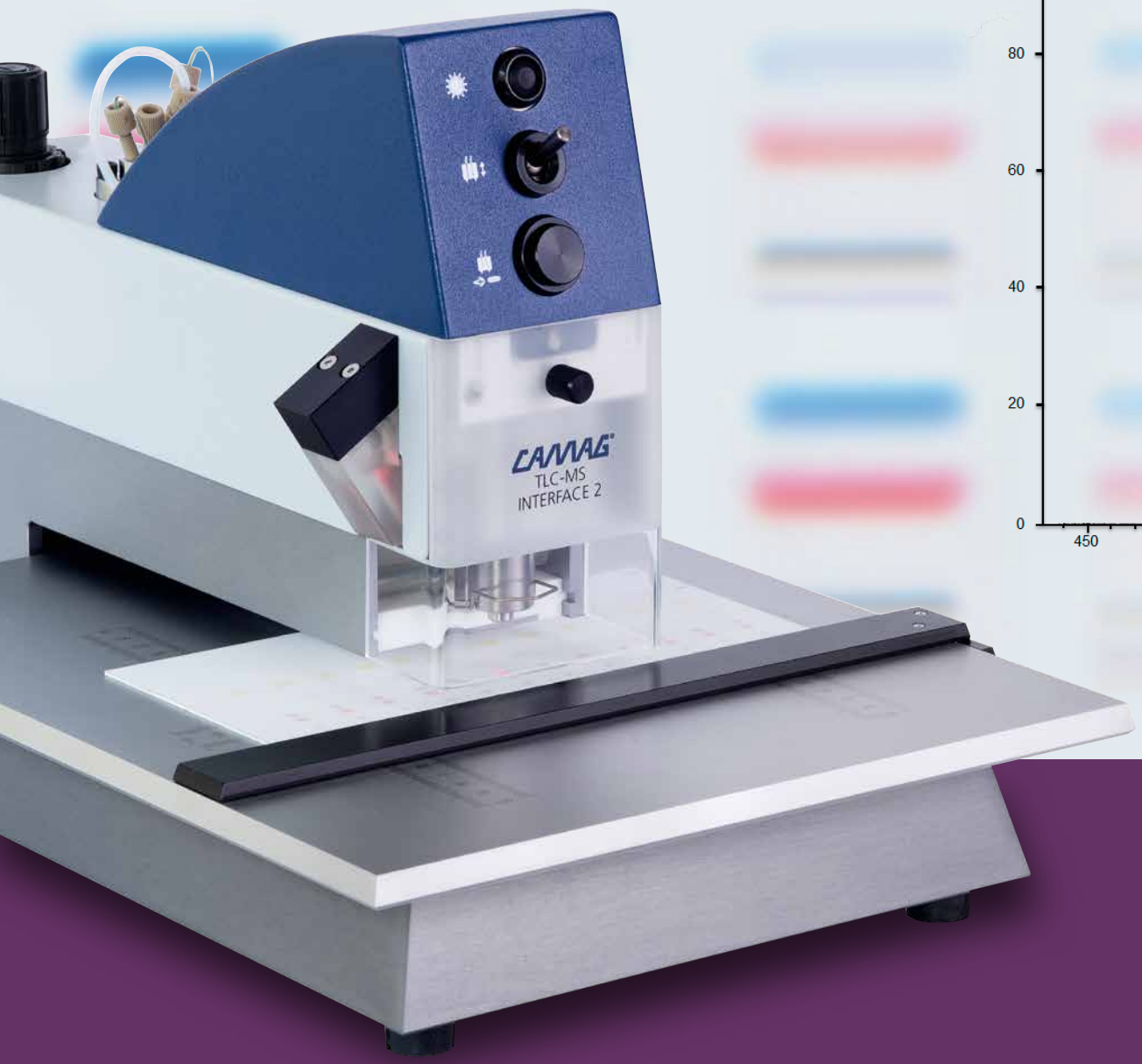
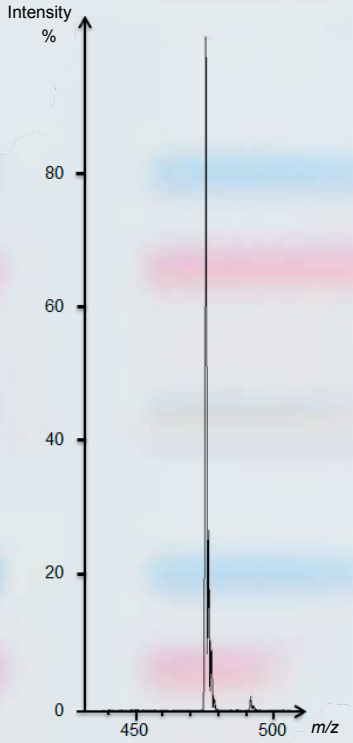
CAMAG
TLC-MS INTERFACE 2





