

Recent Advances in Characterizing Asian Lacquer

Center for Conservation and Preservation, Yale University, July 15 – 19, 2013

SESSION OUTLINE

SESSION TITLE: THM-Py-GC/MS Analysis of Powdered Lacquer Samples

INSTRUCTOR: Michael Schilling

ABSTRACT

Pyrolysis-gas chromatography/mass spectrometry using tetramethylammonium hydroxide for thermally-assisted hydrolysis and methylation (THM-Py-GC/MS) is presently the best analytical technique for comprehensive characterization of organic materials in Asian lacquers. It is capable of identifying numerous organic materials, resolving mixtures, and offers excellent limits of detection for powdered samples roughly 100 micrograms in size. The daunting challenge of systematically sorting through the vast number of resulting chromatographic peaks in order to accurately locate marker compounds that are characteristic of lacquer raw materials can be greatly aided through the judicious use of GC/MS quantitative analysis software. A marker compound calibration table developed at the GCI, which is an essential element of the data processing, makes it possible to produce a peak area report of nearly 400 marker compounds present in lacquer samples. Used in conjunction with a custom Excel worksheet to semi-automatically analyze and interpret the peak area report, it is possible to identify a wide range of Anacard tree saps, drying oils, fats, carbohydrates, proteins, resins, resinous diluents, preservatives and other materials present in samples from individual lacquer layers.

OBJECTIVES

By the end of this session, participants should be able to:

- Understand the fundamentals of Py-GC/MS.
- Recognize the significance of marker compounds for the identification of complex organic materials.
- Assess the effects of thermally-assisted hydrolysis and methylation on Py-GC/MS marker compounds.
- Prepare and analyze powdered samples from individual lacquer layers by THM-Py-GC/MS analysis.
- Detect and measure marker compounds in THM-Py-GC/MS analysis results for lacquer samples using Agilent's Chemstation software with a custom calibration table.
- Identify artists' organic materials present in lacquer samples based on marker compound distributions in THM-Py-GC/MS analytical results through the use of a custom Excel worksheet.
- Prepare and report their complete analytical findings.



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CONTENT OUTLINE

- Basic principles of Py-GC/MS as a method for analyzing large organic molecules.
- Marker compounds in Py-GC/MS and their role in identifying organic materials.
- On-line chemical derivatization using tetramethylammonium hydroxide (TMAH) as a means of improving Py-GC/MS results.
- Analysis of foundation layer and decorative layer samples from a lacquered object using a Frontier microfurnace pyrolyzer and Agilent GC/MS with TMAH reagent.
- Application of Agilent Chemstation quantitative software for evaluating THM-Py-GC/MS test results for lacquer samples, verifying the marker compounds and tabulating them into a peak area report.
- Identification of artists' materials in the lacquer samples through interpretation of the peak area report using a custom Excel data evaluation worksheet.
- Compilation of Excel results into a presentation and poster.

METHODOLOGY

Monday: The lecture will begin with the basics of Py-GC/MS, focusing on the operation of the Frontier microfurnace pyrolyzer, the formation of small molecules from pyrolysis of organic materials, and eventual separation and identification of the molecules by GC/MS. Particular emphasis will be given to the concept that small molecules uniquely formed by a particular organic material, which are called **marker compounds**, can aid in the identification of unknown materials. The benefits of chemical derivation in THM-Py-GC/MS will be then reviewed, with a discussion of how pyrolysis products are changed by on-line reaction with TMAH. The lecture will conclude with a review of specific marker compounds for Anacard tree saps, based upon research from the GCI and other laboratories. Videos will highlight important topics that will be covered in the laboratory sessions.

Tuesday and Wednesday: These laboratory sessions, held in the Organic Materials Laboratory of GCI Science, will consist of THM-Py-GC/MS analysis of the powdered foundation and decorative layer samples that were prepared in the 'layer unpacking' session. While the groups wait for the analysis of their layer samples to finish, they will become familiar with the software and procedures for interpretation that will aid teams in interpreting data and preparing final reports. Training sessions will introduce participants to the Agilent GC/MS Chemstation data evaluation software, the quantitative analysis program and the GCI custom Excel worksheet that will be used during the data processing and interpretation sessions on Thursday.

Thursday: In this laboratory session, held at the Getty's Information Technology computer training laboratory, the groups will analyze the THM-Py-GC/MS analytical results from their foundation and decorative lacquer layers using the concepts developed during the previous laboratory sessions. Each group will analyze their two THM-Py-GC/MS chromatograms, detect and identify marker compounds using the Chemstation software, and prepare peak area reports. After copying the Chemstation reports into the custom Excel worksheet and verifying the marker compounds, the artists' materials present in the two samples will be identified on the basis of the Excel tables and graphs. The session



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will conclude with the groups preparing presentations of their results using the Power Point report template provided by the Getty, and then transferring the completed slides to a poster template for subsequent printing.

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 = Essential reading material

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