



# The “Can Do” Approach to Analytical Standards and Generating Valid Defensible Data

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NEMC Environmental Measurement Symposium, August 4-8, 2014,  
Washington, DC

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environmental clients worldwide.*

# Certified Reference Material

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## Wikipedia Definition

Reference material characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability.

(CRMs) are ‘controls’ or standards used to check the quality and metrological traceability of products, to validate analytical measurement methods, or for the calibration of instruments.

# Reference Material

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Reference materials are particularly important for analytical chemistry and clinical analysis. Since most analytical instrumentation is comparative, it requires a sample of known composition (reference material) for accurate calibration. These reference materials are produced under stringent manufacturing procedures and differ from laboratory reagents in their certification and the traceability of the data provided.

# ISO Guide 34



- Raw materials in the standard must be characterized via qualified methods on qualified instruments
- The reference standard must be produced in an ISO accredited lab under documented procedures
- Reference standard must fall under at least one of the chemical classes for which the lab has been approved under its accreditation

Restek Website

# What If?



You have a request for analytical work and

- The target compound is only reagent grade material?
- The target compound is part of a complex mixture?
- The process by which the target compound(s) are manufactured creates variable amounts?

# Example #1



## Propylene Glycol Monolaurate (PGML)

- Client product contained unknown quantities of PGML as a side product
- Needed to be able to quantify amount of PGML for registration of product
- “Analytical grade” standard not available



# Example #1



Two types of PGML available;

Type I - CAS# 12974-74-4

Type II - CAS# 37321-62-3

Not only contain PGML, but also PGDL  
(propylene glycol dilaurate).

Differ by ratios of each component in mix.

# Example #1



Two types of PGML available;

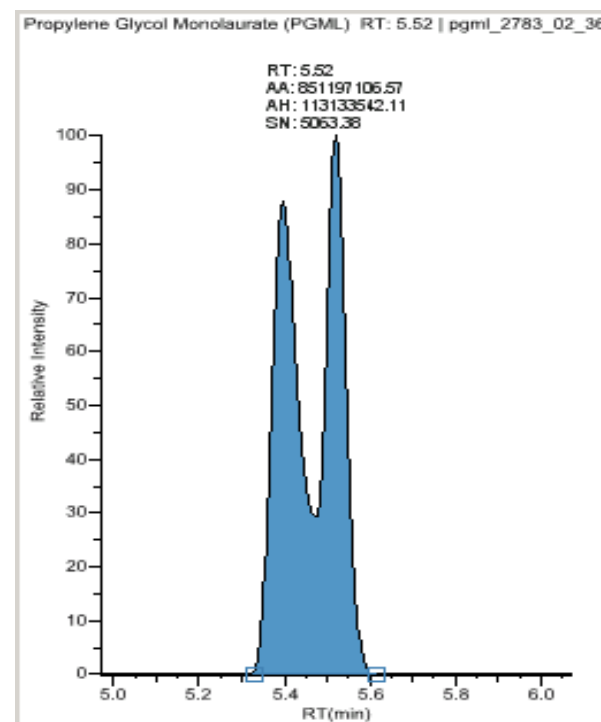
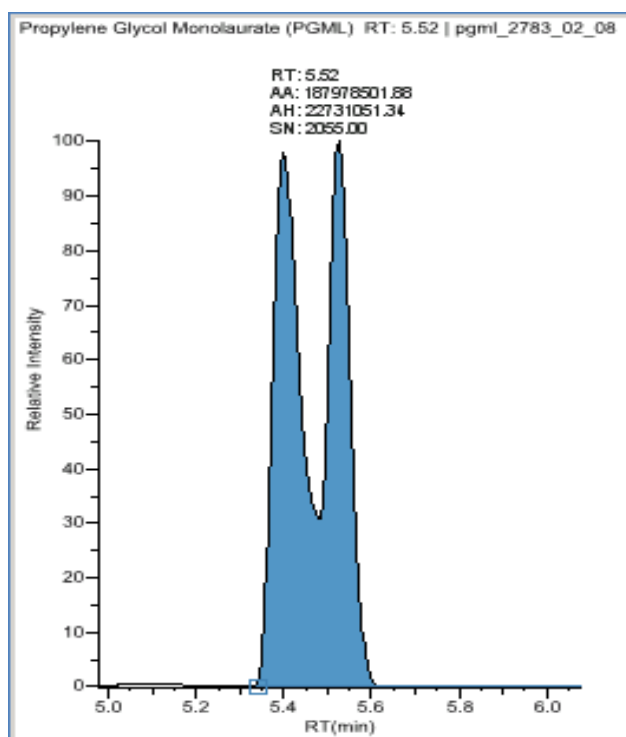
	Content of Monoesters (%)		Content of Diesters (%)	
	Min	Max	Min	Max
Type I	45	70	30	55
Type II	>90			<10



# Example #1



## Examples of PGML from Different Sources



# Example #1



How do we approach an accurate analysis and quantification for PGML?