CAMAG® WORLD LEADER IN PLANAR CHROMATOGRAPHY

IDENTIFICATION AND CONFIRMATION OF UNKNOWN SUBSTANCES

The elution-based TLC-MS Interface 2 is a highly convenient and versatile instrument allowing for rapid and contamination-free elution of TLC/HPTLC zones with online transfer to a mass spectrometer. Through the pioneering concept of hyphenating high-performance thin-layer chromatography with mass spectrometry unequivocal substance identification is possible. The TLC-MS Interface 2 can be installed plug & play with any LC-MS system without adjustments or mass spectrometer modifications. Depending on the MS system, a substance can be identified within a minute via its mass spectrum, or for an unknown substance zone, the respective sum formula can be obtained. Furthermore, interesting zones can be eluted into vials for further investigations with, *e.g.*, NMR, (ATR-)FTIR, ESI-MS, and MALDI-MS.

The chromatogram zones are eluted from the HPTLC plate with methanol or another suitable solvent with the flow speed appropriate for the LC-MS system. The round elution head is used for circular zones and the oval elution head for zones in the form of bands. After elution the eluate is either transferred online to the mass spectrometer or collected in a sample vial for further offline analysis.

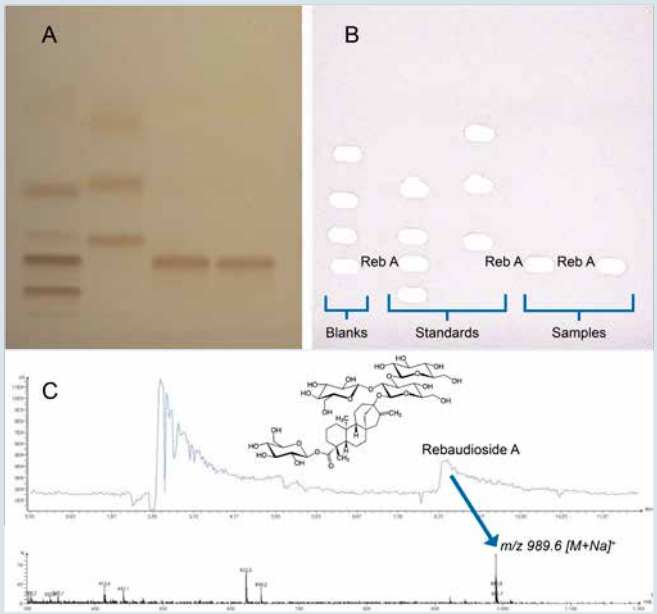
The TLC-MS Interface 2 features a modified elution head and an easily accessible, exchangeable filter, attached directly at the 6 port valve. Cleaning is facilitated as compared to the previous version, making it highly efficient. By pushing a button, the elution path is cleaned of matrix particles with compressed air, increasing the lifetime of the filter and preventing the system from becoming blocked. Filters can be easily replaced without any modification to the elution head.

Plate positioning is significantly simplified: together with the positioning scale and the adjustable plate-stopper, the integrated crosshair laser enables accurate and reproducible positioning of the plate. Alternatively the coordinates determined by the TLC Visualizer or the TLC Scanner can be used to precisely position the HPTLC plate.

