

L-0 | Analytical Challenges within the Carbon2Chem® Approach

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The successful on-line analysis of main and trace compounds in the ppb- and ppt-range contained in metallurgical gases before and after a purification system using PTR-TOF-MS has been demonstrated. However, not all trace compounds can be measured with this technique. Therefore, a complementary technique such as TD-GCMS is being applied to get the “whole” picture of traces, which could be harmful for the subsequent catalysts used for synthesis or even for substances breaking through and accumulating in the methanol product. Since off-line sampling methods are necessary for TD-GCMS analysis, the challenges associated with it have to be faced in order to validate the PTR-TOF-MS data.

Proton-Transfer-Reaction Time-of-Flight Mass Spectrometry

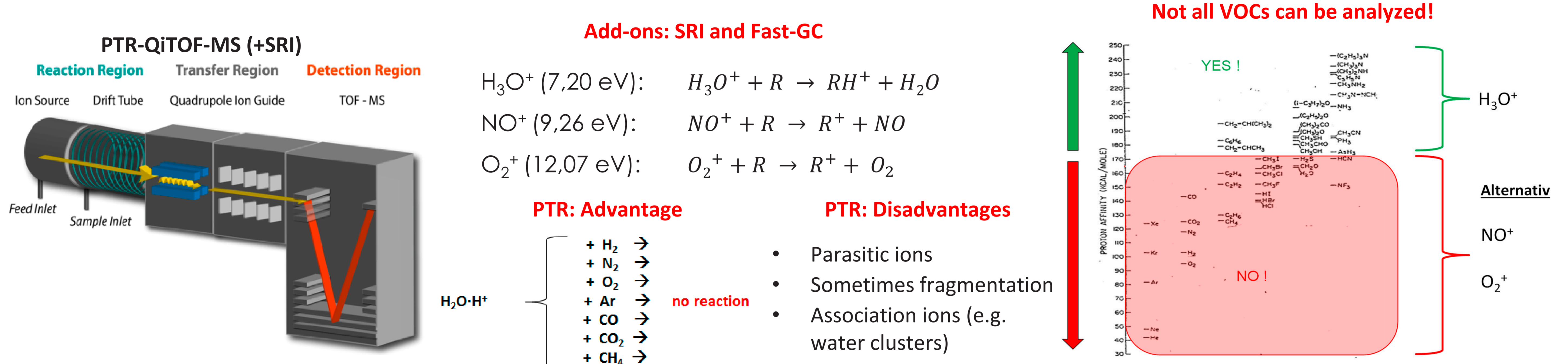


Figure 1 Sketch of the PTR-QiTOF-MS (+SRI) instrument with its advantages and drawbacks.

