Characterising Unknowns: Behind the Scenes of Chemical Investigation

Paul Walker, Socotec. Tel: 0845 603 2112, Email: salesuk@socotec.com

Unknown materials and substances found on-site, in products, or during processing can be a major cause for concern. Being able to effectively and precisely identify them enables public and private sector organisations such as corporations, police, hospitals and councils to take action to prevent risk. Paul Walker, senior development specialist at Socotec, takes us through the processes of a chemical investigation, to show how we move from the unknown to the known.

Unexpected and unidentified substances such as solids, solvents, deposits, oils and dusts can show up in any environment, from large industrial factories to building sites, crime scenes to hospital wards, pharmaceutical clean rooms to everyday households. Probably the most frequently received at the laboratory are unknown dusts, as they clearly can pose an immediate risk to staff health and safety. Other types of deposit often only come to light when they begin to cause an issue and can include: sticky residues causing issues with machinery, solids blocking fuel injector nozzles, unknown particles in vaccines and foods, foreign objects on contact lenses, contamination in packaging, premature blocking of fuel and air filters, and staining on buildings. All these events can have an impact on the SHEQ goals of any organisation or body.

Examples of events impacting SHEQ:
Safety – greasy deposit on brake pads of vehicle, compromising braking efficiency
Health – unknown dust contaminating a work area or house
Environment – suspect fly-tipped material poisoning the environment
Quality – unknown particle in a vaccine

Chemical investigation can be quite a varied job, with a wide diversity of contaminated articles being sent for investigation, such as:

- pieces of plastic/rubber from the breakdown of the conveyer belts found in food
- airborne fibres found floating in medical vaccines
- deposits on medical instruments such as bone fragments and rusting
- deposits affecting delivery of materials via medical needles, catheters etc
- barrels of unknown solids and liquids from fly-tipping or landfill

With this variety of source material, scientists need process to bring clarity. Chemical investigations utilise rapid identification methods which give peace of mind that contaminants will be classified swiftly, reducing process disruption and providing key information for failure investigations, manufacturing issues and health and safety concerns. A mis-identification must be avoided at all costs as it could send a failure investigation down the wrong route and reach the wrong conclusion, and ultimately the wrong solution.

Wherever possible, the article that has been contaminated should be sent in for investigation intact. This will allow the laboratory to take direct samples, and also see how the material lies on the sample. For substances found on surfaces, this could be an important step in determining what the unknown material is and how it has been deposited; for example, if the deposit is in a watermark pattern then it shows the deposit has been formed from the evaporation of liquids on the surface. If the original article itself cannot be sent in then often a sample is placed into a glass container, between glass microscope slides or, failing that, then swabs can be taken. Swabs, however, can limit the type of analysis that can be done as background contamination from the swabs themselves may occur.

Potential contamination, of the investigator or of the article itself, is a real issue and needs to be overcome when working with minute contamination. Work should be carried out in a clean room, preferably with a positive pressure environment to stop dust and fibres from contaminating the surrounding area. In addition to controlling contamination from the laboratory, other basic principles that need to be followed during a chemical investigation include good note keeping and photographic documentation of any physical characteristics identified through the process.

Every investigation has to be bespoke to the sample. Before any investigation is started, the most important thing is to find out as much about the sample as possible such as: where it was found, what work is carried out around the site, how much of the sample is present, is it a recent development or has it been developing over time? Detailed discussions with the client take place to understand the processes that are employed in the area and so potential sources can be identified and taken into account when designing the analytical experiments.

When the sample arrives in the laboratory, the first thing that always happens is a physical examination. This examination will give the first impression of the sample and what analysis should potentially be started with, as well as what analysis is available due to sample size. Extremely small samples might limit the type of analysis that can be performed due to sensitivity of the laboratory instrumentation. Common analysis carried out include Fourier Transform Infrared Spectroscopy (FTIR), Gas Chromatography Mass Spectrometry (GC/MS), Scanning Electron Microscope using Energy Dispersive X-ray analysis (SEM-EDX), Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and Ion Chromatography (IC). The types of laboratory analysis performed on each sample is determined by the expert scientists working on the case.

