Analytical Testing of Pharmaceuticals in Cultural Heritage Collections

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Collections Care for Historical Pharmaceutical Collections Resource Package

February 2024

Over time, products such as pharmaceuticals, pesticides, and scientific chemicals accessioned into cultural heritage collections will degrade – regardless of applied storage techniques. Sometimes these chemicals will reduce in hazard severity, whereas other times their hazard severity will increase. There is no definitive way to confirm this without analytical testing, therefore it is best to practice extreme caution and use appropriate personal protective equipment while handling them. Sometimes the contents of the containers are missing labels, and it is imperative for museums and cultural heritage organizations to determine the contents inside for the purposes of research or exhibitions. External labs specialized in Conservation Science are ideal to assist with analysis, however, there are very few of them. Museum specialists can instead contact external labs specialized in chemical analysis through use of wet chemistry and scientific instruments to perform this analysis.

Questions to ask external labs:

How large does the sample need to be for analysis?

Are there special requirements for the swabs and jars used for sample retrieval? Please provide a link to the preferred container or swab to be used.

What method of sample removal is required?

How many samples are needed?

Is the analysis destructive or non-destructive?

Can the lab come to retrieve the samples themselves? What is the added cost for this service?

How fast is the turnaround time for the analysis?

What information will be provided? Will it include the chemicals identified as well as the concentration of them?

Further Considerations:

Do you need the external lab to sign a non-disclosure agreement prior to analysis?

Certain chemicals are illegal to transport or ship and cannot be sent to a lab for analysis. E.g. DDT

How to find an external lab:

The simplest way to locate an external lab which can perform the appropriate type of analysis is to search by type of instrumental analysis required.

Spectroscopy- Raman and Infrared

The two most common techniques for the identification of organic materials are Fourier Transform Infrared Spectroscopy (FTIR) and Raman Spectroscopy. In FTIR the characteristic absorbances of chemical bonds in the mid-infrared are used to identify molecules. FTIR can be a transmission or surface technique. In Raman a monochromatic laser is directed onto the surface of a sample and a small fraction of the light undergoes inelastic scattering, or Raman scattering. This occurs when the energy of the incident photons is altered by the vibrational energy levels of the molecules in the sample. These vibrations produce a characteristic spectrum allowing materials to be identified, similar to how absorbances are used in FTIR.

Spectroscopy is a valuable analytical technique allowing many materials to be identified quickly, non-destructively and without any prior knowledge of the material allowing fully unknown samples to be examined, but it also has many limitations:

Fluorescence Interference (Raman): Fluorescence from the sample or impurities can interfere with the Raman signal, particularly when using near-infrared excitation wavelengths. This interference can obscure the Raman spectra and affect the accuracy of analysis. Many binders and fillers will fluoresce.

Low Sensitivity for Dilute Samples: low sensitive for dilute samples (<5% of the mass of the sample). This can limit its applicability for trace analysis or samples with low concentrations of analytes. Many drugs have a very low concentration of the active ingredient.

Water Interference: both techniques are sensitive to the presence of water, and water bands can overlap with characteristic peaks for the identification of pharmaceuticals. This can complicate analysis and interpretation for pharmaceutical formulations containing water.

Limited Penetration Depth: Raman spectroscopy and FTIR have a limited penetration depth, typically within a few micrometers into the surface of the sample, so sample shape can have a large effect.

Gas Chromatography Mass Spectrometry (GC-MS)

Gas chromatography (GC) separates compounds based on their differential partitioning between a stationary phase, the column, and an inert gas mobile phase. The sample is vaporized and injected into the GC system all the components are physically separated based on their affinity for the column. Detection is achieved using mass spectrometer (MS), which allows identification of each component as it comes out of the column and are broken into molecular fragments of characteristic weights. This allows complex

mixtures to be identified but is limited as it is a destructive technique (though the sample size can be small on certain instruments) and requires the sample to be vaporized, so large molecules or highly temperature sensitive materials can be difficult to analyze.

Ion Chromatography (IC)

Ion chromatography separates ions based on their interactions with a stationary phase containing charged functional groups. Many pharmaceutical compounds exist as salts, where the active drug molecule is combined with a counterion. IC can separate and quantify these counterions, providing information about the drug formulation. The sample is dissolved in water and injected into a column, where ions interact with the stationary phase and are eluted based on their affinity for the column material. The eluent, typically a buffered aqueous solution, carries the ions through the column and is detected typically using a conductivity detector though more flexible techniques such as mass spectrometry or spectroscopy are also possible. IC is limited due to requiring a large sample size and the ability to dissolve the sample, making its use very limited to identify unknown samples.

This document was written as part of the Collections Care for Historical Pharmaceutical Collections Resource Package created and hosted by the Museum of Health Care at Kingston and

Funded by the Government of Canada | Canada