

Analytical Protocol for Detecting Organic Materials

Samples were analyzed by Py-GC/MS with derivatization of the lacquer samples with tetramethylammonium hydroxide (TMAH) prior to analysis. The rationale for this approach is to convert carboxylic acids, alcohols and phenols to more volatile products prior to analysis. A Frontier Lab PY-2020D double-shot pyrolyzer system equipped was used for pyrolysis. The pyrolysis interface was maintained at 320°C. The pyrolyzer was interfaced to an Agilent Technologies 5975C inert MSD/7890A gas chromatograph/mass spectrometer. A Frontier Ultra ALLOY-5 capillary column was used for the separation (30 M x 0.25 mm x 0.25 µm), with helium carrier gas set to 1 ml/minute. The split injector was set to 320°C with a split ratio of 50:1 and no solvent delay. The GC oven temperature program was 40°C for 2 minutes, then ramped to 320°C at 20°C /minute, followed by a 9 minute isothermal period. The MS transfer line was at 320°C, the source at 230°C, and the MS quad at 150°C. The mass spectrometer was scanned from 33-600 amu at a rate of 2.59 scans per second. The electron multiplier was set to the autotune value. Samples were placed into a 50 µl stainless steel Eco-cup fitted with an Eco-stick, and three microliters of a 25% methanolic solution of TMAH were introduced for derivatization. After three minutes, the cup was placed into the pyrolysis interface where it was purged with helium for three minutes. Samples were pyrolyzed using a single-shot method at 550°C for 6 seconds.

The resulting total ion chromatograms and associated mass spectra are interpreted using a reference table of characteristic marker compounds for a very large selection of raw materials associated with the production of both Asian and European lacquers.

The table, now comprising nearly 300 compounds, was compiled from published and unpublished data as well as in-house analytical results. This reference table is available to interested researchers upon request by contacting Michael Schilling (mschilling@getty.edu).

Protocol developed for Characterization of Asian and European Lacquers, a project of the Getty Conservation Institute, Los Angeles.

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