PTR-TOF-MS Analysis of Process Gases

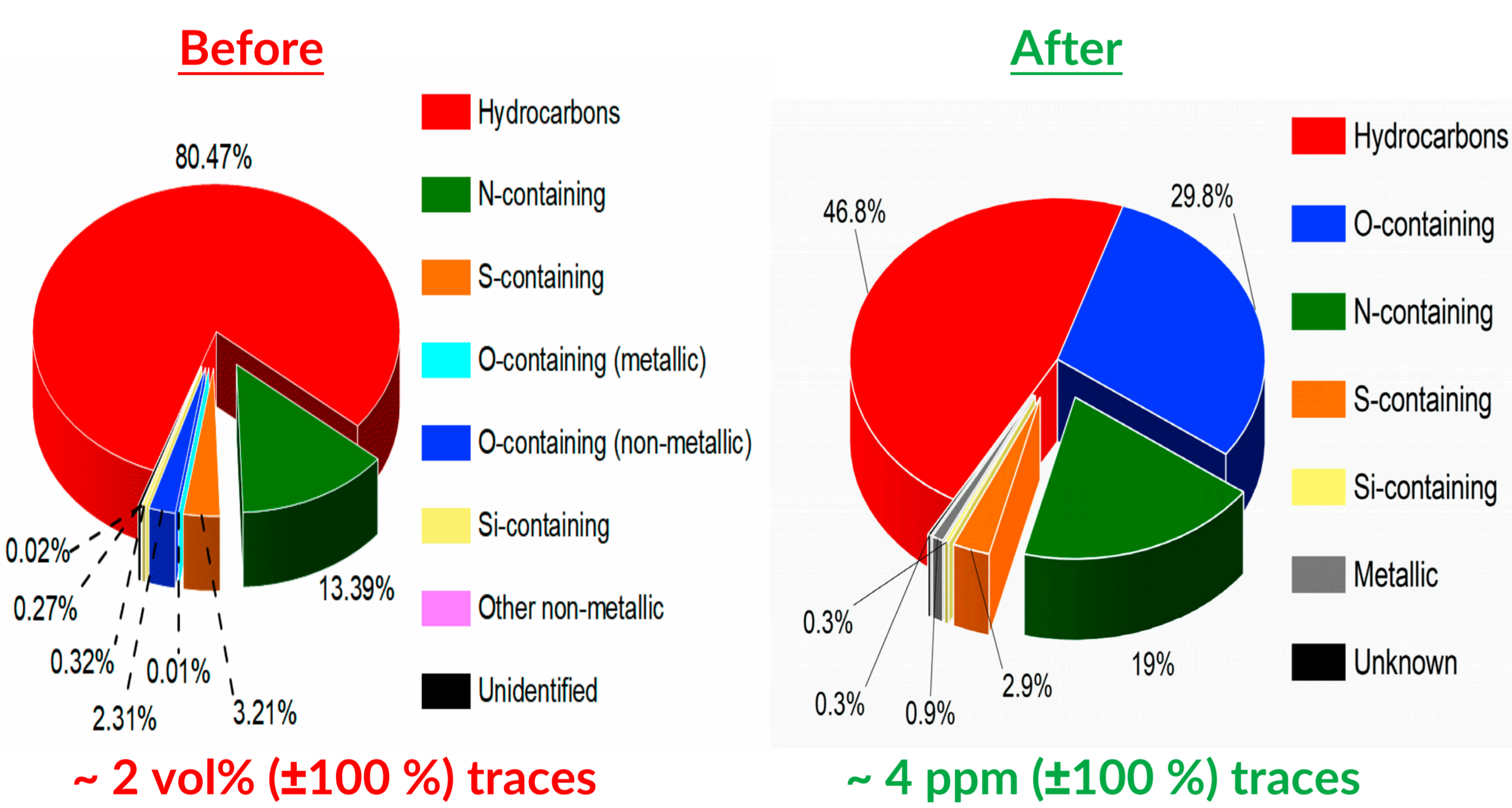


Figure 2 Compound class distribution before and after purification.

Traces in Methanol Product

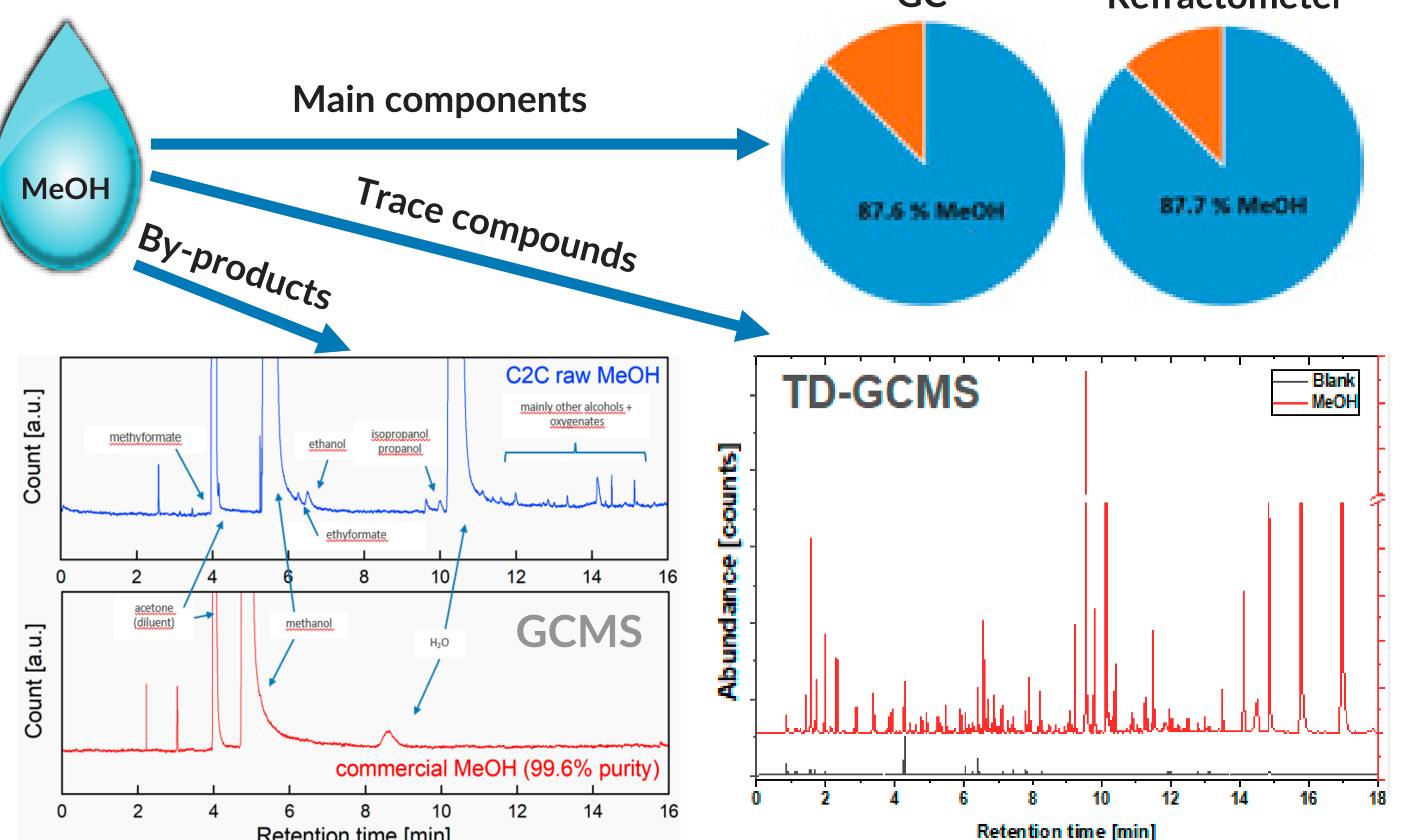


Figure 4 Comparison chromatograms of the higher sensitivity of TD-GCMS towards trace analysis over conventional GCMS.

