

**SYSTEMATIC SAMPLING and
ANALYSIS of TWENTY-ONE
ACRYLATE ESTERS**

by

F.T. Posey and

C.R. Manning

Sampling for ACRYLATES

Problems ...

- **SAMPLE LOSS from Media**
 - decomposition of acrylates on media
- **Low % RECOVERY**
 - hard to extract from media
- **Skewed Chromatography**
 - non-ideal peak shapes

Sampling for ACRYLATES

Problem ...

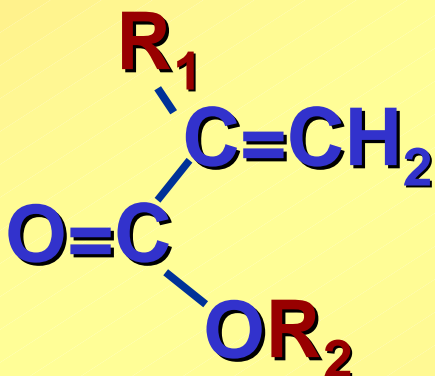
to be efficient, IH & LAB would like to sample many acrylates using a single media and method **BUT...**

	OSHA	NIOSH
ethyl acrylate.....	7, 92	1450
butyl acrylate.....	PV2011	n/a
methyl acrylate.....	7, 92	1459
methyl methacrylate....	94	2537
ethylhexyl acrylate.....	PV2026	n/a
other acrylates.....	n/a	n/a

Non-Polar Acrylates

$R_1 = H$
acrylates

R_2
*ethyl-
methyl-
n-butyl-
iso-butyl-
2-ethylhexyl-*



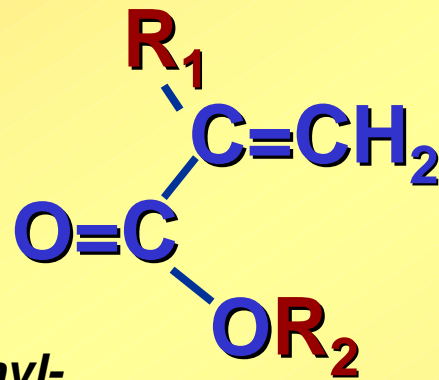
$R_1 = CH_3$
methacrylates

R_2
*allyl-
ethyl-
methyl-
n-butyl-
iso-butyl-
2-ethylhexyl-*

Polar Acrylates

$R_1 = H$
acrylates

R_2
hydroxyethyl-
hydroxypropyl-



$R_1 = CH_3$
methacrylates

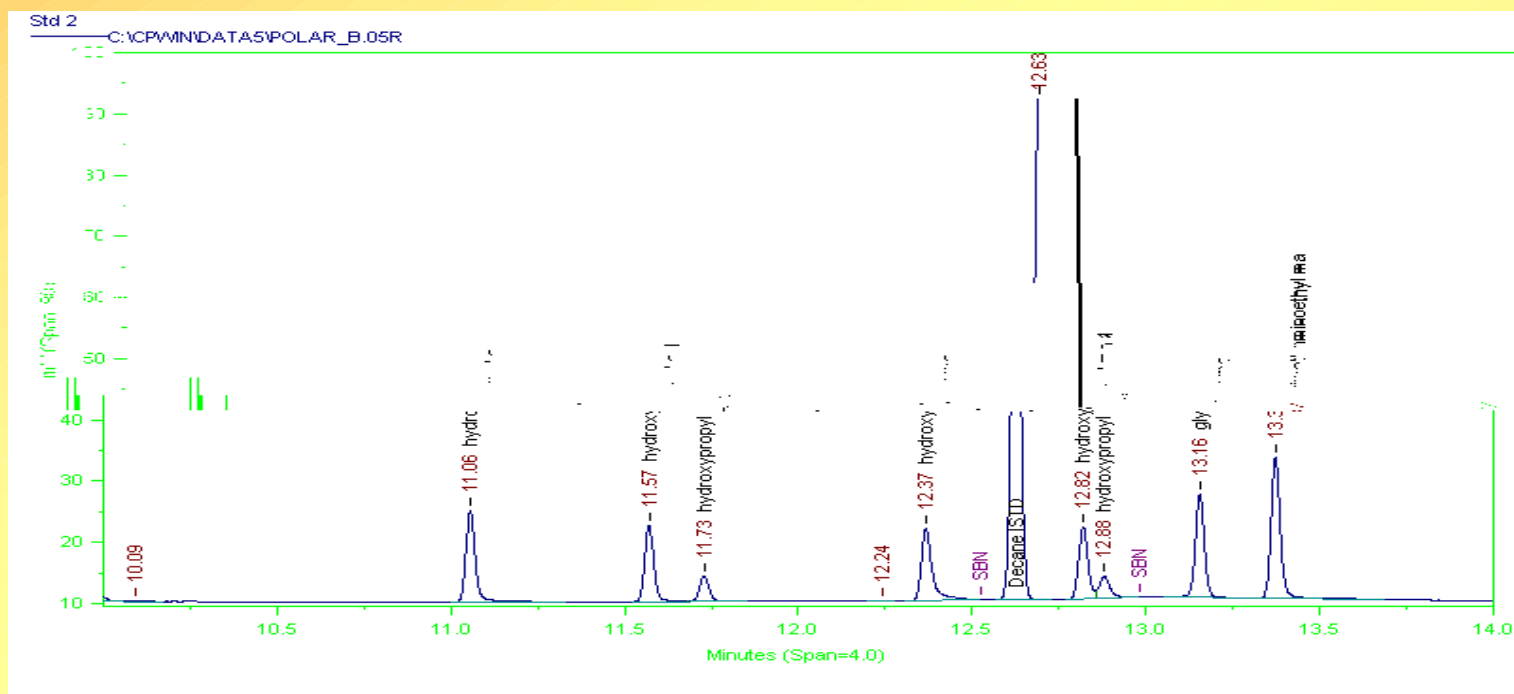
R_2
glycidyl-
hydroxyethyl-
hydroxypropyl-
2-(dimethylamino)ethyl-

