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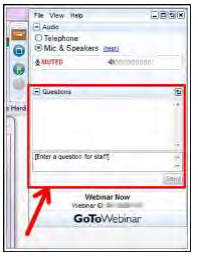

Type them into questions box!

"Why am I muted?"
Don't worry. Everyone is muted except the presenter and host. Thank you and enjoy the show.

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Hundreds of presentations from the best and brightest minds that chemistry has to offer are available to you on-demand. The Archive is divided into 6 different sections to help you more easily find what you are searching.

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From renewable fuels to creating the materials for the technology of tomorrow, chemistry plays a pivotal role in advancing our world. Meet the chemists that are building a better world and see how their science is making it happen.

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- Find a job
- Effectively use research tools like SciFinder® and ACS ChemWorx
- Communicate your science
- Write grant proposals
- Build industry partnerships
- Prepare for a changing employment landscape

http://acsoncampus.acs.org

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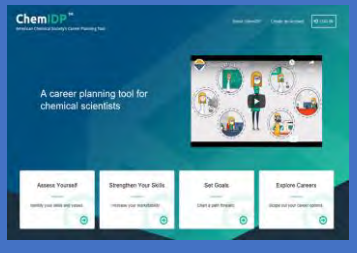

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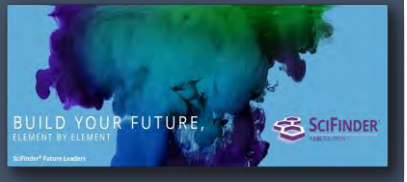

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Free ACS Webinars Every Weekday!

Upcoming Broadcasts

ACS Chemistry for Life®

Monday, June 15, 2020 at 2-3pm ET

Speakers: Marcy Towns, Purdue University / Justin Carmel, Florida International University / Edgar Arriaga, University of Minnesota

Moderator: Celia Arnaud, *C&EN*

Register for Free!

What You Will Learn

- Essential skills and knowledge that students develop in (and only in) laboratory courses
- Strategies for building laboratory curricula that develop essential knowledge and skills
- Approaches for determining what students are gaining from laboratory experiences

Co-produced with: ACS Education and *Chemical & Engineering News*

Wednesday, June 17, 2020 at 2-3pm ET

Speakers: CJ, Chemjobber.com and Kevin Swift, American Chemical Council

Moderator: Linda Wang, *Chemical & Engineering News*

Register for Free!

What You Will Learn

- The current state of the chemistry job market in both industry and academia
- How job seekers at all levels can navigate this new and uncertain job market
- Where experts see pockets of opportunity

Co-produced with: *Chemical & Engineering News*, C&EN Jobs, and ACS Career Navigator

Thursday, June 18, 2020 at 2-3:30pm ET

Speakers: Christopher Bowman, University of Colorado Boulder and Marek Urban, Clemson University

Moderator: Brent Sumerlin, University of Florida

Register for Free!

What You Will Learn

- The current state-of-the-art and future of self-healing polymers
- Physical and chemical processes involved in self-healing of polymers and how to design polymers with these properties
- How dynamic covalent chemistry (DCC) can be used to induce healing, actuation, phase changes and other desirable attributes

Co-produced with: ACS Division of Polymer Chemistry

www.acs.org/acswebinars

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ACS Efforts and Resources on COVID-19

Browse **ACS Resources** and **Initiatives!**

- **YOU MAY RECEIVE A ONE-YEAR WAIVER ON YOUR NATIONAL DUES** If your membership is up for renewal, but you're experiencing a special hardship, such as unemployment, furlough, reduced wages or illness.
- **RECEIVE ACCESS TO LINKEDIN LEARNING THROUGH THE END OF THIS YEAR** This powerful resource includes over 15,000 on-demand courses to support your continued learning and career advancement for active ACS members.
- **INOVA EAP/WORK-LIFE ASSISTANCE PROGRAM** 24/7 assistance on a wide range of issues, such as emotional, relationship, major life, health, wellness, educational and more for ACS members based in the United States. Confidential services are provided via telephone or comprehensive online resources.

www.acs.org/covid-19

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2020 GC&E Virtual Conference

REGISTER FOR FREE!

On June 15-19, from 11am-5:30pm EDT attendees will view and engage with:

- Daily keynote speakers
- 40+ technical sessions
- 80+ posters
- Networking breaks and #gcande happy hour
- Green Expo
- GC&E Fridays: Live technical sessions on Fridays leading up to the conference week

https://www.gcande.org/register

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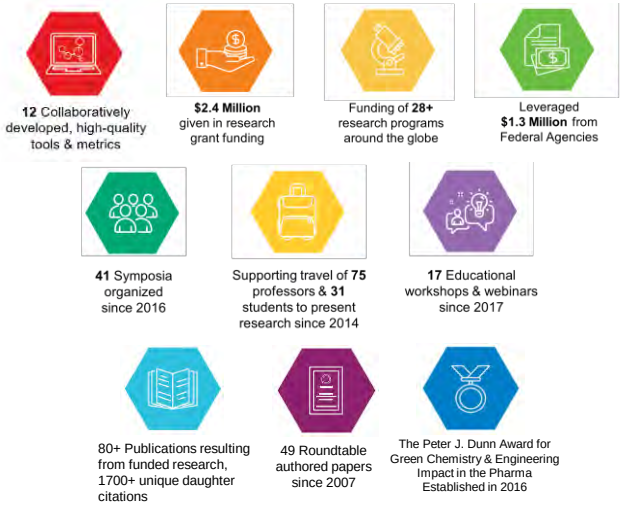
15 YEARS of Catalyzing GC&E in Pharma and Beyond

ACS Green Chemistry Institute Pharmaceutical Roundtable

- Bridging the gap between academics & industry
- Enabling better decisions about chemical selection process design
- A leading voice for GC&E
- Inspiring and educating the next generation
- Recognizing excellence in GC&E



