M24 TEST:REF COMPOUND

TWO TRIALS

PURPOSE

- Determine if a test compound is more or less volatile than a reference compound, for possible exclusion as VOC
- Improve the reproducibility of M24 for this purpose

TERMS

- MP methyl palmitate
- GLY glycerol
- PG propylene glycol
- EG5 pentaethylene glycol
- DBP dibutyl phthalate
- RR response ratio *
- RRR relative response ratio*
- RPD relative percent difference*

TEST PROCEDURE

- Trial 1: 0.20 grams each of test compound and reference compound (MP) (0.40 g total) were dispensed neat into an evaporation pan and mixed. Each compound was prepared and measured in triplicate.

- Mixtures were place in an oven at 110 C for 1 hour per M24

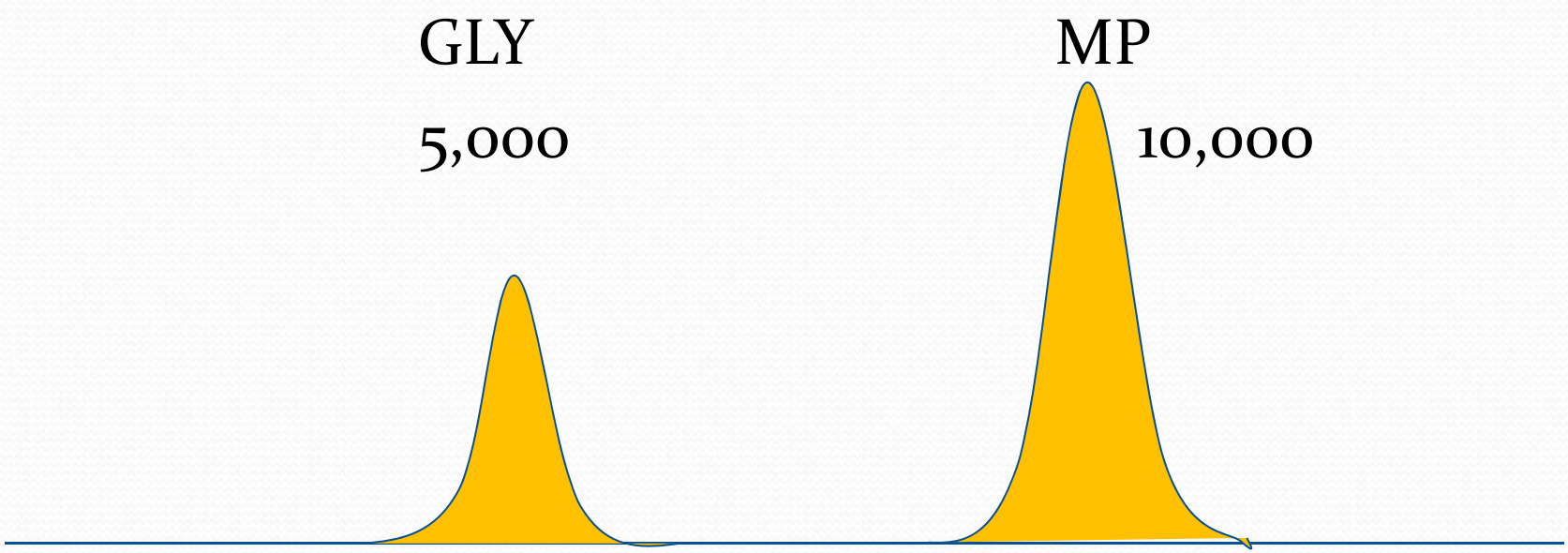
TEST PROCEDURE (CONT'D)

- Pans were removed after one hour, cooled, and weighed, and the non-volatile content was calculated

- The mixture was recovered with solvent and analyzed by FID for relative peak areas compared to a non-evaporated solution.

