

Laurent Calcul, Ph.D.
Director

CPAS operates as a self service core facility and offers its expertise in chromatographic separation and purification (MPLC, HPLC, UHPLC), mass spectrometry detection, identification and quantification (LC-MS SQ, LC-MS QqQ, LC-MS Triple TOF, TDU-GC-MSD, GC-MSD/ μ ECD and GC-MS QTOF [HS-SPME] and MALDI TOF). The services also include Circular Dichroism (CD, VCD), Optical Rotatory Dispersion (ORD), general spectroscopic characterization (UV, FTIR), lyophilization (benchtop and general purpose freeze-dryer), extraction (microwave reactor), synthesis (microwave reactor, hydrogenation apparatus), ADME screening (Parallel Artificial Membrane Permeability [PAMPA - GIT, BBB and skin] and drug solubility evaluation), binding affinity measurement (Microscale Thermophoresis [MST]), and high-throughput screening development capability (liquid handling robot and multimode plate reader). CPAS provides trainings and instruments are available 24/7 in self-service mode and upon reservation for trained users.

Email – calcul@usf.edu; Web – <https://www.usf.edu/arts-sciences/departments/chemistry/research/core-facilities/cpas.aspx>

- Location site 1: 3720 Spectrum Blvd., IDRB, Suite 318 – Tampa, FL 33612 <> Office (813) 974-0112
- Location site 2: 4202 E. Fowler Ave, NES 406 – Tampa, FL 33620 <> Office (813) 974-6031

Liquid Chromatography Mass Spectrometry (LC-MS)

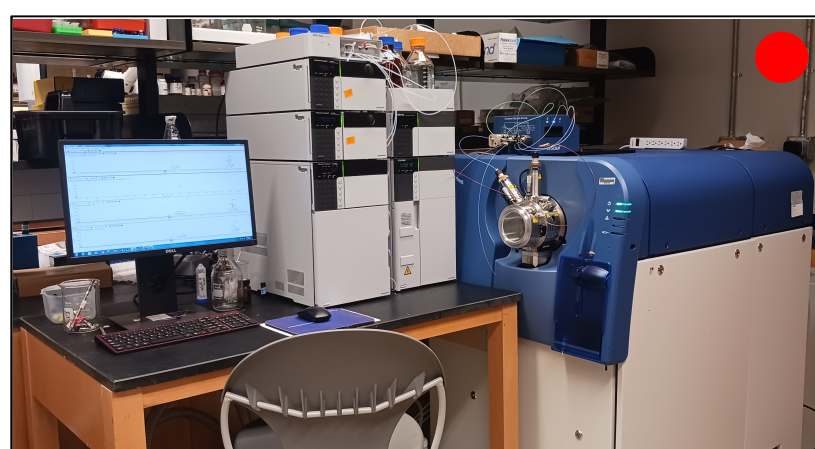
High Resolution Mass Spectrometry (HRMS and accurate MS/MS)

Shimadzu LC-MS Triple TOF AB Sciex 5600TOF

- HRMS for Formula determination (organic small molecules to proteins)
- Identification capability
- Metabolomics capability

Modes and Features:

- ESI source (positive or negative mode)
- APCI source
- Software: Analyst, PeakView, Multiquant



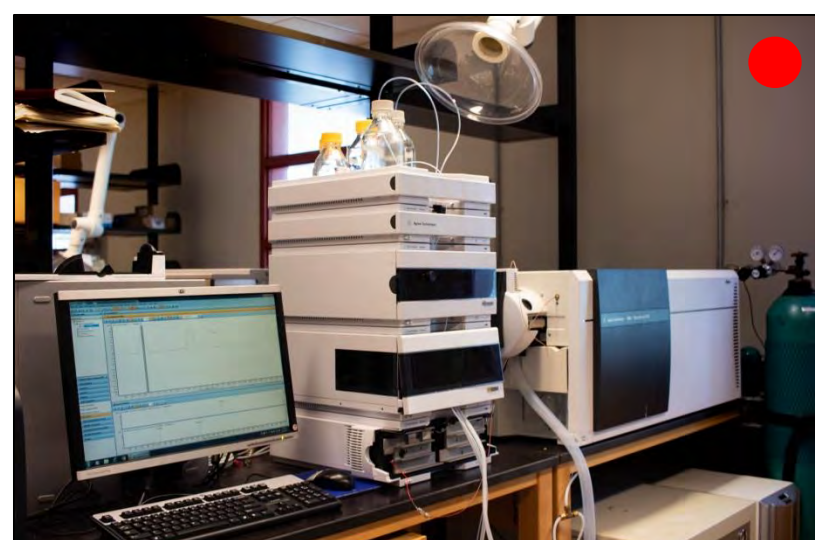
High sensitivity in triple quadruple LC/MS/MS

Agilent 1200 LC/ 6460 QqQ MS

- Multiple reaction monitoring (MRM) ideal for Quantification
- Sensitivity (Femtomole to Attomole range)

Modes and Features:

