



# Leaktight Couplings, The Final Detail For Robust GC Analysis

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Interview with Wim Broer of Alcontrol

"There are many situations in petrochemistry, including large volume injection systems, where capillary GC would benefit from coupling an additional column or a pre-column to the analytical separation column. As a result of the difficulties associated with the use of existing column connectors, however, analysts are reluctant to use coupled column systems. According to Wim Broer, Production Development Chemist from Alcontrol, Hoogvliet, the Netherlands, the new melt-fit technology developed by NLISIS eliminates the drawbacks of the current column connectors available. It improves reliability, saves time and gives better performance. "This is the last detail we needed to have a robust analysis."

Analysts face many problems in their day-to-day GC operation. One of the most common frustrations is the leakage of column connectors, causing bad results and hours of delay. Wim Broer tested an innovative connecting device, the Melfit One from NLISIS, that claims to eradicate all of those problems, and wanted to share his experiences with us. His conclusion was: "In the past we had problems on a regular base with leaking columns. They will leak, and their active surface will decompose the analytes. Now with the use of the Melfit One we always have very reliable connections. The Melfit One is easy to operate and can be used in our labs and in petrochemical routine applications in production labs."

Wim Broer went on to say, "Coupling is a drama for most analysts, some can do it, but most analysts have trouble with it and cannot make a good connection. When we used metal ferrules and Pressfits, the issue was the practical handling, and getting a reliable leak tight connection. If the coupling is not leak tight, the oxygen will disturb your stationary phase and influence your results and you get tailing. The most irritating effect of leakage is that it not only occurs at the wrong moment but also when you least expect it. Often you do not know what the cause of the problem is. You may think it is the injector itself, the liner, a bad column, or a connection which may have an active surface and causes peak distortion. Before you know what it is, it costs you a lot of time."

## Is this a new problem?

"The problem has existed for over twenty years, and you know how it is with those things, you learn to live with it. You work hard to get it leak tight, use good glue and press the coupling very tight. For most analysts it takes a lot of time to learn how to make a good connection, and some never learn. It is irritating, you lose a couple of hours in the day, you get a bad analysis and you lose your sample. We have a hundred GC's running, and we need to make couplings every day and bad connections happen all the time. Also the connection will leak sooner or later, that is the reality. Once that happens you have to start over again which includes cutting the pre-column resulting in a lot of extra work. I saw the Melfit One at "The Instrument", the bi-yearly meeting for new analytical instruments in the Netherlands. The manufacturer NLISIS claimed that the Melfit One would eliminate the problems associated with the use of the usual column connectors and make connections without leaking and I knew this would be the way to solve our coupling problems."

## How do you know if a coupling is leaking?

"We do a leak test with a leak detector, but that is done with the cold coupling. But then when you warm the

column it can start to leak and you see it is sucking in air when using a MS as a detector. This means you lose an hour and you have to start over again. If you are lucky and you see the problem in time it can still cost a lot of time to set up a new analysis. Additionally if you use an FID, you only see it after the first analysis is complete. This is a great loss for a contract lab like ours, we cannot lose that time since we work on the lowest possible costs. In a petrochemical lab in production control the costs are even higher and. If an analysis is incorrect, that causes a lot of trouble."

## How did you test the Melfit?

"We used it specifically for our Time of Flight (TOF) applications. If you have air in the system your Mass Spec does not work anymore. Previously we could not stop the leaking but with the Melfit we could get it leak tight in one go. We applied it to many connections and checked all the connections visually under the microscope and with oxygen sensitive detectors. You can immediately see the effect of leakage and loose connectors when you use oxygen sensitive detectors".

