Chemsultants International

Specialized Analytical Testing Services

Analytical testing methods are used to identify an adhesive or its components, confirm the content of adhesives, and analyze key properties of the adhesive.

Chemsultants International Inc. • 9079 Tyler Blvd, Mentor, OH 44060 • (440) 974-3080 • chemsultants.com

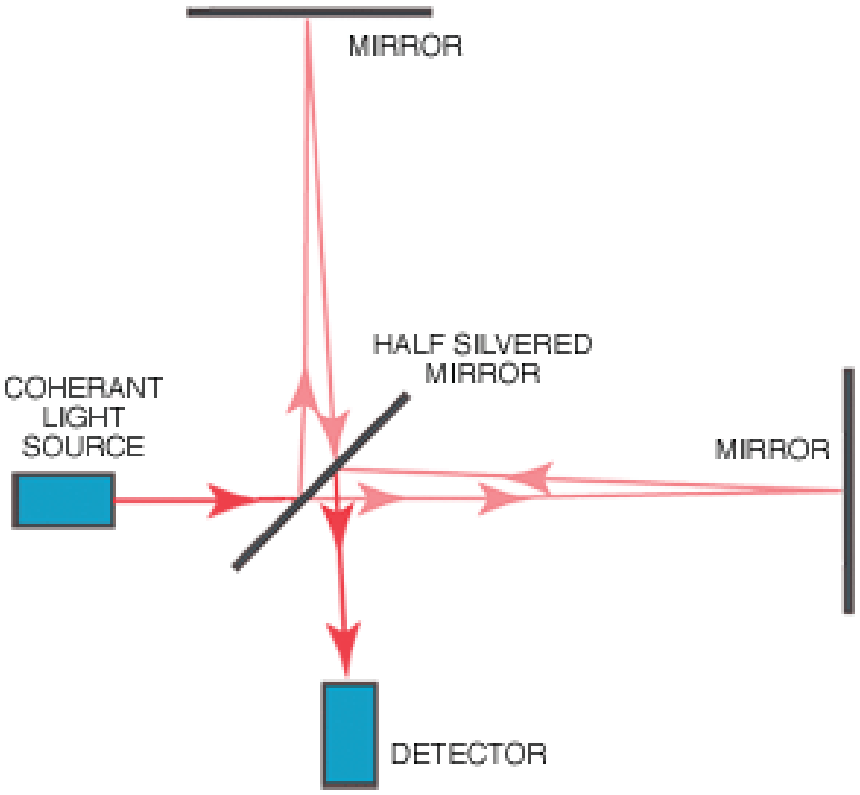
Specialized Analytical Testing Services

Analytical testing methods are used to identify an adhesive or its components, confirm the content of adhesives, and analyze key properties of the adhesive. They can also be used to identify contaminants, and to develop raw material substitutes for adhesives, coatings and sealants. There are a number of useful specific analytical test methods including:

1. FTIR

Continuous wave Michelson or Fourier transform spectroscopy

The Fourier transform spectrometer is just a Michelson Interferometer but one of the two fully-reflecting mirrors is movable, allowing a variable delay (in the travel-time of the light) to be included in one of the beams.The Michelson spectrograph relies on the same principle as the Michelson-Morley experiment. Light from the source is split into two beams by a half-silvered mirror, one is reflected off a fixed mirror and one off a moving mirror which introduces a time delay -- the Fourier transform spectrometer is just a Michelson interferometer with a movable mirror. The beams interfere, allowing the temporal coherence of the light to be measured at each different time delay setting. By making measurements of the signal at many discrete positions of the moving mirror, the spectrum can be reconstructed using a Fourier transform of the temporal coherence of the light.



Pulsed Fourier transform spectrometer

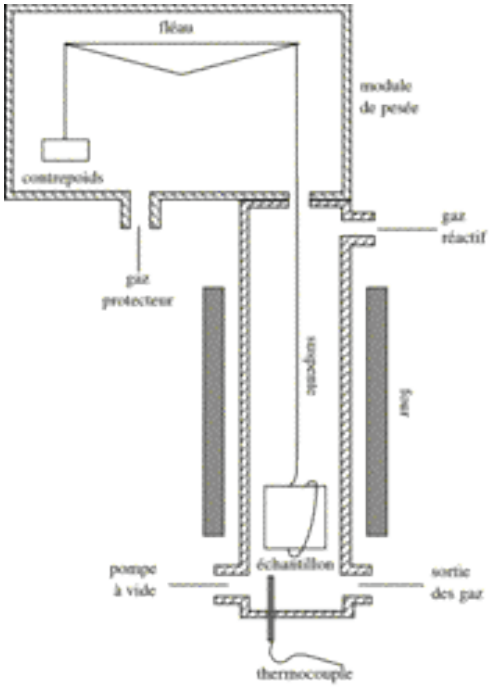
A pulsed Fourier transform spectrometer is usually used to measure the spectrum of the light transmitted through a laboratory sample. In a conventional (or “continuous wave”)