Enabling Better Decisions About Chemical Selection Process

ACS Green Chemistry Institute Pharmaceutical Roundtable

12 Collaboratively developed, high-quality tools & metrics

- SOLVENT GUIDE
- SOLVENT SELECTION TOOL
- PROCESS MASS INTENSITY METRIC (PMI)
- CONVERGENT PMI
- PMI-LCA METRIC
- PMI PREDICTION TOOL
- BIOPHARMA PMI TOOL
- REAGENT GUIDES
- GREEN CHEMISTRY INNOVATION SCORECARD CALCULATOR
- MEDCHEM TIPS AND TRICKS
- BIOCATALYSIS CHEAT SHEET
- **ANALYTICAL METHOD GREENNESS SCORE TOOL**

"They encompass multiples aspects of a chemical process enabling more sustainable outcomes to be achieved in a proactive manner." Paul Richardson, Pfizer

2020 Membership

ACS Green Chemistry Institute Pharmaceutical Roundtable

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THIS ACS WEBINAR WILL BEGIN SHORTLY...

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ACS Webinars
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ACS Chemistry for Life®

A Perfect Match: Vibrational Spectroscopy and Sustainable Chemistry

John Wasylyk
Senior Principal Scientist,
Bristol-Myers Squibb

Bob Wethman
Senior Research Scientist,
Bristol-Myers Squibb

Joe Fortunak
Professor of Chemistry,
Howard University

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This ACS Webinar is co-produced with the ACS Green Chemistry Institute and the ACS GCI Pharmaceutical Roundtable.

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Audience Challenge Question

ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT

How many webinars have you attended in the last 60 days?

- Just this one
- 2 - 10
- 11 - 20
- I have lost count
- None, I can't find "Webinars" on Netflix?

** If your answer differs greatly from the choices above tell us in the chat!*

Instruments

- **Spectroscopy-based Instruments:**
  - FT-IR
  - FTNIR
  - Raman
- **Additional Instruments:**
  - FBRM (Lasentec)
  - Mass Spectrometer
- **Advantage:**
  - ✓ Safety….minimal to no sample handling!
  - ✓ Speed….less than 2 minutes!
  - ✓ Process Knowledge….kinetic profiling!
  - ✓ Non-invasive….you can have your sample back!
  - ✓ Rapid Method Transfer and Validation
  - ✓ Sustainability…little or no sample prep
- **Portable Instruments and Walk-ups Available**

Green Instrument Capabilities

- **Raman** (in-line and off-line)
  - Reaction monitoring and control (replaces HPLC)
  - Solvent exchanges (replaces GC)
  - Form control and determination (replaces PXRD)
- **FT-NearIR** (in-line and off-line)
  - Solvent exchanges (replaces GC)
  - Reagent analysis (replaces titration)
  - Moisture determination (replaces KF)
- **FT-IR** (in-line and off-line)
  - Reaction monitoring and control (replaces HPLC)
  - Foreign material investigations (What are those specs? Is this what I ordered? Is it still good?)
- **UV/VIS** (in-line and off-line)
  - Reaction monitoring and control (replaces HPLC)
- **Mass Spectrometry**
  - Off-gas for reaction monitoring, drying, identification (can replace GC/KF/HPLC)
- **Focused Beam Reflectance Measurement**
  - Particle size and distribution

Spectroscopy Advantages

- **No or Minimal Sample Preparation Required** *Safety
  - Minimize Potential Sources of Error
  - Minimize Sample Exposure
  - Generally less than 2 minutes
  - Non-invasive
- **Robust Instruments** *Green
  - Low Maintenance, Minimal Moving Components
- **No Additional Wastes or Waste Streams** *Green
  - No Gases Involved, No Additional Solvents Required
- **Method Development Time**
  - Equivalent/Slightly Longer Chemometric Model Development Time
- **Transfer of Methods** *Speed & Quality *Green
  - Across Instruments
  - From At-Line to In-Line
  - No Re-development Calibration Curve of Required
    - Matched Instrument
    - Locked Chemometric Models
      - Can Update Models as New Data is Gathered