Application notes can be found on **www.camag.com/applications.**

KEY FEATURES

- Rapid and contamination-free elution of selected zones
- Online transfer to the mass spectrometer
- Plug & play installation
- Compatible with any given HPLC-MS system
- Confirmation of known substances within a minute
- Low solvent consumption
- Highly effective backwashing function prevents the elution path from becoming blocked
- Easy handling ensures accurate and reproducible plate positioning



Characterization of separated compounds by mass spectrometry (Steviol glycosides in Stevia formulations)*

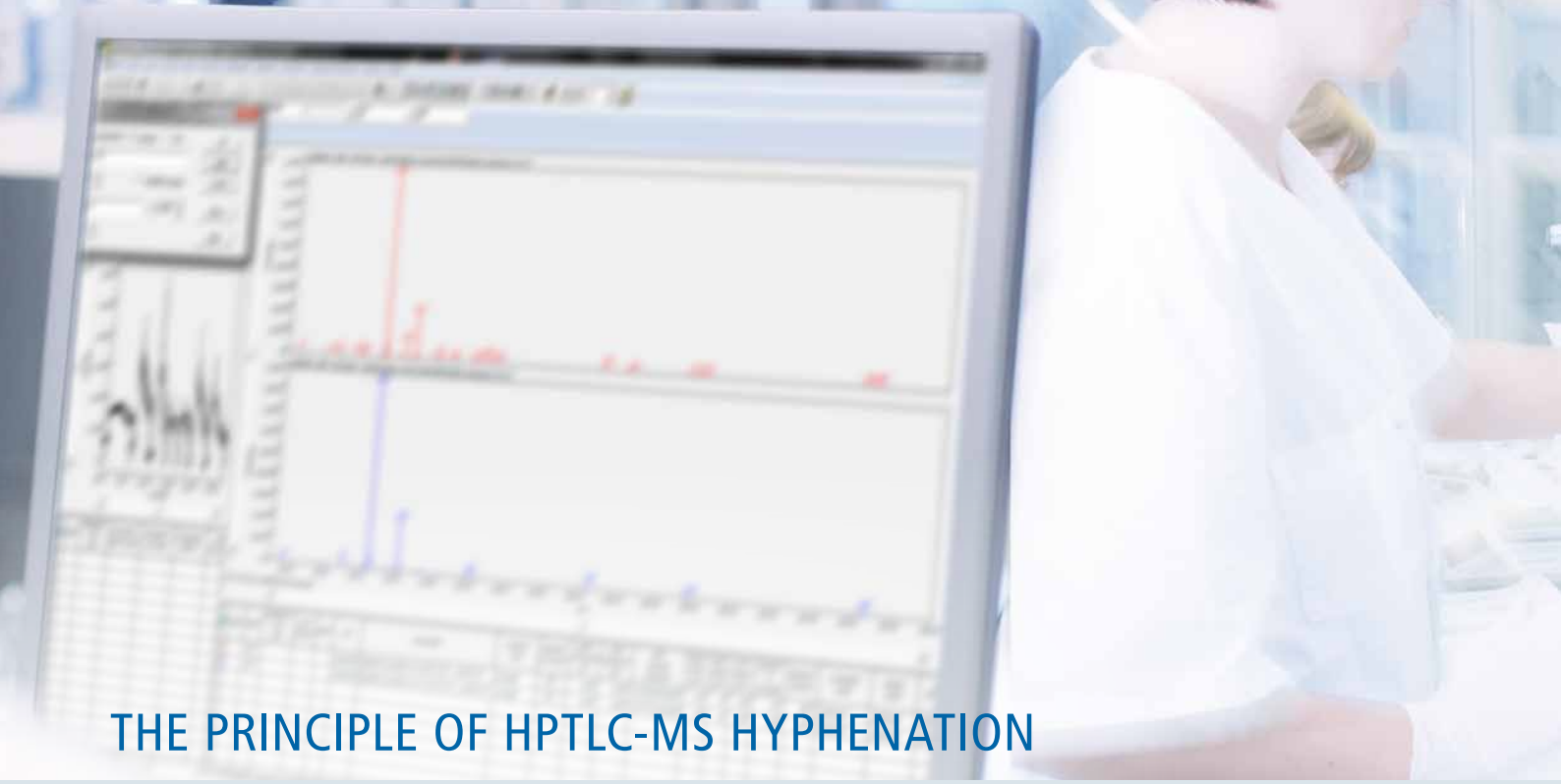
A: Chromatogram for localizing the zones (derivatized with β-naphtol reagent)

B: HPTLC plate after elution of zones with the CAMAG TLC-MS Interface 2

C: HPTLC-ESI-MS spectra of Rebaudioside A, m/z 989.6 $[M+Na]^+$

**Morlock et al., Journal of Chromatography, A, 1350 (2014) 102–111*

For further information, go to **www.camag.com/stevia** or scan the QR code.