Sampling Media

- **Charcoal Sampler**
 - Room Temp & Cold Storage
- **XAD-2**
 - Room Temp & Cold Storage

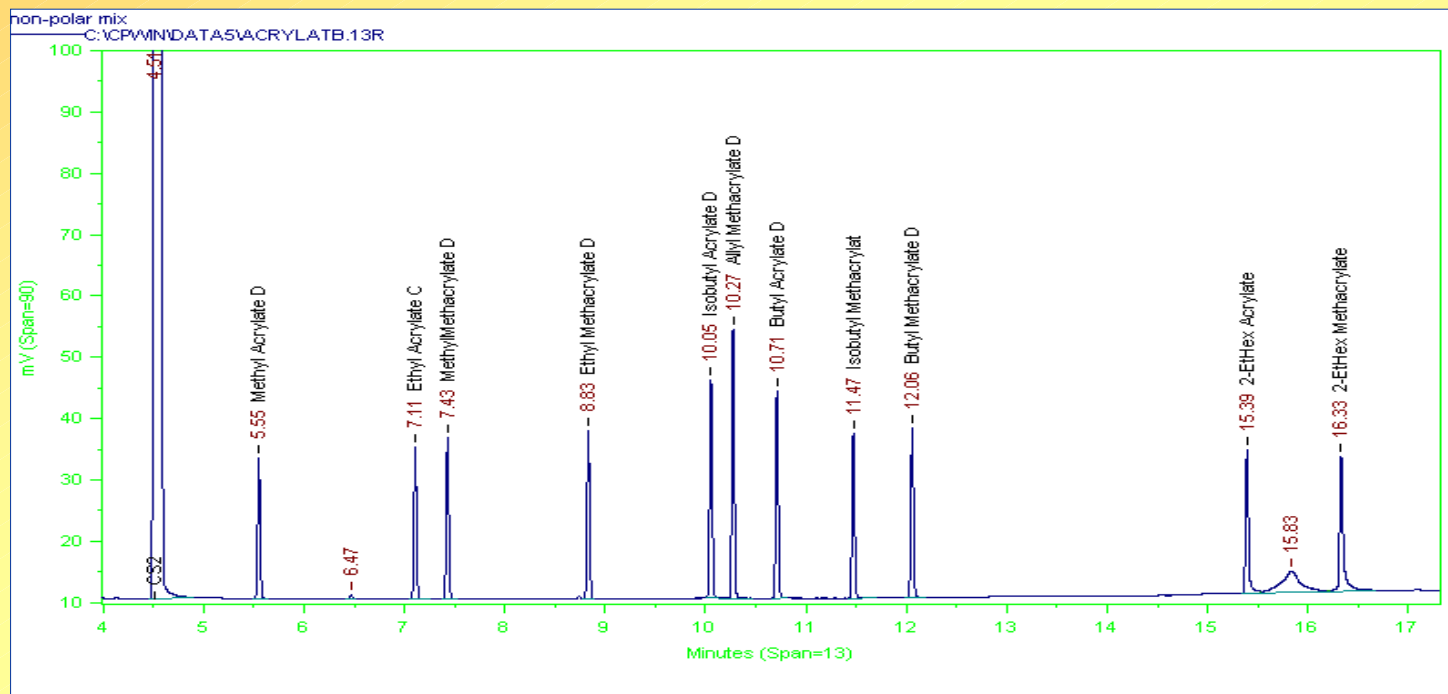
GC Analysis of Polar Acrylates

- Extract Sample with 2.0 mL 97% CS₂ w/ 3% v/v Dimethyl Formamide
 - 1µL Injection in Gas Chromatography Column - Restek RT-1 (Methyl Silicone 1.5µm film) 30 x 0.32mm
 - Split 10:1 Init Temp = 50°, 3 min hold, 15°C per minute to 250°C

