# Developing an Efficient Coordinated Organic Analysis for Returned Samples

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## Introduction

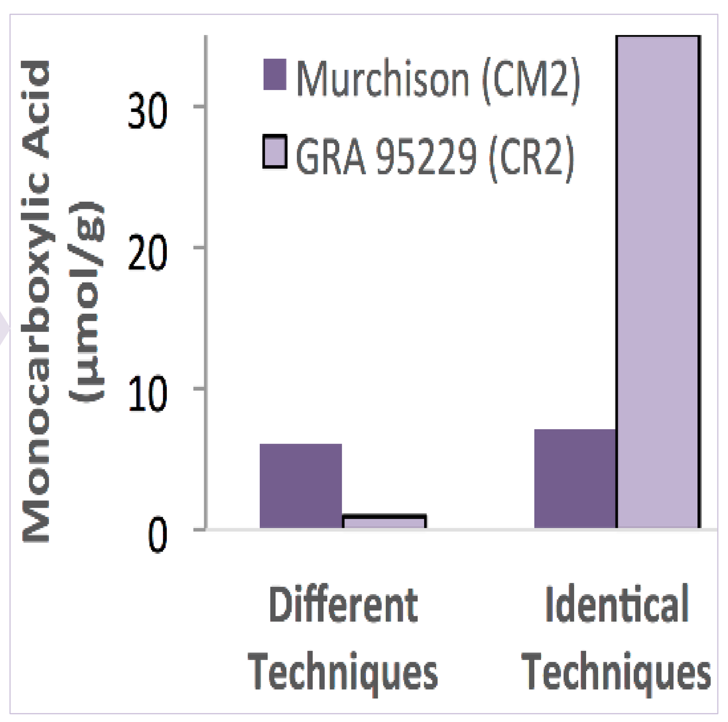
### Problem

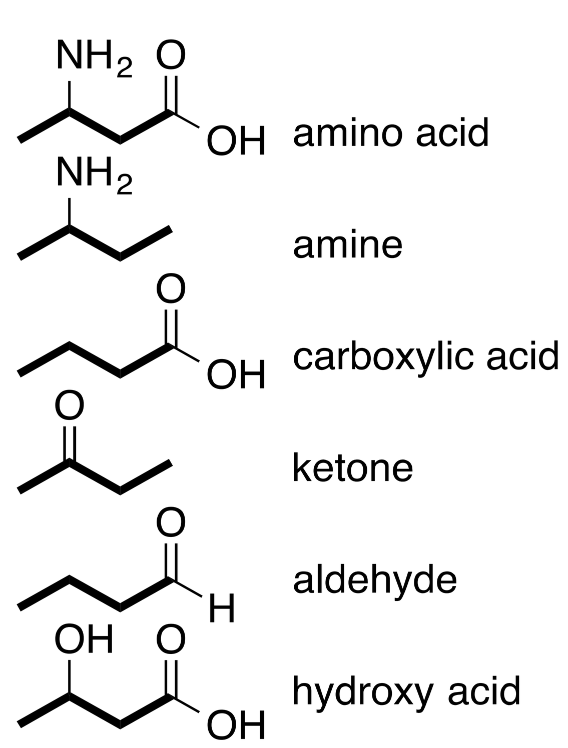
- **Analysis of soluble organics in extraterrestrial samples can introduce contamination, alteration, and loss.**
- **These effects are not always well characterized, making interpretation and comparisons difficult.**

Understanding the organic content of meteorites, returned lunar samples, and other extraterrestrial materials is needed to address cosmochemical and astrobiological questions. The essential first steps of these analyses are **sample extraction and purification**. The preparative steps are when the sample has the greatest **risk of contamination, loss, or alteration.** However, a rigorous analysis of the trade-offs that bias yields of one compound class, let alone many, is rarely performed.

When different methods are used, the differences in the resulting analyses can be substantial.

**Very different conclusions are reached about** relative abundances of carboxylic acids in two meteorites when they are analyzed using different preparative techniques (left, Murchison [1] and GRA 95229 [2]) vs. identical techniques (right, [3]).




We are evaluating and optimizing methods for **six structurally related compound classes** that have been detected in extraterrestrial samples.

**A new coordinated approach** will enable analysis of multiple compound classes, maximize efficiency of sample use, and allow for meaningful comparison of data with different measurements.

### Key questions

- What protocol leads to optimal purification of various compound classes?
- What are the opportunity costs and benefits of different methods?
- How do conditions (e.g., temperature, time) affect aqueous extraction of various soluble organic compound classes?
- How do variables such as pH, time, and temperature affect hydrolysis of extracts?
- What is the most efficient sample allocation and order of analyses to assess the molecular abundance and distributions of organic compound classes in a solid extraterrestrial sample?

