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Selective ionization of marker molecules in fuels by laser-based ion mobility spectrometry (LIMS)[†]Martin Zühlke,^a Leonard Genin, Daniel Riebe^b and Toralf Beitz

Careful quality control of complex matrices such as fuels and food is necessary due to the prevalence of counterfeit and pirated goods in global trade. The addition of taggants (indicator substances) to products or their packaging helps to ensure traceability. In order to prevent the mixing of different liquid products, such as different taxed fuels, invisible labelling (marker) can be used to detect illegal activities. This study investigates the qualitative and quantitative analysis of markers in complex fuel matrices using Resonance-Enhanced Multiphoton Ionization (REMPI) Ion Mobility Spectrometry (IMS). The potential of REMPI as a selective ionisation technique for the detection of markers is highlighted, particularly with respect to minimizing matrix background and the possibility of detection without chromatographic pre-separation. Finding a suitable marker–wavelength combination that provides a suitable marker-to-matrix ratio allows selective ionization of markers while minimising matrix background. Matrix analysis shows that higher excitation wavelengths result in reduced matrix signals, with the low intensities observed at 355 nm for diesel and petrol matrices. Several candidate markers are evaluated based on the criteria of intense signal at 355 nm and non-leachability for the low tax labelling. The analytical performance of selected markers is evaluated, with a focus on the charge transfer reaction (CTR) between markers and matrix components. Our findings demonstrate the potential of REMPI-IMS for marker analysis in fuels without the need for chromatographic pre-separation, providing a promising approach for detecting illegal or fraudulent activities in the supply chain.

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1. Introduction

Complex matrices such as food products, cosmetics or pharmaceuticals require rigorous quality control to ensure the safety and traceability of the products' ingredients.^{1,2} The Organisation for Economic Co-operation and Development (OECD) estimates that counterfeit and pirated goods accounted for up to 2.5% of global trade in 2019, and estimates an increase in subsequent years.³ The loss of tax revenue, the damage to various branded companies and the health risk to the consumers can be immense. To monitor the process chains, indicator substances (so-called taggants) can be added to the ingredients or their packaging at any point in the production process.⁴ In the best case scenario, this ensures complete labelling from the origin of each raw material right through to the product, thus preventing counterfeiting. Products that have been traded illegally (tax-free) or plagiarized can thus be identified and withdrawn from circulation. Tagging options include, for example, magnetic inks, fluorescent labels, photochromic and thermochromic coatings, isotopic tracers and Raman-

active components.^{5,6} Most of these physical methods are designed for labelling solid surfaces or are incorporated directly into the product during manufacture.

Blending of different taxed fuels is a prominent example of tax avoidance worldwide. Mixing untaxed fuels (e.g. from crude oil theft, fuel piracy and illegal trade) with low or high taxed fuels can increase profits enormously. While the regular tax on petrol or diesel is paid by the customer, the full tax has not been previously paid and therefore represents an additional profit for the distributor. As a result, oil producing and processing countries as well as the petrol industry add unique chemical signatures or a concentrate of an indicator solution to a fuel (known as tagging) in order to help track its distribution and identify any illegal or fraudulent activity in the supply chain.^{7–9} Different taggants can be used to label the fuel at various points including refineries, terminals, or blending facilities. The requirements for potential taggants are high: inert, non-leachable, inexpensive, easy to detect, safe to use and without an effect on the product's properties. To detect diluted or contaminated products, the marker must also be quantifiable in the complex matrix and tolerant of matrix changes.¹⁰ Tagging substances can be distinguished between covert (invisible markers) and overt (visible dyes).¹¹ Commonly used qualitative and quantitative laboratory analyses are based on a pre-separation step, usually performed by high performance

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[†] Electronic supplementary information (ESI) available. See DOI: 10.1039/D3AY01994B



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CH Cherryholmes

A red circular graphic with a gradient, appearing as a semi-circle or a partial circle, located to the right of the CH Cherryholmes text.

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m mix m 1 v 1 m 2 v 2 v 1 v 2 1 5 x 480 1 2 x 520 480 520 720 624 1000 1 344 m q5 with increase in temperature which of these changes a molality b fraction of solute present in water

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