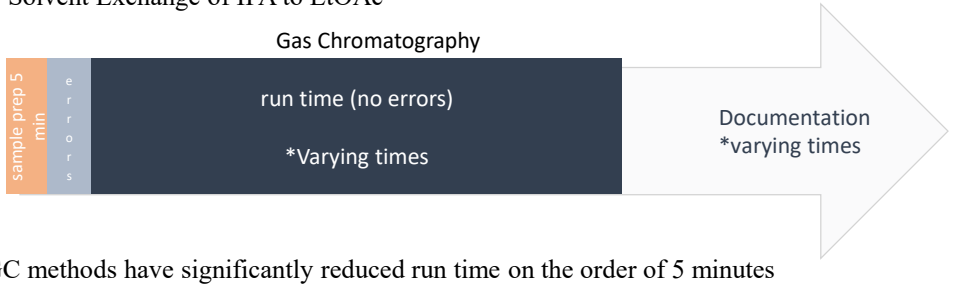
SPECIFICS and EXAMPLES

Benchtop Raman Spectrometer with Non-Contact Sampling

Comparison of GC versus a Spectroscopy-Based Method for a Solvent Exchange

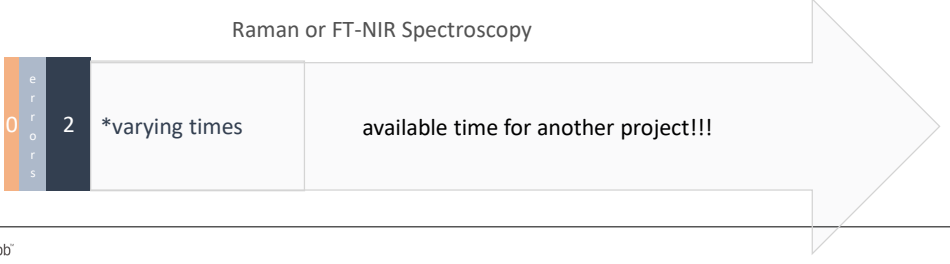
Solvent Exchange of IPA to EtOAc

Gas Chromatography



* Fast GC methods have significantly reduced run time on the order of 5 minutes

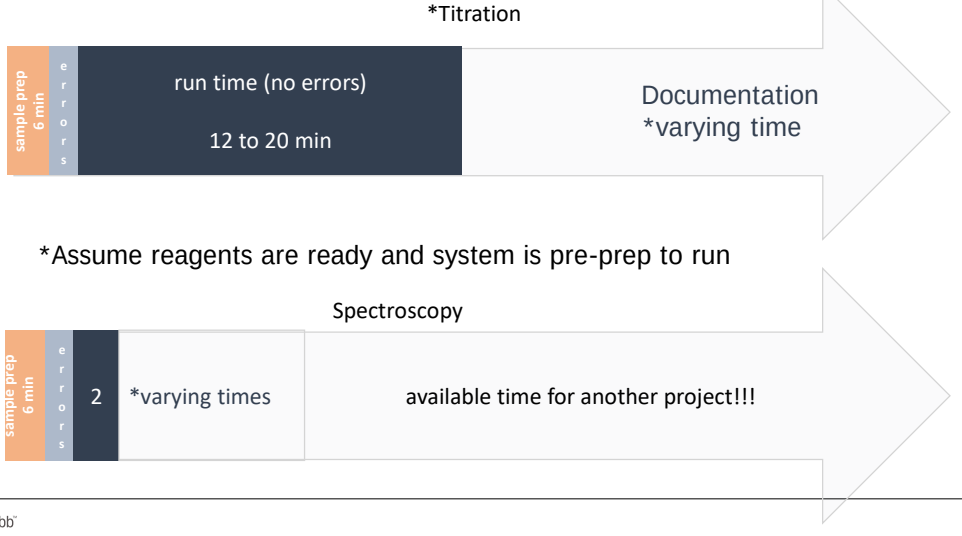
Raman or FT-NIR Spectroscopy



Spectroscopy Methods: Replacement of Traditional Assays

Analyte	Diluent	Instrument Type	Unit
Acetoin	Toluene	Raman	molarity
Acetone	BMS-cpd in MeOH	Raman	weight %
Acetone	IPA	Raman	volume %
Acetone	MTBE	Raman	volume %
Acetone	n-butyl acetate	Raman	volume%
Acetone	DCM	FT-NIR	weight %
ACN	EtOAc	Raman	weight %
ACN	IPAc	Raman	volume %
ACN	MeOH	Raman	volume %
ACN	Toluene	Raman	volume %
ACN	SDA3A	Raman	volume %
DCM	Toluene	Raman	volume %
DCM	THF	Raman	volume %
DCM	ACN	Raman	volume %
DCM	DMF	Raman	weight %
DCM	THF	Raman	weight %
DCM	IPA	Raman	volume %
DCM	Acetone	Raman	volume %
DCM	IPAc	Raman	volume %
DCM	MeOH	Raman	volume %
DCM	EtOAc	Raman	weight %
DCM	IPA-H2O (3:1)	Raman	volume %

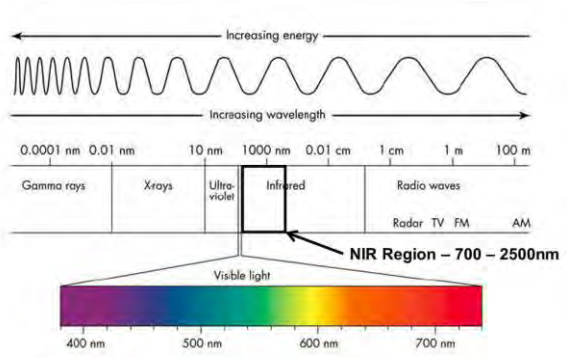
Comparison of a Titration Method Versus a Spectroscopy-Based Method



Fourier Transform Near Infrared Spectroscopy (FT-NIR)

Thermo Electron Corporation. Theory and Utility of FT-NIR Spectroscopy

- **Study of the interaction between matter and electromagnetic waves**
- **NIR absorbers include O-H, C-H, N-H, and S-H bonds**



Karl Fisher Reagent (stabilized)

Used for the Quantitation of Water in Liquids and Solids

Component	CAS-No	Weight %
2-Methoxyethanol	109-86-4	52.25
Pyridine	110-86-1	25.40
Iodine	7553-56-2	14.55
Sulfur dioxide	7446-09-5	7.80

- **Causes skin irritation**
- **Causes serious eye irritation**
- **Harmful if inhaled**
- **May damage fertility.**
- **May damage the unborn child**
- **Causes damage to organs**
- **May cause damage to organs through prolonged or repeated exposure**

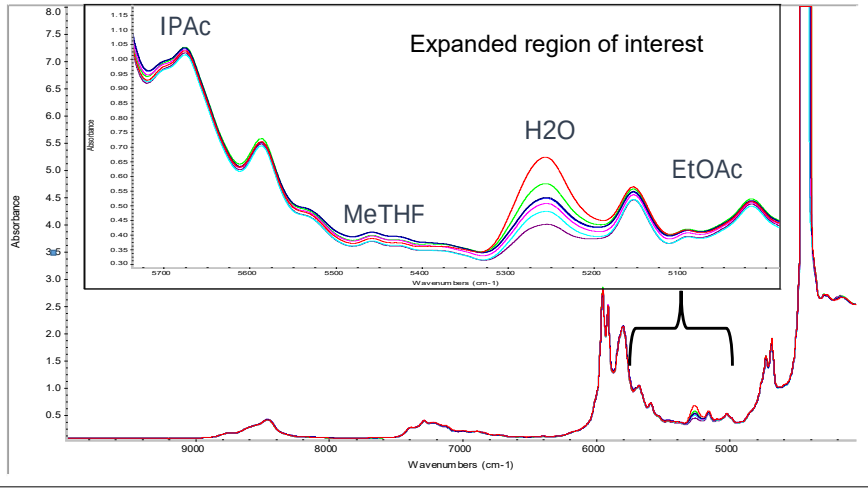
Spectroscopy Methods: Replacement of Traditional Assay (KF)