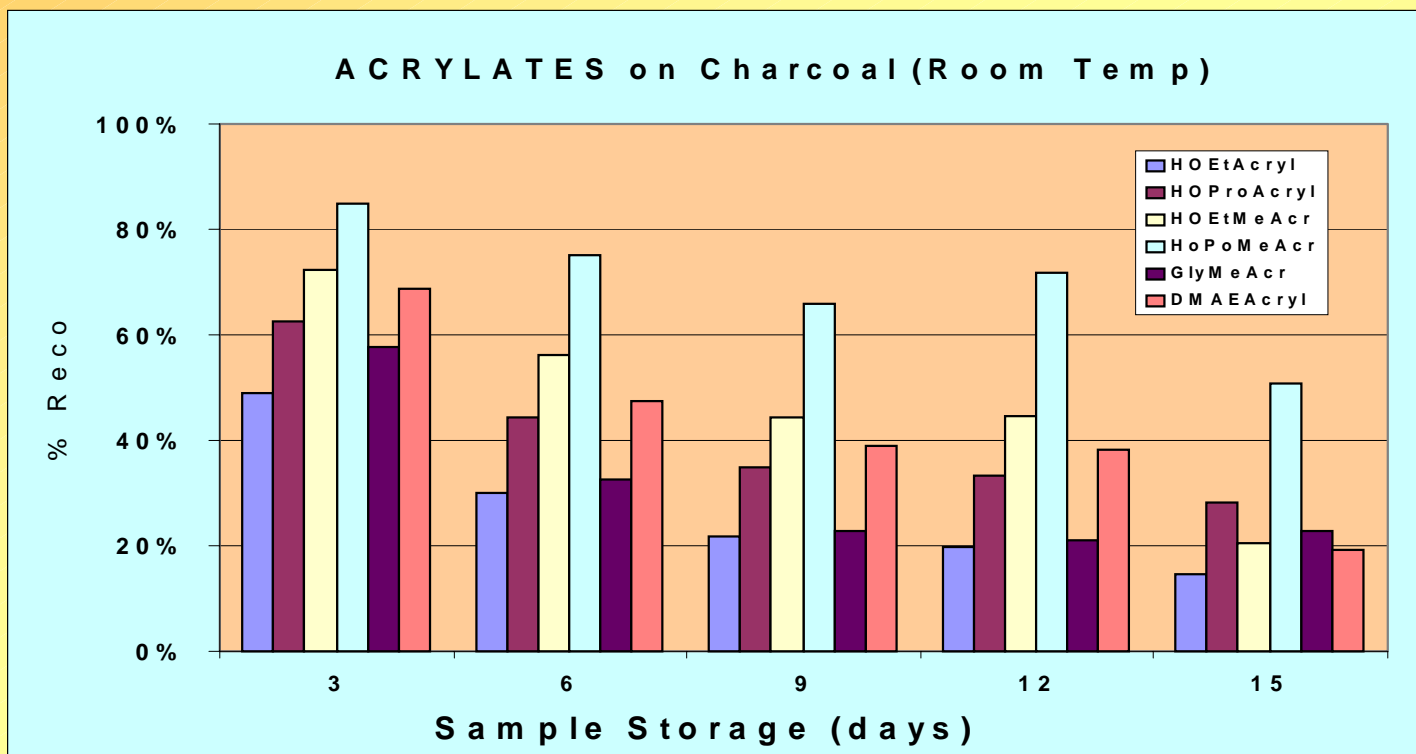


GC Analysis - Non-Polar Acrylates

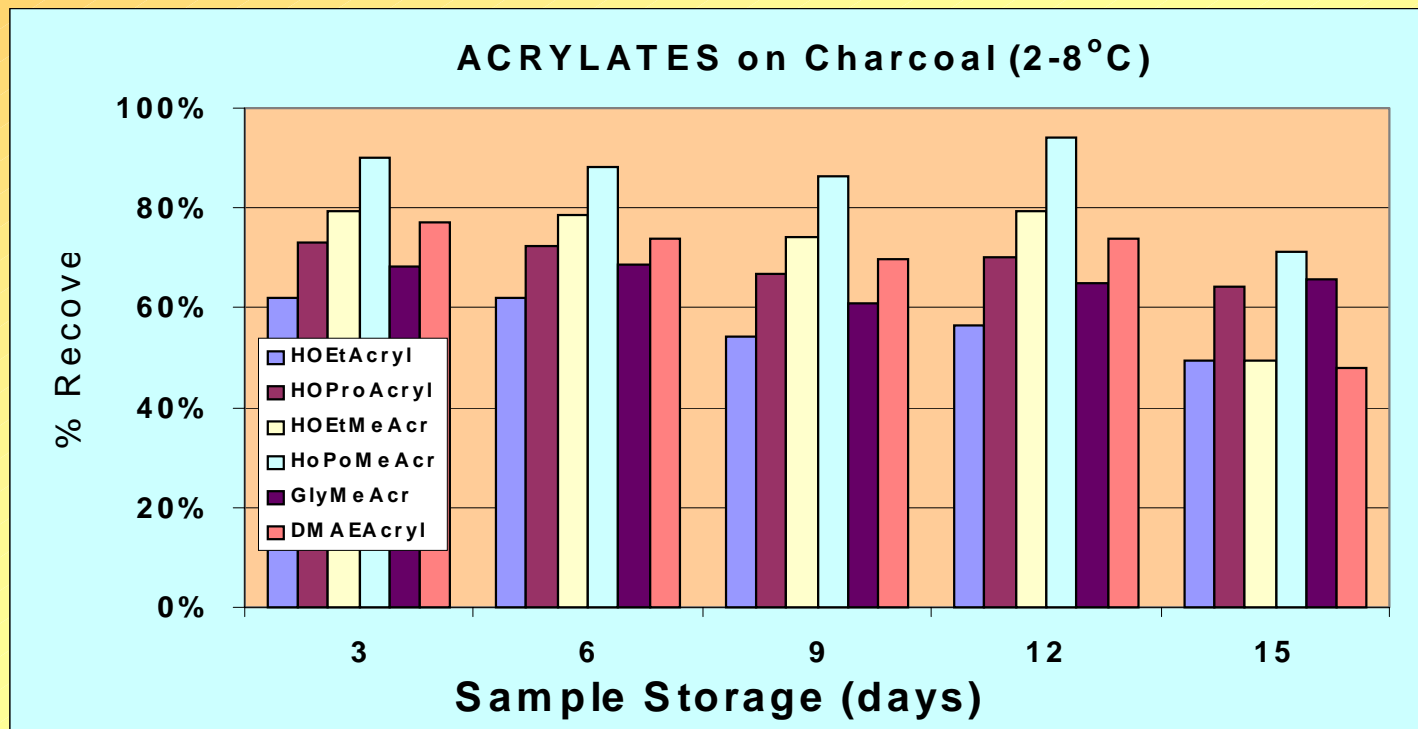
- **Extract Sample with 2.0 mL 97% CS₂ w/ 3% v/v Dimethyl Formamide**
 - 1 μ L Injection Gas Chromatography Column- Restek RT-1 (Methyl Silicone 1.5 μ m film) 30 x 0.32mm
 - Split 10:1 Init Temp = 50°, 3 min hold, 15°C per minute to 250°C