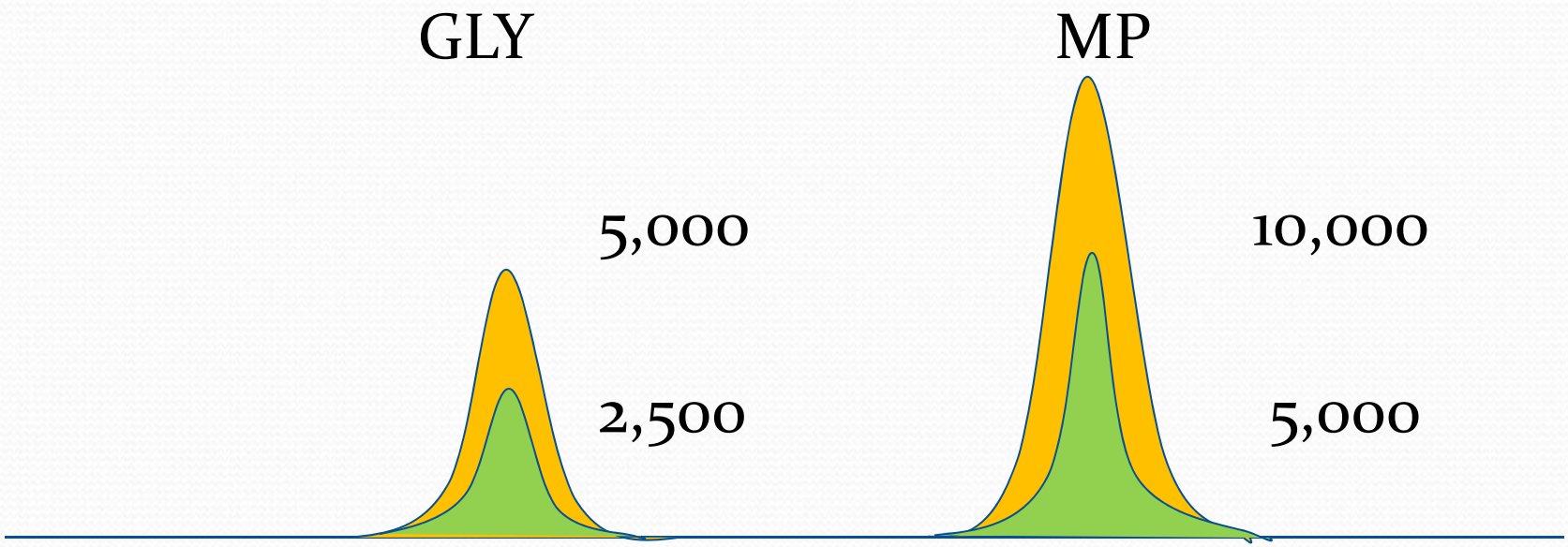
SAMPLES AFTER EVAPORATION

FID PEAK AREAS PRE- EVAP



Chromatographic peaks before evaporation

FID PEAK AREAS PRE- AND POST-



If compounds evaporate at about the same rate (50%)

