



CLEAR CHOICE HIGH PURITY SOLVENTS FOR CHROMATOGRAPHY FROM MERCK

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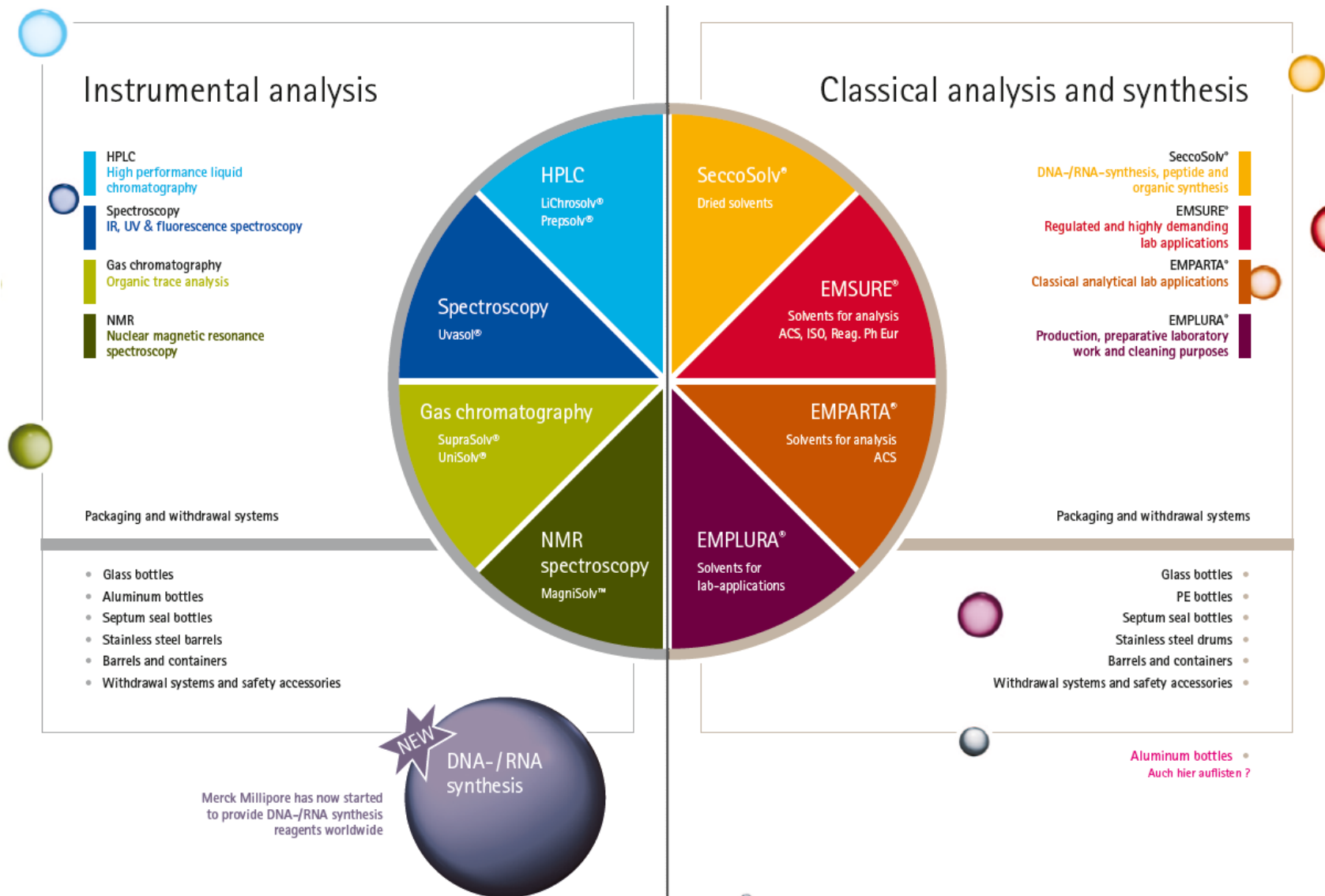
Kaunas

24.10.2017

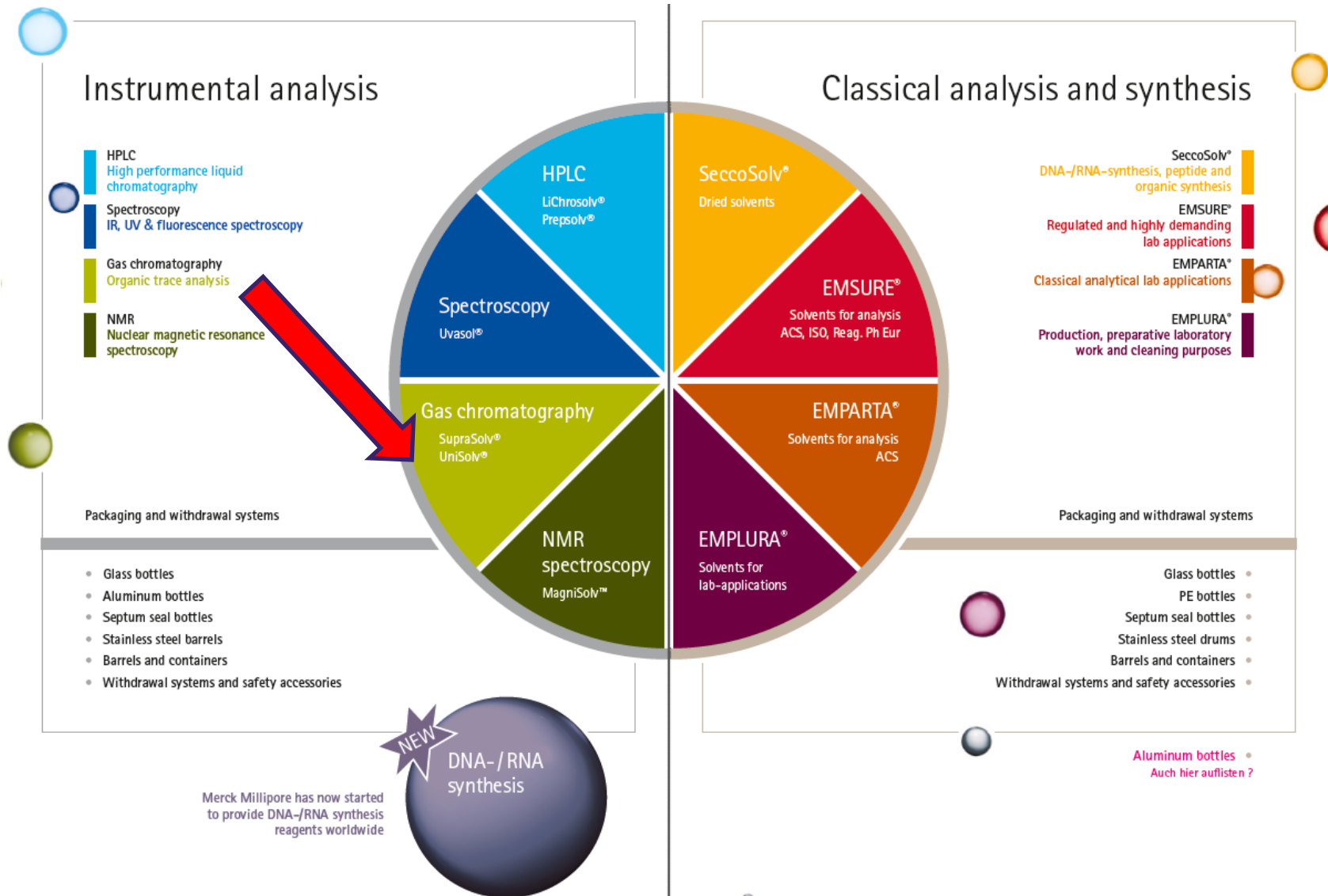


MERCK

Solvents for Instrumental Analysis



Solvents for gas chromatography – Suprasolv® and Unisolv®

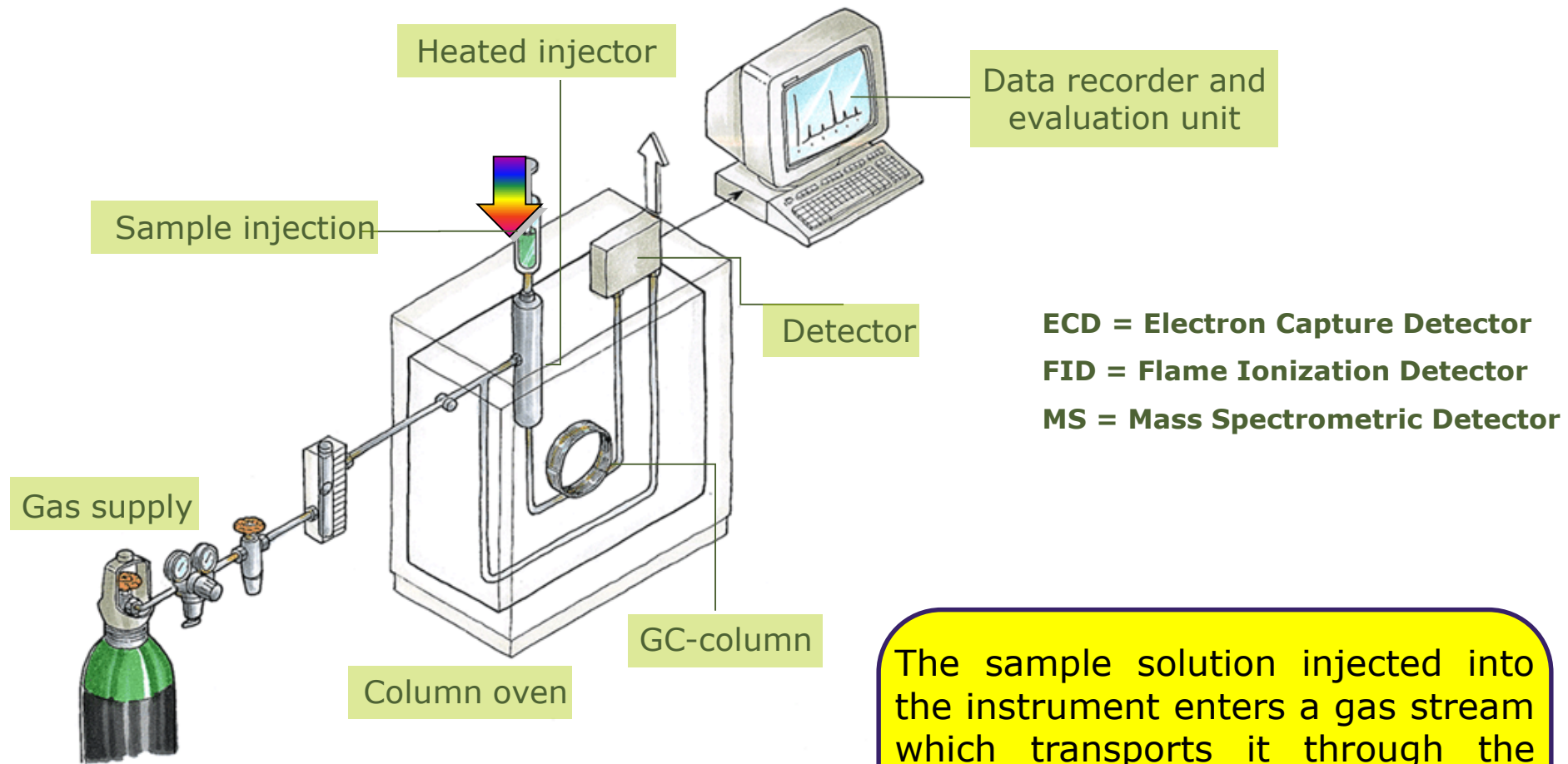


SOLVENTS FOR GASS CHROMATOGRAPHY SUPRASOLV[®] / UNISOLV[®]

