200°C
  - Pressure: 6 to 250 bar.
- Cleaning Mechanism:
  - Tunable solvent cleaning power + physical cleaning action.



CO_2 as a 'Tunable' Cleaning Solvent

- CO2 solubility variable with temperature and pressure variations.
- Three CO2 Solvent Chemistries:
  - CO2 Only (Neat)
    - Silicone / Phthalate Extraction
    - Aerospace Outgassing Alternative
  - CO2 with Co-Solvent
    - Advanced chemistries: alcohols, ketones, others
    - Oil removal, films, stains, disinfection / sterilization
  - HBS prewash with CO2 Rinse
    - HBS moves residue from part into solution, CO2 rinse's part with HBS
    - Effective for: heavy degreasing, dry cleaning, shop rags, polishing compound removal, porous metal manufacturing debris

HSP for CO2 at Various Pressures and Temperatures



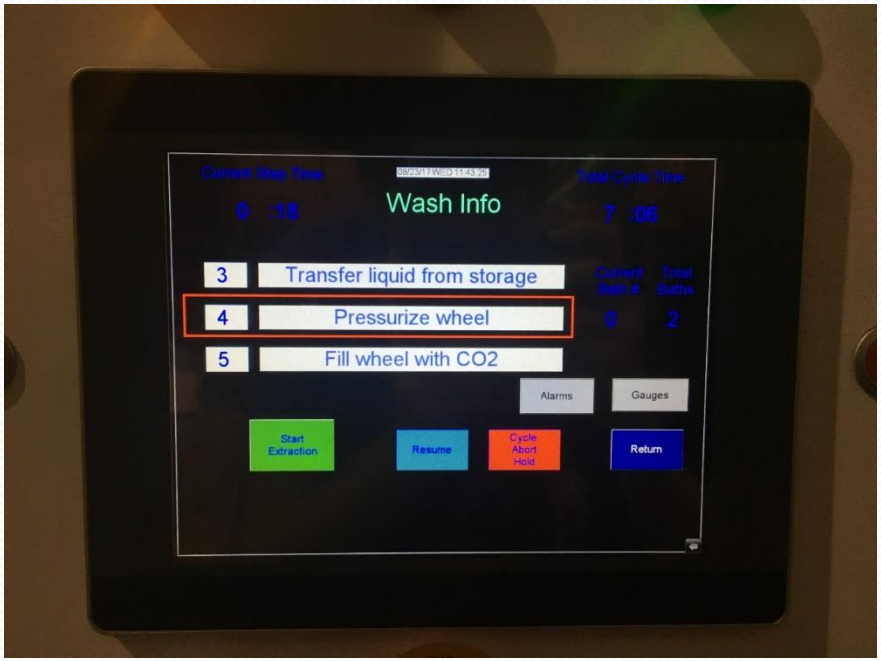
Enertia™ LCO2 Extraction System – CFx - Top Loader

Enertia™ LCO2 Extraction Systems – GFx - Front Loader

LCO2 Level in GFx Cleaning Vessel

Key features of Enertia™ Dense Phase Systems

- Easy operator interface
  - Simple loading/unloading
  - Automatic extract delivery
  - Push-button operation
  - Easy filter cleaning
  - Data logging of run info
- Large capacity extraction vessel volume
  - Ideal for low density botanicals
- Large CO2 throughput
- Effective CO2 Recycling
  - +99% of CO2 Processed is recycled