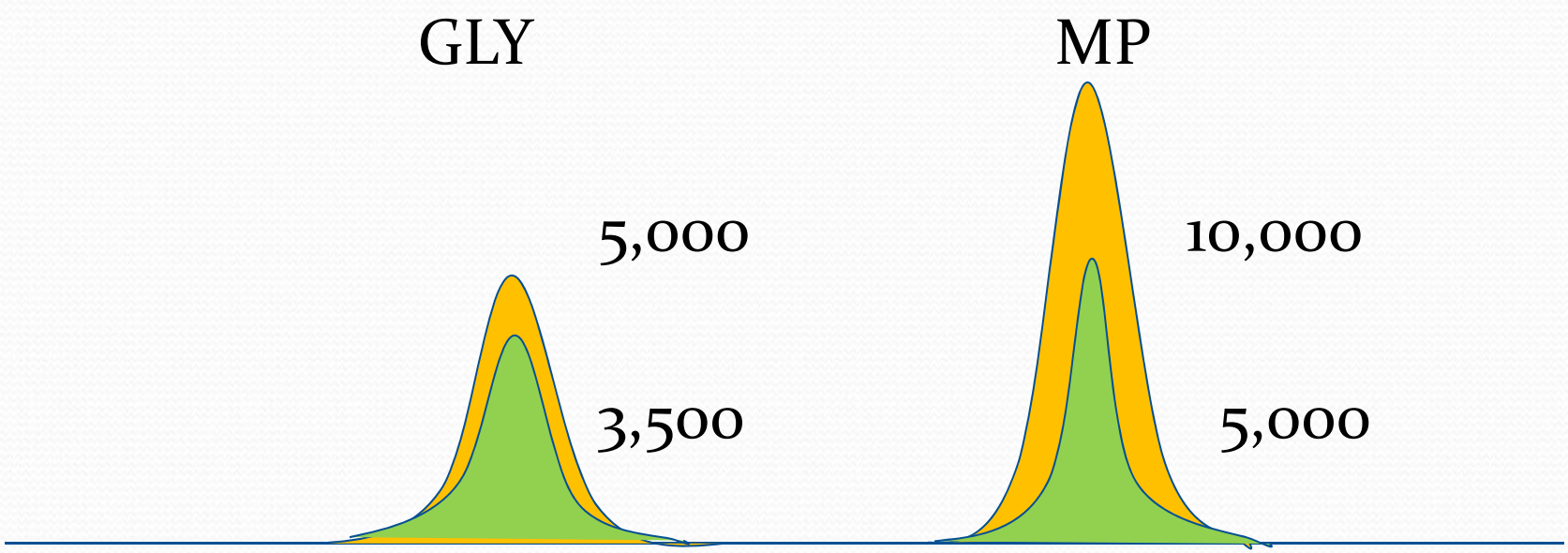
RRR - RELATIVE FID PEAK AREAS, CALC

Example, both evaporate at the same rate

	Area (pre)	Area (post)
GLY	5,000	2,500
MP	10,000	5,000
RR	0.5	0.5

RRR (post/pre) = 0.5/0.5 = 1 (equal)

FID PEAK AREAS PRE- AND POST-



If GLY evaporates more slowly

RRR- RELATIVE FID PEAK AREAS, CALC

Example, GLY evaporates more slowly than MP

	Area (pre)	Area (post)
GLY	5,000	3,500
MP	10,000	5,000
RR	0.5	0.7

RRR (post/pre) = 0.7/0.5 = 1.4 (GLY less volatile)

PRECISION - RPD CALCULATION

Example, GLY:MP

Test 1	1.642
Test 2	1.559
Test 3	1.336
Avg	1.512

RPD = (Highest – lowest value)/ Average * 100

= (1.642 – 1.336)/ 1.512 * 100

= 20.28%

RESULTS TRIAL 1

TEST:REF	%NV	RPD	RRR	RPD
EG5: MP	82.6	11.3	1.52 (less)	21.9
GLY: MP	83.7	4.9	1.51 (less)	20.3
PG: MP	38.2	6.9	0.00	NA (0)

SINGLE COMPOUND	
MP	~60
GLY	~85
EG5	~95
PG	~ 0

TEST PROCEDURE TRIAL 2, MODS

- Reduce variability as much as possible
- Because of solid form at RT and difficulty mixing, MP changed for DBP
- Test compounds and reference compound (DBP) were dissolved in common solvent before evaporation, each compound tested in triplicate, still applying 0.20 g of each compound (0.40 g total)
- Trial 2 samples were photographed after removal from oven

RESULTS TRIAL 2

TEST:REF	%NV	RPD	RRR	RPD
GLY: DBP	62.6	10.6	0.60(more)	18.9
PG: DBP	36.6	6.1	0.00	NA (0)
EG5: DBP	72.8	7.1	1.40 (less)	8.9