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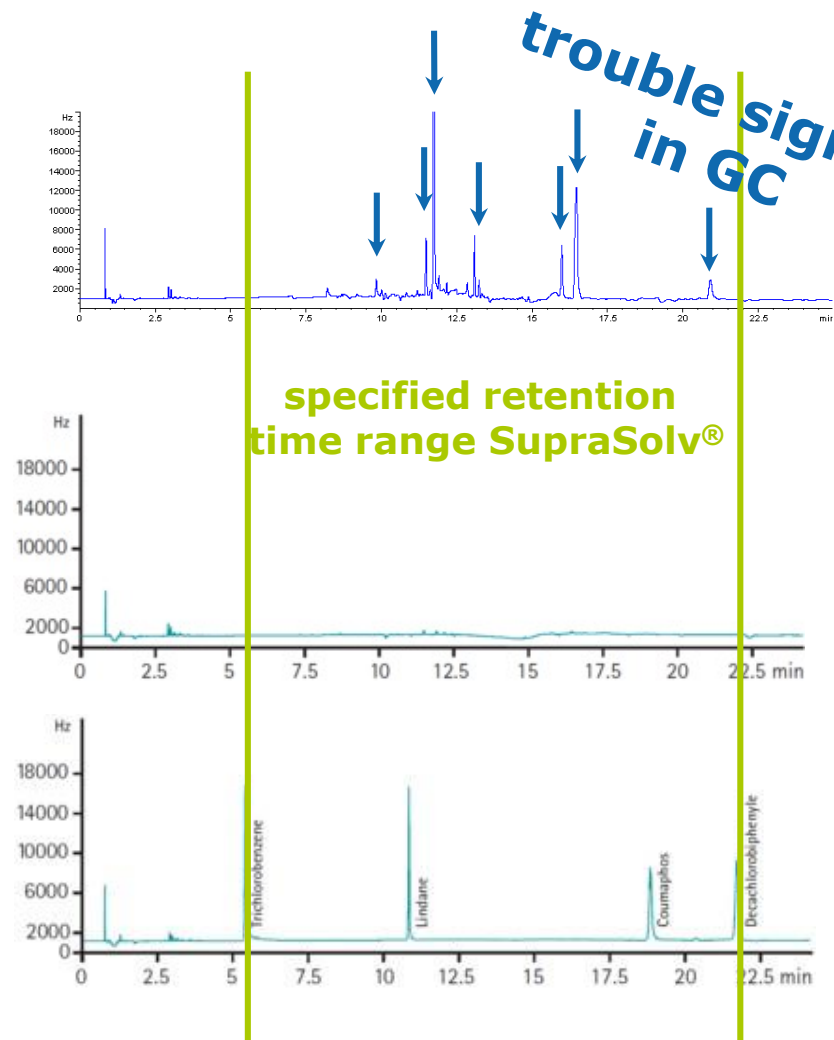
The principle of gas chromatography



ECD = Electron Capture Detector
FID = Flame Ionization Detector
MS = Mass Spectrometric Detector

The sample solution injected into the instrument enters a gas stream which transports it through the column. The column separates the various components. The detector measures the quantity of the components that exit the column.

Why offering a special GC grade?



n-Hexane, purity (GC) 99.9%

Reagent grade
not specified for GC-application

n-Hexane SupraSolv® ECD
and FID, Merck Millipore
1.04371

standard

Benefit:

Reliable and reproducible results due to constant minimal signal-noise-ratio (clear baseline)

SupraSolv® for Gas Chromatography

An Application Oriented program

SupraSolv

For all your needs in gas chromatography

FID

flame ionization detector

- BTX (Benzene, Toluene, Xylene) – highly volatile aromatic hydrocarbons in sewage, ground-water, juices, canned fish etc.
- Hydrocarbon-oil index in water
- Determination of emissions in car cockpit material



ECD

electron capture detector

- Pesticide analysis in fruits and vegetables
- Acrylamide in e.g. potato chips, crisps and crisp bread
- Polychlorinated biphenyls (PCB) in water and sludge
- DDT (preserver and insecticide) in milk, fish, meat, fruits etc.
- Highly volatile halogenated hydrocarbons in water
- Nitrate in lettuce, radish etc.



MS

mass spectrometry

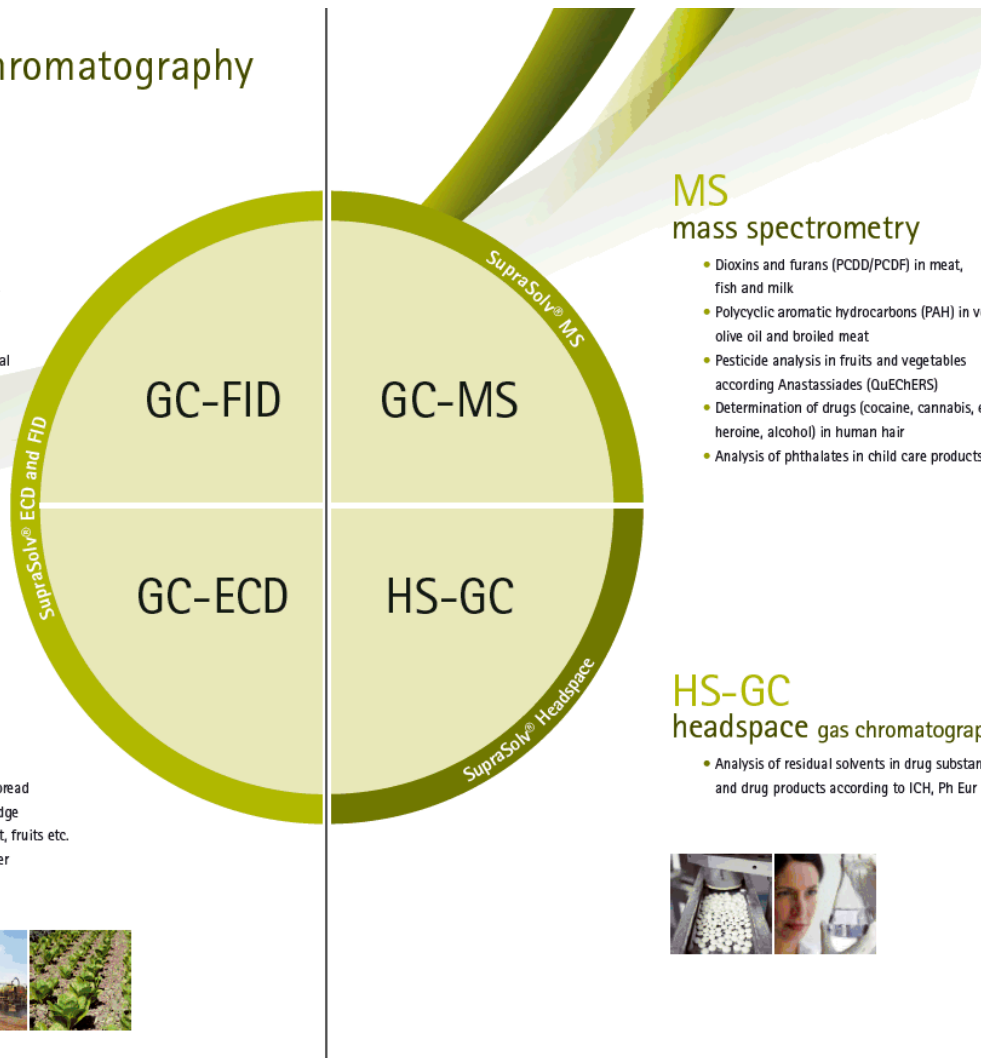
- Dioxins and furans (PCDD/PCDF) in meat, fish and milk
- Polycyclic aromatic hydrocarbons (PAH) in vegetables, olive oil and broiled meat
- Pesticide analysis in fruits and vegetables according Anastassiades (QuEChERS)
- Determination of drugs (cocaine, cannabis, ecstasy, heroine, alcohol) in human hair
- Analysis of phthalates in child care products and toys



HS-GC

headspace gas chromatography

- Analysis of residual solvents in drug substances, excipients, and drug products according to ICH, Ph Eur and USP



Solvents for GC - SupraSolv® & UniSolv®

SupraSolv®

For headspace GC



Application:

Analysis of residual solvents in drug substances and products acc. Ph Eur & USP

UniSolv® for organic trace analysis

For ECD, FID and MS

„One for All“

SupraSolv® ECD and FID

For gas chromatography ECD and FID

Applications

- Pesticide analysis
- Volatile halogenated hydrocarbons in water
- Polychlorinated biphenyls (PCB) in water
- Detection of Acrylamide in food

Applications

- BTX (Benzene, Toluene, Xylene) detection
- Hydrocarbon-oil-index in water
- Determination of emissions in car cockpit materials

SupraSolv®

MS For GC-MS

Applications

- Analysis of Dioxins and Furans (PCDD/PCDF)
- Polycyclic aromatic hydrocarbons
- Pesticide analysis acc. QuEChERS
- Determination of phthalates in plastics

ECD

Electron Capture
Detector

FID

Flame Ionization
Detector

MS

Mass Spectrometric
Detector

Dedicated Specifications SupraSolv® & UniSolv®

1.04371.0000 n-Hexane for gas chromatography ECD and FID
SupraSolv®

	Spec. Values	
Purity (GC)	≥ 98.0	%
Sum of hexane isomers + methylcyclopentane (GC)	≥ 99.8	%
Identity (IR)	conforms	
residue on evaporation	≤ 3.0	mg/l
Water	≤ 0.01	%
Colour	≤ 10	Hazen
GC/ECD (retention range 1,2,4-trichlorobenzene to decachlorobiphenyle individual signals (lindane standard))	≤ 3	pg/ml
GC/FID (retention range n-undecane - n-tetracontane individual signals (n- tetradecane standard))	≤ 3	ng/ml

suitable for residue analysis

1.00795.1000 n-Hexane for gas chromatography MS SupraSolv®

	Spec. Values	
Purity (GC)	≥ 98.0	%
Sum of hexane isomers + methylcyclopentane (GC)	≥ 99.8	%
Identity (IR)	conforms	
residue on evaporation	≤ 3.0	mg/l
Water	≤ 0.01	%
Colour	≤ 10	Hazen
GC/MSD (retention range n-undecane - n-tetracontane; scanning area 30 -600 amu individual signals (n- tetradecane standard))	≤ 3	ng/ml

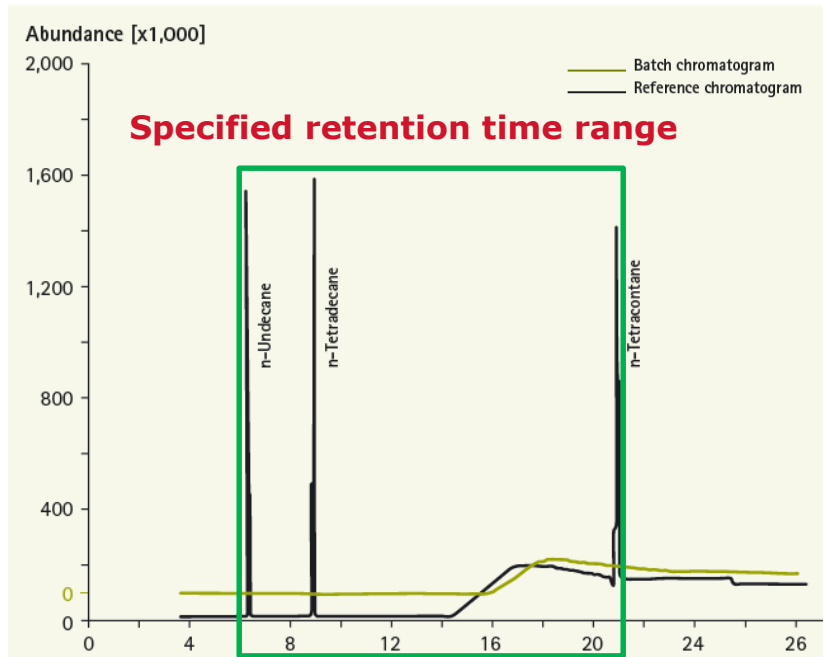
Suitable for residue analysis.

1.04369.0000 n-Hexane for organic trace analysis UniSolv®

	Spec. Values	
Purity (GC)	≥ 99.0	%
Sum of hexane isomers + methylcyclopentane (GC)	≥ 99.9	%
Identity (IR)	conforms	
residue on evaporation	≤ 3.0	mg/l
Water	≤ 0.005	%
Colour	≤ 10	Hazen
GC/ECD retention range 1,2,4-trichlorobenzene to decachlorobiphenyle individual signals (lindane standard)	≤ 2	pg/ml
retention range dichloromethane to 1,2,4-trichlorobenzene individual signals (tetrachloromethane)	≤ 1	ng/ml
GC/FID (retention range n-undecane - n-tetracontane individual signals (n- tetradecane standard))	≤ 2	ng/ml
GC/MSD (retention range n-undecane - n-tetracontane; scanning area 30 -600 amu individual signals (n- tetradecane standard))	≤ 2	ng/ml

Recommended for analysis of polychlorinated Dibenzodioxins and polychlorinated Dibenzofurans (PCDD/F).