spectrometer, a sample is exposed to electromagnetic radiation and the response (usually the intensity of transmitted radiation) is monitored. The energy of the radiation is varied over the desired range and the response is plotted as a function of radiation energy (or frequency). At certain resonant frequencies characteristic of the specific sample, the radiation will be absorbed resulting in a series of peaks in the spectrum, which can then be used to identify the sample. (In magnetic spectroscopy, the magnetic field is often varied instead of the frequency of the incident radiation, though the spectra are effectively the same as if the field had been kept constant and the frequency varied. This is largely a question of experimental convenience.)

Instead of varying the energy of the electromagnetic radiation, Fourier Transform spectroscopy exposes the sample to a single pulse of radiation and measures the response. The resulting signal, called a free induction decay, is a direct measurement of the temporal coherence of the light and contains a rapidly decaying composite of all possible frequencies. Using a Fourier transform of this, the spectrum of the light can be calculated as for the Michelson Fourier transform spectrometer. In this way the Fourier transform spectrometer can produce the same kind of spectrum as a conventional spectrometer, but in a much shorter time.

2. TGA (Thermal Gravimetric Analysis)

Thermo gravimetric Analysis or TGA is a type of testing that is performed on samples to determine changes in weight in relation to change in temperature. Such analysis relies on a high degree of precision in three measurements: weight, temperature, and temperature change. As many weight loss curves look similar, the weight loss curve may require transformation before results may be interpreted. A derivative weight loss curve can be used to tell the point at which weight loss is most apparent. Again, interpretation is limited without further modifications and deconvolution of the overlapping peaks may be required. TGA is commonly employed in research and testing to determine characteristics of materials such as polymers, to determine degradation temperatures, absorbed moisture content of materials, the level of inorganic and organic components in materials, decomposition points of explosives, and solvent residues. It is also often used to estimate the corrosion kinetics in high temperature oxidation.



3. NMR (Nuclear Magnetic Resonance)

NMR spectroscopy is one of the principal techniques used to obtain physical, chemical, electronic and structural information about molecules. It is a powerful technique that can provide detailed information on the topology, dynamics and three-dimensional structure of molecules in solution and the solid state. Also, nuclear magnetic resonance is one of the techniques that has been used to build elementary quantum computers.

By studying the peaks of nuclear magnetic resonance spectra, skilled chemists can determine the structure of many compounds. It can be a very selective technique, distinguishing among many atoms within a molecule or collection of molecules of the same type but which differ only in terms of their local chemical environment.

By studying T2* information a chemist can determine the identity of a compound by comparing the observed nuclear precession frequencies to known frequencies. Further structural data can be elucidated by observing spin-spin coupling, a process by which the precession frequency of a nucleus can be influenced by the magnetization transfer from nearby nuclei. T2 information can give information about dynamics and molecular motion.

Because the nuclear magnetic resonance timescale is rather slow, compared to other spectroscopic methods, changing the temperature of a T2* experiment can also give information about fast reactions, such as the Cope rearrangement or about structural dynamics, such as ring-flipping in cyclohexane.

A relatively recent example of nuclear magnetic resonance being used in the determination of a structure is that of buckminsterfullerene. This now famous form of carbon has 60 carbon atoms forming a sphere. The carbon atoms are all in identical environments and so should see the same internal H field. Unfortunately, buckminsterfullerene contains no hydrogen and so C13 nuclear magnetic resonance has to be used. C13 spectra are more difficult to obtain because carbon-13 is not the common isotope of carbon (unlike hydrogen, where H1 is the common isotope). However, in 1990 the spectrum was obtained by R. Taylor and co-workers at the University of Sussex and was found to contain a single peak, confirming the unusual structure of C60.