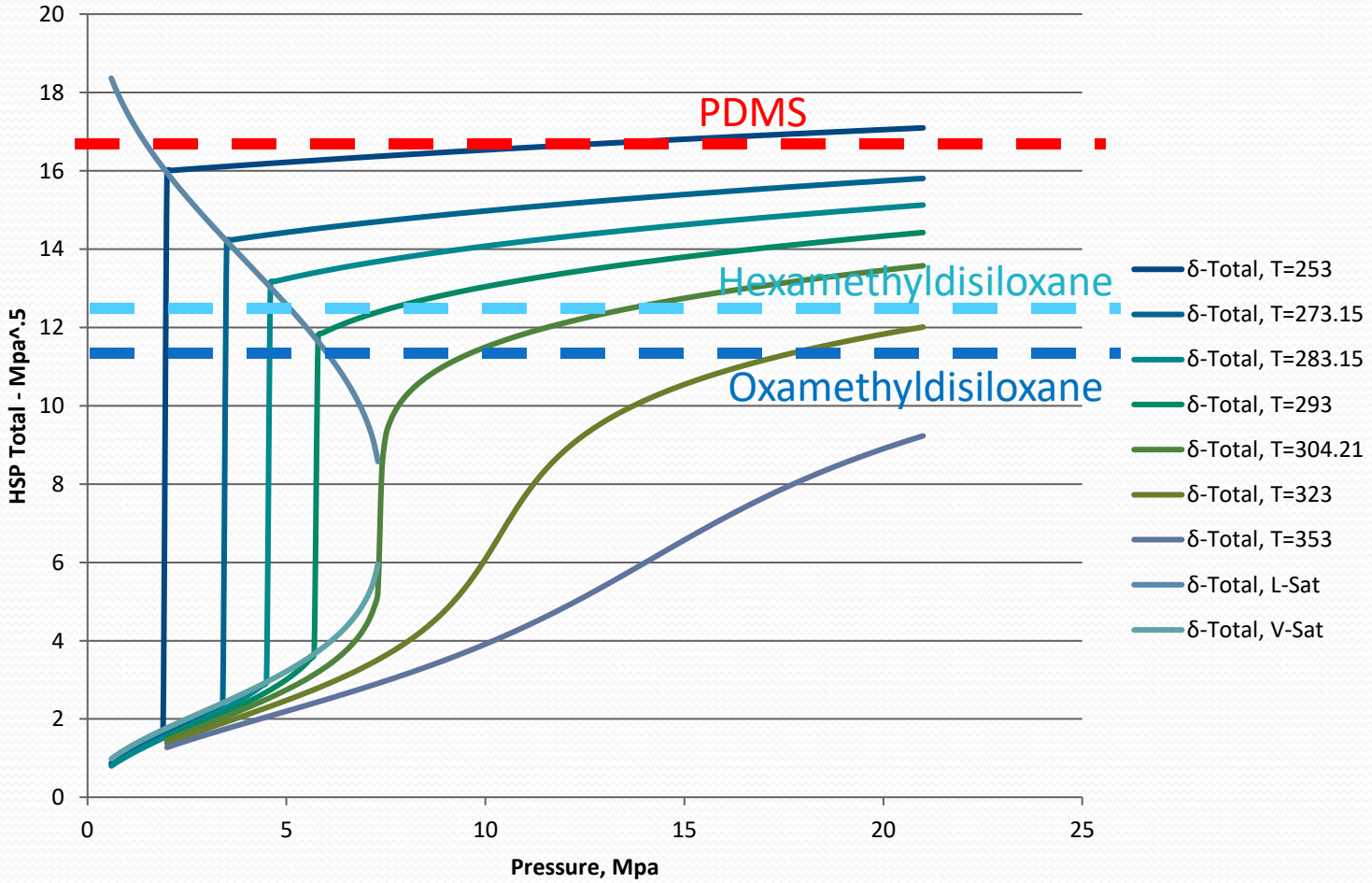


LCO2 Extraction of Silicones

LCO2 Extraction is an alternative to TVB

- Uses beneficial LCO2 – silicone monomer solubility to enable rapid and effective extraction of volatile compounds.
- LCO2 solubility can be ‘tuned’ to optimize process.
- Uses beneficial LCO2 diffusivity / transport characteristics to penetrate substrate to solubilize volatiles.

