The Use of Focused Non-Dispersive Infrared Spectroscopy (NDIR) in Total Organic Carbon (TOC) Analysis

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Non-dispersive infrared spectroscopy (NDIR) is the most widely used detection method employed by commercially available TOC analysers in the laboratory. This detection technique enables both, a selective and sensitive detection of the carbon dioxide (CO_2) formed from the various carbon compounds during the TOC analysis. However, CO_2 and carrier gas (synthetic air or pure oxygen) are not the only gases or components, which are coming into contact with such a NDIR detector. In many TOC applications corrosive gases are released from the matrix of the combusted samples (aqueous or solids such as soils, waste, sludges, sediments) which, if not effectively removed prior to detection, can cause considerable damage to the sensitive detector. Even with the conventional TOC determination in aqueous samples by the direct method (EN 1484), also known as the NPOC method (Non-Purgeable Organic Carbon), the hydrochloric acid added to separate the inorganic carbon (carbonates and hydrogen carbonates) is nearly entirely released as corrosive HCl gas during the high temperature catalytic combustion. In fact this is considered welcome from the combustion point of view, since the volatility of the hydrochloric acid, unlike the oxide forming phosphoric acid, means there are significantly less unpleasant deposits remaining on the active catalyst surface, thus extending the lifetime of the catalyst. However, from the point of view of the detector, this HCl gas is a major threat and must be quantitatively removed before reaching the detector by means of so-called halogen traps, typically active metal surfaces that bind the HCl chemically in the form of chlorides.

Frequent Sensitivity Losses of Conventional NDIR Detectors and their Causes

In the case of TOC measurement in samples of extreme matrix composition, such as sea water, brine, or even concentrated acids, just as in combinations of a liquid TOC device with a solid module for digestion of acidified solid samples in accordance with the direct method B (EN 13137 or EN 15936), there is still corrosion in the detector, even if the halogen trap filling is regularly replaced, which is associated with severe losses of sensitivity up to the total failure of the NDIR detector. Why is this and can it be avoided?

To this end, let us first consider the design and functioning of conventional NDIR detectors.

In principle, NDIR detectors consist of the following three essential components: a radiation source that emits broadband IR radiation, a cuvette through which the sample gas flows, and a sensor that detects the change of the radiation intensity after absorption by the analyte (CO_2). As well, the detector for a TOC device must be designed to be as sensitive as possible (detection threshold) while simultaneously having a large dynamic measuring range and ideally long-term stability.

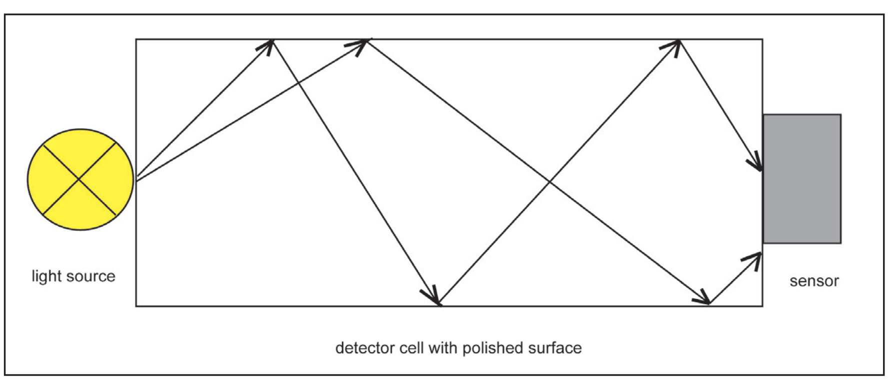

Figure 1: Schematic structure of a conventional NDIR detector (image: Analytik Jena)

Many NDIR detectors feature reflections of the IR radiation at the walls of the cuvette, caused by the construction and material, which account for the measurement result to a significant extent. On the one hand, using the total reflection at the walls of the cuvette increases the sensitivity due to the extension of the path length. On the other hand, however, not all of the IR radiation reaches the sensor as a result of the reflection processes, thereby reducing the radiation power at the detector. An optimum wall reflection is reliant on using polished, mirror-bright metallic materials. The detector's sensitivity falls significantly as soon as there are any changes at the cuvette surfaces that affect the reflection, for example as a result of the effect of corrosive gases such as HCl or the depositing of dust and other precipitates.