SupraSolv® MS – marks the difference



Specification

1.00837.2500 Methanol for gas chromatography MS SupraSolv®

Specification

Purity (GC)	≥ 99.8	%
Identity (IR)	conforms	
Evaporation residue	≤ 3.0	mg/l
Water	≤ 0.1	%
Colour	≤ 10	Hazen
GC/MSD (retention range n-undecane to n-tetracontane, scanning area 30 - 600 amu, individual signals (n-tetradecane standard))	≤ 3	ng/ml

Suitable for residue analysis.

- **Clear baseline** – accurate, reliable & reproducible results (no risk of misinterpretation, no loss of valuable samples, no need for repeat analysis)
- **Batch-to-batch consistency** – time and cost saving
- **Application tested quality** – application security

Target groups

SGS



eurolins

Contract Labs

Food analysis
Environmental analysis
Pharma analysis



Water & Environmental

Drinking water, (waste) water and soil analysis

Nestlé

kraft foods
make today delicious

MARS



Food & Beverage

Quality Control (e.g. pesticide analysis, dioxin detection, etc.)

Coca-Cola



sanofi aventis



Pfizer

Roche

EMDSerono
Living science, transforming lives

MSD

Pharmaceutical Industry

Quality Control & R&D
(e.g. analysis of residual solvents)



P&G

DOW

Chemical Industry

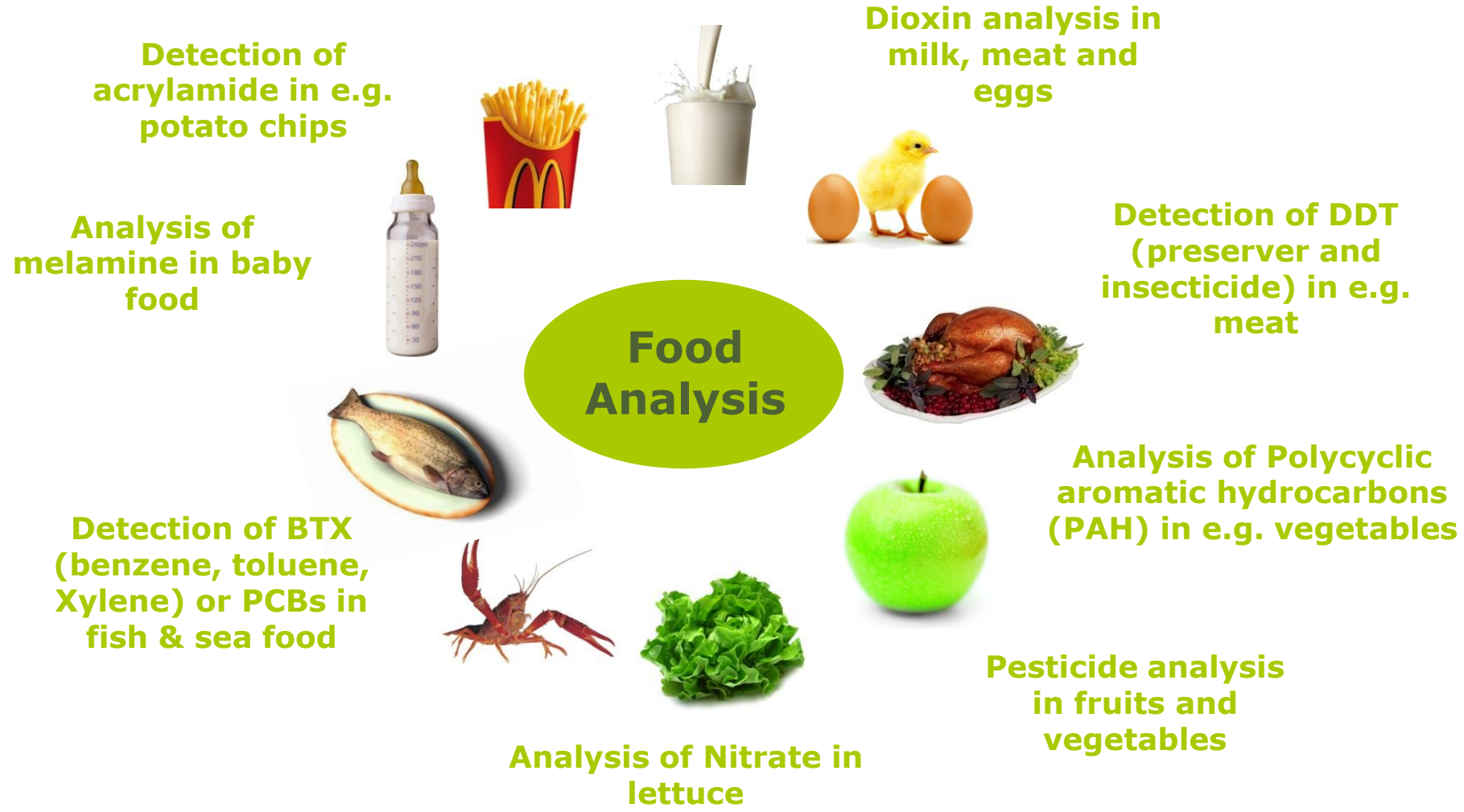
QC, R&D
Waste water control



Academia

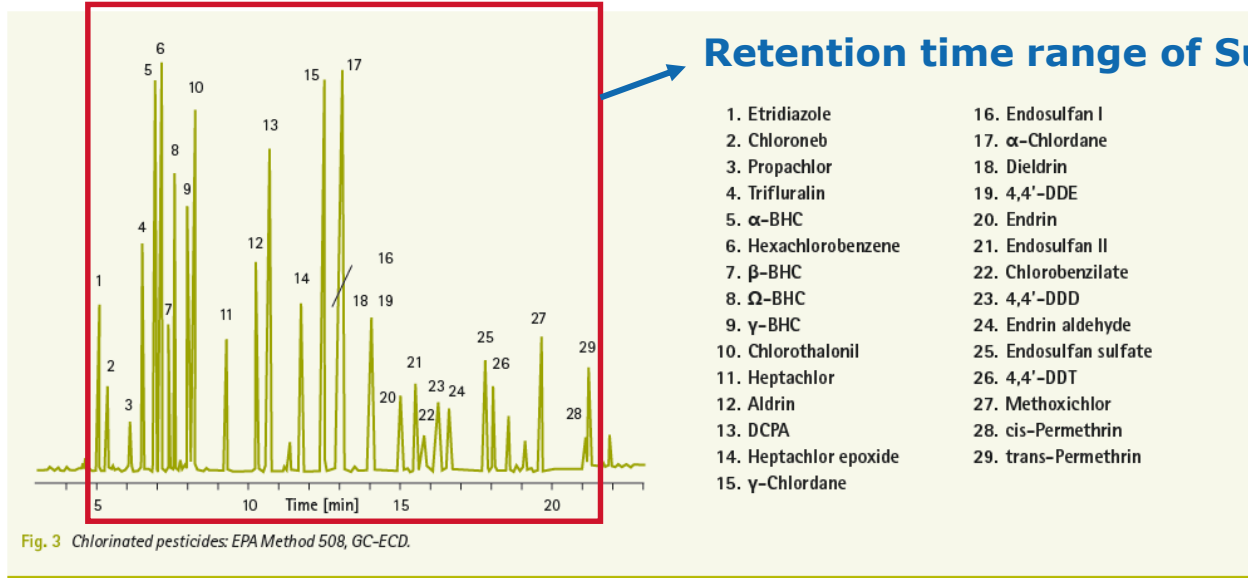
R&D

GC applications Food & Beverage Industry



Determination of pesticides in food & water according EPA method 508 by GC-ECD

EPA Method 508: Determination of chlorinated pesticides in water, standard chromatogram



■ Solvents:

- 1.00012 Acetone ECD and FID SupraSolv®
- 1.06054 Dichloromethane ECD and FID SupraSolv®
- 1.01995 tert-Butyl methyl ether ECD and FID SupraSolv®



Determination of Pesticides in Crops using the QuEChERS method



- Method: GC-MS, LC/MS-MS
- Regulation: EN 15662:2007 : Foods of plant origin – Determination of pesticide residues using GC-MS and/ or LC-MS/MS - QuEChERS-method
- Solvent: 1.00665 Acetonitrile MS SupraSolv

- QuEChERS-method:

- Worldwide used method for food control
- Introduced in 2003 for pesticide residue analysis
- Provides high quality results in a fast, easy, and inexpensive approach
- Follow-up studies have further validated the method for > 200 pesticides

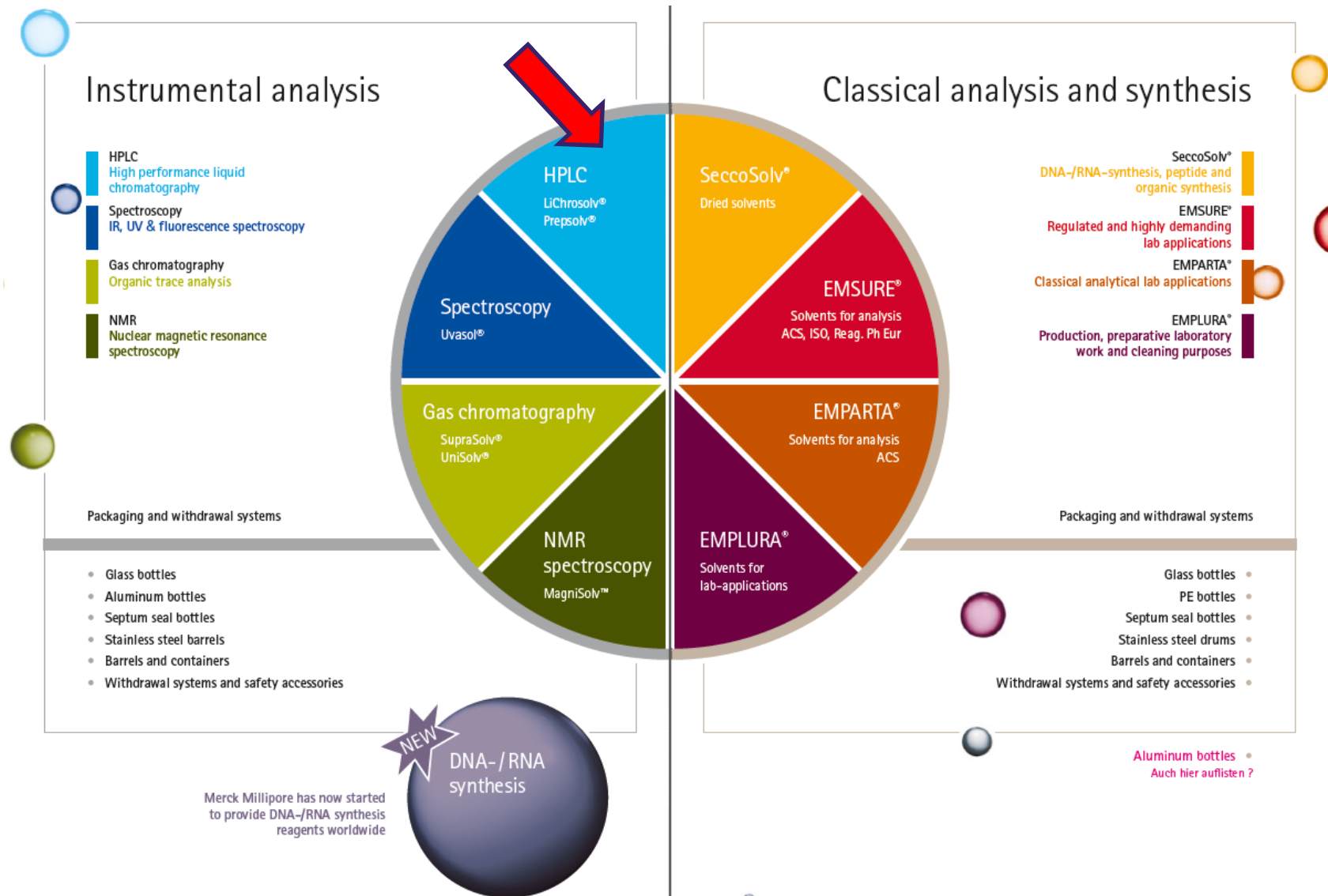


SupraSolv® & UniSolv® mark the difference

- **Lowest impurities/ clear baseline** – accurate, reliable & reproducible results (no risk of misinterpretation, no loss of valuable samples, no need for repeat analysis)
- **Widest retention time range** – most comprehensive range of applications
- **Batch-to-batch consistency** – time and cost saving (avoidance of analysis repetition)
- **Application tested quality** – application security
- **Flexibility** – Top-products are also offered in 10L stainless steel barrels as standard pack size
- **UniSolv®** is suitable **for all three main detection methods ECD, FID & MS** – one solvent quality for all applications

UNIQUE !