THE PRINCIPLE OF HPTLC-MS HYPHENATION

Solvent

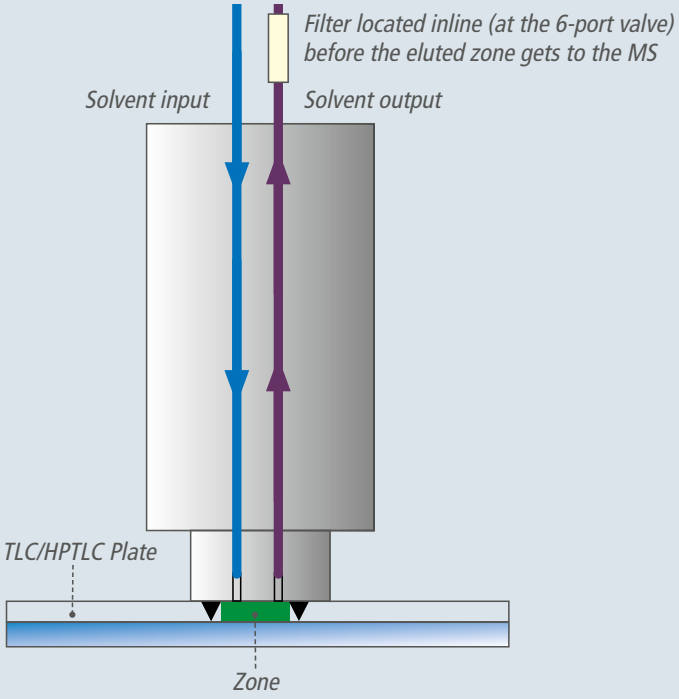
HPLC-Pump
Flow rate 50–300 µL/min

MS-System

TLC-MS Interface 2

THE PRINCIPLE OF THE TLC-MS INTERFACE 2

After lowering the elution head and switching the valve to the elution position, the solvent moves through the elution head and elutes the zone. The eluate is directed through a capillary and an in-line filter to the MS system. To activate the backwashing function that prevents the elution path and the filter from blocking the valve must be switched to the "bypass position" after the elution.



Technical specifications

Dimensions and weight with standard plate support
Width: 275 mm, depth: 425 mm, height: 275 mm
Net weight: 14.5 kg

Laser crosshairs
Laser: 5 mW, class 2M, battery operated (two batteries 1.5 V, AA or LR6), operating time on batteries: up to 100 hours

Materials
- Elution head: made of passivated stainless steel, resistant to all common solvents
- Filter: Column Saver 2 µm

Requirements
- Gas connection: compressed air or Nitrogen 4–6 bar
- Solvent flow rate: 50–300 µL/min
- Pressure of elution head onto HPTLC plate: max 400 N

Elution heads

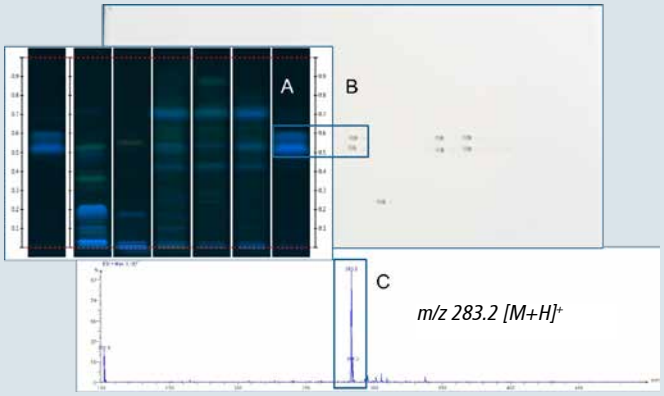
Elution head oval, 4 x 2 mm
for layer thickness up to 0.25 mm