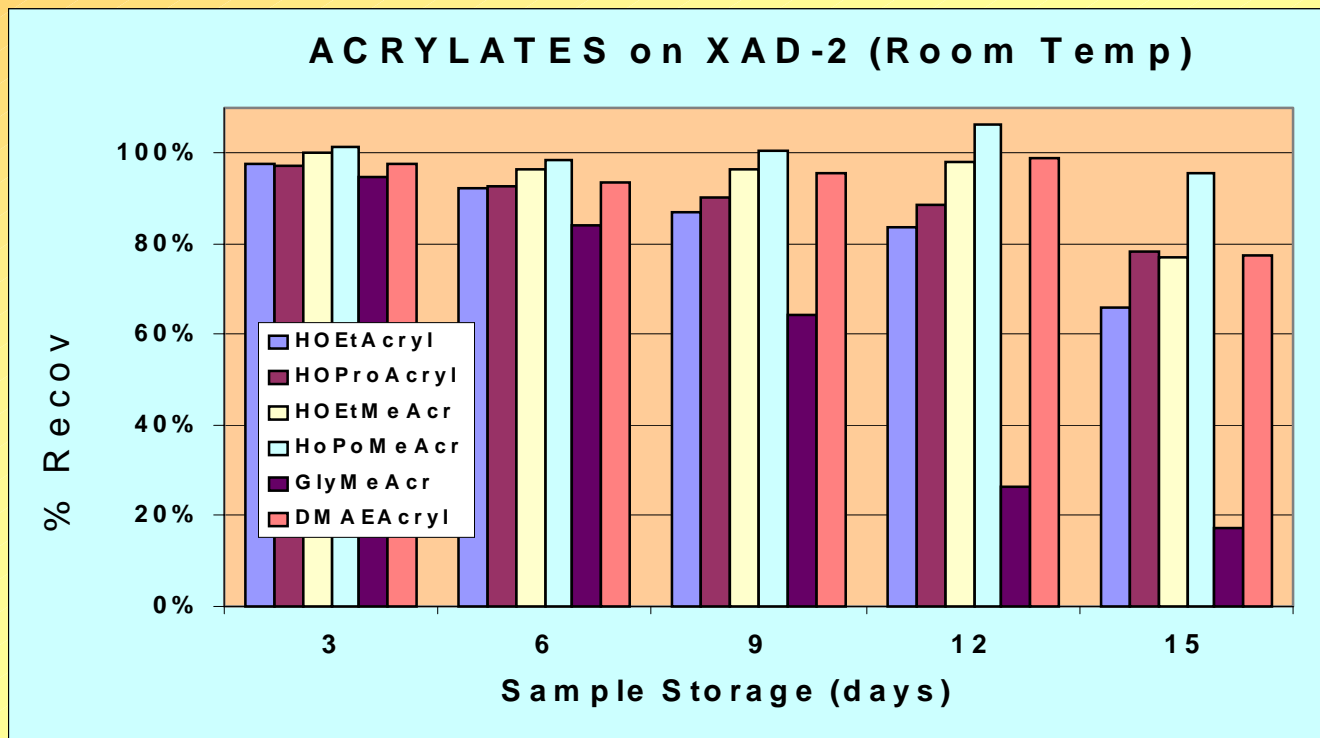
Stability of Polar Acrylates on Charcoal Sampler (RT)



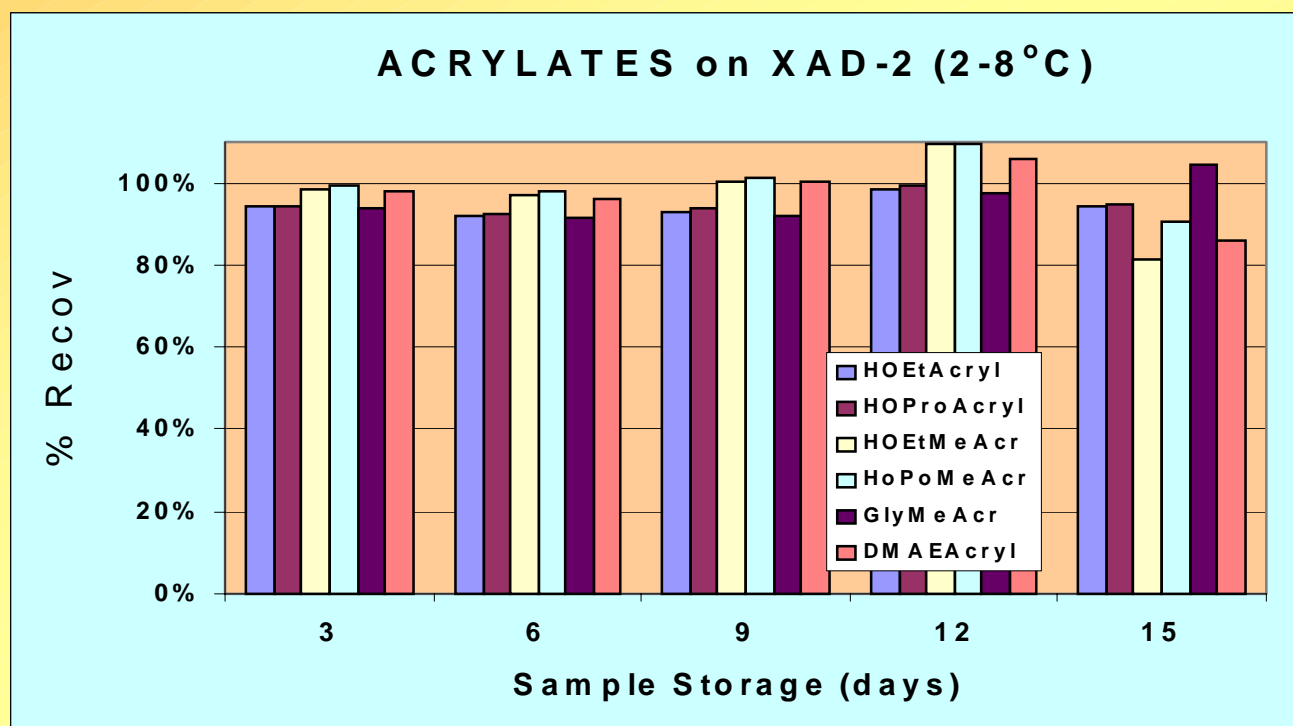
Stability of Polar Acrylates on Charcoal Sampler (cold)