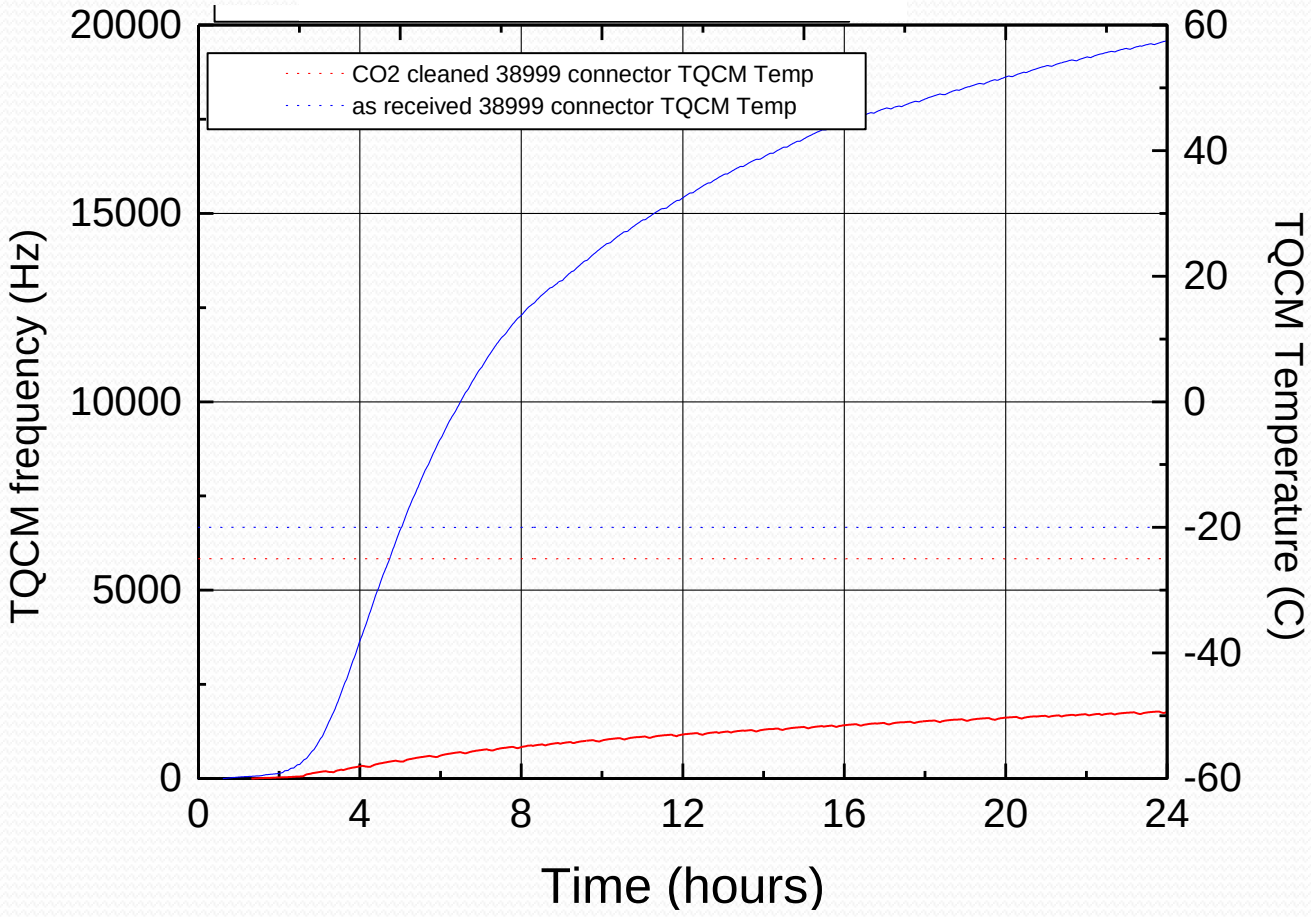
CO2 for Extraction Solubility of Representative Silicones



LCO_2 Outgassing: Advantages / Disadvantages

- Advantages:
  - Room temperature extraction (0 - 30ºC)
  - Typically, minimal impact on elastomeric materials - with careful depressurization
  - Much faster than vacuum bake-out - 3 hrs vs 24 - 72 hrs
  - Faster turn-around time for chamber between runs
  - More variable scheduling for crucial projects - no long process times
- Disadvantages:
  - New application for some material – may need confirmation testing
  - LCO2 Outgassing not necessarily an accepted / qualified process
    - Can be used as a 'pre-step' to compliant outgassing process
  - Impact of LCO_2 at extraction pressure on substrate needs to be confirmed
  - Chamber size limitations
  - Part configuration must accommodate pressures up to 70 atmospheres.

Effect of LCO_2 Cleaning on Outgas Potential of MIL-C-38999 Connector Saver



Effect of Processing on Outgassing Potential

Laird Tech T-Flex 6100 / 6120

Material/Test conditions	Condition	(TML)	RML (TML - WVR)	CVCM
T-Flex 6100	Baseline (no processing)	0.939	0.927	0.283
T-Flex 6100	48 hr/125°C VB	0.204	0.194	0.134
T-Flex 6120	72 hr/125°C VB	0.130	0.096	0.072
T-Flex 6120	**2 hr LCO_2 cleaning**	**0.111**	**0.091**	**0.035**
T-Flex 6120	**4 hr LCO_2 cleaning**	**0.040**	**0.024**	**0.004**

4-hour LCO_2 processing produced vastly better outgassing results than 72 hours of vacuum bakeout

TML – Total Mass Loss
CVCM - Collected Volatile Condensable Materials
WVR – Water Vapor Regained

Effect of Processing on Weight Loss

	THERMAL VACUUM BAKEOUT*	2-hour LCO_2 cleaning	4-hour LCO_2 cleaning
% Weight loss	-0.81	-5.96	-8.11

*125 °C for 48 hrs at pressure < $5x10^{-5}$ torr

- Silicones in gap pad materials migrate and contaminate surrounding surfaces.
- As-received material had been observed to leave a “potato-chip” stain assigned to low MW silicones
- This impact should be considered when selecting silicone-based materials.
- Extensive LCO2 cleaning can change material consistency (less sticky, harder)

LCO_2 removes mobile material much more effectively than TVB.