Solvents for analytical liquid chromatography - LiChrosolv®



HPLC : High Performance (Pressure) Liquid Chromatography

Analytical technique to separate, identify & quantify chemical & biological compounds.



Separation is depending on:

- Type of analysed substance
- Type of column
- Temperature
- pH value
- Flow rate of mobile phase
- Composition of mobile phase (solvent)

HPLC – what can you do with it?

Qualitative analysis

Identification of compounds by their retention time (= time it takes to elute from the column after injection) and/or spectra in MS/UV detector

Quantitative analysis

Measurement of compound concentration via peak height and peak area

Preparative chromatography

Preparation of pure compound by collecting / concentrating the compound for further studies

Trace analysis

Of extremely low concentrations of harmful or toxic compounds with high resolution separations and very sensitive detectors

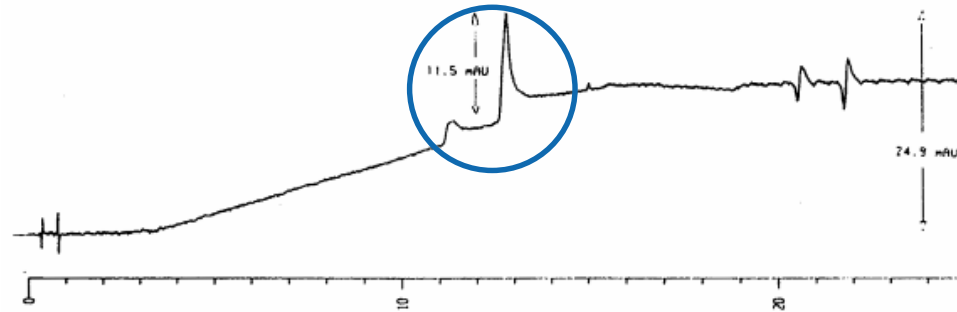
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SOLVENTS FOR LIQUID CHROMATOGRAPHY LICHROSOLV® AT A GLANCE



Why using a special HPLC grade?

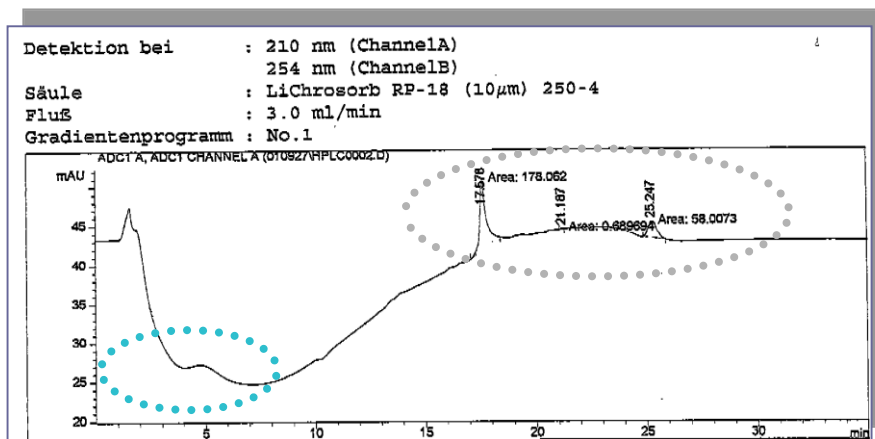
Solvent impurities can result in ghost peaks !



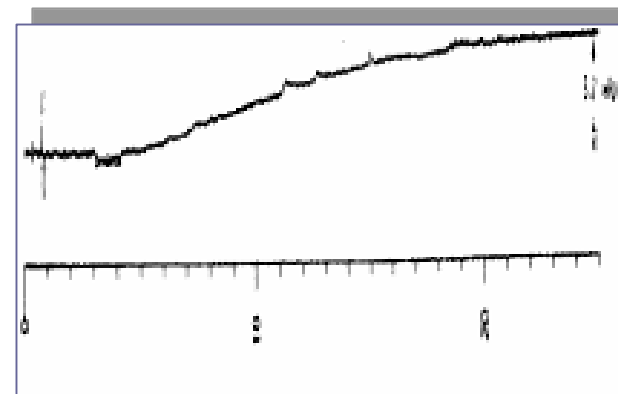
→ HPLC peak resolution & quantitative results are effected by solvents with lower quality

Compounds producing artefacts: Additives, Peroxides, Phtalates

Risks „using non suitable Acetonitrile @ 210 nm“



Risk due to enrichment process of impurities



Baseline drift due to trace impurities

Solvent impurities

No reliable HPLC result
Loss in separation performance
Reduced column lifetime

3 Grade-Program: Application Oriented

LiChrosolv®
Isocratic Elution

▪ **Detection method: UV**

- For the analysis of simple matrix
- Applications: sample preparation & isocratic separation of similar polar, non polar compounds in QC

- Customer Segment: Food & Beverage, Environmental, Pharma, Chemical Industry

LiChrosolv® Gradient
Grade
Gradient Elution

▪ **Detection method: UV, Fluorescence**

- Applications: sample preparation & analysis of complex, highly sensitive & demanding quality control, impurity profiling in QC, R&D for mixed samples of polar /non-polar compounds

- Customer Segment: Pharma, Chemistry, Cosmetics

LiChrosolv®
hypergrade
LC-Mass Detection

▪ **Detection method: UV, Fluorescence, Mass**

- Applications: Protein Profiling
Pesticide Analysis
PAHs
Proteomics LC-MS
LC-MS routine analysis

- Customer Segment: Pharma, mainly R&D, Environmental QC

3 Grade – Application Oriented Program

LiChrosolv® Isocratic Elution

Specification

1.1401.2500 Acetonitrile isocratic grade for liquid chromatography (LiChrosolv®)

Specification

Acidity (GC)	≤0.1	%
Acidity (HPLC)	≤0.05	μg/mL
Evaporative residue	≤40	mg/L
Water	≤0.05	%
Acidity	≤0.005	mg/g
Acidity	≤0.002	mg/g
Transmittance (at 210 nm)	≥75	%
Transmittance (at 220 nm)	≥80	%
Transmittance (at 254 nm)	≥85	%

Filtered by 0.2 μm filter

LiChrosolv® Gradient Grade Gradient Elution

Specification

100200 Acetonitrile gradient grade for liquid chromatography (LiChrosolv® Reag. Ph. Eur.)

	Spec. Value	
	min	max
Acidity (GC)	≤0.1	%
Acidity (HPLC)	≤0.05	μg/mL
Evaporative residue	≤10	mg/L
Water	≤0.02	%
Colour	≤0.1	Platinum
Density (at 20°C/15°C)	0.78	
Refractive index (at 20°C)	1.36	
Bioassay (at 20°C)	≤10	μg/mL
Acidity	≤0.002	mg/g
Alkalinity	≤0.002	mg/g
Gradient grade		
at 210 nm	≥1.0	μg/L
at 220 nm	≥1.0	μg/L
Transmittance		
at 210 nm	≥1.0	μg/L
at 220 nm	≥1.0	μg/L
at 254 nm	≥1.0	μg/L

Filtered by 0.2 μm filter
Suitable for HPLC / UHPLC / UHPLC - instruments
Complies to requirements for chromatography and bioassay (EU) according to Reag. Ph. Eur.
Complies to the requirements of ACQ for liquid chromatography suitability

LiChrosolv® hypergrade LC-Mass Detection

Specification

1.00020.2500 Acetonitrile hypergrade for LC-MS (LiChrosolv®)

Specification

Purity (HPLC)	≥99.9	%
Acidity (HPLC)	≤0.05	μg/mL
Evaporative residue	≤1.0	mg/L
Water	≤0.01	%
Colour	≤0.10	Platinum
Acidity	≤0.0001	mg/g
Alkalinity	≤0.0001	mg/g
Ac (Phosphorus)	≤0.00	μg/L
Ac (Sulfur)	≤0.10	μg/L
Ac (Nitrogen)	≤0.10	μg/L
Phosphorus (at 210 nm)	≤0.10	μg/L
Phosphorus (at 220 nm)	≤0.10	μg/L
Phosphorus (at 254 nm)	≤0.10	μg/L
Phosphorus (at 210 nm)	≤0.10	μg/L
Phosphorus (at 220 nm)	≤0.10	μg/L
Phosphorus (at 254 nm)	≤0.10	μg/L
Suitability for bioassay analysis	complies	
at 210 nm	≥1.00	μg/L
at 220 nm	≥1.00	μg/L
at 254 nm	≥1.00	μg/L
at 210 nm	≥1.00	μg/L
at 220 nm	≥1.00	μg/L
at 254 nm	≥1.00	μg/L
Suitability for mass detection	complies	
at 210 nm	≥1.00	μg/L
at 220 nm	≥1.00	μg/L
at 254 nm	≥1.00	μg/L

Filtered by 0.2 μm filter
Suitable for UHPLC / UHPLC - UHPLC - instruments

Solvents for liquid chromatography

LiChrosolv®

Different applications require different purity

LiChrosolv®							
Hypergrade							
Gradient grade							
Isocratic grade							
Application	Isocratic HPLC	Far UV HPLC	Gradient Elution	Protein Profiling	Pesticide Analysis	PAHs	Proteomics LC-MS application



Specification

1.00030.2500 Acetonitrile gradient grade for liquid chromatography LiChrosolv® Reag. Ph Eur

Specification

Purity (GC)	≥ 99.9	%
Identity (IR)	conforms	
Evaporation residue	≤ 2.0	mg/l
Water	≤ 0.02	%
Colour	≤ 10	Hazen
Density (d 20 °C/20 °C)	0.78	
Refractive index (n 20/D)	1.344	
Boiling range (80-82°C)	≥ 95	% (v/v)
Acidity	≤ 0.0002	meq/g
Alkalinity	≤ 0.0002	meq/g
Gradient grade (at 210 nm)	≤ 1.0	mAU
◆ Gradient grade (at 254 nm)	≤ 0.5	mAU
Fluorescence (as quinine at 254 nm)	≤ 1.0	ppb
◆ Fluorescence (as quinine at 365 nm)	≤ 0.5	ppb
Transmission (at 193 nm)	≥ 60	%
Transmission (at 195 nm)	≥ 80	%
◆ Transmission (from 230 nm)	≥ 98	%

Filtered by 0.2 µm filter.
Suitable for UPLC / UHPLC /Ultra HPLC - instruments.
Conforms to Acetonitrile for chromatography and Acetonitrile R1 according to Reag.Ph Eur;
conforms to the requirements of ACS for liquid chromatography suitability.

What's different ?