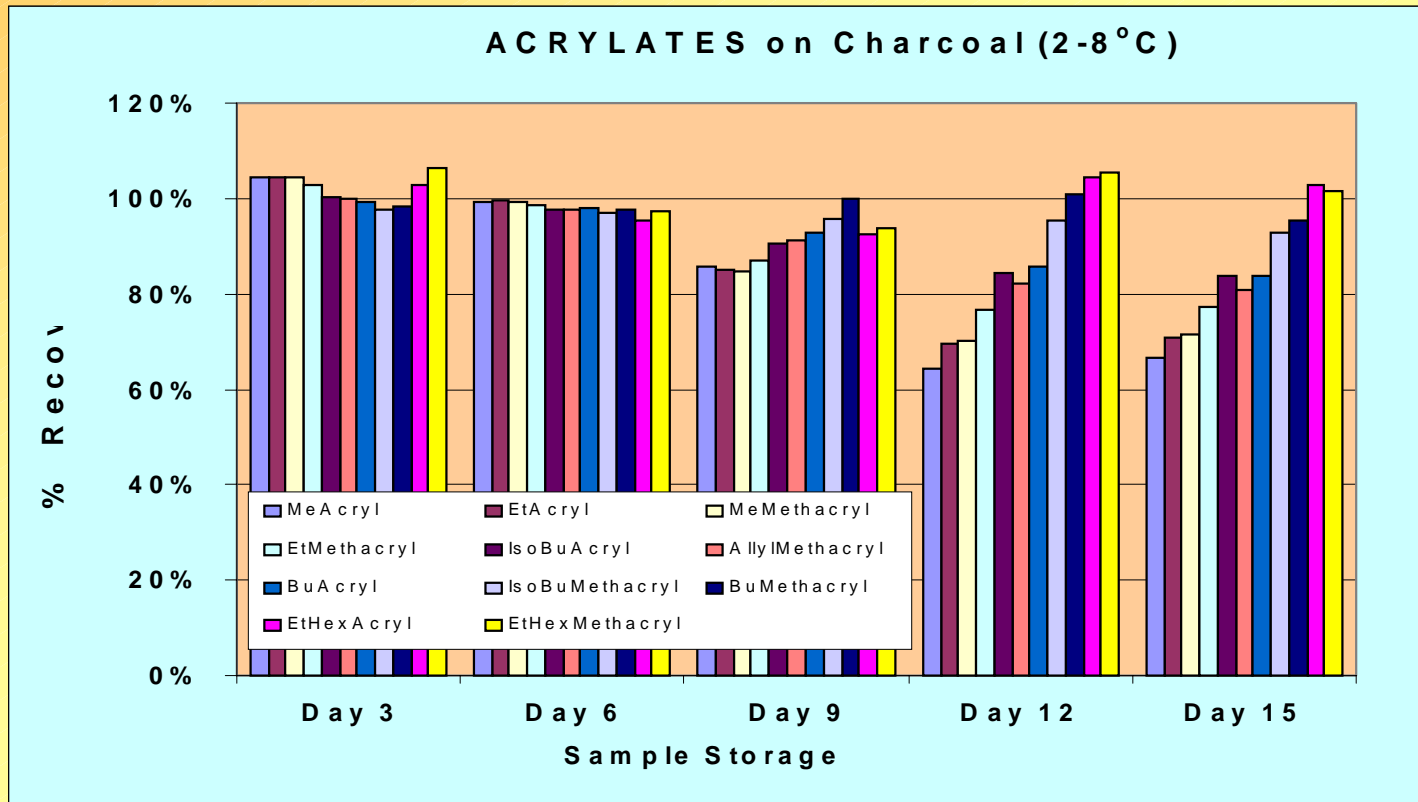
Stability of Polar Acrylates on XAD-2 Sampler (RT)