H2O	ACN with MeOH	FT-NIR	weight %
H2O	THF	FT-NIR	weight %
H2O	DCM	FT-NIR	ppm
H2O	nBuOH	FT-NIR	weight %
H2O	ACN (with drug product present)	FT-NIR	weight %
H2O	EtOAc	FT-NIR	weight %
H2O	N-methyl imidazole	FT-NIR	weight %
H2O	IPA	FT-NIR	weight %
*H2O	MeTHF/IPAc/EtOAc	FT-NIR	weight %
H2O	ACN	FT-NIR	weight %
H2O	MeOH	FT-NIR	weight %
H2O	IPAc	FT-NIR	ppm
H2O	2-MeTHF	FT-NIR	weight %
H2O	2-MeTHF (with drug product present)	FT-NIR	weight %
H2O	EtOH	FT-NIR	weight %

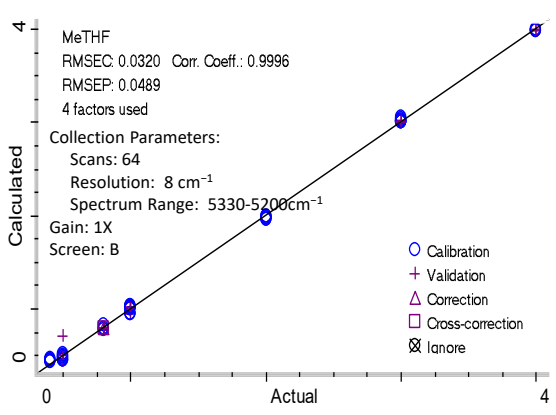
***ONE Spectrum Collected….FOUR Results**

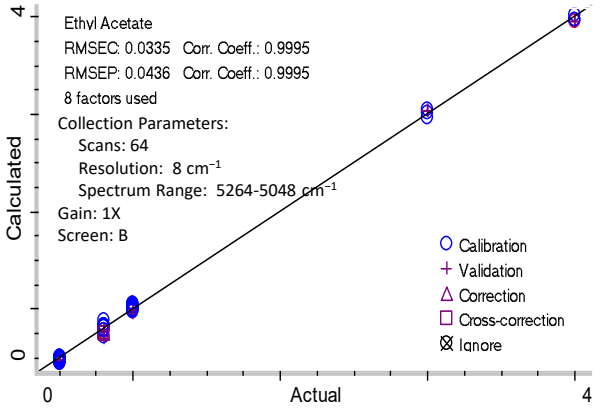
FT-NIR: Overlay of Spectra for MeTHF/EtOAc/H2O/IPAc

Methyl Tetrahydrofuran/Ethyl Acetate/Water/Isopropyl Acetate



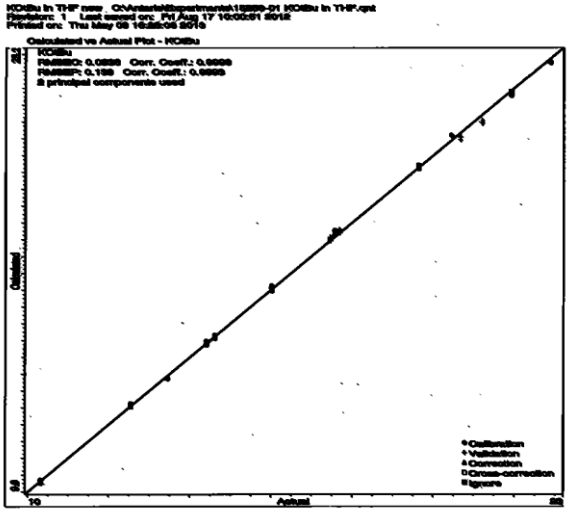
FT-NIR: Linearity for MeTHF and EtOAc in MeTHF/EtOAc/H2O/IPAc





Linearity of K-tBuOx in THF via FT-NIR

- 2 principal components
- PLS model
- Range up to 23 wt%
  - (1.8M)
- Sample run ‘as is’
- No Reagents!



Spectroscopy Facilitates Lean Analysis.” John Wasylyk, Ming Huang, Bob Wethman, Kieran O’Connor. *Pharmaceutical Technology*. 43(2) 35–39 (2019).

Spectroscopy Methods: Replacement of Traditional Assays

Analyte	Diluent	Instrument Type	Unit
HCl	MeOH	FT-NIR	molarity
HCl	EtOH	FT-NIR	molarity
HCl	IPA	FT-NIR	molarity
Hydrazine	H2O	Raman	molarity
KEtOx	EtOH	Raman	molarity
KEtOx	EtOH	FT-NIR	weight %
KtBuOx	THF	FT-NIR	weight %
KtBuOx	THF	Raman	molarity
LitBuOx	THF	Raman	molarity
NaEtOx	EtOH	Raman	molarity
NaHMDS	THF	Raman	molarity
NaEtOx	EtOH	FT-NIR	weight %
NaOMe	CH3OH	Raman	molarity
NaOMe	CH3OH	FT-NIR	weight %
Red-Al	Toluene	Raman	molarity

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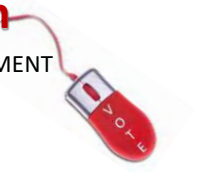
Audience Challenge Question

ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT



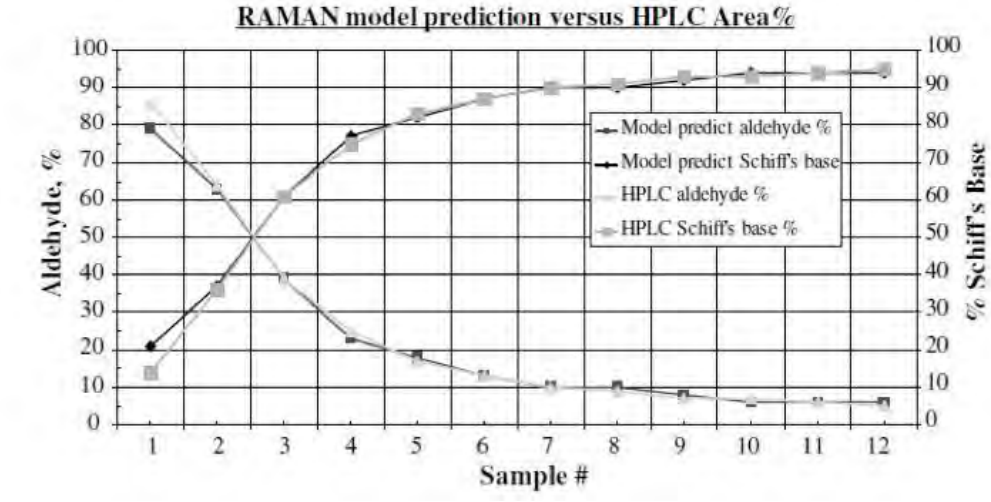
What is your primary go-to method for following a chemical process:

- HPLC
- LC-MS
- NMR
- Pass the samples to someone else, let them worry about it
- use an in-line based system

** If your answer differs greatly from the choices above tell us in the chat!*

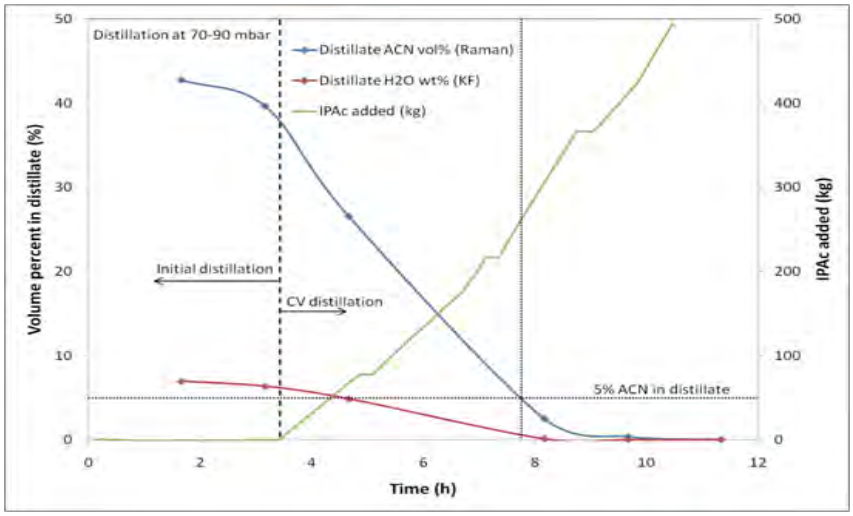
Production-based In-line Raman

Typical Reaction: In-line Raman versus Grab Samples

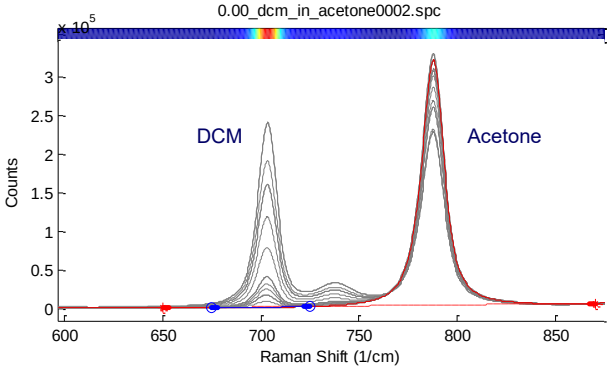


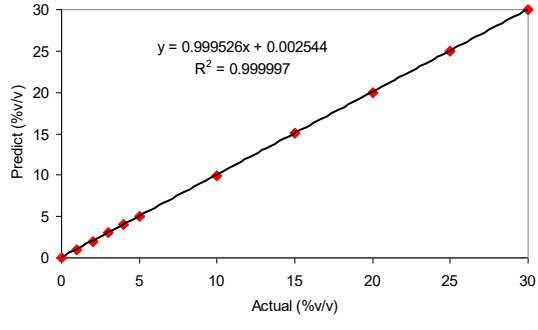
Process Knowledge Gained, Sampling Eliminated

DynoChem Model for the Distillation of IPAc/H_2O /ACN
Two ways to sample: (1) Off-line; (2) In-line



Overlay of 0-30% DCM in Acetone and Linearity



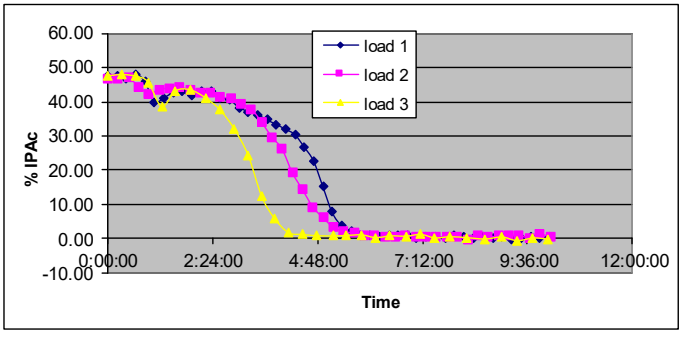


Ensuring Product Quality with Process Raman and FT-IR Spectroscopy." John Wasylyk. *Spectroscopy*, **31**(5) 33-35 (2016).