1. Develop a method with the required specificity for PGML  
GC/MS full scan
2. Sum peaks for PGML and treat as one
3. Determine percent composition by calculating ratio of PGML to total area response
4. Do this for several suppliers of PGML

# Example #1



## Standards of PGML Selected for Assay and Comparison

PGML – Type I, USP Reference Standard, 100%, Lot F0E202

PGML – Type I, BOC Sciences,  $\geq$  95%, Lot B13W1027

PGML – Type I, Dr. Ehrenstorfer, 99%, Lot 403102

PGML – Type II, ABITEC, 94.0%, Lot 111121-TMC

PGML – Type II, USP Reference Standard, 100%, Lot F0G355

# Example #1



- For the assay, each standard material weighed out five separate times.
- For each weighing of each material, the ratio of PGML peaks to total area response determined
- Average and %RSD calculated for each of 5 different sources
- No %RSD greater than 2.0%

# Example #1



## Results of Assays

Product	Compound	Average Ratio
USP Type I	PGML	0.489
BOC Type I	PGML	0.621
Dr. Ehrenstorfer Type I	PGML	0.846
ABITEC Type II	PGML	0.902
USP Type II	PGML	0.980

## Example #2

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- January 9 spill at Freedom Industries into Elk River in Charleston, WV.
- Crude MCHM leaked from Tank #396
- Samples of the material that was in the tank collected by WV National Guard
- MSDS for product listed multiple components

## Example #2



4-methylcyclohexanemethanol

MW = 128.2 g/mole

Density = 0.9074 g/ml

BP = 202 C

Soluble in water



## Example #2

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- Assayed crude material for possible constituents
- Performed library searches of detected peaks and compared presumptive identities with what was listed on MSDS for Tier II site materials
- Used TIC responses to approximate percent MCHM content

