

### TOC Analysis: New Approaches to Detection – FR-NDIR Detector

#### Introduction

Non-dispersive infrared spectroscopy (NDIR) is the most widely used detection method employed by commercially available TOC analyzers in the laboratory. This detection technique enables both, a selective and sensitive detection of the carbon dioxide (CO<sub>2</sub>) formed from the various carbon compounds during the TOC analysis. However, CO<sub>2</sub> and carrier gas are not the only gases or components that are in contact with the NDIR detector. In many TOC applications corrosive gases are released from an aqueous or solid sample matrix during the oxidation process which, if not effectively removed prior to detection, can cause considerable damage to the sensitive detector.

#### Your Benefits

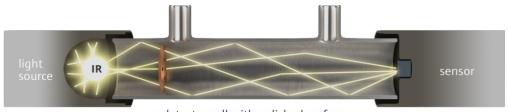
- No corrosive parts in the gas way
- No maintenance requirements
- Extreme long-term stability
- One-channel wide range detector for maximum sensitivity and widest working range

Even with the typically used direct TOC determination in aqueous samples by the NPOC (non-purgeable organic carbon) method according to DIN EN 1484 or DIN EN ISO 20236<sup>2)</sup> for catalytic combustion systems, hydrochloric acid is added to eliminate the inorganic carbon (carbonates and hydrogen carbonates) by a gas purge treatment. This volatile acid is almost entirely released as corrosive HCl gas during sample combustion. From the combustion point of view this is appreciated since the catalyst is not spoiled by residues and surface deposits, which is the case with the oxide-forming phosphoric acid. Thus HCl acidified samples are extending the lifetime of the catalyst. However, from the NDIR detector point of view, this HCl vapor is a major threat and must be quantitatively removed before reaching the detector by means of a halogen trap.



Frequent sensitivity losses of conventional NDIR detectors and their causes

NDIR detectors consist of 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 IR absorption by the CO<sub>2</sub>. Also the detector for a TOC device must be designed to be as sensitive as possible (detection limit) while simultaneously having a large dynamic measuring range and ideally long-term stability.



detector cell with polished surface



In TOC testing of samples of extreme matrix composition, such as sea water, brine or concentrated acids, as well as for liquid/solid TOC combinations, where acidified solid samples are measured according the direct method B (DIN EN 13137<sup>3</sup>) or DIN EN 15936<sup>4</sup>), a high risk of detector corrosion is given, even if the halogen trap filling is regularly replaced. Such corrosion processes are associated with severe losses of sensitivity up to the total failure of the NDIR detector. This is due to the functioning of conventional NDIR detectors, which is based on the reflection of the IR radiation at the inner surface of the gas cuvette.

The reflection of the IR radiation is supported by the polished metal material of the construction, which has a considerable influence on the measurement result. On the one hand, using the surface reflection inside the detector cell increases the sensitivity by the extension of the radiation path length. On the other hand, however, not all of the IR radiation reaches the sensor because of the reflection processes, thereby reducing the radiation intensity at the sensor. An optimum wall reflection is reliant on using polished, high-gloss metallic materials. The detector's sensitivity drops significantly as soon as there are any changes at the cuvette surfaces that affect the reflection, for example as a result of corrosion processes 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, up to the complete replacement of the gas cuvette or the entire detector assembly. For users, this translates into additional work load, analytical unreliability, downtime of the TOC instrument and significantly higher operating costs.



#### A new standard in NDIR detection: Focus Radiation NDIR

The measurement principle of the Focus Radiation NDIR Detector developed by Analytik Jena avoids corrosion and prevents sensitivity loss of IR radiation intensity by means of a specific optical arrangement. The design ensures the parallel alignment of the IR radiation before it enters the gas cuvette. In combination with a specially designed focusing lens, the IR radiation is focused towards the sensor, where it arrives without loss and with high intensity, guaranteeing the high sensitivity of the detector.

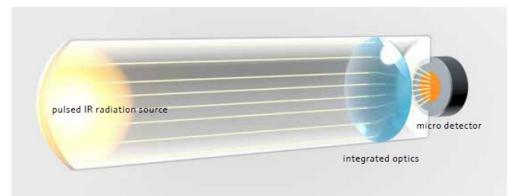


Fig. 2: Schematic structure of the Focus Radiation NDIR Detector (image: Analytik Jena)

In addition, the parallelized IR radiation enables the second and crucial advantage of the FR-NDIR detector over conventional NDIR detectors - the gas cuvette and its inner surface are no longer involved in the optical process, any deposits no longer affect the sensitivity of the detector. This allows the use of non-corroding glass instead of corrosion-prone metallic materials. The result is an extremely robust and long-term stable NDIR detector. Thanks to the outstanding features of the FR-NDIR detector, Analytik Jena can confidently offer its customers a unique 5-year long-term warranty on the detector assembly.

The Focus Radiation NDIR Detector has even further key advantages. Its pulsed IR radiation source eliminates the need for mechanical moving parts such as a chopper mechanism (motorized perforated disk) to interrupt the radiation beam, making the detector much more robust.

The sensor in combination with advanced signal processing and signal evaluation provides a wide dynamic measurement range from the detection limit up to 30,000 mg/L for undiluted aqueous samples and up to 500 mg of carbon absolute for solids analysis. This eliminates the need for measurement channel switching to cover the required concentration range of a TOC analyzer and frequent re-calibration.



#### Conclusion

With its unique long-term stability, robustness and comfortable dynamic measurement range, the Focus Radiation NDIR Detector contributes significantly to increasing productivity and reducing operating costs in TOC analysis.

To view a summary of the enhancements the Focus Radiation NDIR Detector offers compared to common NDIR detectors, have a look at our tutorial video available on <u>YouTube</u>.

#### References

- 1) DIN EN 1484: Water analysis Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon
- 2) DIN EN ISO 20236:2023-04 "Water quality Determination of total organic carbon (TOC), dissolved organic carbon (DOC), bound nitrogen (TN<sub>b</sub>) and dissolved bound nitrogen (DN<sub>b</sub>) after catalytic oxidative high-temperature combustion (ISO 20236:2018)"
- 3) DIN EN 13137: Characterization of waste Determination of total organic carbon (TOC) in waste, sludges and sediments
- 4) EN 15936: Sludge, treated biowaste, soil and waste Determination of total organic carbon (TOC) by dry combustion

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