Specification

1.00030.2500 Acetonitrile gradient grade for liquid chromatography LiChrosolv® Reag. Ph Eur

Reag. PhEur and ACS Specification

Specification

Purity (GC)
Identity (IR)
Evaporation
Water

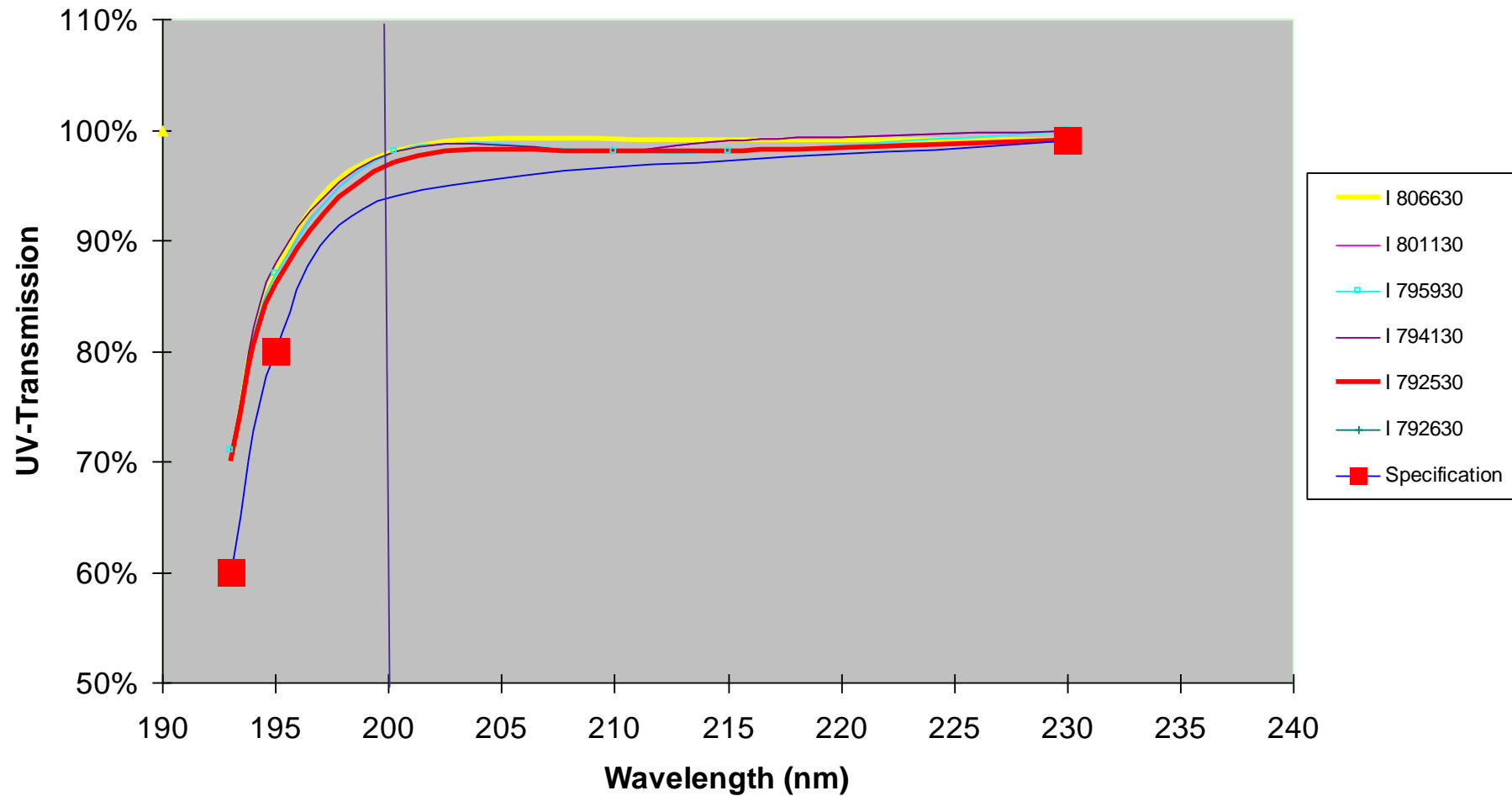
Conforms to Acetonitrile for chromatography and Acetonitrile R1 according to Reag.Ph Eur; conforms to the requirements of ACS for liquid chromatography suitability.

Colour	≤ 10	Hazen
Density (d 20 °C/20 °C)	0.78	
Refractive index (n 20/D)	1.344	
Boiling range (80-82°C)	≥ 95	% (v/v)
Acidity	≤ 0.0002	meq/g
Alkalinity	≤ 0.0002	meq/g
Gradient grade (at 210 nm)	≤ 1.0	mAU
Gradient grade (at 254 nm)	≤ 0.5	mAU
Fluorescence (as quinine at 254 nm)	≤ 1.0	ppb
Fluorescence (as quinine at 365 nm)	≤ 0.5	ppb
Transmission (at 193 nm)	≥ 60	%
Transmission (at 195 nm)	≥ 80	%
Transmission (from 230 nm)	≥ 98	%

Filtered by 0.2 µm filter.
Suitable for UPLC / UHPLC / Ultra HPLC - instruments.
Conforms to Acetonitrile for chromatography and Acetonitrile R1 according to Reag.Ph Eur;
conforms to the requirements of ACS for liquid chromatography suitability.

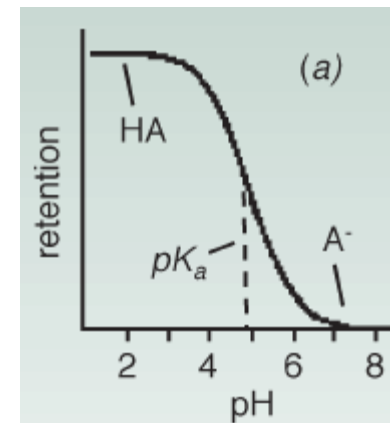
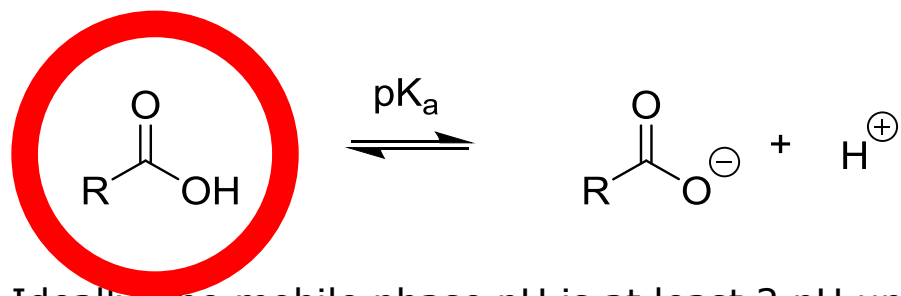


Quality Assurance (1) - UV Transmission



Why using buffers at all in HPLC and LC-MS...?

- Buffers are used in RP-HPLC separations to control the retention of ionizable compounds.
- This is to suppress ionization of analytes in order to maximize sample retention.



- Ideally, the mobile phase pH is at least 2 pH units below (acids) or above (bases) the sample pK_a .
- For the most effective buffering, a buffer should be carefully chosen and used within ± 1 pH unit of the buffer's pK_a .
- For LC-UV assays, phosphate and acetate buffers are most popular.
- For LC-MS applications, the buffer must be volatile. Various combinations of formate, acetate, ammonia and bicarbonate are most popular for LC-MS work.

Lichropur® HPLC Reagents

Product No.	Name	Description	Package Size
5.43804	Formic acid	100%	100, 250 ml
5.43808	Acetic acid	100%	100, 250 ml
5.43827	Sulfuric acid	96%	100, 250 ml
5.43828	o-Phosphoric acid	85%	100, 250 ml
5.43830	Ammonia solution	25%	100, 250 ml
5.43832	Sodium chloride		100, 250 ml
5.43833	Sodium acetate trihydrate		100, 250 ml
5.43834	Ammonium acetate		100, 250 ml
5.43835	Ammonium hydrogen carbonate		100, 250 ml
5.43837	Ammonium dihydrogen phosphate		100, 250 ml
5.43838	Di-Sodium hydrogen phosphate anhydrous		100, 250 ml
5.43839	Di-Potassium hydrogen phosphate anhydrous		100, 250 ml
5.43840	Sodium dihydrogen phosphate anhydrous		100, 250 ml
5.43841	Potassium dihydrogen phosphate anhydrous		100, 250 ml

Lichropur[®] HPLC Reagents

HPLC Reagents

We offer LiChropur[®] reagents ideal for HPLC analysis.

These reagents are tested for inorganic impurities, including Al, Ca, Cu, Fe, K, Mg and Na.

- QC performed using a HPLC suitability gradient test at 220, 254 and 280 nm.
- Filled under clean room conditions
- Extensive impurity profile of the product on the Certificate of Analysis

**Al: ≤ 0.050 ppm; Ca: ≤ 0.2 ppm; Cu: ≤ 0.020 ppm; Fe: ≤ 2.0 ppm;
K: ≤ 0.10 ppm; Mg: ≤ 0.50 ppm; Na: ≤ 0.50 ppm**

HPLC-gradient suitability test (220, 254, 280 nm)
chromophoric impurities based on 4'-hydroxyacetophenon



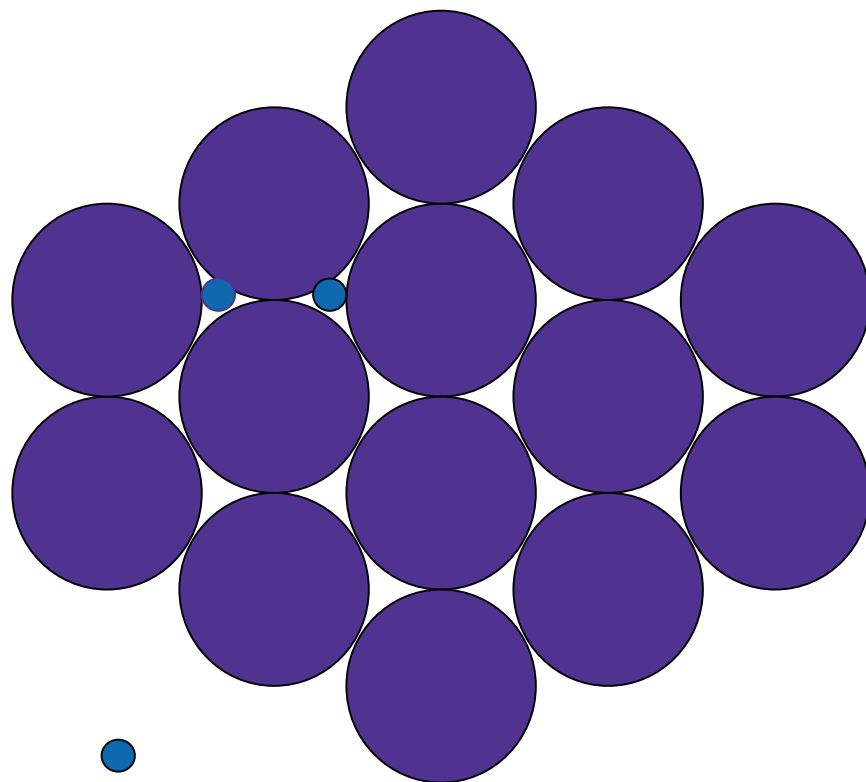


02b

**SOLVENTS FOR
LIQUID
CHROMATOGRAPHY
LICHROSOLV® - FAST
CHROMATOGRAPHY**

Fast chromatography – UHPLC

UHPLC column $1,7\mu\text{m} / 6 < 0,3\mu\text{m}$ **means high risk of blockage**



Faster
Short & thin columns
Higher separating capacity
Particle diameter $> 2\mu\text{m}$

Columns packed with $1.7\mu\text{m}$ particles tend to clog easily, because the space in between the particles is only $0.3\mu\text{m}$

1.00030.2500 Acetonitrile gradient grade for liquid chromatography
LiChrosolv® Reag. Ph Eur

	Spec. Values	
Purity (GC)	≥ 99.9	%
Identity (IR)	conforms	
residue on evaporation	≤ 2.0	mg/l
Water	≤ 0.02	%
Colour	≤ 10	Hazen
Density (d 20 °C/20 °C)	0.78	
Refractive index (n 20/D)	1.344	
Boiling range (80-82°C)	≥ 95	Vol%
Acidity	≤ 0.0002	meq/g
Alkalinity	≤ 0.0002	meq/g
Gradient grade		
at 210 nm	≤ 1.0	mAU
at 254 nm	≤ 0.5	mAU
Fluorescence		
as quinine at 254 nm	≤ 1.0	ppb
as quinine at 365 nm	≤ 0.5	ppb
Transmission		
at 193 nm	≥ 60	%
at 195 nm	≥ 80	%
from 230 nm	≥ 98	%

*Filtered by 0.2 µm filter
Suitable for UPLC / UHPLC / Ultra HPLC - instruments*



O2C

**SOLVENTS FOR
LIQUID
CHROMATOGRAPHY
LICHROSOLV® LC –
MASS DETECTION**

LC-MS Method

Is it necessary to have a better grade of solvent and better column purity for LC-MS?

