Manufacturers are constantly developing their GC’s and their equipment, such as detectors, to get lower detection limits in order to trace the slightest amounts of target compounds.

For decades and through the whole laboratory industry, chromatography has been a widely used separation technique for analytical and preparation purposes. Chromatography refers to the words chromos (Latin for colour) and graphic (visual registration). Lines or dots of, for example, a green or orange marker on a wet sheet of paper, divided into their primary colours yellow/blue and yellow/red. This is the basic process that takes place during chromatography.

There are several chromatographic techniques like Gas Chromatography (GC), High Performance Liquid Chromatography (HPLC), Thin Layer Chromatography (TLC), Gel Permeation Chromatography (GPC) or Chemical Electrophoresis (CE).

This article will focus on GC and GCMS (GC combined with a Mass Selective detector) analysis in the environmental industry.

Instruments and usage for environmental analysis

GC is a common analysing technique to separate organic compounds that are volatile, without decomposing, out of a complex mixture from other substances like lipids, acids, minerals and proteins. To achieve reliable results of the amount of target compound(s), a GC contains various basic elements.

In the beginning, samples were injected manually into an injector of a GC. To manage larger amounts of samples, to increase accuracy and efficiency and to automate pre-treatment handling, manufacturers developed different kinds of robotic samplers called Auto Samplers.

For the separation column in a GC, there can be two kinds available, capillary (used for sample solutions or extracts) or packed (used for gases). A column is packed or coated with a stationary phase. In this phase the separation takes place by difference in affection of the target compound(s) with the stationary phase. This phase is mostly a cross-linked polymer.

The carrier gas (mobile phase) transports the target compound and the other substances through the separation column (stationary phase). This gas must be inert because otherwise it will react with the target compound or materials in the GC or cause problems in the detector.

Helium is commonly used for GC analyses thanks to several benefits like costs and being safer to use. Hydrogen and nitrogen are also used. For hydrogen you have to take extra safety arrangements like a hydrogen detector. Nitrogen is less used, creating longer analysis.

The separation column is placed into the temperature programmable oven. The chromatographic separation can be optimised using a temperature programme for critical pairs of target compounds which separate at a constant temperature.

The detector counts the target compounds coming out of the column. For GC there are two kinds of detectors, specific (like the Electron Capture Detector ECD, very sensitive for halogen containing target compounds) and non-specific (like the Mass Selective Detector MSD or MS, every molecule or ion has a mass).

To calculate results a computer is required to handle the data, especially for a GCMS. In some situations a recorder is enough for registration or report purposes.

Before a GC analyses can be performed on an environmental sample, the sample needs to be pre-treated to be suitable for analyses. In general, analysis by a GC (GCMS) is performed as follows. The sample extract is injected into a preheated injector. The organic solvent, target compounds and other substances are vaporised into the injector. This vapour will be, partly or in total lead over the separation column by the carrier gas. All the different substances, including the target compounds, will be separated from each other, supported by the heat of the GC oven in the column. Finally the target compounds will be detected by a detector. For calculations and reporting of the results, the data is sent to a PC or recorder.

Modern gas chromatography was invented in 1952 and in 1954 with
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Depending on the purpose, there are various standard methods
available for different kinds of compounds suitable for several
The leading aim for GC (GCMS) methods is to achieve the
required detection limit to get reliable results from the target
compounds in relevant environmental samples. The method is built
up in reverse order.
You select a suitable detector which is sensitive and selective
easily enough to detect the target compound(s). The separation column
must have enough affinity with the target compound to get the
required separation of the target compounds and substances.
Manufacturers of columns have a large variety of length, internal
diameter, stationary phase and column material available. The GC
oven has to be powerful enough to ramp the temperature (heating
up) to what is needed to separate critical pairs which can be
difficult to separate.
Which injector type is suitable for the method? The choice is
dependent on how many target compounds need to be brought into
the GC system to get a signal that fits the range for the
detector. A PTV injector is used when you are expecting a low
presence of target compounds. You can inject more extracts, for
example 50 µl compared to a S/L injector (1 or 2 µl). The injection technique
is also dependent on the target compound. When you have
target compounds with high boiling points, you can pre-
separate the target compound from the compound and solvent
with a much lower boiling point using a PTV injector.
Finally a suitable auto sampler needs to be chosen to complete the
configuration of your GC(GCMS) system. For environmental use
there are three options;
• An auto sampler for liquid samples extracts of samples for semi
and less volatile compounds)
• An auto sampler for Head Space (HS) samples (vapour sample
above a liquid sample for volatile compounds)
• A hybrid auto sampler like the Combipal sampler from CTC
This is a XYZ robotic auto sampler and very versatile. There are
different modules available such as a liquid module, a HS
module and a Solid Phase Extraction (SPE) module.
The first two types of auto samplers are dedicated samplers and
are economically the best choice and most suitable for routine
analyses. The third type of auto sampler like the, Combipal, is more
expensive but is also suitable for routine analyses. The strength of
the Combipal is the significant possibility to automate pre-
treatment. SPE for example is a pre-treatment method to extract
your target compounds out of a liquid sample. A liquid sample is then
poured into a column with the solid phase. When the solid phase is
nearly dry, the target compounds are simply washed of the solid
phase with an organic solvent. A Combipal can do all this for you and
more, such as adding injection standard solution to the extract before
injection or derivatisation. Derivatisation is a chemical modification method
to make target compounds suitable for GC analysis.

