GCM Lab

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THE CSI EFFECT

The American television show CSI (Crime Scene Investigation) has not only had a lot of success around the world but it has also greatly increased the interest for high-tech scientific methods. In the mass media, the term "CSI effect" has mainly been used to illustrate the influence that the show has had on the judicial system (for example jurors asking why the police did not dust off the lawn for fingerprints – something that was never presented on TV in the first place). However, series based on forensic science have also had the positive effect of showing the power of the scientific method since, in reality, strict procedures and a combination of different analytical techniques must be followed to obtain reliable results.

At the GCM, similar scientific procedures are used to solve other types of problems: the presence of contaminants, a manufacturing defect, a handling error, etc… In both cases however, it is critical to benefit from the most up to date instruments and highly qualified personnel able to extract as much information as possible from the results.

To illustrate this point, a quick example[1]:

The crime: an inefficient industrial process

The location: a large plant

The Exhibit: A powdery industrial residue of unknown composition but that probably contains organic and inorganic phases.

The objective: Find the chemical composition of the powder to improve the process.

The procedure: Use different analytical techniques to obtain complementary information.

The detail of the different instruments used:

X-RAY DIFFRACTION (XRD)

One of the most well-known and established technique. It allows the identification of crystalline phases (generally but not exclusively composed of inorganic compounds) in a quick and effective way. Since precise phase are detected (ex : CaSO4), a lot of useful information is obtained about the chemistry of the sample. However, this method is at best semi-quantitative (5% detection limit) and amorphous phases are invisible. Furthermore, if the sample is very complex (numerous phases) it will often be difficult to differentiate the numerous diffraction peaks and to indentify one compound in particular.

In the case of the mystery sample, three inorganic phases were detected. This was obviously and interesting breakthrough but, as a fraction of the sample appeared to be organic, this single measurement was not sufficient.

FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

This method allows for the identification of chemical bonds present in molecules (mainly organics). The spectrums can then be analysed using databases to indentify the compounds (or family of compounds) present in the samples. The analysis is fast and relatively inexpensive but generally qualitative only and difficult to analyse if complex mixtures are present. It is thus possible to compare this method with X-ray diffraction (that gives similar information but mainly for inorganic samples).

The analysis of the industrial powder showed the presence of different families of aromatic hydrocarbons, confirming the significant presence of organic material. However, the quantitative composition of the sample was still unknown.

[1] Any resemblance with past or present persons or situations is purely coincidental.

X-RAY PHOTOELECTRON SPECTROSCOPY (XPS)

This technique, probably one of the most versatile, allows for the determination of two types of data: the elemental composition (up to 0.1 atomic %) and the nature of the chemical bonds present. Using this information, it is generally possible to quantitatively indentify the molecules present in the sample, a feat that very few techniques can achieve. However, the depth of analysis is quite limited (around 10 nanometers) and this can cause problems if the surface is heavily contaminated. Also, volatile samples are difficult (if not impossible) to analyses since the analysis is performed in a vacuum.

The analysis of the mysterious powder helped confirm the results obtained previously, to discover the presence of another inorganic phase (that had not been identified by XRD) and to obtain the quantitative chemical composition of the sample. However, a long degassing time was required before the measurement because of the volatile nature of the organic compounds present.

SCANNING ELECTRON MICROSCOPY (SEM)

If is often very useful to image samples under high magnification or to determine if they are chemically homogeneous. Electron microscopy is particularly appropriate for this task since magnifications up to 500 000x are possible and the use of backscattered electrons allows for the formation of images where the contrast indicates differences in chemical composition. It is also possible, using an x-ray detector, to determine (qualitatively or semi-quantitatively) the elemental composition of very small areas. However, as in the case of XPS, the analysis is performed under a high vacuum, causing problems with volatile samples.

The observation of the industrial powder gave the opportunity to study the morphology of the particles and to see its heterogeneity. Using the EDS detector, it was found possible to obtain the chemical composition of most particles and thus identify them.

As the previous descriptions demonstrate, it is often very useful to combine different analysis technique to obtain comprehensive results at a reasonable cost. For example, if little information is available about a sample, it is generally advantageous to start with relatively simple and inexpensive techniques such as XRD and FTIR and use more advanced instruments if needed. Furthermore, it is important to note that good results analysis is also extremely important since it often makes the difference between a useless hodgepodge of graphs and a wealth of useful information. Also, the expertise offered by the GCM can often lead to the root cause of the problem, thus helping identify the culprit!

Furthermore, as the previous sample showed, it is unfortunately impossible to answer all the questions, starting from almost nothing, very rapidly and at a negligible cost (as opposed to what CSI-type shows lead to believe). In reality, the universal instrument that can give all the answers for all types of instruments has not yet been invented. We are working on it day and night though!

To conclude, to obtain the best service possible, it is useful to:

- Obtain as much information as possible about the samples and share it with the wonderful GCM personnel.
- Have a good idea of the results you are after (elemental composition, nature of the compounds, concentration, morphology, etc.)

To find out what GCM special agents at can do for you, we invite you to contact:

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