BECAUSE:

YES!

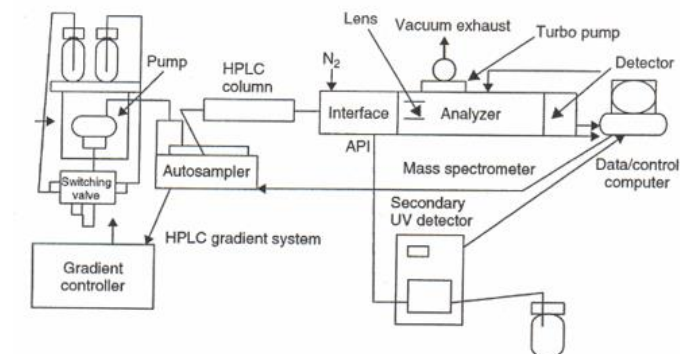
With standard solvents & columns, trace impurities cause unwanted background signals in LC-MS which reduce sensitivity and cause complex spectra with low reproducibility - compared to HPLC with standard UV detectors.



LC-MS requires improved procedures, compared to HPLC with UV detectors

Criteria LC-MS / Optimized Performance

- High ionisation efficiency
- High reproducibility of the ionisation signal
- Low „background“ signals and low adduct formation
- Low „ion suppression“ due to solvents impurities
- Low contamination of the ionisation source



3 Grade – Application Oriented Program

LiChrosolv® Isocratic Elution

Specification

1.94291.2500 Acetonitrile isocratic grade for liquid chromatography LiChrosolv®

Property	Specification	Units
Purity (GC)	≥ 99.9	%
Residue (HPLC)	≤ 0.05	mg/ml
Evaporation residue	≤ 0.02	mg/l
Water	≤ 0.05	%
Acidity	≤ 0.005	mg/g
Alkalinity	≤ 0.005	mg/g
Transmission (210 nm)	≥ 95	%
Transmission (254 nm)	≥ 90	%
Transmission (280 nm)	≥ 85	%

Filtered by 0.2 µm filter.
Suitable for HPLC (DAD, DRI, UV, MS) - instruments.
Complies to acceptance for chromatography and acetonitrile (AC) according to Reg. Ph. Eur. conforms to the requirements of AC for liquid chromatography suitability.

LiChrosolv® Gradient Grade Gradient Elution

Specification

100000 Acetonitrile gradient grade for liquid chromatography LiChrosolv® Reg. Ph. Eur.

Property	Specification	Units
Purity (GC)	≥ 99.9	%
Residue (HPLC)	≤ 0.05	mg/ml
Evaporation residue	≤ 0.02	mg/l
Water	≤ 0.02	%
Color	≤ 0.01	mg/ml
Residue (HPLC) (210 nm)	≤ 0.01	mg/ml
Residue (HPLC) (254 nm)	≤ 0.01	mg/ml
Residue (HPLC) (280 nm)	≤ 0.01	mg/ml
Acidity	≤ 0.005	mg/g
Alkalinity	≤ 0.005	mg/g
Conductivity	≤ 0.05	µS/cm
Resistivity	≥ 20	Ω·cm
Evaporation residue (210 nm)	≤ 0.02	mg/l
Evaporation residue (254 nm)	≤ 0.02	mg/l
Evaporation residue (280 nm)	≤ 0.02	mg/l
Transmission (210 nm)	≥ 90	%
Transmission (254 nm)	≥ 85	%
Transmission (280 nm)	≥ 80	%

Filtered by 0.2 µm filter.
Suitable for HPLC (DAD, DRI, UV, MS) - instruments.
Complies to acceptance for chromatography and acetonitrile (AC) according to Reg. Ph. Eur. conforms to the requirements of AC for liquid chromatography suitability.

LiChrosolv® hypergrade LC-Mass Detection

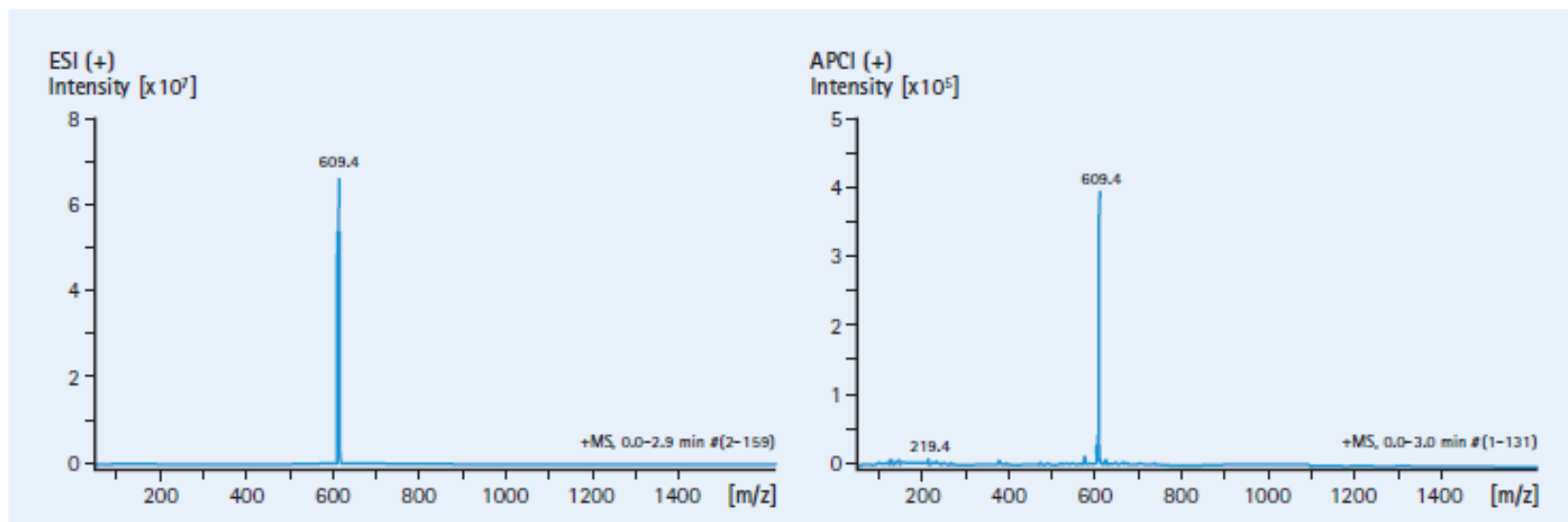
Specification

1.00029.2500 Acetonitrile hypergrade for LC-MS LiChrosolv®

Property	Specification	Units
Purity (GC)	≥ 99.9	%
Residue (HPLC)	≤ 0.01	mg/ml
Evaporation residue	≤ 0.01	mg/l
Water	≤ 0.01	%
Color	≤ 0.01	mg/ml
Acidity	≤ 0.0001	mg/g
Alkalinity	≤ 0.0001	mg/g
Res (HPLC)	≤ 0.001	mg/ml
Res (HPLC) (210 nm)	≤ 0.001	mg/ml
Res (HPLC) (254 nm)	≤ 0.001	mg/ml
Res (HPLC) (280 nm)	≤ 0.001	mg/ml
Fluorescence (excitation at 254 nm)	≤ 0.1	µg/l
Fluorescence (excitation at 280 nm)	≤ 0.1	µg/l
Substrate for fluorescence detection	complies	
Substrate for fluorescence detection (in an acetonitrile/water 50/50 v/v v/v)	complies	
Substrate for fluorescence detection (in an acetonitrile/water 50/50 v/v v/v) with 100 µg/ml of the substance according to the Reg. Ph. Eur. - 101.01 in relation with the substance detection	complies	
Substrate for fluorescence detection (in an acetonitrile/water 50/50 v/v v/v) with 100 µg/ml of the substance according to the Reg. Ph. Eur. - 101.01 in relation with the substance detection	complies	
Fluorescence (excitation at 210 nm)	≤ 0.01	µg/l
Fluorescence (excitation at 254 nm)	≤ 0.01	µg/l
Fluorescence (excitation at 280 nm)	≤ 0.01	µg/l
Fluorescence (excitation at 310 nm)	≤ 0.01	µg/l
Fluorescence (excitation at 330 nm)	≤ 0.01	µg/l
Substrate for HPLC analysis (HPLC) (fluorescence detection)	complies	
Substrate for HPLC analysis (HPLC) (UV detection)	complies	
Substrate for HPLC analysis (HPLC) (MS detection)	complies	
Substrate for LC-MS detection with ion trap MS/MS (fluorescence detection)	≥ 1	µg/l
Substrate for LC-MS detection with ion trap MS/MS (fluorescence detection)	≥ 1	µg/l
Substrate for LC-MS detection with ion trap MS/MS (fluorescence detection)	≥ 1	µg/l

Filtered by 0.2 µm filter.
Suitable for HPLC (DAD, DRI, UV, MS) - instruments.

Example: Acetonitrile LiChrosolv® hypergrade



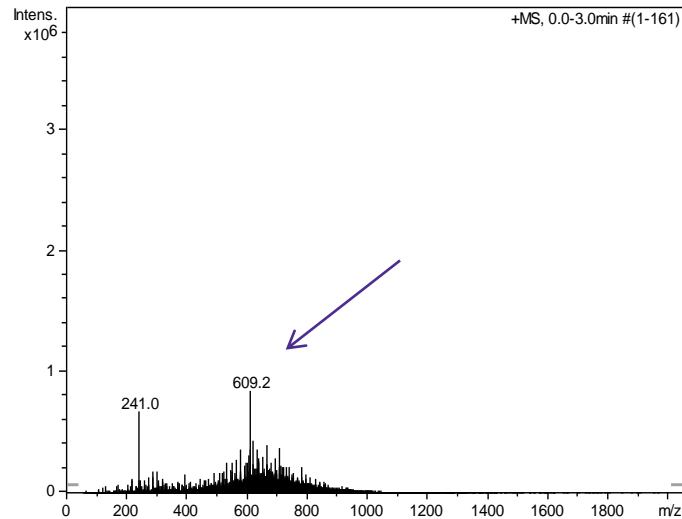
Mass spectrum of LiChrosolv® Acetonitrile hypergrade (100029). Mobile phase Acetonitrile special LC-MS grade. Intensity of single background mass peak based on reserpine standard (m/z 609.4) in e.g. ESI (+) and APCI (+) mode.