SINGLE COMPOUND

MP	~60
GLY	~85
EG5	~95
PG	~ 0

PRECISION BOTH TRIALS

	NV	RRR
NEAT	RPD	RPD
• GLY:MP	4.9	20.3
• PG:MP	6.9	NA (0)
• EG5:MP	11.3	21.9
SOLN		
• GLY:DBP	10.6	18.9
• PG:DBP	6.1	NA (0)
• EG5:DBP	7.1	8.9

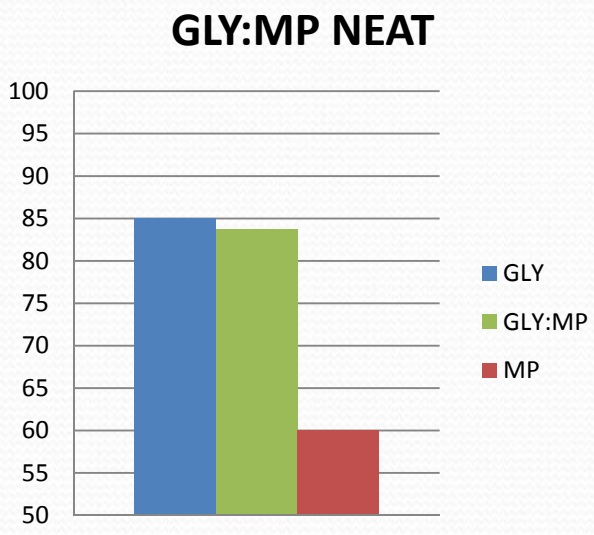
“BEADING”

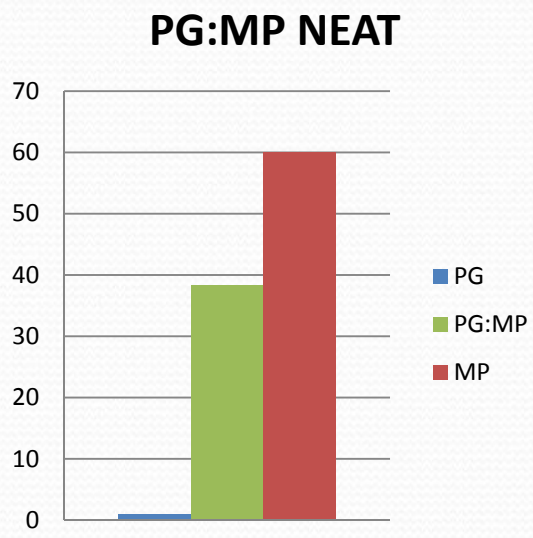
ADDITONAL CONCERN

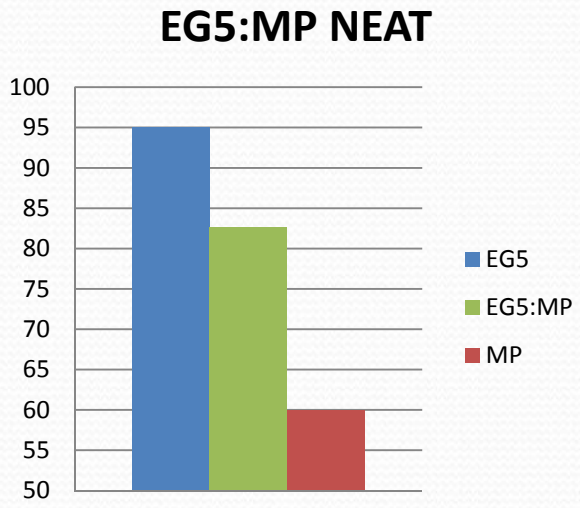
- Unexpected %NV and RRR results, compared to previously-determined %NV of neat (single) compounds

EXPECTED V FOUND %NV - NEAT

When added neat, the %NV of the mixture is higher than the average of the individual components