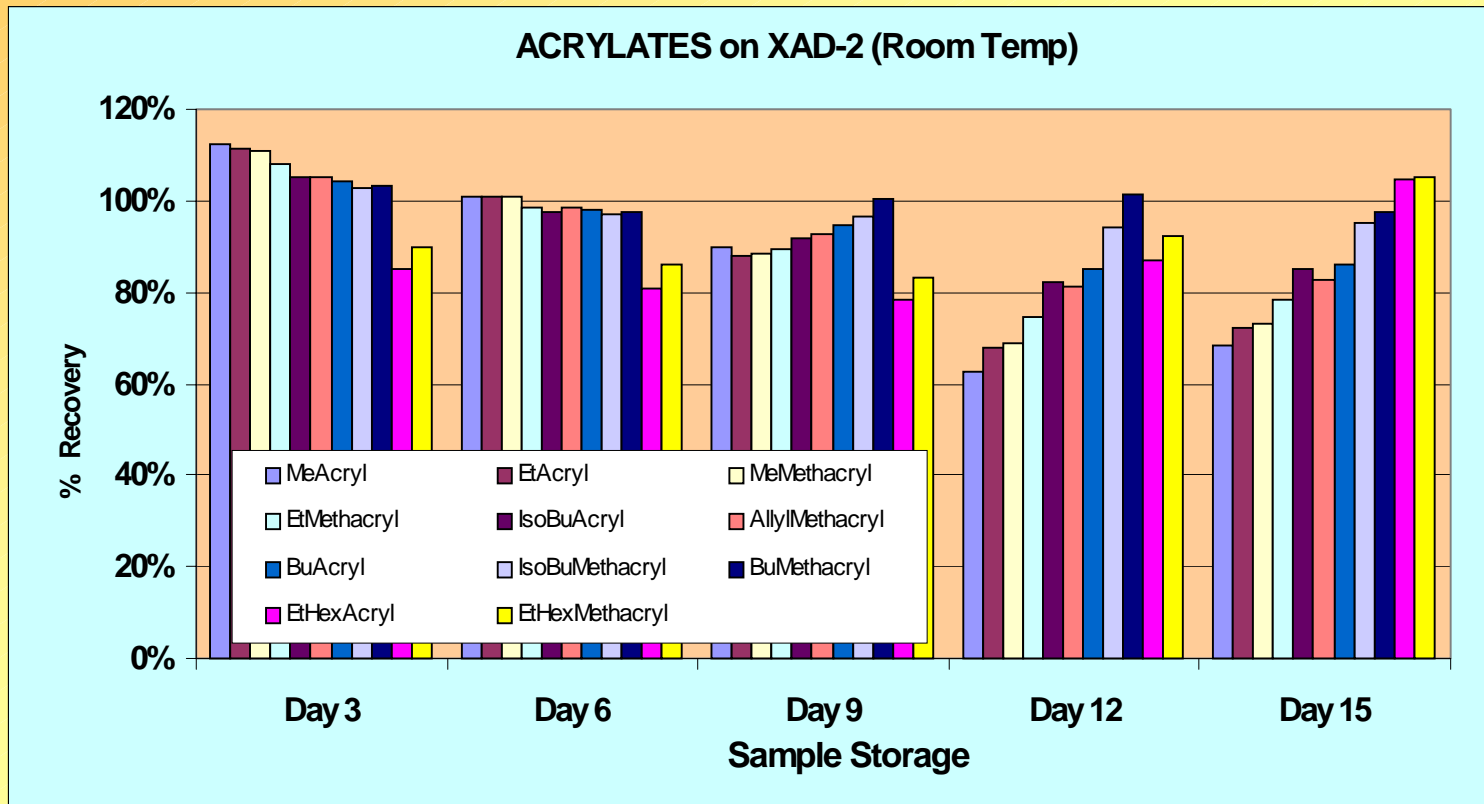
Stability of Polar Acrylates on XAD-2 (cold)