ESI = Electron Spray Ionization

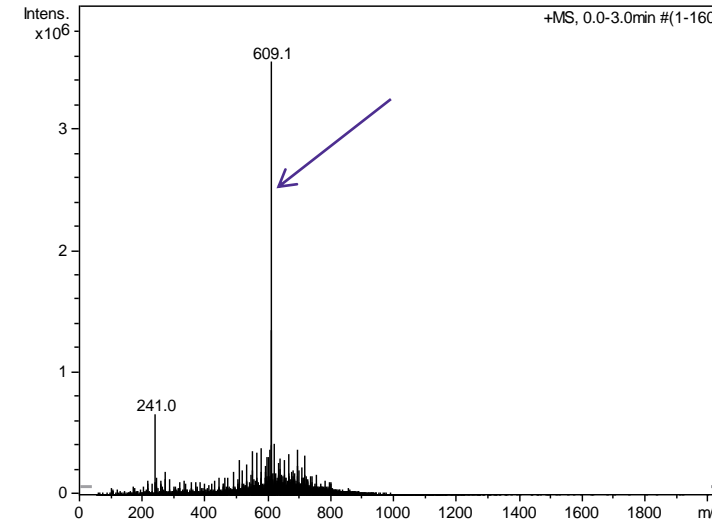
APCI = Atmospheric Pressure Chemical Ionization

Main problem: low ionization efficiency

Gradient Grade



Hypergrade LC-MS Grade



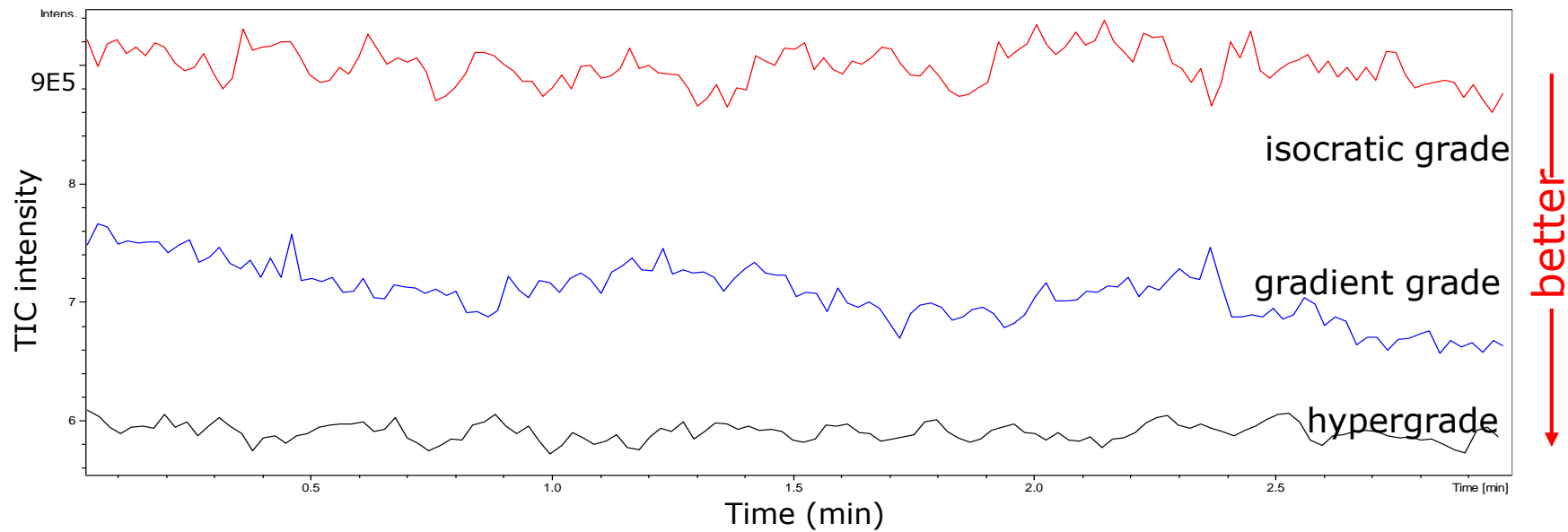
- The mass spectra of different acetonitrile grades show clearly the variation in the intensity of the **reserpine signal** ($[M+H]^+ = 609$) as well as the extent of the background signals.
- The differences in the intensity of the reserpine signal are caused by **ion suppression**. This effect occurs due to interfering trace contaminants that can be present in acetonitrile.

TIC (Total Ion Current) in different eluent qualities

MS grade solvents prevent contamination

- Minimization of contaminant peaks & ion suppression
- Maximization of sensitivity (low background noise)

Combined TICs of the analysis of three different acetonitrile qualities:



Merck: Specification for ACN, Methanol, Water for LC-MS suitability

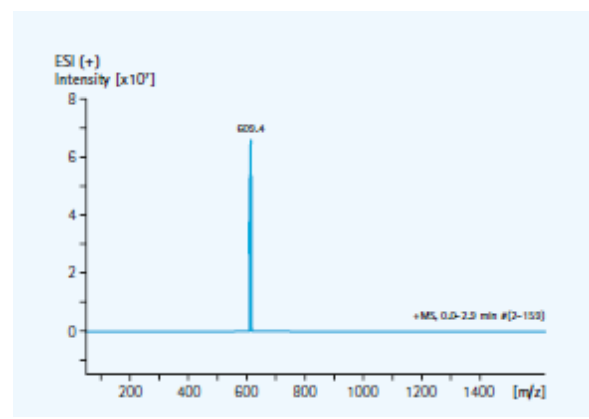
Acetonitrile hypergrade LC-MS suitability	Cat. No. 100029 Spec. values	Methanol hypergrade LC-MS suitability	Cat. No. 106035 Spec. values
Purity (GC)	≥ 99.9 %	Purity (GC)	≥ 99.9 %
Identity (IR)	conforms	Identity (IR)	conforms
Residue on evaporation	≤ 1.0 mg/l	Residue on evaporation	≤ 1.0 mg/l
Water	≤ 0.01 %	Water	≤ 0.01 %
Color	≤ 10 Hazen	Color	≤ 10 Hazen
Acidity	≤ 0.0001 meq/g	Acidity	≤ 0.0002 meq/g
Alkalinity	≤ 0.0002 meq/g	Alkalinity	≤ 0.0002 meq/g
Al (Aluminum) *	≤ 10 ppb	Al (Aluminum) *	≤ 10 ppb
Ca (Calcium) *	≤ 10 ppb	Ca (Calcium) *	≤ 10 ppb
Fe (Iron) *	≤ 10 ppb	Fe (Iron) *	≤ 10 ppb
Mg (Magnesium) *	≤ 10 ppb	Mg (Magnesium) *	≤ 10 ppb
Na (Sodium) *	≤ 50 ppb	Na (Sodium) *	≤ 100 ppb
K (Potassium) *	≤ 5 ppb	K (Potassium) *	≤ 5 ppb
Every other single metal (ICP-MS) *	≤ 5 ppb	Every other single metal (ICP-MS) *	≤ 5 ppb
Gradient grade		Gradient Grade	
at 210 nm	≤ 0.8 mAU	at 220 nm	≤ 2.0 mAU
at 254 nm	≤ 0.3 mAU	at 235 nm	≤ 1.0 mAU
Fluorescence		Fluorescence	
as quinine at 254 nm	≤ 1 ppb	as quinine at 254 nm	≤ 1 ppb
as quinine at 365 nm	≤ 0.5 ppb	as quinine at 365 nm	≤ 0.5 ppb
Transmission		Transmission	
at 191 nm	≥ 25 %	at 210 nm	≥ 35 %
at 195 nm	≥ 85 %	at 220 nm	≥ 60 %
at 200 nm	≥ 96 %	at 230 nm	≥ 75 %
at 215 nm	≥ 98 %	from 260 nm	≥ 98 %
from 230 nm	≥ 99 %	Suitability for LC-MS (tested with ion trap MS): Intensity of single background mass peak based on reserpine:	
Suitability for PAH analysis (HPLC fluorescence-detection)	conforms	Mode: ESI 200 µl pos APCI 200 µl pos	≤ 2 ppb
At an excitation between 240 and 600 nm (with t _{ΔA} = 10 nm) the emission intensity in the range of 250 – 700 nm is smaller then the following standards: Chinin-Standard (1 ng/ml; 0.05 mol/l H ₂ SO ₄), PAH Standard (1:100,000, Acetonitrile; NIST SRM 1647B)		Mode: ESI 200 µl neg APCI 200 µl neg	≤ 20 ppb
Suitability for pesticide analysis (HPLC UV-detection)	conforms	Filtered by 0.2 µm stainless steel filter Suitable for PAH-analysis Suitable for UPLC UHPLC Ultra Fast HPLC-instruments * = enhanced specifications	

New enhanced specification

New enhanced specification



Ion / Metal - information



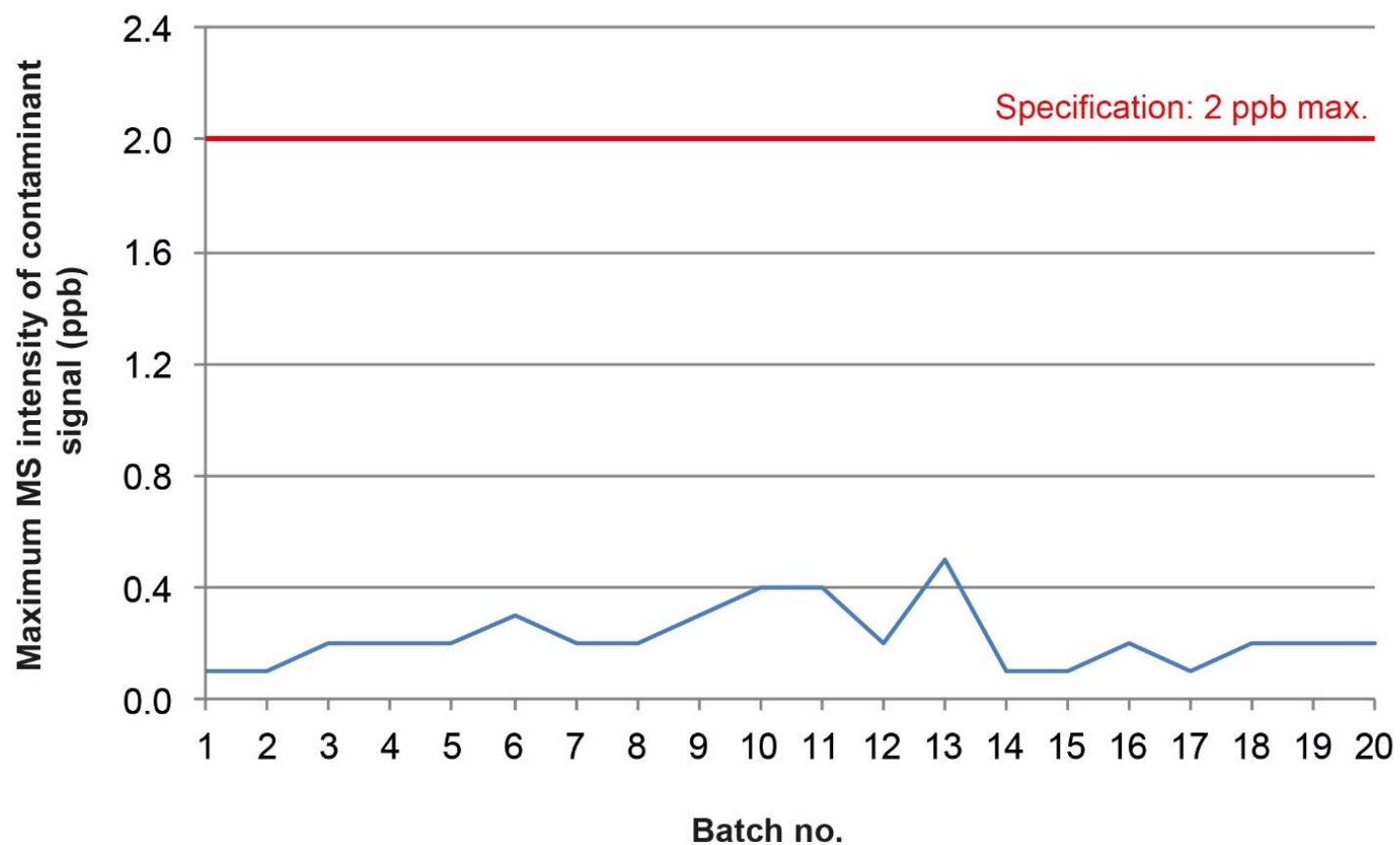
Suitability for LC-MS (tested with ion trap MS): Intensity of background mass peak based on reserpine:	
Mode: ESI 200 µl pos APCI 200 µl pos	≤ 2 ppb
Mode: ESI 200 µl neg APCI 200 µl neg	≤ 20 ppb

Suitability ESI/APCI (+) & ESI/APCI (-) mode

ESI = Electron Spray Ionization
APCI = Atmospheric Pressure Chemical Ionization

Sensitivity in MS

Batch-to-batch reproducibility of acetonitrile hypergrade for LC-MS LiChrosolv® in 20 consecutive batches



reproducibly low background noise
no or weak additional peaks/
signal suppression

Conditions

System Bruker Esquire 6000plus
Detection Pos. ESI-MS,
m/z range 50-2000
Flow rate 0.2 mL/min via syringe
pump
Temp. 25 °C

LiChrosolv® hypergrade Portfolio – pure solvents

1.00029	Acetonitrile LiChrosolv® hypergrade for LC-MS
1.03649	Ethyl acetate LiChrosolv® hypergrade for LC-MS
1.03654	Heptane LiChrosolv® hypergrade for LC-MS
1.03701	Hexane LiChrosolv® hypergrade for LC-MS
1.06035	Methanol LiChrosolv® hypergrade for LC-MS
1.02781	2-Propanol LiChrosolv® hypergrade for LC-MS
1.15333	Water for chromatography LiChrosolv® (LC-MS grade)



LiChrosolv® hypergrade Portfolio- ready to use blends

1.59004	Acetonitrile with 0.1% (v/v) Acetic acid hypergrade for LC-MS LiChrosolv®
1.59002	Acetonitrile with 0.1% (v/v) Formic acid hypergrade for LC-MS LiChrosolv®
1.59014	Acetonitrile with 0.1% (v:v) Trifluoroacetic acid hypergrade for LC-MS LiChrosolv®
1.59007	Water with 0.1% (v/v) Acetic acid hypergrade for LC-MS LiChrosolv®
1.59013	Water with 0.1% (v/v) Formic acid hypergrade for LC-MS LiChrosolv®
4.80112	Water with 0.1% (v:v) trifluoroacetic acid hypergrade for LC-MS LiChrosolv®



Lichropur® LC-MS Reagents

Product Overview

Product No.	Name	Description	Package Size
5.33001	Acetic acid	100% for LC-MS Lichropur®	50 ml
5.33002	Formic acid	98-100% for LC-MS Lichropur®	50 ml
5.33003	Ammonia solution	25% for LC-MS Lichropur®	50 ml
5.33004	Ammonium acetate	for LC-MS Lichropur®	50 ml
5.33005	Ammonium hydrogen carbonate	for LC-MS Lichropur®	50 ml

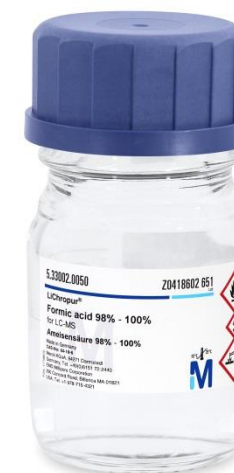
Lichropur® LC-MS Reagents

LC-MS Reagents

We offer LiChropur® products specific for LC-MS analysis.

