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Non-radioactive Electron Capture Detection for Process and Quality Control in Brewery Production

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Summary:
In beer fermentation, diacetyl and 2,3-pentanedione levels must be precisely controlled, with thresholds below 0.10 mg/L. Due to this low concentration, the Central European Brewing Analysis Commission recommends gas chromatography coupled to an electron capture detector as a sensitive analytical method. The new non-radioactive X-ray ECD achieves the necessary detection limit and linearity comparable to conventional radioactive ECDs, eliminating regulatory burdens tied to radioactive sources and allowing continuous concentration monitoring over the entire fermentation process.

Keywords: Gas chromatography, Non-radioactive Electron Capture Detector, X-ECD, Brewery, Food

Introduction

In beer production, managing diacetyl (2,3-butanedione) and 2,3-pentanedione levels is crucial, as concentrations exceeding 0.10 mg/L cause undesirable off-flavors. These compounds are by-products of yeast metabolism and undergo gradual breakdown during fermentation. However, due to variations in natural ingredients, breweries without analytical monitoring instruments often extend fermentation times, impacting process efficiency. To improve monitoring accuracy, the Central European Brewing Analysis Commission recommends gas chromatography with an electron capture detector (GC-ECD) [1]. Traditional ECDs, however, rely on radioactive sources, raising regulatory and operational challenges for breweries.

A non-radioactive ECD, the X-ECD, was recently developed at Leibniz University Hannover [2]. It offers sensitivity and linearity comparable to traditional radioactive detectors but avoids regulatory restrictions for handling radioactivity. This study aims to validate the detector's performance and suitability for its use in brewery applications, showing a safer, more efficient option for in-production monitoring of critical fermentation compounds.

Experimental

The X-ECD is based on a compact high energy photoionization source, enabling ionization of the carrier gas (nitrogen or argon) to generate the necessary free electrons, similar to traditional radioactive sources. The X-ECD was coupled with a Shimadzu GC 2010, while incubation and injection of headspace samples were managed through a Shimadzu AOC-5000 Plus autosampler into a split injector, with detailed operational parameters listed in Table 1. A short, heated transfer line at 100 °C connected the GC column to the X-ECD. Helium served as the carrier gas, and Argon was added as a make-up gas through a T-connector.

Calibration was performed using solutions of diacetyl and 2,3-pentandione in water with 5% ethanol, replicating typical beer conditions. These solutions allowed precise testing of the detector's response to various concentration levels.

Additionally, real beer samples were analyzed under the same conditions to prove the absence of any disturbing matrix effects from coeluting substances.

Tab. 1: Operational parameters of the GC and the X-ECD.

Vial volume	20 mL
Liquid sample	10 mL
Incubation	10 min at 60 °C
Head space injection volume	1 mL
Injector temperature	150 °C

Split	1:5
GC-Column	HP5-MS (30 m, 0.25 mm, 50 µm)
Carrier Gas	0.9 mL/min Helium
Oven temperature	40 °C (3 min hold) 10 °C/min 80 °C (2 min hold)
Transfer temperature	100 °C
X-ECD temperature	100 °C
Make-up gas flow	50 mL/min Nitrogen
ECD modus	Constant-Current
ECD pressure	Open / atmosphere

Results

Figure 1 shows the chromatograms of headspace injections of a water ethanol mixture (95/5) containing different concentrations of diacetyl and 2,3-pentandione. Both compounds can be clearly identified by two clearly separated peaks. A response of the high amount of injected oxygen and ethanol appears much earlier than the analyte peaks, ensuring no baseline interference in the target area.

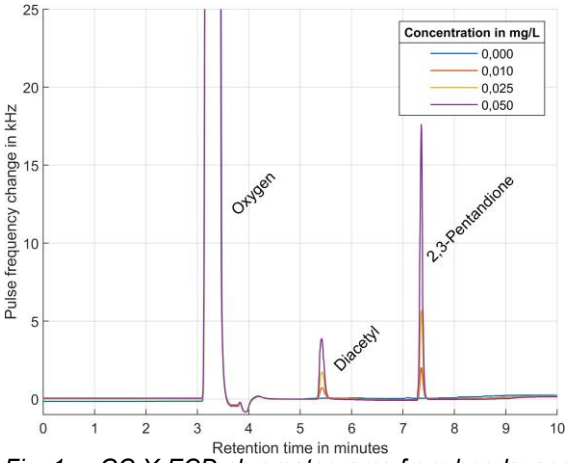

Fig. 1. GC-X-ECD chromatograms from headspace analysis of diacetyl and 2,3-pentanedione in an aqueous solution containing 5% ethanol, simulating a beer-like mixture.

The GC-X-ECD system detects concentrations as low as one-tenth of the threshold (0.1 mg/L). Our current work focuses on method development, enabling the full potential of the X-ECDs high dynamic range (starting already at a noise floor around 10 Hz in pulse frequency change) to cover the entire concentration range of the brewing process. This could enable real-time fermentation monitoring to adjust process parameters based on ingredient quality. Figure 2 shows measurements of two beer samples: one finished beer from the supermarket and one so called "young beer" with incomplete fermentation.

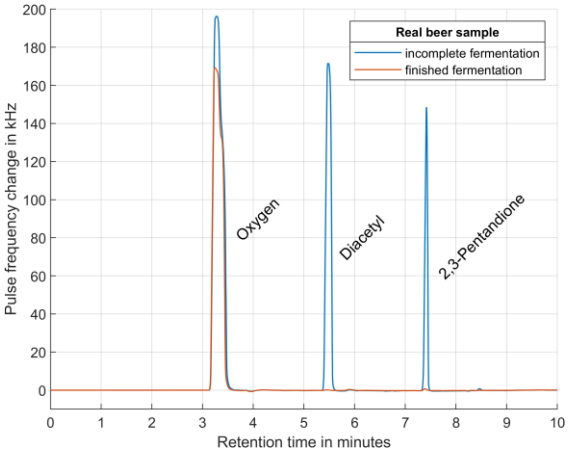

Fig. 2. GC-X-ECD chromatograms from headspace analysis of two real beer samples, comparing their profiles after incomplete and completed fermentation processes.

This illustrates the concentration variability present throughout the brewing process. Furthermore, the measurements demonstrate that no interfering substance peaks in real beer samples obstruct the identification and subsequent quantification of the two target compounds. In addition to achieving the required sensitivity and dynamic range, these results confirm the advantages of using an ECD over non-selective GC detectors, which may encounter challenges with coeluting substances within the complex beer matrix. Ongoing work aims to optimize method development to fully characterize the GC-X-ECD system, focusing on its limit of detection and linearity.

Acknowledgement

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