Elution head round, Ø 4 mm
for layer thickness up to 0.25 mm

APPLICATION EXAMPLES

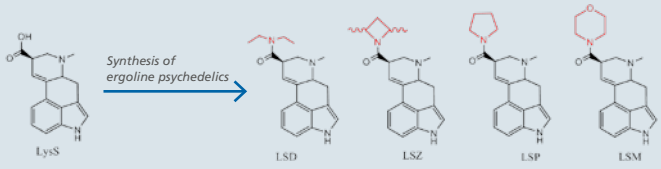
Rapid HPTLC-MS identification of synthesis products

HPTLC-MS is a very fast way for confirmation of substances during chemical synthesis and with low consumption of solvents.



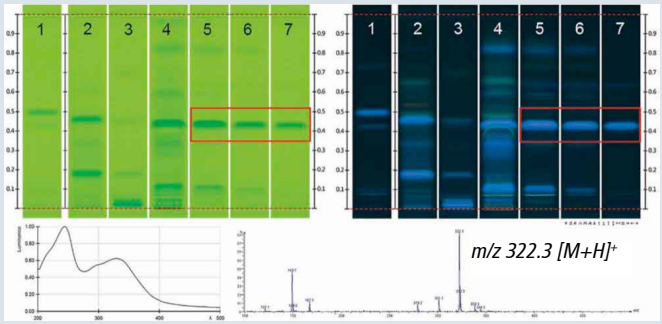
Identification of ergoline psychedelics during chemical synthesis and purification
A: Chromatogram of LSZ synthesis samples under UV 366 nm for localizing the zones
B: HPTLC plate after elution of zones with the CAMAG TLC-MS Interface 2
C: HPTLC-ESI-MS spectra of LSZ, m/z 283.2 [M+H]+

Synthesis of ergoline psychedelics: in a first step the lysergic acid (LysS) is produced with ergotamine as raw material. Then the formation of different amides takes place, with LSD, LSZ, LSP, and LSM as finished products. During column purification, a purity > 99% is achieved. The identity can be rapidly confirmed by HPTLC-MS.



LysS is the starting material for the synthesis of LSD, LSZ, LSP, and LSM; different side chains are highlighted in red

HPTLC is performed on silica gel 60 F_{254}, the zones are localized under UV 366 and then directly eluted with the TLC-MS Interface 2 and methanol (with 0.1 % formic acid, flow rate 0.2 mL/min) to any ESI-MS. The obained mass spectra shows the expected mass for LSP (*m/z* 322.3 [M+H]+).



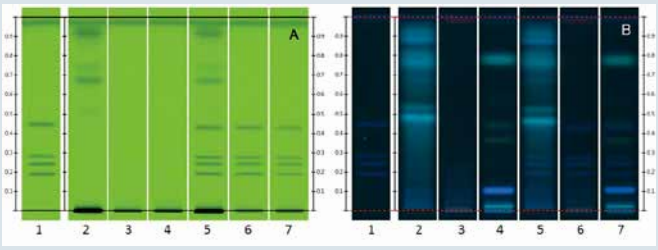
In-process control of the LSP: Chromatograms under UV 254 nm and UV 366 nm show the sequence of the synthesis and the purification steps (track 1: LSD as reference, 2: digest of the lysergic acid from ergotamine, 3: purified lysergic acid (starting material for the chemical synthesis), 4: crude synthesis product (LSP and side products), 5, 6: column purification steps, 7: finished product as well as the UV spectra and the mass spectra of the eluted LSP zone at m/z 322.3 [M+H]+

According to the chromatograms the improved purity grade during the purification workup can be easily observed in the example of the LSP. During process development for a new synthesis product, valuable information about the individual process steps can be obtained by HPTLC. Visual evaluation of the chromatograms enables a rapid observation on the formation or up-/downscaling of the different components during each step.

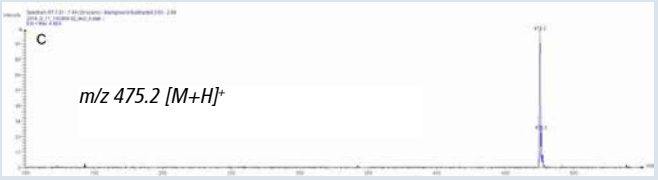
Collaboration between Lipomed AG, Switzerland and CAMAG

For further information, go to
www.camag.com/cbs115
or scan the QR code.