Once all this information is collated, we can start to make hypotheses about what might have happened and how the unknown substance could have come to occur. It is important that assumptions do not start to take root, as these can influence the interpretations rather than simply drawing on the analytical results.

Each stage of the investigation tells us a bit more about the sample such as GC/MS gives the volatile/semi-volatiles organics, the SEM-EDX giving the elemental composition, the IC analysis giving the anions and so on. It is very rare that just one analytical technique will allow you to confidently state what the sample is. Instead, each stage builds on the one before to form a picture of what the sample is. We are also looking to see if the different techniques confirm what each one is telling us or question what the initial results were, and it usually takes several days to form a complete picture.

Case Study: Theory in Practice

It's amazing what can be found on the UK's railways and when there are issues, they are not always easy to identify.

Our chemical analysis laboratory received an engine turbo air filter which had become mysteriously blocked by an unknown deposit. The premature blocking of the filter had led to damage of the engine turbo on class 172 vehicles due to reduced airflow through the turbo, which in turn, resulted in a loss of power to the engine.

Our inquisitive specialist chemistry team set to work using the following steps to uncover the mystery.

First impressions count: visual examination

Our senior chemical analysis team has a vast amount of experience in the characterisation of unknown materials so, more often than not, they can usually make an educated guess by examining the substance by look, feel and smell. At the very least, this examination allows us to decide on the most suitable suite of tests to be carried out and take photographs to record the appearance of the sample in the client report.

It was clear from visual examination that the deposit had hardened itself to the outside of the filter and included debris such as bird feathers and leaves. This showed that there was certainly an amount of material that had originated from the outside environment.

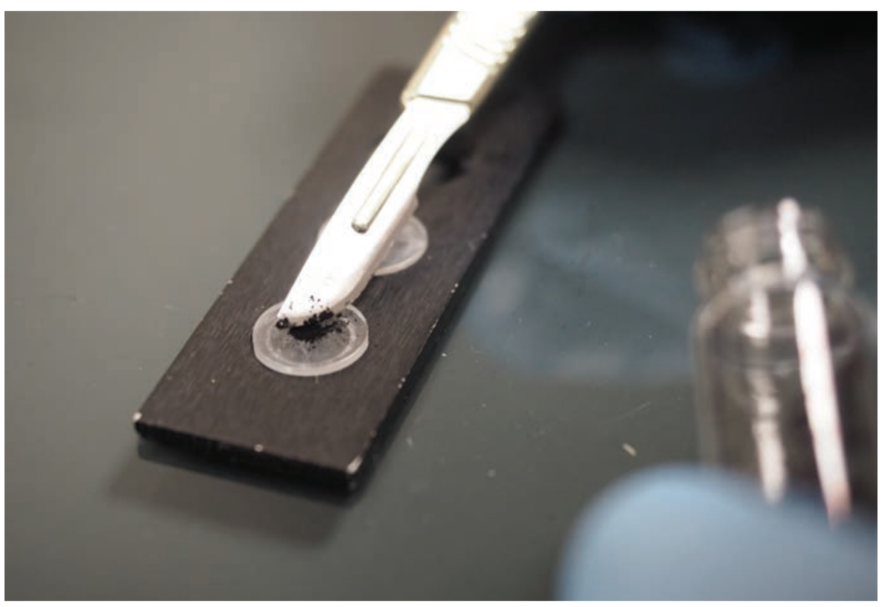
When touched, the substance left a sparkling residue on our gloves indicating the presence of a fine metallic powder.

Taking a closer look: optical microscopy

The next step was to carry out optical microscopy in order to take a closer look into the debris. Under the microscope, along with the leaves, feathers and metallic dust we found a matting of fine fibres along with seed pods.