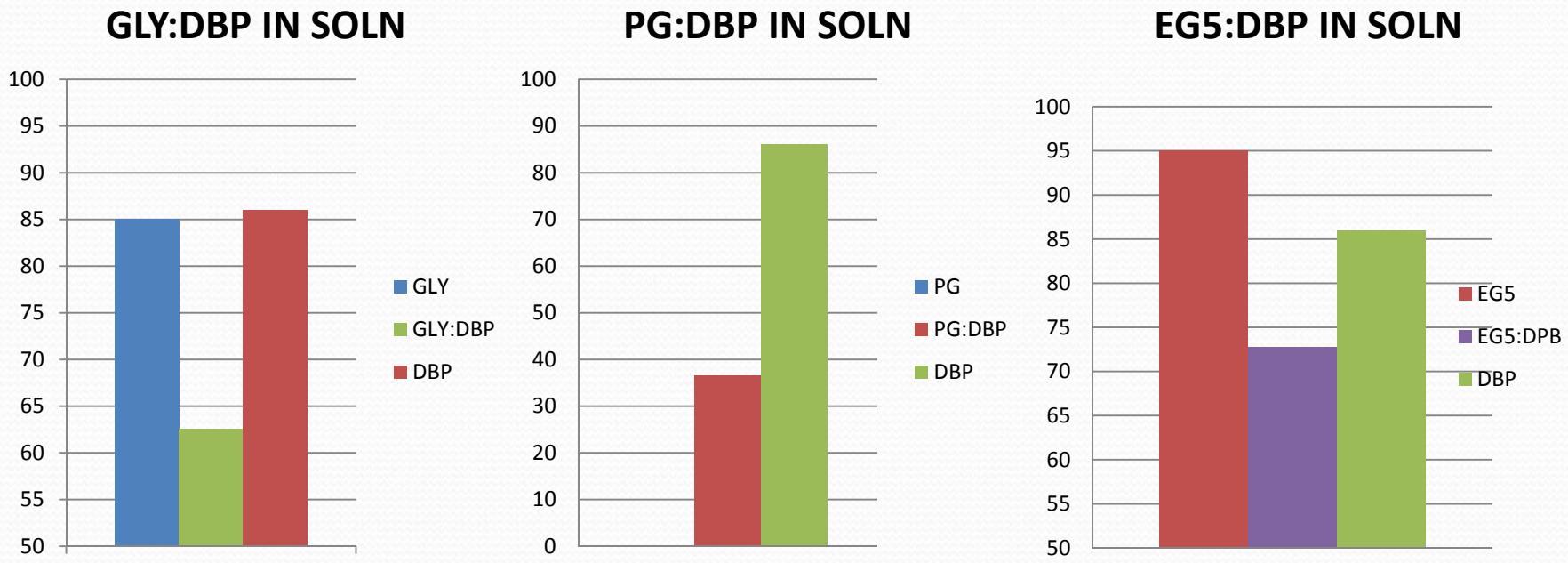




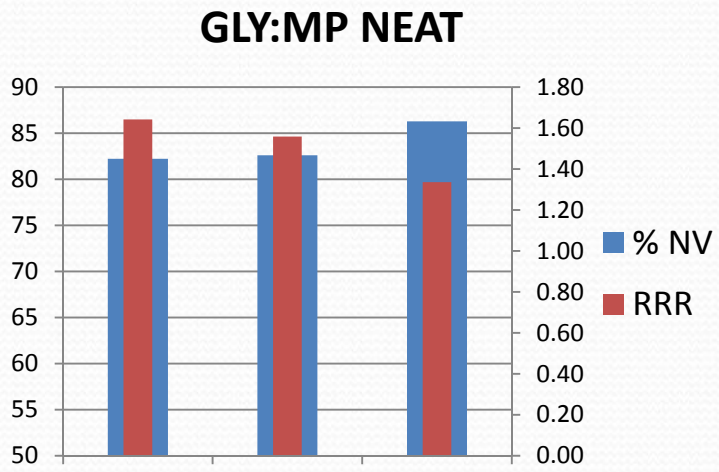


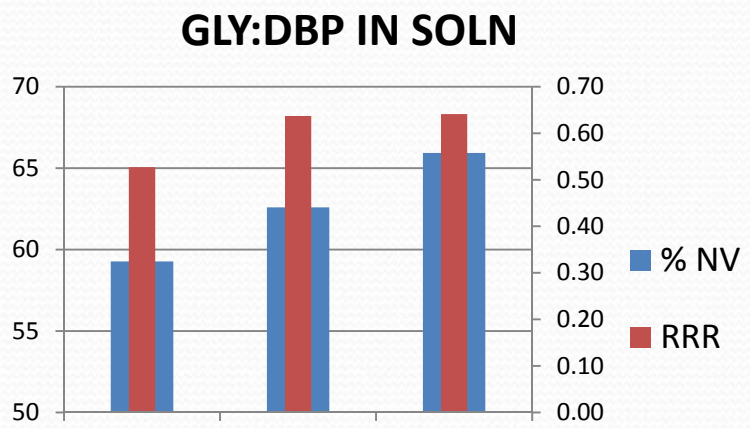
EXPECTED V FOUND %NV- SOLN

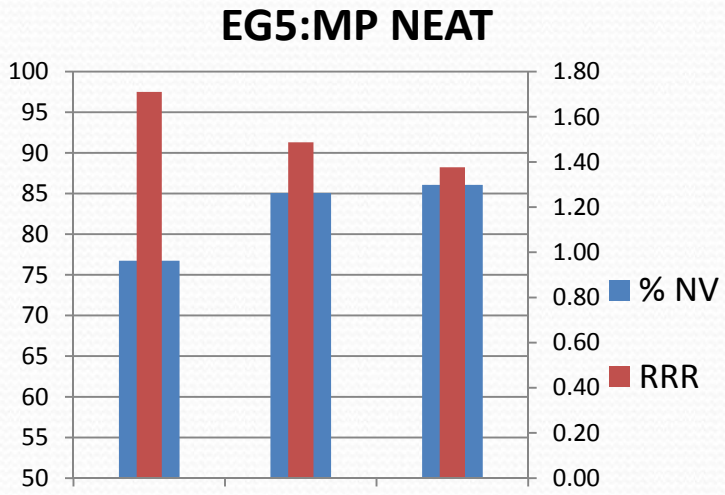
When added in solution, the %NV of the mixture is significantly lower than the average of individual components