## Evaluation of Analytical Methods

**We are evaluating and optimizing four components of soluble organic analysis:**

**1. Extraction**, typically the first step in soluble organic analysis

Test effects of:
- Temperature
- Time of extraction
- Air vs. vacuum
- pH

**2. Hydrolysis**, often used to liberate or generate molecules from precursors

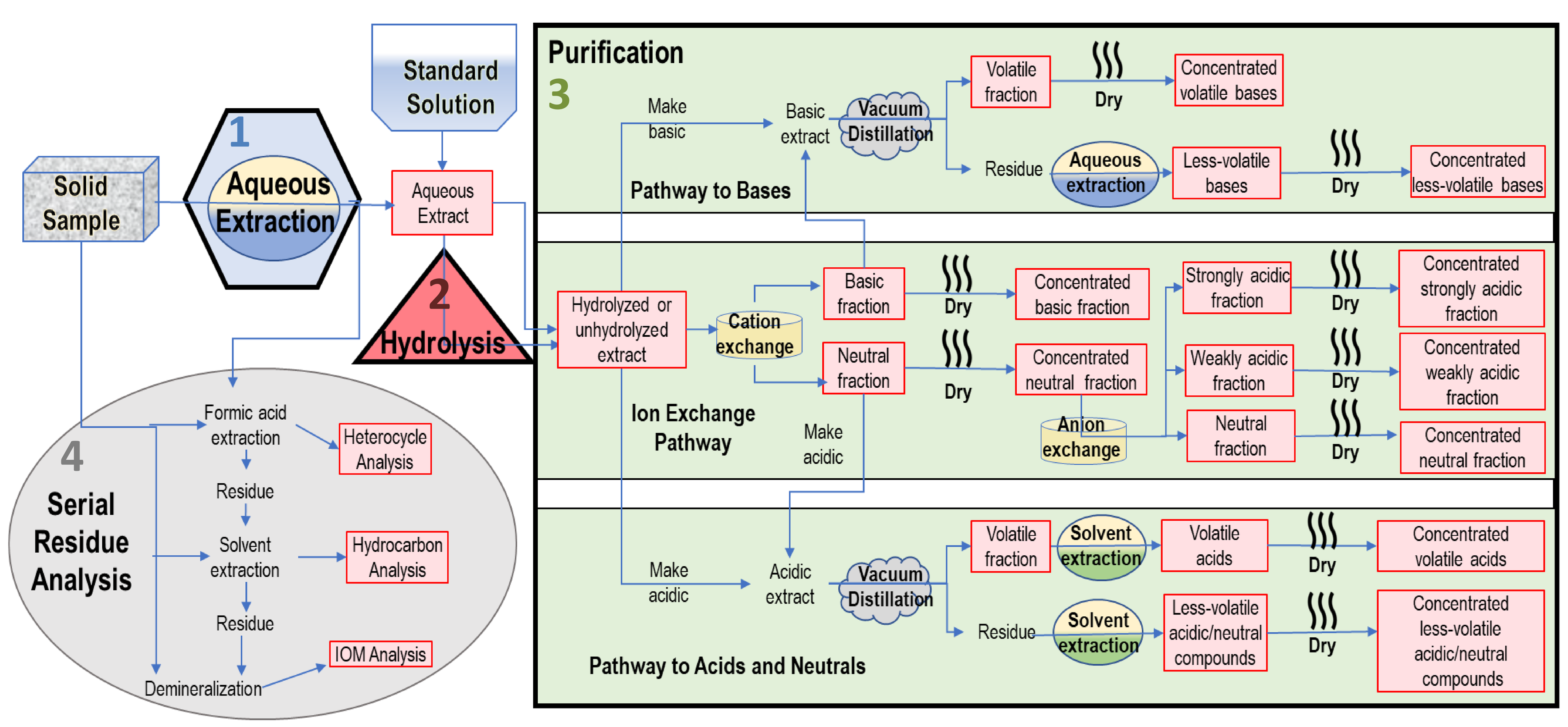
Test effects of:
- Temperature
- Time
- Liquid vs. vapor hydrolysis
- Acid vs. base

**3. Purification**, to separate and prepare individual compound classes for analysis:

Characterize and optimize methods for:
- Cation or anion exchange to separate acidic, neutral, and basic compounds
- Drying techniques for concentration of samples: centrifugal vacuum (Centrivap), freeze-drying, nitrogen blow-down
- Solvent extraction to separate compound classes by solubility
- Vacuum distillation to separate by volatility

**4. Serial Residue Analysis**, for analysis of non-aqueously extractable compounds

Determine efficiency and biases in analyzing acid- or organic-soluble compounds from residues that have previously been aqueously extracted.



