FILM EXTRACTION EXPERIMENTS

Film Extraction Goals

- To develop a film-extraction method that can be used for Compliance
  - Minimum number of solvents
  - Robust
  - Feasible
    - Reproducible

Method Development

- Initial strategy
  - Use an extraction marker
  - Test coatings with different matrices
  - Extraction with methanol and tetrahydrofuran
  - Extract with sonication
  - Extraction set-up includes cut-up pans with cured sample

Glycerin in Different Samples/Solvents

- \>98% of film used for extraction attempts

- 5 sample coating types

- MeOH/THF/MeCl$_2$ – poor recovery success
  - Pool Paint 67%(THF), 13%(MeCl$_2$)

- MEK/Acetone – somewhat better recoveries

Glycerin in Different Samples/Solvents

		Acetone	MEK
Coating Type	**Total VOC Material (g/L)**	**% glycerol recovered**	**% glycerol recovered**
High Gloss Sealer	25	12	12
Elastomeric Deck Coating	110	39	0
Stain	55	5	4
Swimming Pool Paint	95	62	59
High Gloss Sealer	25	4	10

- Extraction marker is extracted differently from different matrices by the same solvent
    - Glycerin maybe atypical as an extraction marker

Method Development

- Modified strategy:
  - Try a different extraction marker
  - Extraction from a simpler matrix
  - Try different solvents and a solvent combo
  - Increase extraction time (sonication then shaking)
  - Add mixing beads
  - Exclude aluminum pans in the extraction set-up

Glycerin to Pentaethylene Glycol

Method 24, Neat Compounds

Methyl Palmitate
64% NV

Pentaethylene Glycol
94% NV

Aluminum Pans

Pentaethylene Glycol

Aluminum Pans

Pool Paint Results

Solvent	EOEOE % Recovery	Pentaethylene Glycol % Recovery
MeCl$_2$	198	55
ACN/Water (5%)	95	102
DMF	99	82

Latex Semi-Gloss Results

Solvent	EOEOE % Recovery	Pentaethylene Glycol % Recovery
MeCl$_2$	208	30
ACN/Water (5%)	101	43
DMF	74	60

- unpredictable PEG recoveries
- unreliable extraction precision
- replicate analysis showed 20% RPD

w/o Aluminum Pans

Pool Paint Results

Solvent	EOEOE % Recovery	Pentaethylene Glycol % Recovery
ACN/Water (5%)	97	78
DMF/Water (1%)	95	65

Latex Semi-Gloss Results

Solvent	EOEOE % Recovery	Pentaethylene Glycol % Recovery
ACN/Water (5%)	149	50
DMF/Water (1%)	97	58
DMF/Water (1%)	95	70

Flat Interior Results

Solvent	EOEOE % Recovery	Pentaethylene Glycol % Recovery
DMF/Water (1%)	99	40
DMF/Water (1%)	95	32

8 Replicates (Flat), 1 Solvent (ACN/water)

Polyethylene glycol Recoveries	
Average	25.3%
Precision (99%)	11.9%
Lower Limit	19.4%
Upper Limit	31.3%

What We Know

- Recovery is not repeatable (M24 parameters)
- Recovery was low under tested conditions
- Different solvent maybe needed for each coating
- Trial-and-error extraction
- Labor-intensive procedure
- Not a promising Compliance method

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