Processing Impact on Laird T-Flex 600 Thermal Conductivity

Material Conditioning	% Change from Baseline	Change in Thermal Conductivity Value *, W/m-K
48 hr Vacuum Bake	-12.2	-0.23
72 hr Vacuum Bake	-1.3	-0.03
2 hr LCO_2 Cleaning	+3.0	+0.06
4 hr LCO_2 Cleaning	+16.9	+0.32

* - Thermal conductivity measured with guarded plate test method (ASTM C 177 “Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus”)

Cost Comparison of LCO_2 Extraction vs TVB

- System prep time:
  - LCO_2 - 3 hrs system prep (pressurization, temperature);
  - TVB – 8-12 hrs system prep (evacuation, temperature).
- Sample Prep, Reporting Time:
  - Similar for both methods.
- Processing time:
  - LCO_2 process time proportional to material thickness (as for VB);
  - One-hour LCO_2 nominated as equivalent to 24 hours vacuum bake.
- Operational costs:
  - LCO_2 process - CO_2 + electricity - $20-50 per cycle, depending on length of extraction;
  - TVB process – Electricity - $???.

LCO_2 Extraction Substantially Faster than System Prep and Processing Time Savings

Outgassing of Aerospace Components

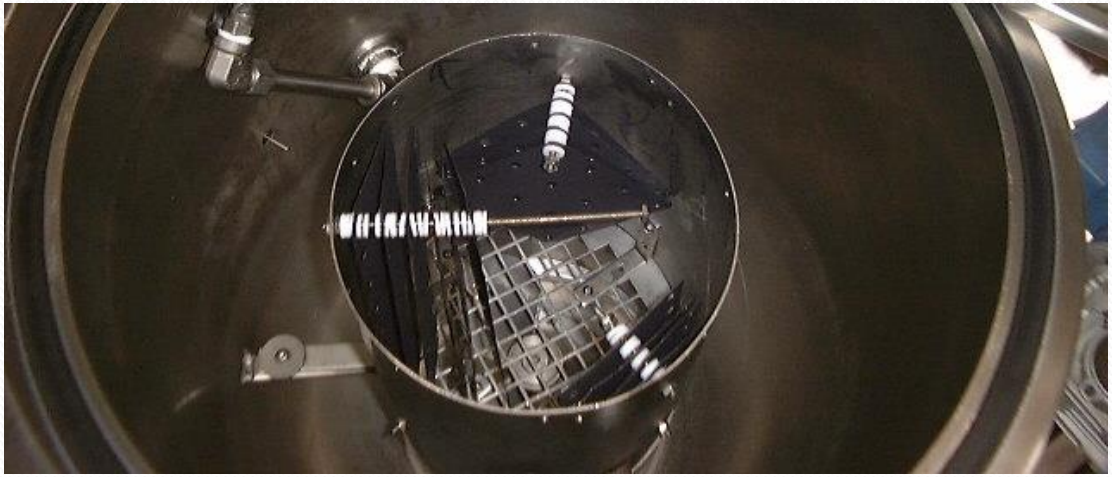
- Objective:
  - Extract volatiles and condensables from aerospace elastomers.
- Results:
  - LCO2 Extraction per Customer Spec;
  - Extraction duration: 2 hrs vs 36 hrs;
  - Outgassing confirmed by ASTM 595.

Assembled Cables

Satellite Heaters and Connectors

Extraction of Volatile Compounds from Aerospace Elastomers

- Objective:
  - Extract volatiles and condensables from aerospace elastomers;
  - Alternative to traditional vacuum bake-out procedure (lasting 24 - 72 hours).
- Method:
  - Enertia liquid phase extraction, 1000 psi for 30 - 120 minutes.
- Results:
  - Meets ASTM E595 outgassing standard by factor of 6.