Enhanced Productivity

- Reduced overall production cycle time by 1 hr 25 min (equivalent to 2.5%) from the original 42 hrs per batch.
- The projected production cycle time savings is approximately 100 hrs for a year long campaign of 98 batches.
- This single method change saved enough time to produce 2 extra batches.
  - ✓Increased safety and productivity
  - ✓Decreased waste, and cost

Centrifuge Washing

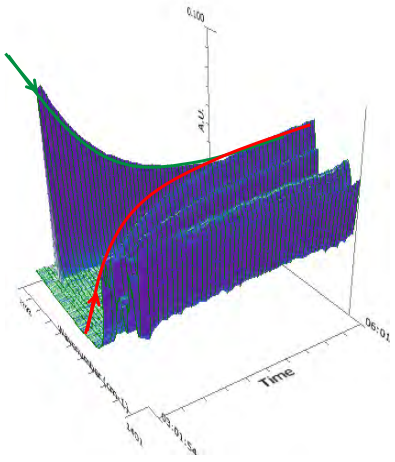


Monitor removal of residual solvent via anti-solvent cake washing

- Confirm low solvent level vs. anti-solvent (increased volatility) to minimize drying time
- Minimize washing cycle time and volumes
- 20 second feedback

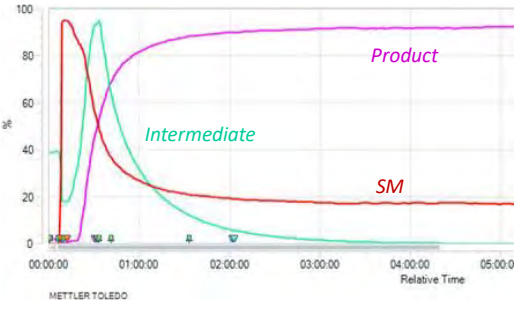
Sustainability!!!

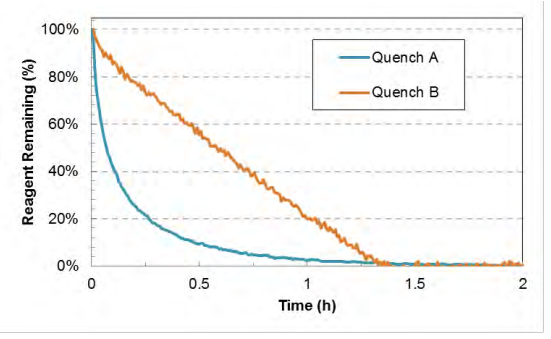
FT-IR: In-line Approach



Plant Mid-IR System

Application: Reaction Kinetics

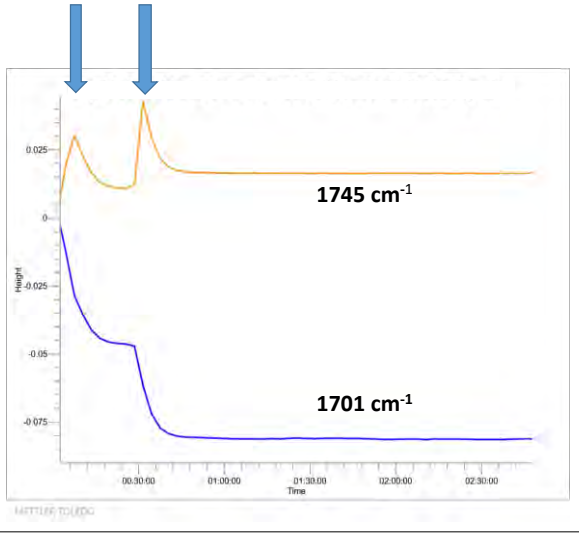




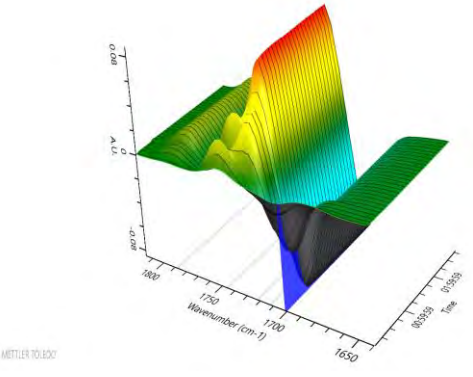
- In early development, PAT often provides the most flexible analytical tool for endpoint determination (before good HPLC methods are available)
- Even simple kinetic analysis can give you important mechanistic insights