HPTLC-MS for identification of PDE5-Inhibitors in lifestyle products



Chromatogram under (A) UV 254 nm and (B) UV 366 nm, track 1: sildenafil, propoxyphenyl hydroxyhomosildenafil, homosildenafil, hydroxyhomosildenafil with increasing hR_f; tracks 2–4: plants extracts; tracks 5–7: spiked plant extracts



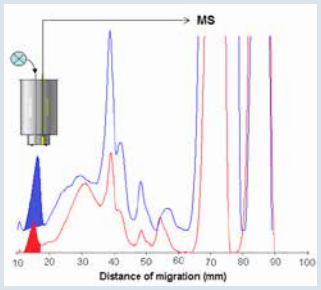
(C) HPTLC-ESI-MS spectra of sildenafil

The sale of "Natural Aphrodisiacs" seems to be a prospering business. Frequently these products are adulterated with synthetic phosphodiesterase type 5 inhibitors (PDE5-Inhibitors), including the active principles of Viagra® (sildenafil), Levitra® (vardenafil), and Cialis® (tadalafil).

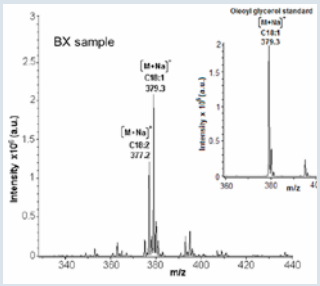
HPTLC can be used for rapid screening of commercial products for adulteration with 11 known PDE5-Inhibitors. It is applicable to a variety of finished products without interference from matrix and excipients. The identity of PDE5-Inhibitors can be confirmed by mass spectrometry using the TLC-MS Interface 2 and any ESI-MS.

For further information, go to
www.camag.com/cbs114
or scan the QR code.

Determination of monoacylglycerides in biodiesel by HPTLC-MS



Densitogram comparison of B5 (red) and B20 (blue), both 5000 µg/band: monoacylglyceride bands at 16 mm were eluted via the TLC-MS Interface 2 into the ion trap MS