Benefits
There are many benefits when using GC or GCMS for
environmental analysis such as;
• High throughput of routine samples. Environmental contract
laboratories are capable of keeping operating costs low by
automating and optimising their analyses.
• High accuracy and sensitivity. GC's are highly sophisticated.
Manufacturers are continuously developing their GC's and all
new models have new features and better performance.
• Multiple parameter analysis. It's possible with a GC to quantify
several kinds of different compounds in one single run like
Organic Chlorinated Pesticides (OCP's) and Poly Chlorinated
Biphenyl's (PCBs) or over a hundred different organic compounds.
• Qualification. Besides the usual quantification of target
compounds, you can use a GCMS to quantify unknown compounds.
• Low detection limit. A GC is capable of quantifying tar-
get compounds at a very low level e.g. Parts Per Trillion (ppt).
• Second life. A GC is flexible. In many cases a second hand GC
can be easily adapted to fit for another purpose by changing
detector(s), auto samplers or injectors.
• Costing. For specific purposes, GC's are sometimes coupled to
other analytical instruments like Inductive Coupled Plasma-MS,
ICP-MS (instrument to analyse elements) and LC (coupled to the
front of a GC in combination with large volumes injected to
perform clean-up of the extract).

Opportunities and
developments for the future
Besides all the benefits of using GC or GCMS, it may be obvious
that there are still some opportunities for improvements and
development with GC or GCMS analysis in general, but also for
environmental use too.
In general GC analyses is used for compounds with a boiling point
of 500 degrees Celsius. This range could be extended in the future
by using other injection techniques.
Water can be disastrous for the stationary phase of the separation
column. Other injection techniques can prevent water reaching the
column or even make it possible to inject aquatic samples. At
the same time manufacturers could improve their column to be
less sensitive for water.
Some target compounds, compounds that are familiar to the
target compound or other compounds which are present in the
sample (matrix) are hard to separate from each other. In that case
these compounds leave the column (almost) at the same time the
target compounds leaves. This effect is called co-elution and affects
the result even causing a false positive result. When optimising the
oven program, changing columns or other qualifiers (specific
fragment for a target compound when using an MS) doesn't help,
you can look for better pre-treatment techniques or use GC x GC.
Two GC's are coupled to each other. The compounds that co-elute
are lead to another GC with another column, where the separation
starts over to desperate them.
Depending on the target compounds, separation and numbers of
compounds, a GC run can take a view minutes to more than an
hour. Fast GC analysis performed in TV CSI episodes are an illusion.
When you want to analyse more samples or analyses in shorter
time, manufacturers are developing small GC oven with heating
coils on the outside or columns on a chip. The benefit of this is that
the column is heated in seconds to reach a preset temperature
compared to a couple of minutes with a regular oven. This kind of
development has lead to accurate GC analyses in less time.
More data of characteristic materials or samples will enable the
(future) analyst to perform better identification. With
comprehensive GCMS (a 3D plot of a sample) or pyrolysis
(burn a material or solid sample) give characteristic information
of a sample.
Manufacturers are constantly developing their GC's and their
equipment, such as, detectors, to get lower detection limits in order
to trace the slightest amounts of target compounds. MSD's are
developing like Triple Quad MS (Three Quadrupoles placed in
series) or Time Of Flight MS (measures the time of flight of the
ions). Flame Ionisation Detectors (FID) are also gaining sensitivity.
GC analysis is commonly performed in a laboratory on a bench top
GC system in order to achieve correct results. GC (GCMS) analysis is commonly performed in a laboratory on a bench top
GC system in order to achieve correct results. GCMS in the future. GC's and GCMS are essential multi-tools for
evironmental analysis, both now and in the future.

In the University of Amsterdam there is a faculty under the
supervision of Professor Peter Schoeakers. They are studying
deways to increase the working field of chromatography like GC and
GCMS. These studies will lead to more applications for GC and
GCMS in the future. GC and GCMS are essential multi-tools for
environmental analysis, both now and in the future.