Halogenation with N-chlorosuccinimde

NCS delivered in two charges

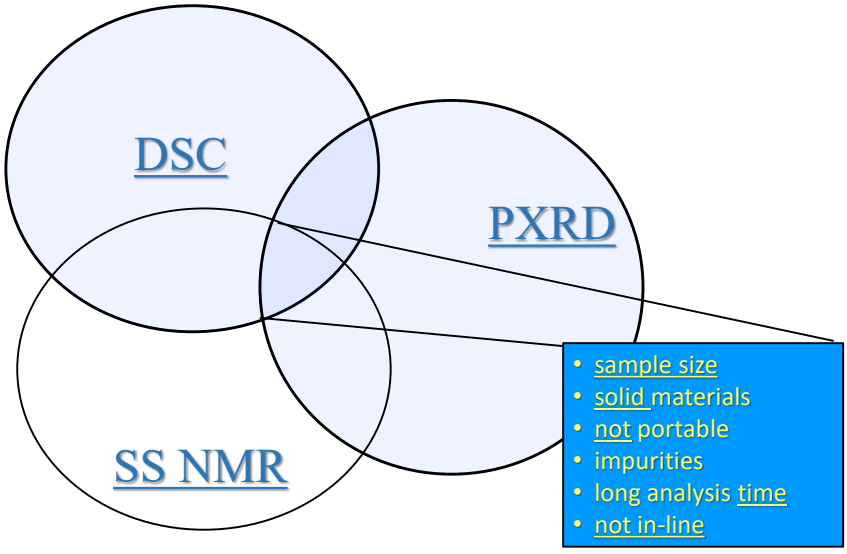


3D Plot of key spectral bands

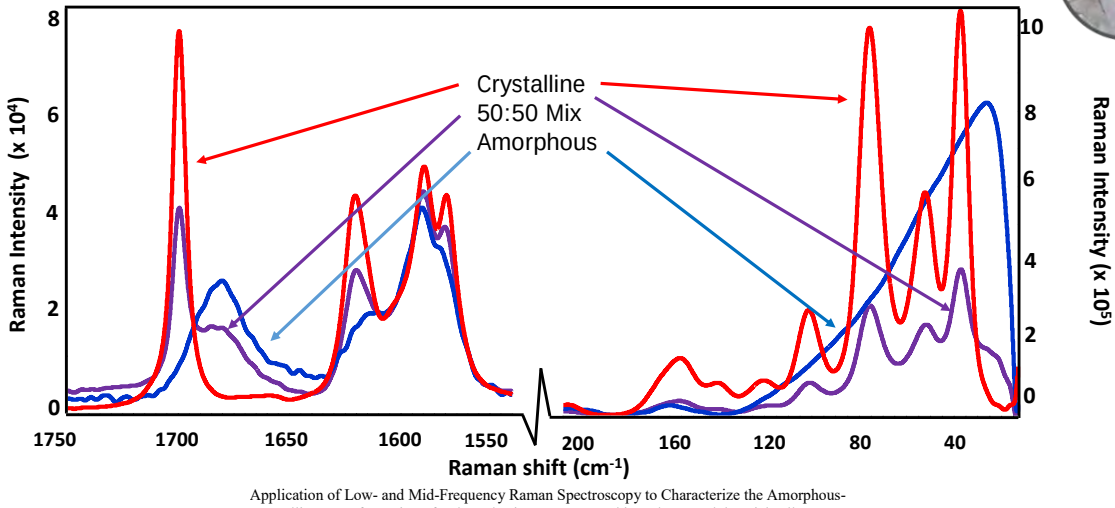


To be presented at: ACS GCI's 24th Annual Green Chemistry & Engineering Virtual Conference. 15-19 June 2020.

3 Common Crystal Form Identifiers

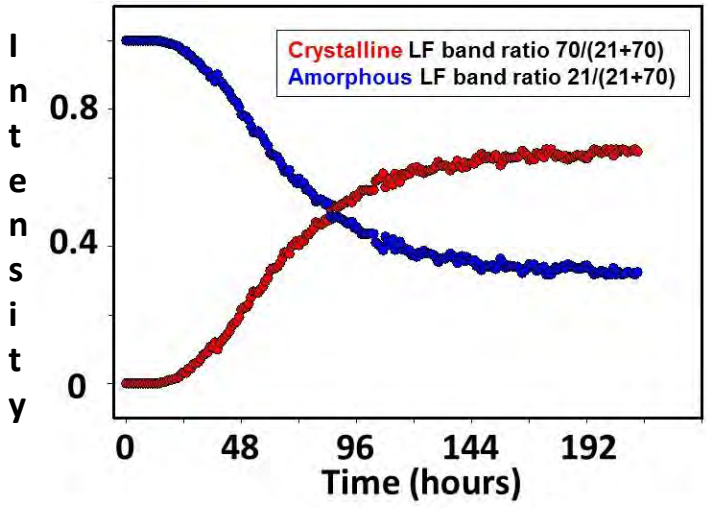


Low Frequency Raman: Amorphous and Crystalline Material

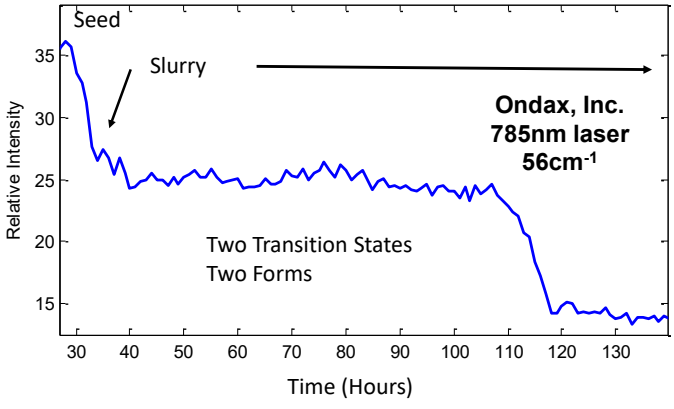


Application of Low- and Mid-Frequency Raman Spectroscopy to Characterize the Amorphous-Crystalline Transformation of Indomethacin." Peter J. Larkin, John Wasylyk, Michaella Raglione. *J. of Applied Spectroscopy*. **69**(11) 1217-1228 (2015).

Tracking the Change



In-line Trend After Initiation of Crystallization at 57 C



Capture the transitions without taking samples!