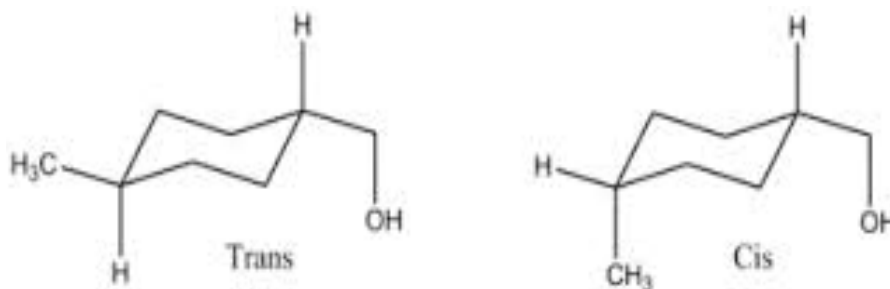


## Example #2



Analytical Grade standard found from  
TCI America

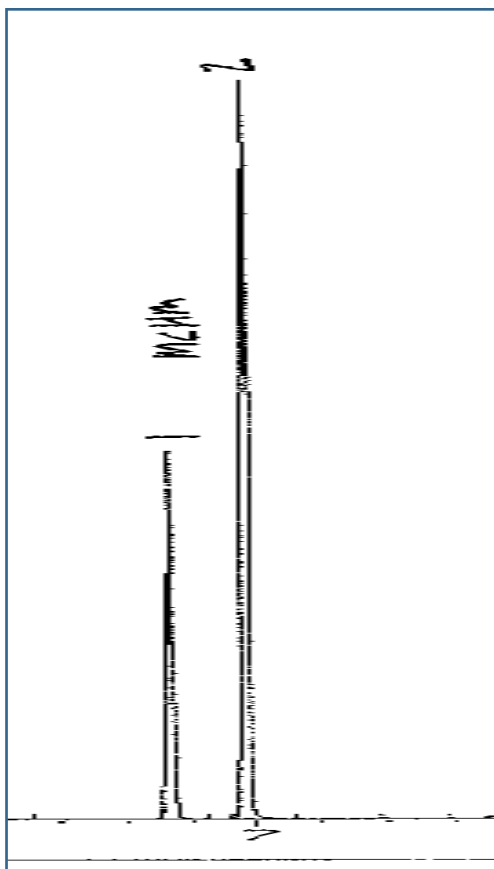
Purity listed as 98% by GC



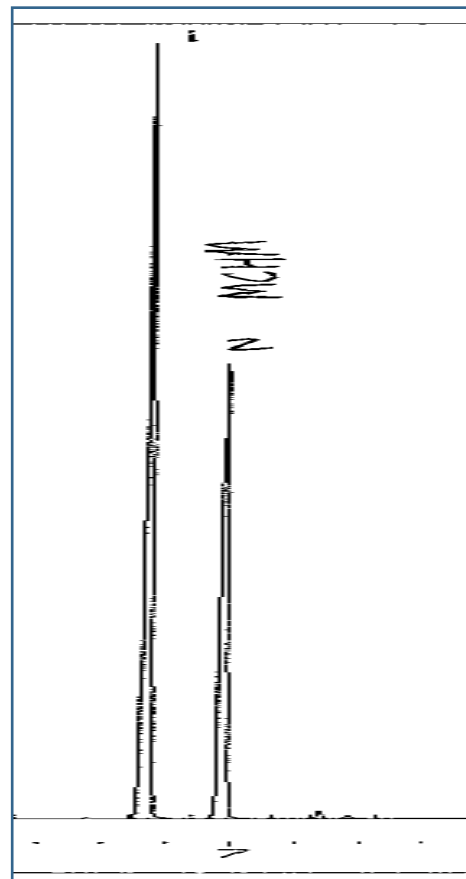
# Example #2



TCI America Standard



Crude MCHM



## Example #2

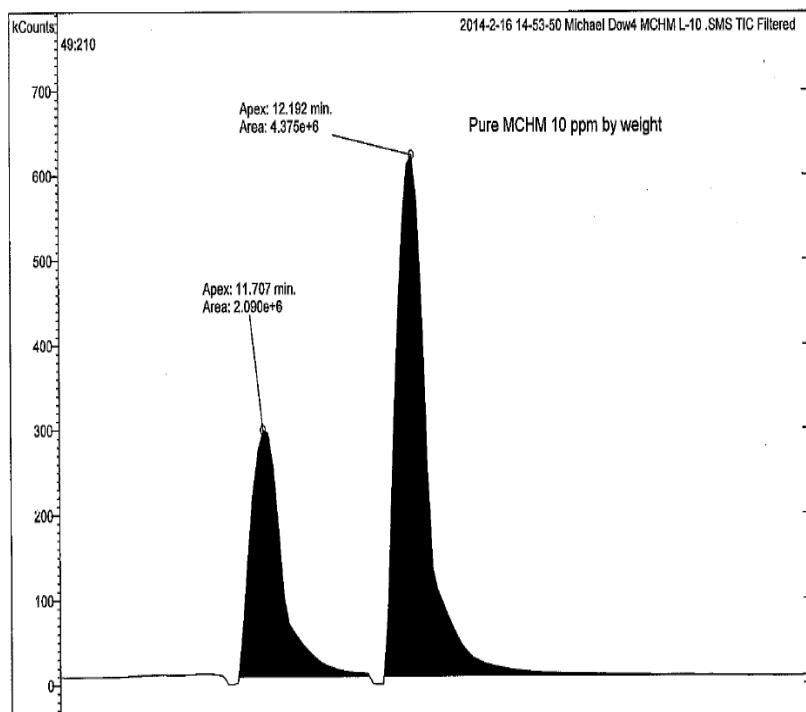


- Through work with WV TAP team able to collaborate with Dr. Mel Suffitt and group at UCLA working on oxidation by-products
- Independently, had acquired same standard material from TCI America.
- Also had aliquot of same crude material recovered from spill site
- Also assayed crude material for constituents

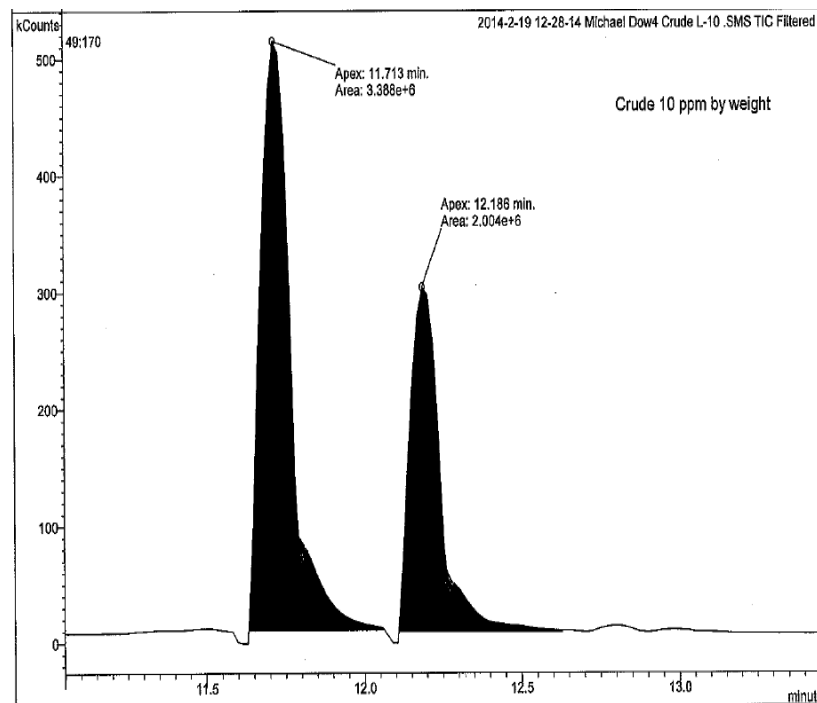
# Example #2



TCI America Standard



Crude MCHM



# Summary

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- Independent researchers confirmed same constituent contents
- Confirmed same approximate % MCHM content of crude MCHM
- Confirmed same composition of isomers and ratio of isomers in both crude MCHM and analytical standard obtained from TCI



# Questions?