"We found no leakage at all and in addition we proved that shorter stabilisation times for oxygen sensitive detectors and lower detection limits could now be reached. Such reliable couplings increase the reliability of our analysis. With these couplings we prevent the loss of time and valuable sample material". "I cannot express it in money, but the main gain is in the time we would otherwise lose in solving problems or repeating our analysis. Another advantage is that there is no longer a dead volume in our GC-system. The dead volume is reduced by a factor of ten, in essence reducing it to zero. In this set up, there is no more contact of the analytes with the glass or metal of the coupling, so there are no more disturbances. We apply it for our polar and slightly polar compounds such as anilines, phenols and PAK's and this is a sensitive measurement, where you need stable analysis conditions. We saw the sensitivity for polar compounds improve considerably. The chromatograms show there is less dead volume and fewer active spots where the ferrules decompose the analytes. We also found that analyses were improved. The peak performance is better. We see better Gaussian shapes and no more tailing. For instance, the chromatograms of Benzo(ghi)perylene, 4-Nitrofenol and 2-nitroaniline show better results with the Melfit."

## Will you be using the Melfit?

"We will use the Melfit One in all cases where we now use other connectors. It is especially helpful in GC/MS, where we couple columns of different size with retention gaps and where we interface with valves and detectors.

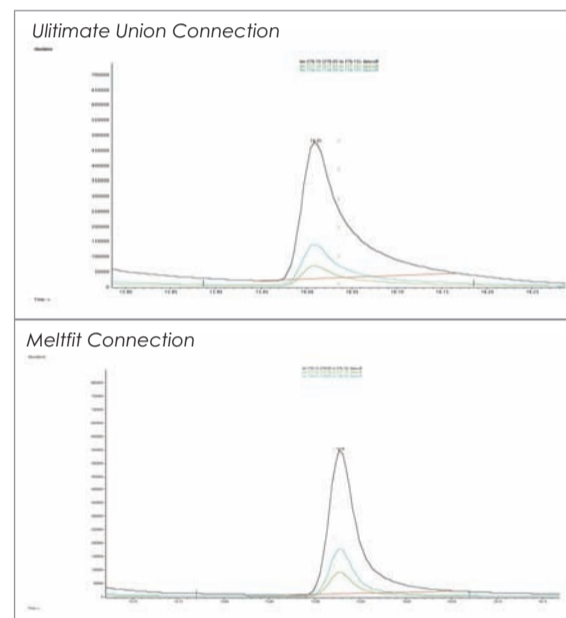


Figure 1: Benzo(ghi)perylene

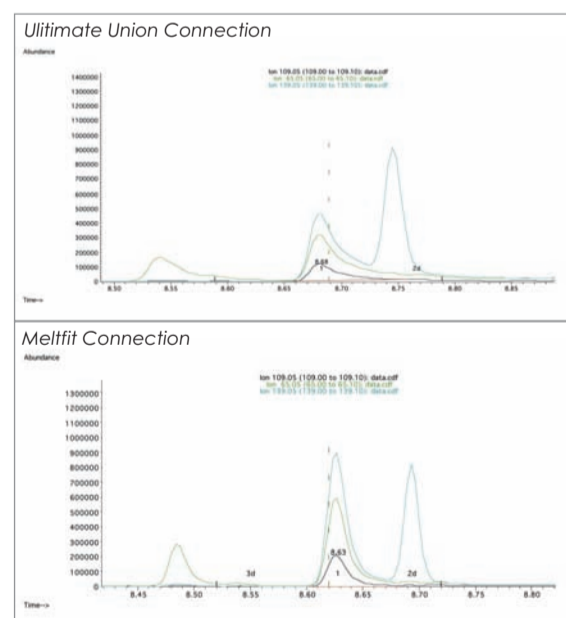


Figure 2: 4-Nitrofenol

In addition, many more GC applications will now be possible such as coupling an additional column or a pre-column to the analytical separation column. Due to the difficulties associated with the use of column connectors so far, we have been reluctant to use coupled column systems. The use of the Melfit One will allow many users in our labs and production labs in petrochemistry to take advantage of coupled columns in GC."

