Updated Trends in Materials’ Outgassing Technology

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History

- Predessor Joe Colony reported on chemical analysis from GSFC chambers in 1979 in NASA TM 80585
- TM covered 8 years of reports from 1970-1978
- Utilized FORTRAN source code on 80 deck cards to extract information out of each report to create the data bank.
- Looked to identify common outgassing chemical species from flight hardware, spacecraft, and ground support equipment (GSE) from Thermal vacuum exposure.
- Tallied 102 different chemical species from 1163 reports.
- Typically only reviewed data from Cold Fingers and when depositions tallied above 10 mg.
- Together with Fred Gross developed library of FTIR plots and mass spectrum data from GC.
Updated Trends

- Chemical Analysis reports (CARs) are created in MS Word by GSFC Materials Engineering Branch, Code 541, and are electronically mailed to various project personnel.
  - Cut and pasted chemical species as text into rows into Excel spreadsheet and hand entered other pertinent information report.
- Only reviewed CARs that I had received and that had the Scavenger Plate (SP) or the chamber’s cryocoil activated.
  - covered ~3 years of reports from late 2007 - mid 2010, tallying 152 reports out of 713 total reports with >100 identified species.
  - No residue limitations.
- Utilized database of reports to identify common outgassing chemical species from flight hardware, spacecraft, and ground support equipment (GSE).
Chamber Mass Collectors

- All GSFC TV chambers have a Cold Finger (CF)
  - Typically ~16 in² of active area, with drain
  - Liquid Nitrogen (LN2) flows through at end of test, typically after hardware had achieved desired test criteria, for an 8 hour duration
- Most GSFC TV chambers have a Scavenger Plate (SP)
  - Most are 1 foot square with both sides active
  - LN2 flows through the SP continuously during TV testing of article.
  - Has a trough mounted underneath to collect excess residue.
- 2 GSFC Chambers have cryocoils in the door.
  - Assists with “pumping” the chamber down to high vacuum.
  - Are not insulated and are several feet in total length.
  - LN2 flows through coils continuously, acts like a SP.
  - Chamber 245 and 281
- Chamber vacuum pump
  - Most chambers have cryopumps that have chevrons in front of pumping exit.
- Quartz Crystal Microbalances (QCMs)
  - Mounted on flexible fluid lines; able to be moved anywhere in TV chamber
  - Able to be set at any temperature, typically -20°C.
Chamber Mass Collectors

Typical CF and SP location inside chamber
Chamber Mass Collectors
Sample collection

- Residue samples are rinsed off SP and CF, collected in clean/furnace baked collection bottles.
- Rinsate is typically Spectra or Optima-grade IPA or chloroform.
- Sample is evaporated on foil dish and weighed on 5 decimal place scale (0.01 mg)
Chemical Analyzers

Fourier Transform Infra-Red (FTIR) Spectrometer

- Sample placed on ZnSe crystal
- Scans from 2.5 to 20 microns (4000 to 500 cm\(^{-1}\))
- Takes about 10 minutes per scan, plots in a minute or 2.
- Can File Save scan and compare with library spectra to identify species
- More qualitative analysis, but can yield quantitative estimates
- An older Nicolet 380 apparatus, from Thermo Scientific, is shown on right.
Chemical Analyzers

- FTIR scan (Hydrocarbons, Lauric acid, DC704 peaks)
Chemical Analyzers

Gas Chromatograph/Mass Spectrometer (GC/MS)
- Requires UHP Helium, slight vacuum pulled internally
- Sample introduced as liquid (1 microliter)
- Thermal extraction
- Ramped rate from 25C to 300C
- Takes 25 minutes/scan
- More post processing involved to ID species
- Shimadzu GC-2010 shown
Chemical Analyzers

- GC output scan, species fragment/outgas at specific temps, which is related to a retention time.
- Lighter more volatile species peak early, heavier weighted molecules peak later (higher temp)
Chemical Analyzers

- Mass Spectrum output scan from a selected peak at time 20.14 minutes.
- Primary peak at 326 m/z with secondary peak at 77 m/z yielded triphenyl phosphate (TPP).
Database inputs