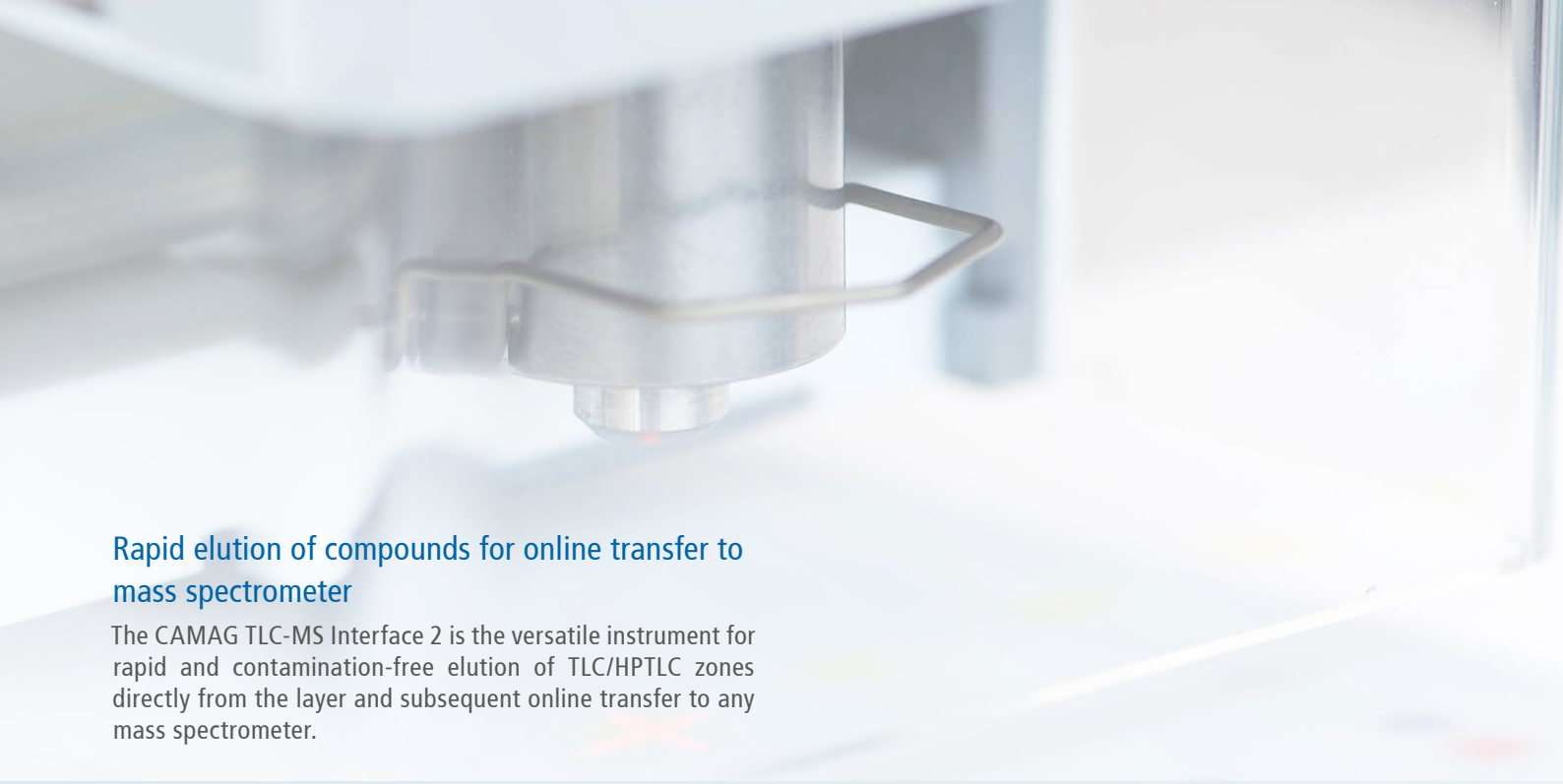
HPTLC-MS full scan mass spectrum of the monoacylglyceride band in the B100 (16 mm) sample and of the oleoyl glycerol standard

A reliable characterization of impurities in biodiesel (according to the European standard UNE EN 14214:2013) and the concept for identity confirmation of monoacylglycerides by mass spectrometry, which allows origin identification are presented.

A fatty-acid methyl ester content lower than 98 wt% indicates inappropriate reaction conditions for biodiesel production, and therefore, the presence of impurities in the finished product. One impurity class in biodiesel is monoacylglycerides, which can produce obstruction in fuel filters.

The zones for MS are localized by the coordinates obtained with the TLC Scanner 4 (fluorescence detection) or with the TLC Visualizer under UV 366 nm. Zones are eluted with methanol via the TLC-MS Interface 2 into an ion-trap MS operating in positive ionization mode.

For further information, go to
www.camag.com/biodiesel
or scan the QR code.



Rapid elution of compounds for online transfer to mass spectrometer

The CAMAG TLC-MS Interface 2 is the versatile instrument for rapid and contamination-free elution of TLC/HPTLC zones directly from the layer and subsequent online transfer to any mass spectrometer.

Ordering information

022.8440 **CAMAG® TLC-MS Interface 2**
including *oval elution head 4 x 2 mm* (mounted), for elution of substances from TLC/HPTLC layers, semi-automatic instrument involving automatic elution head movement, cleaning of the elution path with compressed air, manual positioning and switching

022.8441 **CAMAG® TLC-MS Interface 2**
including *round elution head Ø 4 mm* (mounted), for elution of substances from TLC/HPTLC layers, semi-automatic instrument involving automatic elution head movement, cleaning of the elution path with compressed air, manual positioning and switching

022.8445 Elution head oval 4 x 2 mm, h = 0.25 mm, for CAMAG® TLC-MS Interface 2

022.8446 Elution head round Ø 4 mm, h = 0.25 mm, for CAMAG® TLC-MS Interface 2

022.8450 Filter: Column Saver 2 µm, SST Frit Interface 2

022.8460 "Push Start" for MS data acquisition to CAMAG® TLC-MS Interface 2

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