LCO_2 Outgassing of Silicone Sheet

- Outgassing tests were tested on samples (thickness = 0.075) A-A-59588 CLASS 3B - GRADE 50:
  - Grey silicone of the “red” and “grey” silicone rubber samples;
  - Required ‘aging’ 70 hours at 100˚C (212˚F).
- LCO2 Extraction per HPR46035:
  - 2 hr extraction, 900 psig, 70F Vs 24-hour vacuum bake-out;
  - Some blistering of grey sample, red sample showed no blistering;
  - Both samples passed ASTM E-595.

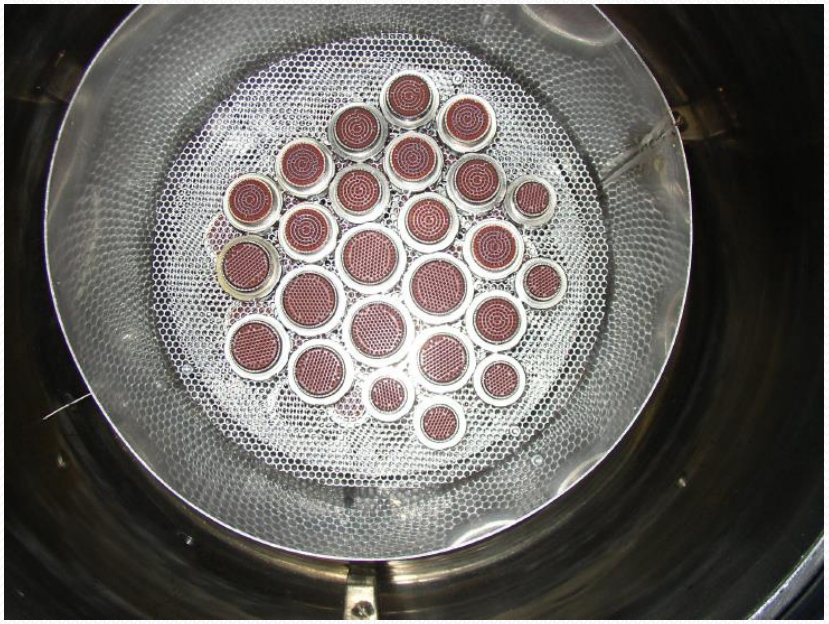
SAMPLE	Observations	AS RECEIVED		CO_2 CLEANED		VACUUM BAKEOUT	
		TML, %	CVCM, %	TML, %	CVCM, %	TML, %	CVCM, %
A.D. RUBBER-grey	Some blistering observed	0.3730	0.1730	0.0920	0.0040	0.0600	0.0080
STILLMAN SEAL-red	No blistering obserrved	0.4910	0.0960	0.4220	0.0050	0.3820	0.0060
ASTM E 595 screening level standard for rejection of spacecraft materials		1.0000	0.1000	1.0000	0.1000	1.0000	0.1000
Vacuum bakeout parameters 24 HRS., 125C, 1 x 10^{-5} torr							

Cleaning of Aerospace Cable Assemblies

- Objective:
  - Extract volatiles and condensables;
  - Remove particles;
  - Alternative to solvent wash / wipe and traditional vacuum bake-out procedure (lasting 24 - 72 hours).
- Method:
  - Enertia™ liquid phase extraction, 1000 psi for 120 - 240 minutes.
- Results:
  - Reduces outgassing time by half;
  - No impact electrical characteristics, (continuity, isolation, dielectric strength).

Aerospace Connector Outgassing

- Objective:
  - Extract solvent soluble compounds from connectors.
  - Traditional vacuum bake-out requires 100's of hours.
- Method:
  - Enertia™ liquid phase extraction, 1000 psi.
- Results:
  - 4 - 6 hour LCO2 process reduced acceptable bake-out target by 100 hours.
  - Components used in aerospace flight hardware.



Cleaning of Thermal Straps

- Objective:
  - Extract solvent soluble compounds from thermal straps.
  - Traditional vacuum bake-out takes too long.
- Method:
  - Carbonated solvent soak followed by LCO2 extraction, 1000 psi, 120 minutes .
- Results:
  - Passed outgassing test.
  - Cleaned components used in aerospace manufacturing.

Summary

- LCO2 Extraction systems are an effective way to remove volatile silicone compounds from aerospace materials.
- LCO2 shows wide variation in solubility parameter values over a relatively small pressure range.
- LCO2 solubility compares favorably with solubilities of many important silicones.
- LCO2 extraction equipment has been outfitted to automatically control all relevant extraction parameters for repeatable results.

For Further Information . . .

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