Table 1: Wim Broer conclusions and comments

Pro and cons	Melfit	Pressfit	Metal connectors
<b>Easy to make a connection</b> <b>Leak tightness</b> <b>Risk of down time</b> <b>Dead volume</b> <b>Inertness</b> <b>Extra column band broadening</b> <b>Thermal degradation</b> <b>Size</b> <b>Mechanical stability</b>	<b>Easy</b> <b>Tight</b> <b>Eliminated</b> <b>No</b> <b>Excellent</b> <b>No</b> <b>No</b> <b>Small</b> <b>Good</b>	<b>Experienced operator</b> <b>Prone to leaking</b> <b>Considerable</b> <b>Minimal</b> <b>Good</b> <b>Possible</b> <b>Sometimes</b> <b>Small</b> <b>Poor</b>	<b>Normal</b> <b>Tight</b> <b>Eliminated</b> <b>Yes (moderate)</b> <b>Poor</b> <b>Possible</b> <b>No (Yes for certain compounds)</b> <b>Medium</b> <b>Good</b>
<b>Overall result</b>	<b>Prevents time loss improved reliability &amp; robustness</b>	<b>Needs good check for leak tightness and poor mechanical stab</b>	<b>Thermal mass is bigger than other systems. No visibility check for connection possible poor inertness</b>

### Principle of the Melfit device

Columns are inserted into a small glass tube made of low-melting proprietary glass (softening temperature 380 °C). After heating, a small gas pressure (typically between 1 and 2 bar) is applied. The softened glass then closes tightly around the connector. The insert shows a schematic drawing of a connection and a photograph of a real connector.

In our laboratory the Melfit connectors were subjected to a series of critical tests. The test protocol included tests for leak tightness, inertness (both in terms of adsorption as well as with regard to surface catalysed degradation) and dead-volume.

### Alcontrol

Alcontrol is a service lab in Fuel, Environmental and food testing, with locations in several countries in Europe.

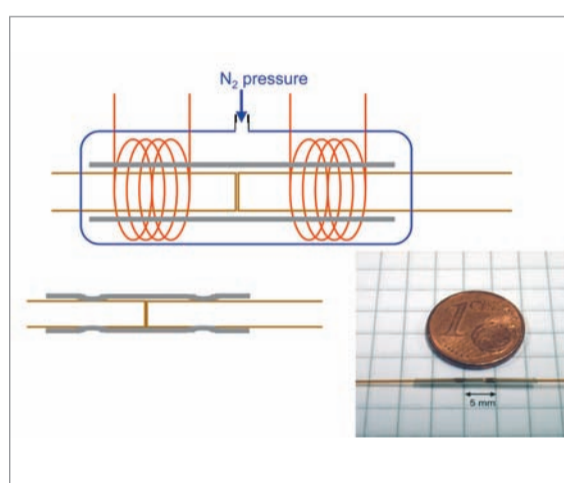


Figure 3: Principle of the Melfit device

The international group is located in the UK, and has over 2000 employees, performing 1500000 samples per year.

Wim Broer is the GC specialist in the Hoogvliet location in the Netherlands. His job is to develop new analysis methods based on the demands of the customer and as a result from regional or European legal obligations. He is located in the Environmental lab of Alcontrol in Hoogvliet, the Netherlands, where analyses are done for a variety of companies in area such as environmental, food, health and safety,, and petrochemistry. The group develops techniques and new methods for Alcontrol, in close communication with their customers. For example the methods for the new European regulations, ISO and work with CEN. They also develop the techniques for the analysis of mineral oil for environmental applications. Recently his group developed an analysis for the determination of straight run hydrocarbons in mineral oil in air and surface water. This will be applied in their routine analyses in their labs. Furthermore they do environmental analyses in water, soil and air and have a large number of techniques available, such as GCMS, LC, LCMSMS, ICP, discrete analysers and more.

### NLISIS in the Petrochemical Industry

The Melfit One was developed and is manufactured by NLISIS Chromatography, a company based in Veldhoven in The Netherlands. The petrochemical industry is an important market for NLISIS. Shell and ExxonMobil were among the first companies to take advantage of this innovative connection technology. Mike Ophield is responsible for worldwide sales and is based in Houston, Texas explains the relevance of the Melfit One for the Petrochemical Industry: "In the petrochemical industry in particular time equals money. Time spent preparing samples and carrying out sample analysis is wasted if a bad column connection ruins the results. This is particularly true when you take into consideration the wide use of GC/MS and GC x GC in this industry. The Melfit One removes the worry associated with leaking connections and therefore saves valuable time and money as well as improving the chromatography."