- ESI (positive or negative mode)
- Agilent Jet-stream Electrospray Ionization
- Software: Mass Hunter, MS/MS optimizer



Low Resolution Mass Spectrometry

1) Analytical Agilent 1100 LC/ G1956A SQ MS

- Robust Low resolution mass measurement
- ESI (positive mode)
- APCI capability

2) Preparative Agilent 1200 infinity HPLC/ DAD/ SQ MS

- Fast switching ESI (+/-)
- Diode array detector (DAD, 190-600 nm)
- Autosampler and fraction collector



Gas Chromatography Mass Spectrometry (GC-MS)

High Resolution Mass Spectrometry (HRMS and accurate MS/MS)

Agilent 7890A GC/ 7200 QToF MS

- HRMS for Formula determination
- Identification
- Metabolomics capability
- Quantification capability

Modes and Features:

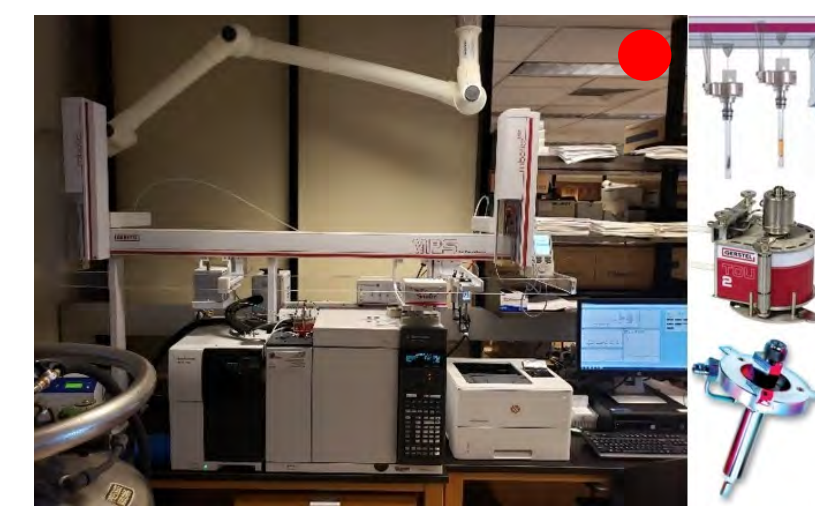
- EI, PCI and NCI
- 10 μ L syringe, HS (2.5mL syringe or SPME fiber)



Low Resolution Mass Spectrometry (LRMS)

Agilent 7890B GC/ 5977B SQ MS

- Selected ion monitoring (SIM)
- Gerstel MultiPurpose Sampler (MPS):
 - Dual headed for sample preparation, 10 μ L syringe, HS (2.5mL syringe or SPME fiber)
 - cold injection system (CIS) using LN₂
 - TDU using ALEX for Twister/ Stir Bar SBSE, TF-SPME and desorption liner



Low Resolution Mass Spectrometry (LRMS)

Agilent 7890B GC/ 5977B SQ MS/ μ ECD

- 2-inlet system:
 - Multi-mode inlet (MMI) with LCO₂ for automated (150-vial sampler) injection to the MSD
 - Split-splitless inlet (SSI) for manual injection connected to the Electron captor detector (μ ECD)



Note: All GC-MSs contain NIST library

Medium to Ultra High Performance Liquid Chromatography

- Large scale from 0.5 to 300 g
TELEDYNE-Isco Torrent (UV 200-760 nm)
- Scale from 200 mg to 33 g:
- TELEDYNE-Isco Rf (UV 200-360 nm)
- Shimadzu semi-preparative HPLC for sample fractionation and purification (UV, ELSD and RID)
- Fisher Scientific Dionex Ultimate 3000 UHPLC (DAD)



Matrix Assisted Laser Desorption Ionization (MALDI)

MALDI TOF and TOF/TOF technology

Bruker UltraFleXtreme MALDI

- Protein, peptide, lipids, small molecule and polymer
- MALDI Top down sequencing (TDS)
- Imaging (MALDI-MSI)

Modes and Features:

- MS (linear, reflector), MS/MS (LIFT) modes
- Smartbeam II, 2GHz for MS and 1GHz for MS/MS
- Resolution > 45,000



ADME (PAMPA), solubility and HTS capability

- PAMPA (3 sets: Skin, Gastrointestinal tract [GIT] and Blood-brain barrier [BBB])
- Solubility evaluation
- Tecan Freedom Evo 150 automated liquid handler (MCA96, LiHa 8-span, RoMa) with infinite M-1000 Pro, multimode microplate reader (excitation/emission, absorbance, fluorescence, fluorescence polarization and luminescence) for HTS development.