This inevitable aging process, which can only be partially offset by a reference sensor, leads to unpleasant drift effects, sensitivity losses, and increasing 'blindness' of the NDIR detector. This is the main reason for the generally poor robustness and long term stability of reflection-based NDIR detectors. The consequences are frequently required re-calibration of the system, frequent detector maintenance for cleaning and/or readjustment of the detector's electronic signal gain, through to the complete replacement of the gas cuvette or the entire detector assembly. For users, this means a high degree of additional work, unreliability of the measurement results, downtime of the TOC instrument, and significantly higher operating costs.

NDIR Detection with Focused Radiation and its Benefits

The problems described above can now be put to an end thanks to the newly developed measurement principle of the Focus Radiation NDIR Detector®. This principle is applied right from the radiation source, preventing the loss of IR radiation intensity by means of a specific optical arrangement that ensures the parallel alignment of the IR radiation before it enters the gas cuvette. In combination with a specially designed converging lens, the IR radiation is focused towards the sensor, where it arrives without loss and with high intensity, guaranteeing the high sensitivity of the FR NDIR detector.

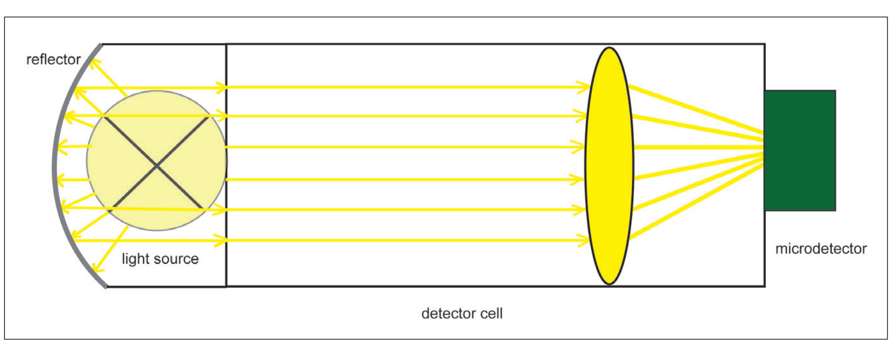

Figure 2: Schematic structure of the Focus Radiation NDIR Detector® (image: Analytik Jena)

The parallelised IR radiation means the gas cuvette and its inner surface are no longer involved in the optical process. This now opens up the design freedom to move away from corrosion-prone metallic materials and towards non-corroding glass. Since in this detector design the wall reflection is no longer relevant, any deposits no longer affect the sensitivity of the detector either. The result is an extremely robust and long-term stable NDIR detector, which Analytik Jena has been putting to good use for a number of years in the multi N/C® series with great success.

Considering the outstanding properties of this FR NDIR detector, Analytik Jena is now offering a unique long-term guarantee of 10 years on the detector assembly.

The Focus Radiation NDIR Detector® has other key advantages. This detector, with its pulsed IR radiation source, does away with the otherwise common interval-like interruption of the beam path by a chopper mechanism (motorised perforated disk), making it much more robust. The sensor used in conjunction with advanced signal processing and signal evaluation allows the detector to have a wide dynamic measurement range. The working range in the TOC measurement ranges from the detection limit to 30,000 mg/l for undiluted aqueous samples and up to 500 mg of carbon absolute for solids analysis. It is thus characterised as a long-term stable, easy-to-use wide range detector, without measurement channel switching to cover the required working area of a TOC analyser and without the need for frequent re-calibration of the measurement system.

The field of TOC analytics has entered new territory with the development of the Focus Radiation NDIR Detector®. The long-term stability and robustness, in addition to the convenient measuring range, contribute to increasing productivity and to reducing the operating costs of a TOC analyser.