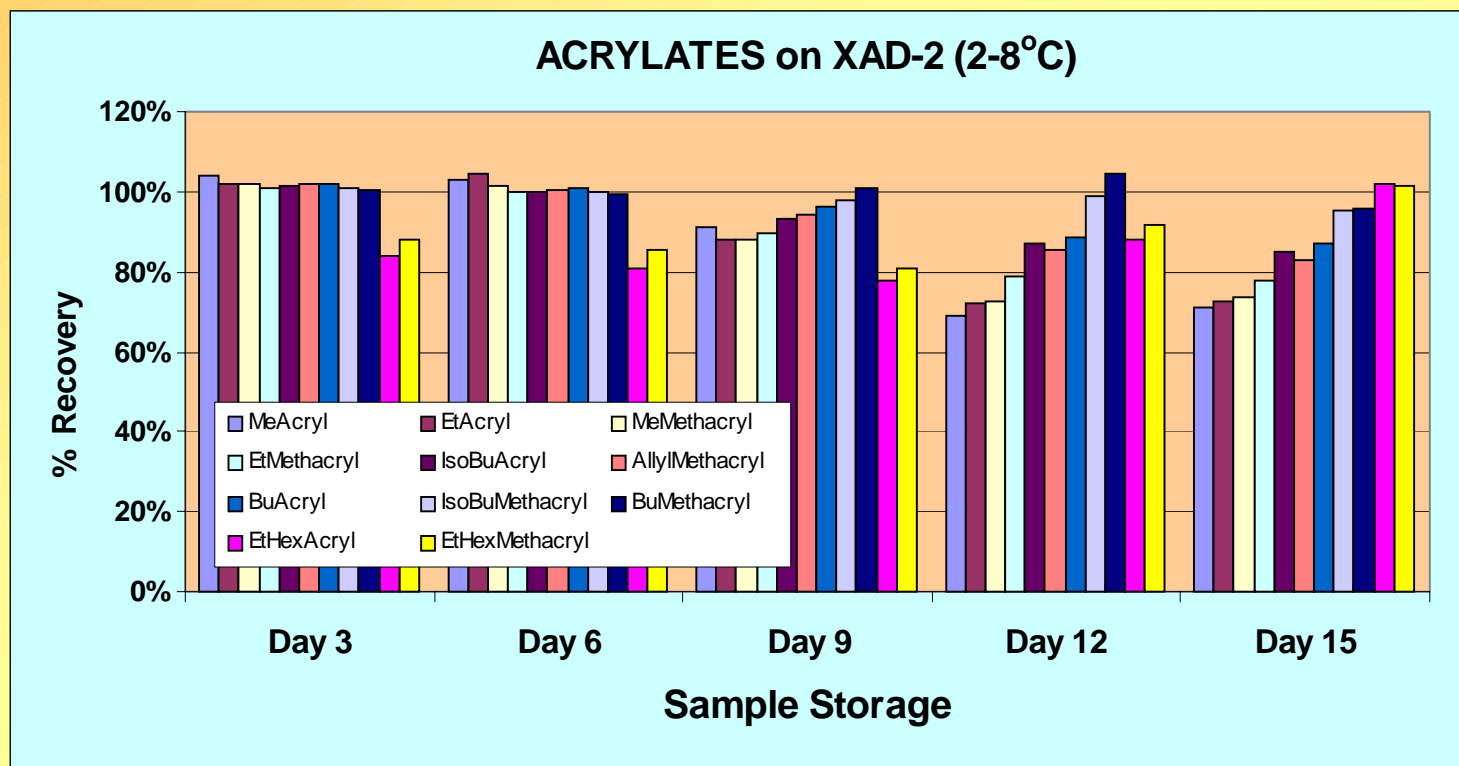
Stability of Non-Polar Acrylates on Charcoal Sampler (cold)



Stability of Non-Polar Acrylates on XAD-2 Sampler (RT)



Stability of Non-Polar Acrylates on XAD-2 Sampler (cold)



CONCLUSIONS

SAMPLING & HOLDING

17 Acrylates can be collected on the same Sampling Media
Charcoal or XAD-2

XAD-2 is Preferred for Sample Stability

16/17 Acrylates stable on XAD-2 for 1 week

all Acrylates stable on cold XAD-2 for 9 days

11/11 Non-Polar Acrylates stable on Charcoal for 1 week

EXTRACTION and ANALYSIS

17 Acrylates can be extracted with 97% CS₂ + 3% DMF

11 Non-Polar Acrylates can be resolved in a single GC Scan

6 Polar Acrylates can be resolved in single GC Scan