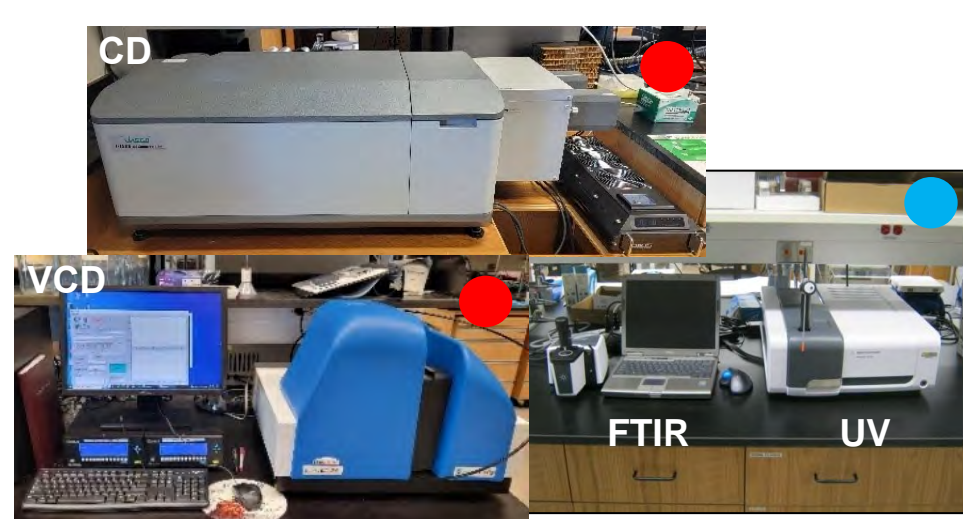
MicroScale Thermophoresis (MST)

- Molecular interaction, binding affinity measurements (K_d)
- Fluorescence channels: RED & BLUE
- Dynamic range 1nM to μ M
- Up to 24 samples/ run
- Small molecules, proteins, DNA, ...



Additional resources provided by the CPAS core facility

CPAS is a convenient platform to assist investigators in natural product chemistry (extraction, lyophilization, chromatography, spectrometry, spectroscopy), medicinal chemistry (microwave assisted reaction, ADME screening), as well as biology (storage, HTS development) for Drug Discovery:



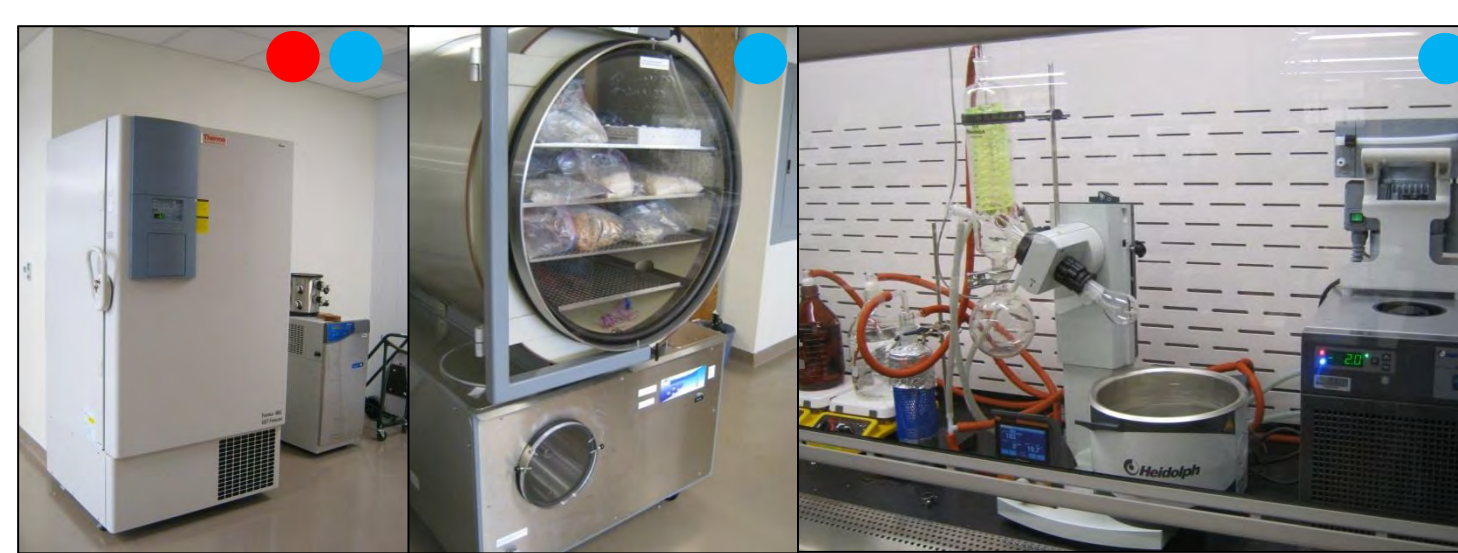
Spectroscopy

- CD (JASCO [E]CD, Biotoools VCD)
- FTIR (Agilent Cary 630, ATR)
- UV (Agilent Cary 60, fiber optic)



Synthesis, extraction activities

- Solvent purification
- Anton Paar Monowave 300
- Hydrogenation apparatus
- Accelerator solvent extractor 350



Storage and drying capabilities

- -80°C freezer, -20°C Cold room, -20°C freezer
- Benchtop and high capacity (GPFD) freeze-dryers
- Rotary evaporator



General lab use:

- Bench
- Fume hood