Additional in-line tools to enhance sustainability

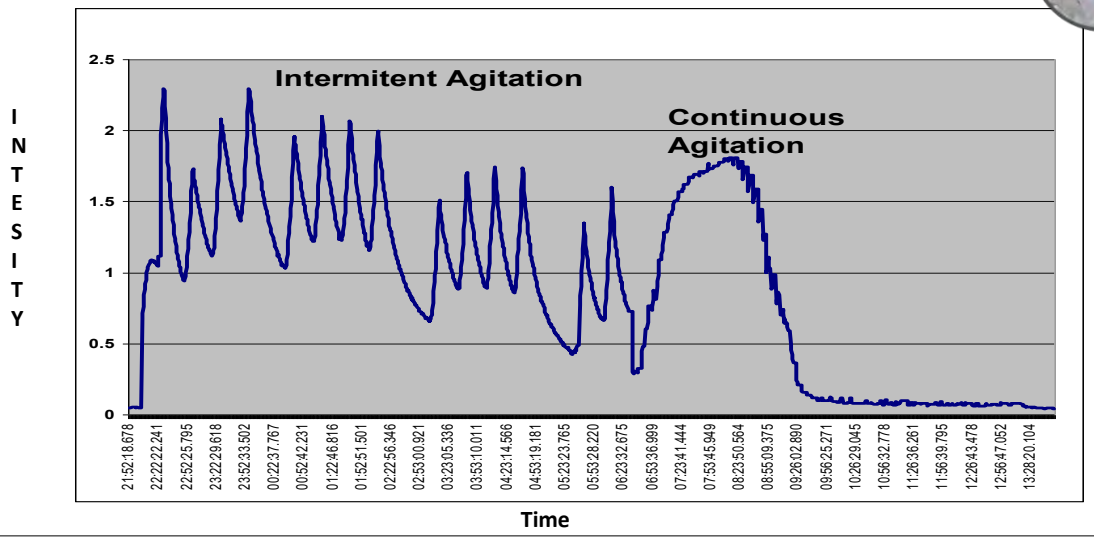
Non-vibrational spectroscopy-based instruments

In-line Mass Spectrometry

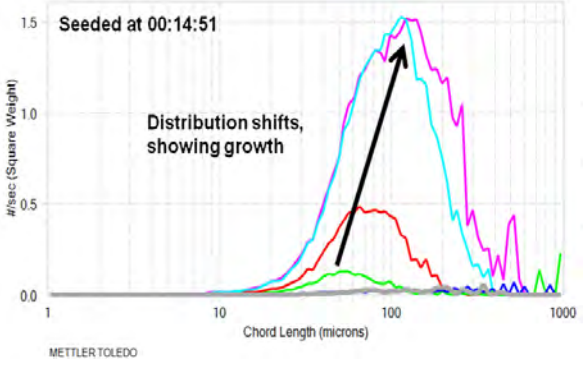
- **In-line Mass Spectrometer**
  - Confirmation of removal of solvent during the drying process
  - Scrubber efficiency
  - Specific off-gases
  - Unknown off-gases

  - **Unique applications:**
    - Can use a gas sampling bag
    - Sample from a rotovaporator
    - Off-gas from reagents
      - Stability
      - Contamination

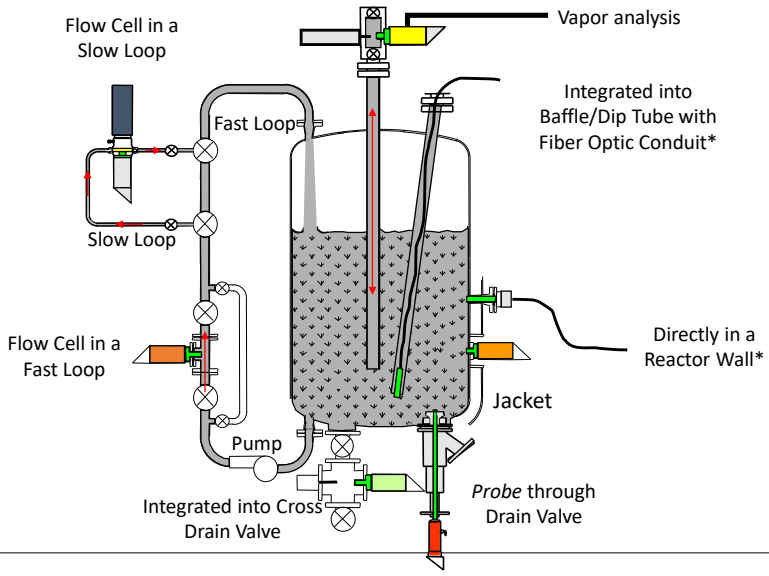
In-Line Mass Spectroscopy: Removal of Solvent



Focused Beam Reflectance Measurement (FBRM): Crystallization



Interface Overview for a Reactor



Collaborative Efforts During Method Validation

- **Develop method** (Site 1)
  - Use *pure solvent* as standard to build calibration set
  - Use *pure solvent* to build validation set for testing
  - Test method on a second instrument for Instrument-to-instrument robustness
  - Sent method (Experiment and method calibration files) to Site 2 partner
- **Test method for distillate or pot samples** (Site 2)
  - Verify the method works for distillate/pot sample vs GC results
- **Modify method if needed for pot samples** (Site 1)
  - Modified peak regions to avoid interference from API if needed
- **Develop validation protocol** (Site 1 / Site 2)
- **Execute validation** (Site 1 / Site 2) **– no tech transfer required**
- **Co-validation**

Selected Spectral References:

- Larkin, P.J., Infrared and Raman Spectroscopy: *Principles and Spectral Interpretation. Elsevier: Oxford, UK 2017.*

- *Workman, J., Weyer, L., Practical Guide to Interpretive Near-Infrared Spectroscopy, CRC Press: Boca Raton, FL 2008.*

- *Bakeev, K.A., Process Analytical Technology. Blackwell Publishing: Oxford, UK 2008.*

Thank you!

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- SOLVENT GUIDE
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- CONVERGENT PMI
- PMI-LCA METRIC
- PMI PREDICTION TOOL
- BIOPHARMA PMI TOOL
- REAGENT GUIDES
- GREEN CHEMISTRY INNOVATION SCORECARD CALCULATOR
- MEDCHEM TIPS AND TRICKS
- BIOCATALYSIS CHEAT SHEET
- **ANALYTICAL METHOD GREENNESS SCORE TOOL**

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