Challenges off-line sampling for TD-GCMS

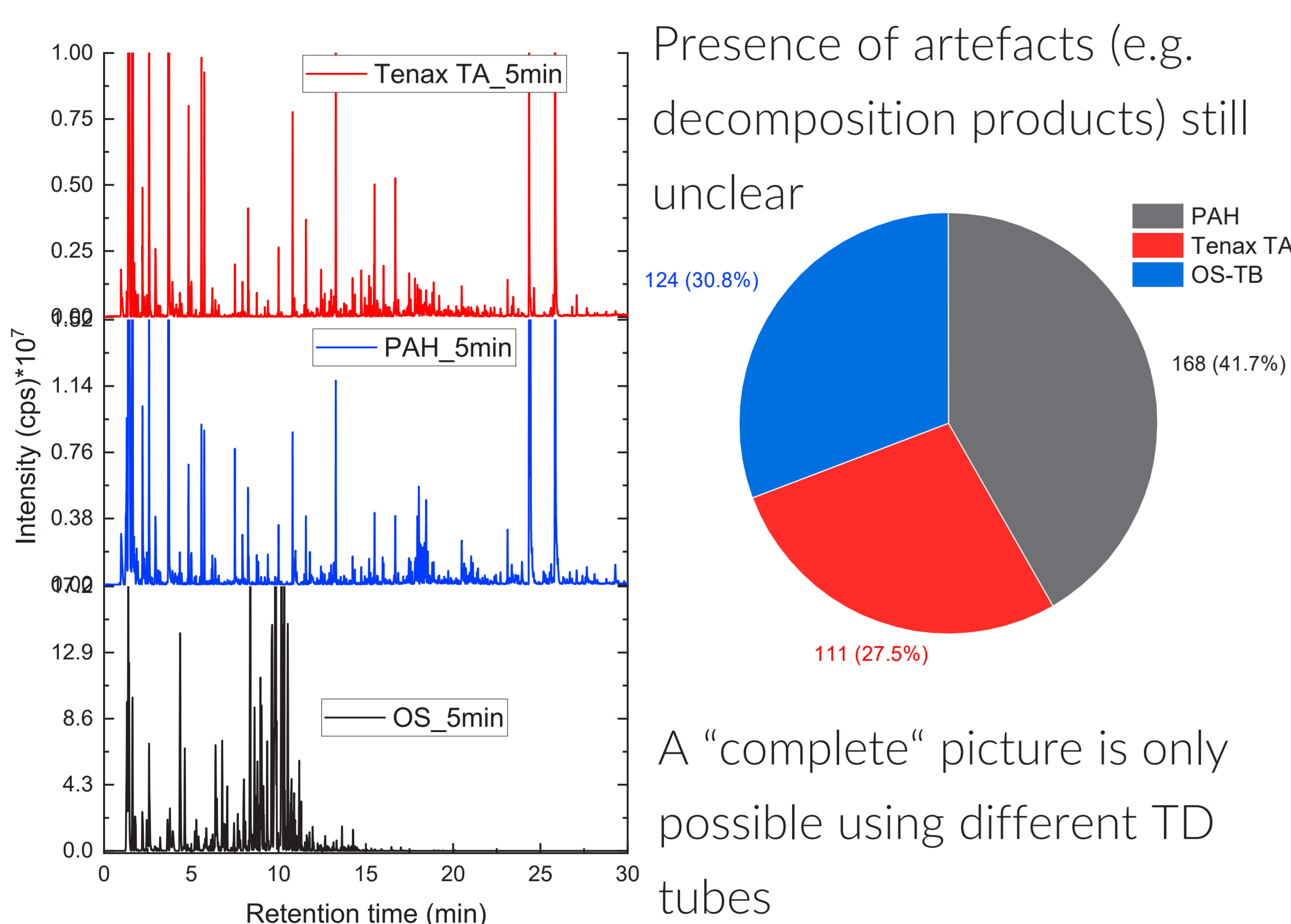


Figure 3 Chromatograms of the different selectivity of TD-tubes towards trace compounds in raw metallurgical gases.

- Only a few traces detectable with GCMS, mainly by-products
- Over 150 traces in commercial methanols
- About 500 traces in raw C2C methanol

Method Validation: TD-GCMS & Valibration Gas Generator



Figure 5 TD-GCMS (left) and calibration gas generator (right) used for method validation in Carbon2Chem®-Laboratory (Oberhausen).

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