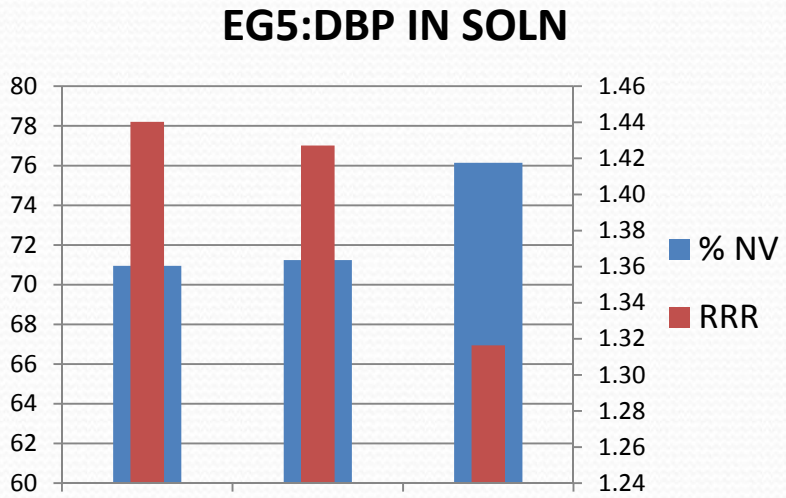


%NV v RRR NOT PREDICTABLE





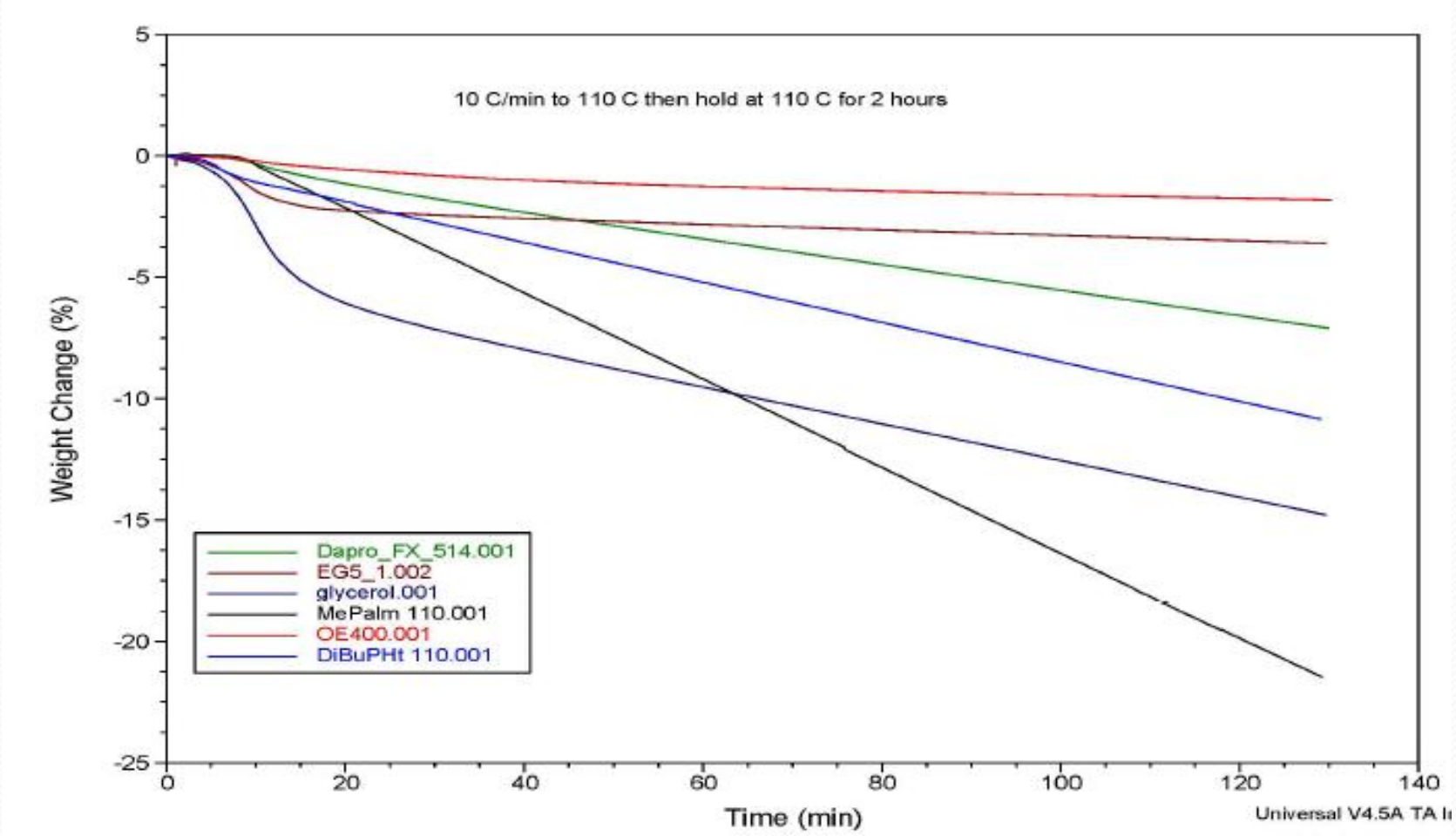




CONCLUSIONS

- Large variation in GC results. RPDs of approximately 20% are typical.
- Lower, but still large, variation in gravimetric results. RPDs above 10% occur. However, %NV unusable “as is”.
- Effect may be from “beading” of one compound on another, as photographed.
- Presence of reference compound and/or solvent introduces matrix effects.

TGA (Cal Poly SLO)



M24 TEST:REF

- Recommend investigating another method; suggest investigating to see if M24-equivalent results can be obtained via TGA