- Data extracted from a GSFC Chemical Analysis report (CAR) and insert into an Excel spreadsheet
- Column 1-Test report number
- Column 2-Spacecraft Project acronym
- Column 3-Scavenger plate or Cryocoil
- Column 4-Chamber (located in GSFC Bldg 7)
- Column 5-Subsystem of project hardware
- Column 6-generalized test article
- Column 7-Chamber/Hardware temperature (deg C)
- Column 8-Residue collected in milligrams (mg)
- Column 9-Chemical Specie rank in report (1-10)
- Column 10-Chemical specie
Database CAR listing

Copied species as text into Excel, hand entered single line of other CAR info and then created a Sort header
### Database DEHP listing

Sorted by chemical species and rank, newest to oldest report #

<table>
<thead>
<tr>
<th>Report #</th>
<th>Project</th>
<th>Chamber</th>
<th>Chemical Constituents</th>
</tr>
</thead>
<tbody>
<tr>
<td>10-115</td>
<td>GPM</td>
<td>yes</td>
<td>Di (2-ethylhexyl) phthalate (DEHP) – plasticizer</td>
</tr>
<tr>
<td>10-088</td>
<td>JWST</td>
<td>yes</td>
<td>Di (2-ethylhexyl) phthalate (DEHP) – plasticizer</td>
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<td>10-032</td>
<td>JWST</td>
<td>yes</td>
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<td>10-025</td>
<td>GPM</td>
<td>yes</td>
<td>Di (2-ethylhexyl) phthalate (DEHP) – plasticizer</td>
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<tr>
<td>09-094</td>
<td>ELC</td>
<td>yes</td>
<td>Di (2-ethylhexyl) phthalate (DEHP) – plasticizer</td>
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<tr>
<td>09-091</td>
<td>LRO</td>
<td>yes</td>
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<td>LRO</td>
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<td>08-159</td>
<td>SAM</td>
<td>yes</td>
<td>Di (2-ethylhexyl) phthalate (DEHP) – plasticizer</td>
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</table>
1979 Top 20

1. ALHYD-Aliphatic Hydrocarbons (185)
2. MESIL-Methyl Silicones (129)
3. DEHP-Di (2-ethyl hexyl) phthalate (114)
4. ESTER-Esters (100)
5. DBP-Dibutyl phthalate (27)
6. PHEST-Phosphate esters (24)
7. ARMHY-Aromatic Hydrocarbons (23)
8. MPHSI-Methyl Phenyl Silicones (19)
9. URETH-Urethanes (18)
10. HMBZP-Hydroxy Methoxy Benzophenones-antioxidant (17)
11. BHT-Butylated Hydroxy Toulene(16)
12. DC704-DC 704 Diffusion Pump Oil (15)
13. RTV56-Methyl Phenyl Silicones from RTV-560 or -566 (12)
14. ORGAC- organic or fatty acids (9)
15. TCEPH-Tris(2-chloroethyl) phosphate (8)
16. DEHAZ-Di(2-ethyl hexyl) azelate (6)
17. TPP-Triphenyl phosphate (6)
18. DTAMQ-2,5 Ditert amyl quinone (5)
19. PCB-Polychlorinated biphenyls (5)
20. DEHAD-Di(2-ethyl hexyl) adipate (4)
2010 Top 20

1. HYD-Various Hydrocarbons (151)
2. DEHP- Di (2-ethyl hexyl) phthalate-Plasticizers (135)
3. DBP-Dibutyl Phthalate- Plasticizers (93)
4. MESIL-Methyl Silicones (74)
5. ORGAC-Organic acids-surfactants (62)
6. DEHAD-Di(2-ethyl hexyl) adipate-plasticizer (46)
7. CHAF-Cyclohexylamine or formamide-boiler steam additives (44)
8. ALLYL-Tris(allyl) cyanurate(TAC)- wire insulation (40)
9. ESTER-Esters (38)
10. BNZE-Benzoate esters –plasticizers (36)
11. RTV56-Methyl Phenyl Silicones (34)
12. TPP-Triphenyl Phosphate-fire retardant (25)
13. BNZA-Benzoic acids-surfactants (24)
14. DINO-Dinonyl phthalates- Plasticizers (23)
15. BTC-Butyl Compounds-detergents (21)
16. PHEN-Phenols-antioxidants (19)
17. DEHS-Di(2-ethyl hexyl) sebacate (19)
18. OCTE-Octanoate Esters-plasticizers (18)
19. BTYLP-Butyl palmitate-surfactants (18)
20. ACRE-Acrylate ester-cable contaminant (16)
Common species -MLI

