Measurement of trace impurities in multiple bulk gases

**LDETEK SOLUTION:**

*The MultiDetek2 (compact gas chromatograph)*

This compact gas chromatograph can handle up to 3 detectors with 6 parallel chromatographic channels through a network combining up to 10 diaphragm valves and 12 chromatographic columns. It opens the doors to many applications requiring multiple bulk gases analysis using the same compact analytical instrument.

One configuration (method) is pre programmed for each bulk gas analysis requirement. This way, the user can simply load the desired configuration and the analyser is ready to start the analysis. As well, the impurities can be independently configured with specific ranges and minimum detection limits.

No external PC is required since the robust industrial PC is mounted in the instrument and gives access to a data storage capacity. The instrument can be controlled remotely through the Ethernet connectivity. All industrial communication protocols are accessible. Automatic or manual report printing can be handled.
The LDGSS (stream selector)

Multiple bulk gases analysis usually requires a stream selector system being able to be controlled locally or remotely. A manual switch is available on the front to select manually the desired stream. Otherwise, the stream can be selected via the MultiDetek2 interface. The interface gives the possibility to edit the names of the streams, to program multiple sequences and to set specific purging time between each stream. The streams can also be selected and identified by a standard DCS system through a dry contacts network.

This ultra high purity system offers a quick response time due to its dead volume free design. An adjustable sample purge valve and flow meter is independent to each stream to adjust the gas line purging before the stream selector. A purge gas being the same of the carrier gas of the MultiDetek2 is used to keep the ambient air away of the analytical flow path to ensure ultra high purity analysis gas for all the streams. Specifically when trace impurity of a bulk gas is also the pure gas of its neighbor stream that was just analysed in the previous analysis. The design of a back purged stream selector system is very important and this is what is included in this solution.

For this application note, since the analysis of hydrogen and oxygen is required, 2 LDGSS systems have been used to avoid mixing of both gases together. The purge gases are independent to each stream selector to avoid any chance of mixing oxygen and hydrogen. The LDGSS used for Oxygen can be certified for O2 Clean.

The COMPACT-LDP1000 (gas purifier)

The gas purifier generates ultra high purity carrier gas certified for a total of 10ppb total impurities. This compact high capacity gas purifier keeps the system clean and intact even if carrier gas cylinders purity can vary from batch to batch introducing a variation of baselines which has impact on the accuracy of the analytical device. Or in another instance, if by mistake, an air contamination gets in the carrier flow path during the process of changing the carrier gas cylinder.

The gas purifier can also communicate in real-time with the MultiDetek2 to inform about its status. In the event of a default, it generates an alarm in the MultiDetek2 to automatically advise about the fault.

The LDRACK (certified cabinet)

The complete solution comes assembled and certified in a cabinet. Such system is fully tested to certify the analytical response time when switching between streams. This is more than necessary when low ppb analysis of nitrogen and oxygen are targeted. It doesn’t only guarantee that the sample gas lines offers no dead volume and leaks, but also guarantees that the carrier gas lines are properly mounted. It is the best practice to get the best analytical performances especially when traces of impurity at ppb level are also present in ambient air must be detected.
THE COMPLETE SOLUTION

MULTIDETEK2 (COMPACT GAS CHROMATOGRAPH)

LDGSS (COMPACT ULTRA HIGH PURITY STREAM SELECTOR)

LDRACK (INTEGRATED CABINET)

COMPACT LDP1000 (COMPACT GAS PURIFIER)

MOUNTED ON THE BACKSIDE OF THE MULTIDETEK2 PANEL

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Figures 1 to 12 show some examples of results that such configuration can achieve for measuring low ppb/ppm concentrations of multiple trace impurities in multiple bulk gases.

In this example, trace impurities H2, Ar, O2, N2, CH4, CO, CO2 in bulk gases Argon, Carbon dioxide, Hydrogen, Helium, Nitrogen and Oxygen were measured.

An optimized method for high range (HR) covering up to 1000ppm and another method for low range (LR) to ensure good peak detection and stability down to 10ppb are used. This dual methods technique gives the possibility to extend the dynamic range as desired. Conventional analysis techniques will tend to have some limitations on the low ppb detection when a high ppm analysis is required. The linearity and accuracy of our system is improved by having an optimized method for a specific analysis range.

**RESULTS:**

Figure 1: chromatogram of trace impurities in Argon (High range ppm)

Figure 2: chromatogram of trace impurities in Argon (Low range ppb/ppm)
Figure 3: chromatogram of trace impurities in Carbon Dioxide (High range ppm)

Figure 4: chromatogram of trace impurities in Carbon Dioxide (Low range ppb/ppm)

Figure 5: chromatogram of trace impurities in Hydrogen (High range ppm)
Figure 6: chromatogram of trace impurities in Hydrogen (Low range ppb/ppm)

Figure 7: chromatogram of trace impurities in Helium (High range ppm)

Figure 8: chromatogram of trace impurities in Helium (Low range ppb/ppm)
Figure 9: chromatogram of trace impurities in Nitrogen (High range ppm)

Figure 10: chromatogram of trace impurities in Nitrogen (Low range ppb/ppm)

Figure 11: chromatogram of trace impurities in Oxygen (High range ppm)
<table>
<thead>
<tr>
<th>COMPONENT</th>
<th>CONCENTRATION</th>
<th>PEAK HEIGHT</th>
<th>NOISE</th>
<th>LDL (3X NOISE)</th>
</tr>
</thead>
<tbody>
<tr>
<td>H2 (fig.8)</td>
<td>10.000 ppm</td>
<td>1621 mV</td>
<td>0.7 mV</td>
<td>12.9 ppb</td>
</tr>
<tr>
<td>Ar (fig.4)</td>
<td>10.000 ppm</td>
<td>1780 mV</td>
<td>0.4 mV</td>
<td>6.7 ppb</td>
</tr>
<tr>
<td>O2 (fig.4)</td>
<td>10.045 ppm</td>
<td>1320 mV</td>
<td>0.6 mV</td>
<td>13.7 ppb</td>
</tr>
<tr>
<td>N2 (fig.2)</td>
<td>10.006 ppm</td>
<td>1821 mV</td>
<td>0.5 mV</td>
<td>8.2 ppb</td>
</tr>
<tr>
<td>CH4 (fig.6)</td>
<td>10.089 ppm</td>
<td>1612 mV</td>
<td>0.6 mV</td>
<td>11.3 ppb</td>
</tr>
<tr>
<td>CO (fig.10)</td>
<td>10.000 ppm</td>
<td>1806 mV</td>
<td>0.9 mV</td>
<td>14.9 ppb</td>
</tr>
<tr>
<td>CO2 (fig.6)</td>
<td>10.282 ppm</td>
<td>1823 mV</td>
<td>0.8 mV</td>
<td>13.5 ppb</td>
</tr>
</tbody>
</table>

Note: other LDL could be obtained with different injection volume and chromatographic condition

**Figure 12**

**CONCLUSION:**

Our complete integrated gas analysis solution guarantees the best performances and robustness for any applications combining multiple impurities in multiple streams and/or bulk gas types. It is compact and compatible with standards of the industry.