On the right wavelength: Fourier Transform Infrared Spectroscopy (FTIR)

We then moved on to analyse the sample by Fourier Transform Infrared Spectroscopy (FTIR) in order to identify the presence of materials such as polymers, adhesives, fabrics or inorganic salts. The FTIR produces an infrared spectrum by measuring how well the material absorbs the infrared beam of light at different wavelengths. This analysis identified the matted material as being a collection of cellulose fibres.



Establishing the pattern: Gas Chromatography Mass Spectrometry (GC/MS)

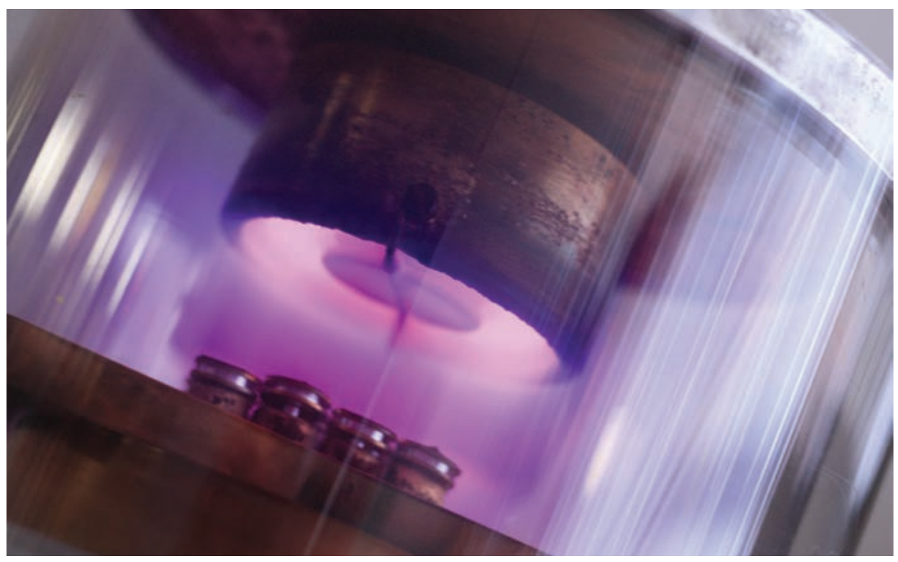
To check for the presence of trace organics, we used Gas Chromatography Mass Spectrometry (GC/MS). This technique will separate out the organic species and detect the fragments from the ionisation of the molecules. These fragments form a unique pattern which is used to identify the organic compound.

We extracted a portion of the debris in dichloromethane and analysed it by GC/MS. No evidence of any oils or fuels was detected. A negative result can be just as important as a positive one, in this case proving the deposit was not being caused by leaking engine oils or fuels. The leaking of oils can often cause an absorbent surface for solid debris to stick to. When the debris was in the solvent, the fine metallic particles sank to the bottom and were able to be isolated for further examination.

Elemental my dear Watson: Scanning Electron Microscopy (SEM) Analysis

Scanning Electron Microscopy using Energy Dispersive X-ray analysis (SEM-EDX) determines the elemental composition of the unknown material. An X-ray beam is fired onto the surface of the material which excites the atoms. This change in energy state produces X-rays which are measured by the spectrometer, allowing us to identify the elements present.

After analysis, the fine metallic powder recovered from the filter was found to be iron, along with smaller amounts of silicon.



Case closed: technical report

Following the completion of an unknown material analysis, the allocated senior scientist provides a full technical report on their findings. Each characterisation is treated uniquely, as one set of analysis is not always applicable to every type of sample.

In this case, we found that the debris present on the exterior of the air filter was fibrous and made up of a combination of natural airborne materials such as cellulose (from plants), feathers, seeds and leaves. Adhered to this was a brown/black coloured iron powder, probably originating from wheel, rail or brake wear debris. The silicon we found could have originated from ballast dust and general siliceous material from the environment. The build-up of this material would certainly have led to a reduction in airflow through the filter, in turn reducing the power of the turbo.

The solution? Our client was advised to look at redesigning the filter housing in order to reduce exposure to the external airborne contamination or to implement a change in frequency of the filter replacement.