MLI-Germanium Black Kapton, Kapton, VDA mylar layups with polyester scrim, finished w/thread and velcro & acrylic-backed tapes

- DEHP-vinyl plasticizer
- Adipate esters- plasticizer, from thermal blankets
- Organic Acids- from fingerprints and cottonseeds
- Various hydrocarbons-greases from threading, transfer contaminant
- Isophthalate esters- from thermal blankets
Common species - Structures

Graphite epoxy structural panels (GrEp on Al honeycomb), tubes, booms for HGA, film adhesives, bonding epoxies

- DEHP-vinyl plasticizers
- Various hydrocarbons-greases, oils, byproducts
- DBP-plasticizers
- TPP-flame retardant
- Bisphenol-A and amine based curing agents- from epoxies
Electronic Box TV
Common species –
Electronic units

Electronic boxes, PWBs, thermal padding, harnesses
- DEHP-vinyl plasticizers, light oils
- Various hydrocarbons-greases, machining oils, byproducts
- DBP-plasticizers, paint byproducts
- TPP-flame retardant, paint byproducts
- Bisphenol-A and amine based curing agents- from epoxies
- Methyl Phenyl silicones- elastomers on connectors and thermal padding (Choseal, chotherm, CV products)
- Aromatic Amides- from PWBs and electronics
Harness Bakeout
Harness Bakeout
Common species – Harnesses, cables

GSE & Flight harnesses and cables

- Various hydrocarbons-greases, machining oils, byproducts
- DEHP-vinyl plasticizers, light oils
- Tri (allyl) cyanurate (TAC) cross-linking agents from wire insulation of Tefzel cables
- Organic Acids-handling contaminants
- Fatty acid esters-surfactants
- Phenol-based compounds-antioxidants (prevent oxidation)
- Urethanes-from encapsulants, potting
- Methyl silicones-mold releases, elastomers, greases
Unique species

From hardware, airborne
- Triphenyl Phosphates- flame retardant plasticizer
- Cyclohexylamine (CHA)- boiler steam/anti-scaling additive, airborne
- Hexa (methoxymethyl) melamine (HMMA)-resins from calrods taken over 300C.
- Butoxy (or ethoxy) compounds –detergents, used to clean GSE, hardware
- Squalene- from skin oil, handling/transfer contaminant
- Caprolactum- bagging contaminant from nylon products
Methods to Reduce NVR

- Review Materials list for flight and GSE hardware.
- Pre clean your hardware with approved cleanroom supplies.
  - Precision clean, NVR sample, possibly reclean, resample.
- Visually inspect critical hardware with white and black light.
- Bake hardware at as high of a temperature as allowed, as long as feasible.
- Store cleaned hardware in low outgassing bagging material (ULO polyethylene or Llumalloy).
- Use red non-flight covers whenever possible.
- Handle hardware with low NVR gloves (Nitrile or polyethylene)
- Store cleaned and baked out hardware in cleanrooms or GN2 purged dry boxes.
- Ship cleaned and baked out hardware doubled bagged and purged with dry filtered air or GN2.
Acknowledgements

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• Special thanks goes to the personnel over the years from the GSFC Materials Branch whom have honed the standards for chemical analytical testing at GSFC.

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• Thanks to the personnel that have worked and managed the operation of the GSFC TV chambers in Building 7 (Ed Packard, Leon Tilwick, Mike Bullinger, & Mike Schools).

• Co-author, Tony, for his review of material contained within the paper and presentation.
  – Refer to his tutorial presented herein at the 26th SSC.