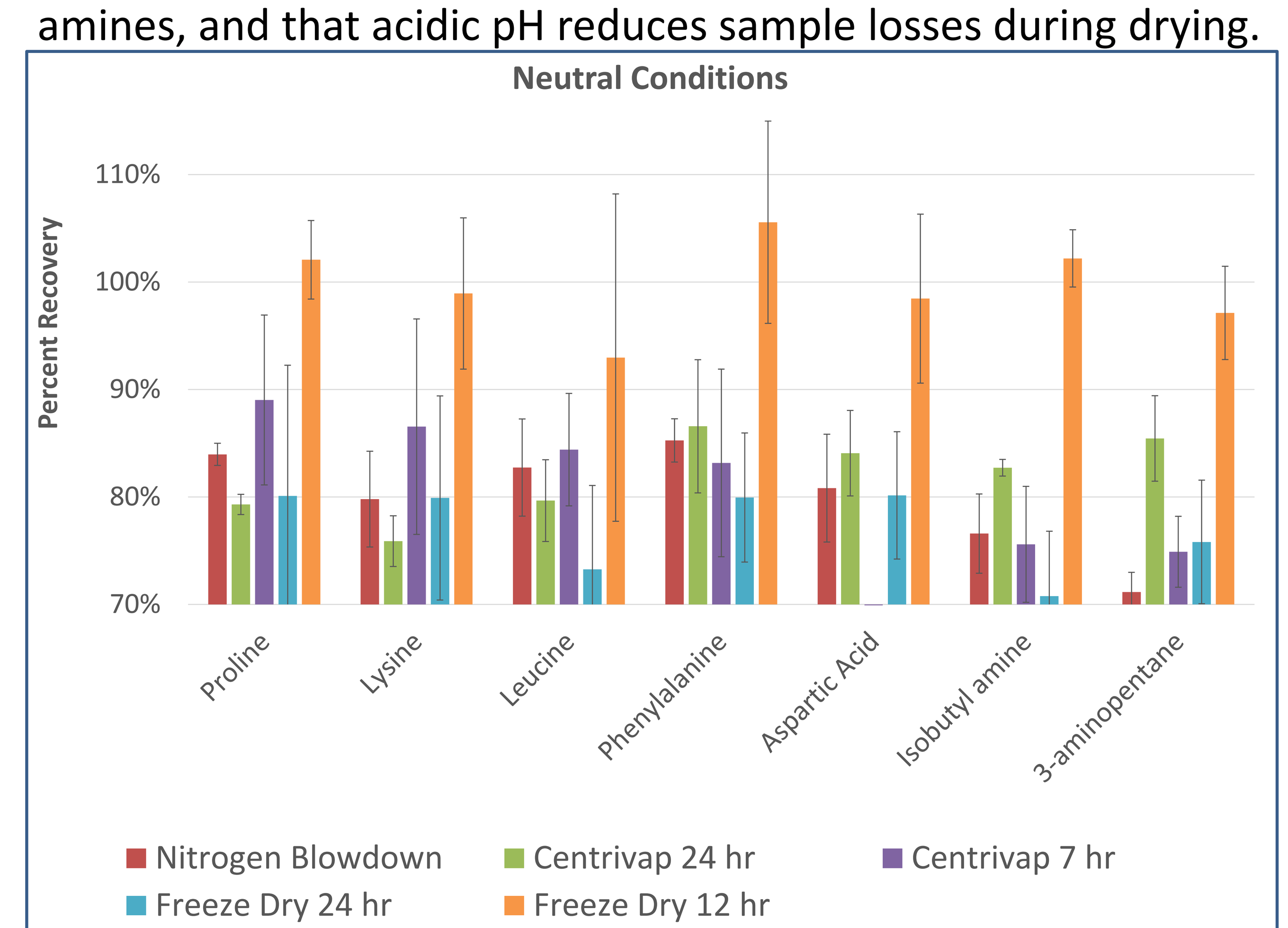

### Testing Plan

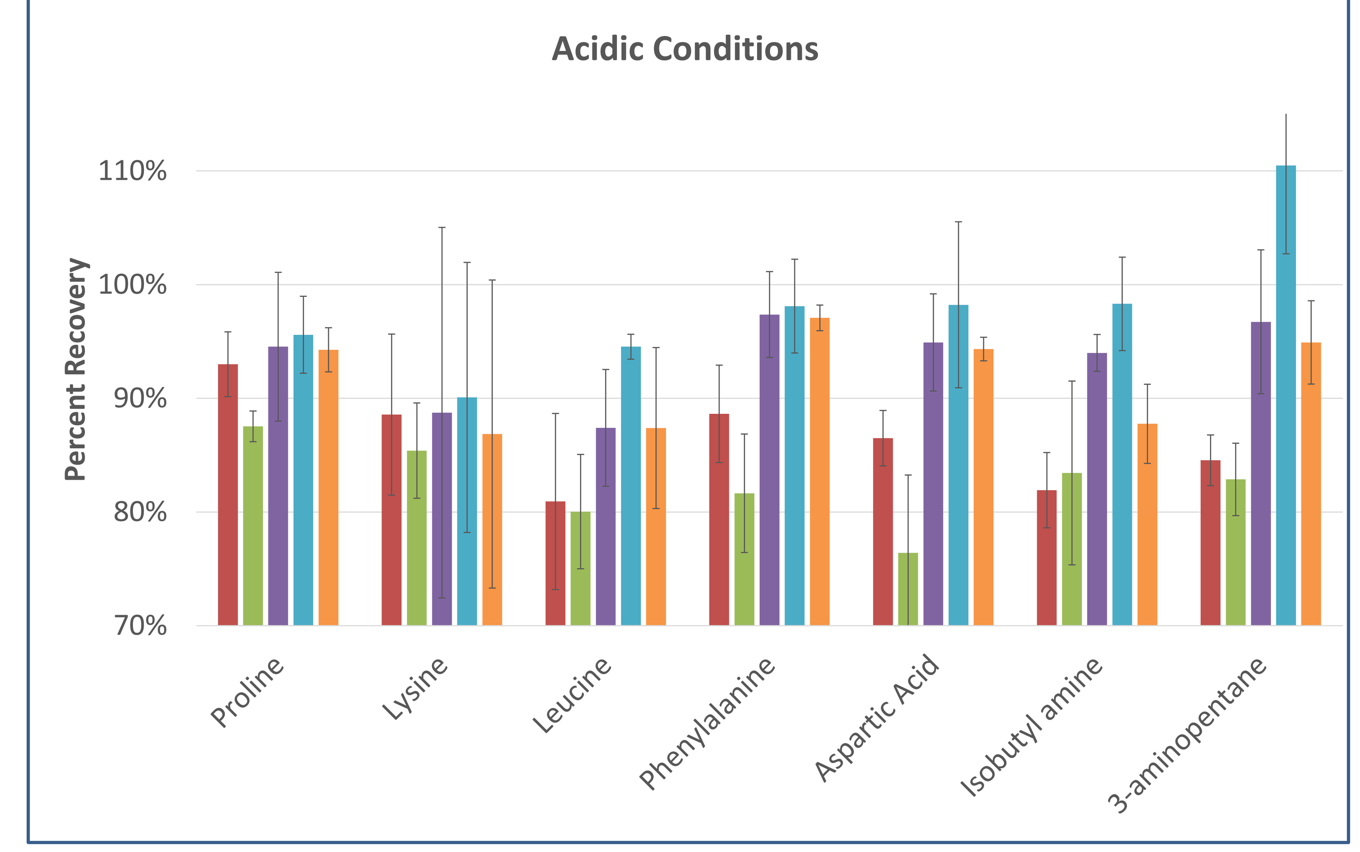
1) Evaluate purification methods using:
   a) A screening mix with 1-2 representatives of each compound class
   b) Compound-class specific mixtures of various volatilities and structures
   c) Standard mix with all compound classes and range of structures
   d) Hot-water extracts of Murchison meteorite
2) Explore hydrolysis efficiency using extracts of the Murchison meteorite and compound-class standard mixes
3) Optimize extraction procedures using homogenized Murchison meteorite powder
4) Test efficiency of analyzing non-aqueous extracts using both fresh and previously extracted samples of Murchison meteorite

## Early Results

Initial efforts to rapidly screen multiple compound classes with liquid chromatography-electrospray ionization mass spectrometry produced inconsistent results caused by ionization effects. We are now using compound-specific analytical techniques to evaluate methods and conditions for sample concentration and drying.

Preliminary results suggest that 12-hour freeze drying is the most effective method for concentrating amino acids and amines, and that acidic pH reduces sample losses during drying.




Near-term work will expand this testing to other compound classes. We will then use the best concentration methods to evaluate cation and anion exchange methods of purification.

## References/ Acknowledgements

[1] Huang Y., Wang Y., Alexandre M. R., Lee T., Rose-Petruck C., Fuller M., and Pizzarello S. (2005) Molecular and compound-specific isotopic characterization of monocarboxylic acids in carbonaceous meteorites. *Geochimica et Cosmochimica Acta* **69**(4), 1073-1084.

[2] Pizzarello S., Huang Y., and Alexandre M. R. (2008) Molecular asymmetry in extraterrestrial chemistry: Insights from a pristine meteorite. *Proceedings of the National Academy of Science* **105**(10), 3700-3704.

[3] Aponte J. C., Woodward H. K., Dworkin J. P., and Elsila J. E. (in preparation) Quantitation and isotopic ratios of monocarboxylic acids in carbonaceous chondrites.

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