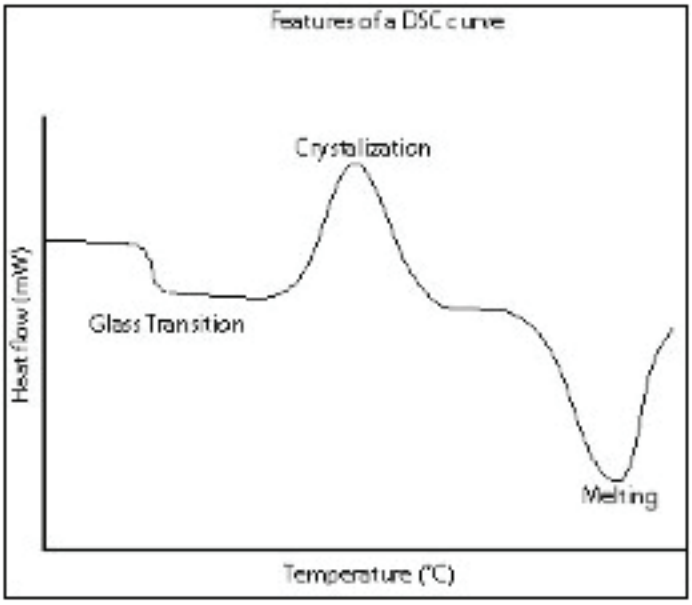
4. DSC (Differential Scanning Calorimetry)

Differential scanning calorimetry or DSC is a thermoanalytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference are Measured as a function of temperature, both the sample and reference are maintained at very nearly the same temperature throughout the experiment. Generally, the temperature program for a DSC analysis is designed such that the sample holder temperature increases linearly as a function of time.
The reference sample should have a well-defined heat capacity over the range of temperatures to be scanned.

Principle underlying this technique is that, when the sample undergoes a physical transformation such as phase transitions, more (or less) heat will need to flow to it than the reference to maintain both at the same temperature.

Whether more or less heat must flow to the sample depends on whether the process is exothermic or endothermic. For example, as a solid sample melts to a liquid it will require more heat flowing to the sample to increase its temperature at the same rate as the reference. This is due to the absorption of heat by the sample as it undergoes the endothermic phase transition from solid to liquid. Likewise, as the sample undergoes exothermic processes (such as crystallization) less heat is required to raise the sample temperature. By observing the difference in heat flow between the sample and reference, differential scanning calorimeters are able to measure the amount of heat absorbed or released during such transitions. DSC may also be used to observe more subtle phase changes, such as glass transitions.

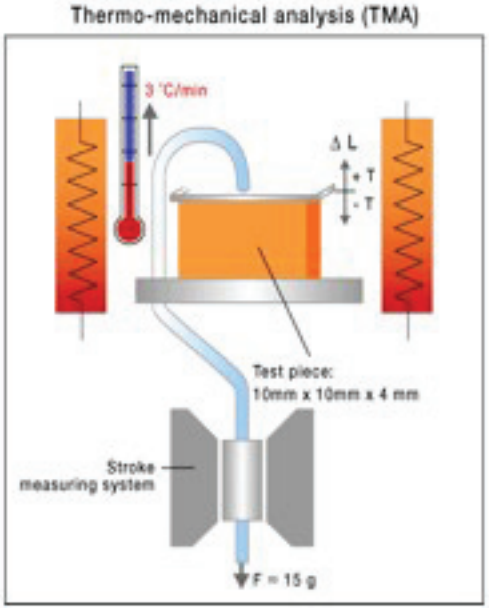
DSC is widely used in industrial settings as a quality control instrument due to its applicability in evaluating sample purity and for studying polymer curing. DSC is widely used in the pharmaceutical and polymer industries. For the polymer chemist, DSC is a handy tool for studying curing processes, which allows the fine tuning of polymer properties. The cross-linking of polymer molecules that occurs in the curing process is exothermic, resulting in a positive peak in the DSC curve that usually appears soon after the glass transition.



5. TMA (Thermal Mechanical Analysis)

Thermo-mechanical analysis (TMA) is used to determine the deformation of a sample (changes in length or thickness) as a function of temperature. The measuring range may extend from -150 °C to +600 °C.

1 Tension/strain in films
2 Coefficient of linear expansion
3 Penetration measurements



For more information or assistance with your analytical testing requirements please contact our Laboratory Services Group at:

Email: info@chemsultants.com
Telephone: 440.974.3080 / FAX: 440.974.3081
Click here to Request a Quote on Testing and Product Development