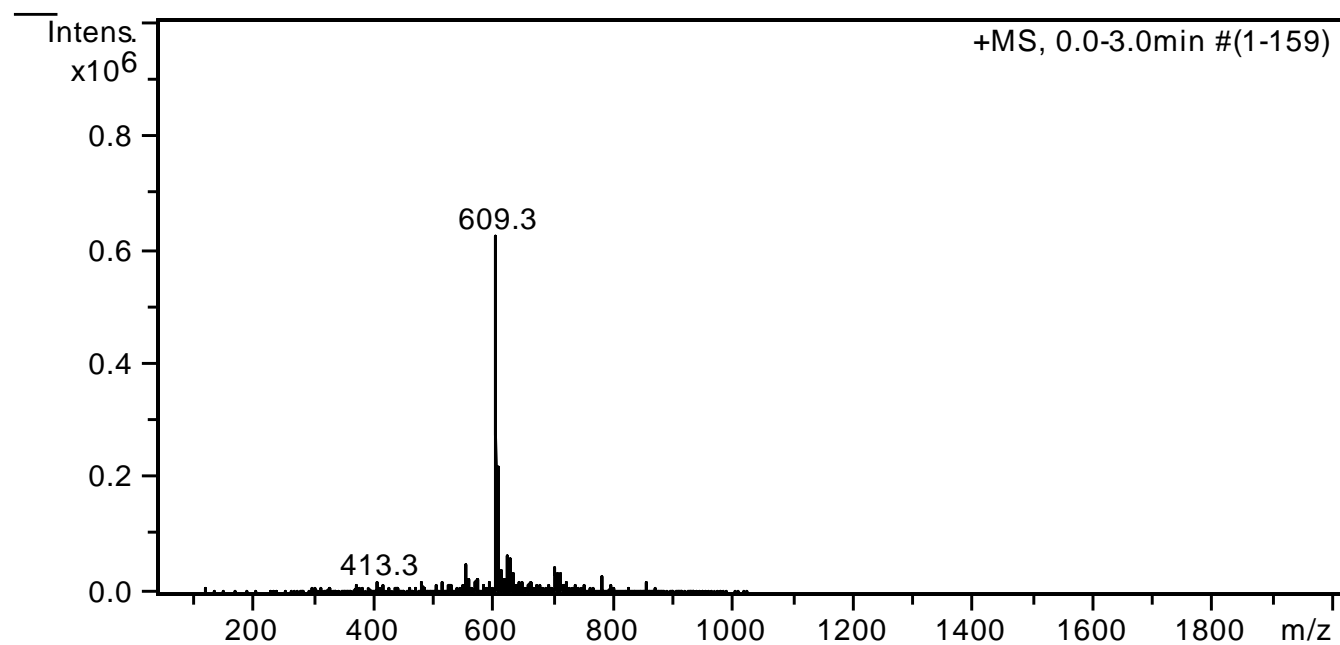
These reagents are tested for inorganic impurities, including Al, Ca, Cu, Fe, K, Mg and Na.

- QC performed using a standard reserpine test
- Filled under clean room conditions
- Extensive impurity profile of the product on the Certificate of Analysis
- Assay (acidimetric): $\geq 98.0\%$
- Colour: ≤ 10 Hazen
- Residue on ignition: ≤ 2 ppm
- Cation traces:
 - **Al: ≤ 5.0 ppb; Ca: ≤ 10.0 ppb; Cu: ≤ 1.0 ppb; Fe: ≤ 5.0 ppb; K: ≤ 5.0 ppb; Mg: ≤ 2.0 ppb; Na: ≤ 5.0 ppb; NH_4^+ : ≤ 10 ppm**
- LC-MS suitability test

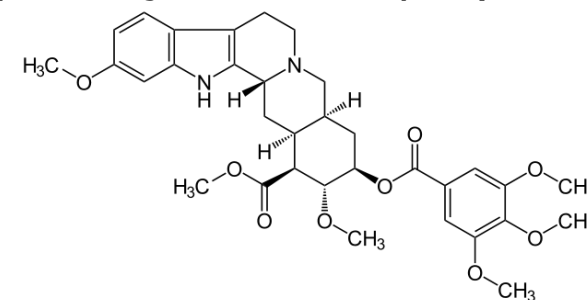


Lichropur® LC-MS Reagents

Reserpine test



Mass spectrum ESI positive
(flow injection analysis)



Reserpine

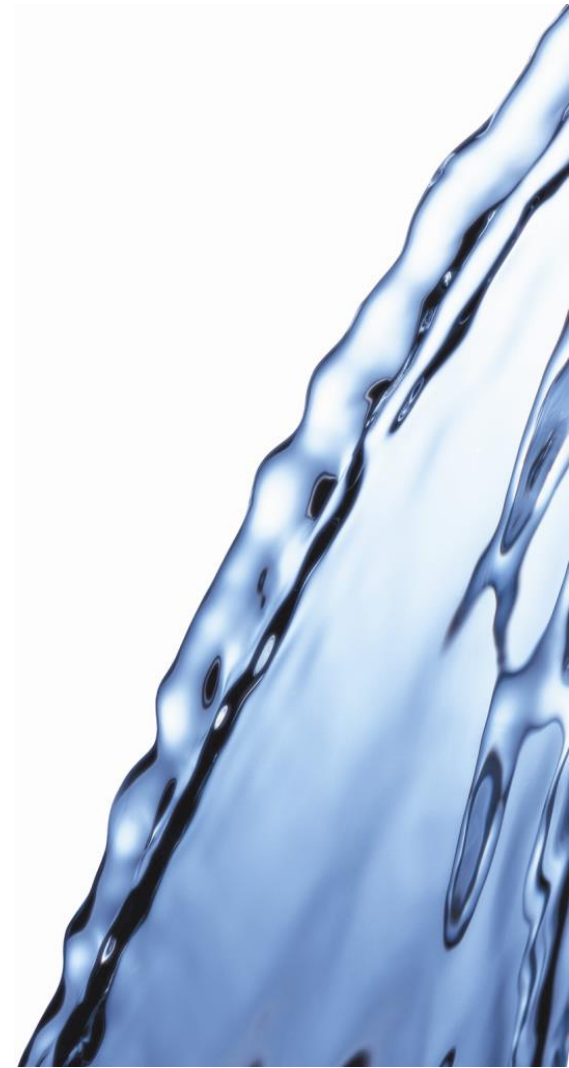
Measuring parameter	Specification value
ESI positive	< 2 ppb
ESI & APCI negative	< 20 ppb

No signal should be
greater than $[M+H] = 609$

LiChrosolv® hypergrade marks the difference

- High ionization efficiency
- Minimal baseline noise
- Low level of ionic background
- Reduced metal adduct formation

➔ Superior resolution & sensitivity





withdrawal



disposal

1.03830.0001 / 1.03831.0001
HPLC adapters for S40
(supply / disposal)



Supporting Literature



Accuracy you can count on.
Tailor-made solvents in tailor-made packaging

2014 update

Merck Millipore is a division of **MERCK**

01

Mobile Phases & Reagents

First stop: Merck Millipore laboratories, Darmstadt, Germany. Here you are bound to find many steady hands and agile minds - busy producing breakthrough mobile phases and reagents world-wide.

Lichrosolv®
analytical chromatography

Modern analytical HPLC often uses gradient methods, which require higher solvent quality compared to isocratic methods. For this reason, we provide many Lichrosolv® solvents in both isocratic and gradient quality.

Lichrosolv® high purity solvents are available in an extensive product range: volumes of 1 liter, 2.5 liters and 4 liters are available in glass bottles, 5 liters in aluminum bottles and 10 liters, 30 liters and 188 liters in returnable stainless steel barrels. Higher volume vessels are available on request. The advantages of such barrels are described in our product information brochure "Accuracy - you can count on".

For information on safe and contamination-free solvent withdrawal from bottles and barrels, please refer to the action "Solvent Management System".

Lichrosolv® is available in **hypergrade** for LC/MS/MS applications in **100 L** specially treated amber glass bottles.

ChromBook
The world of chromatography in your hands.

Merck Millipore is a division of **MERCK**



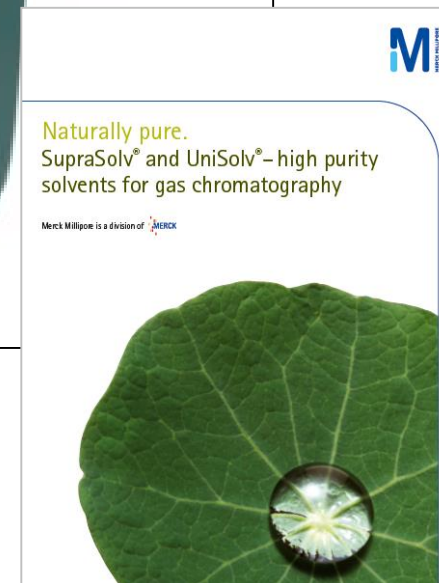
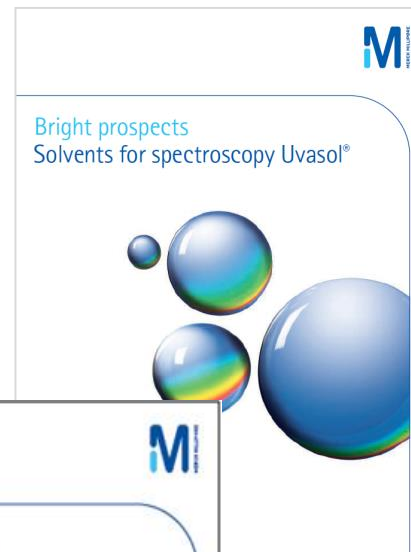
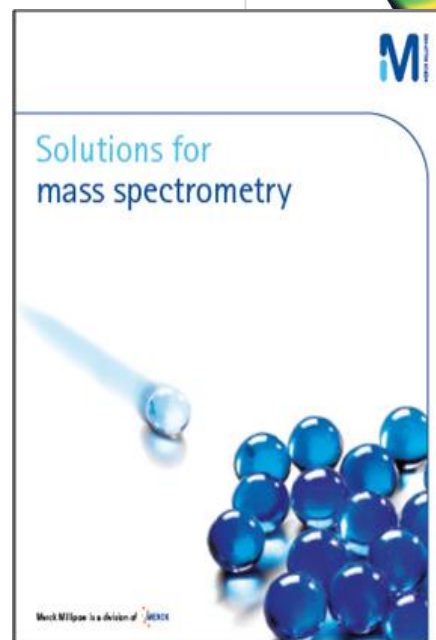
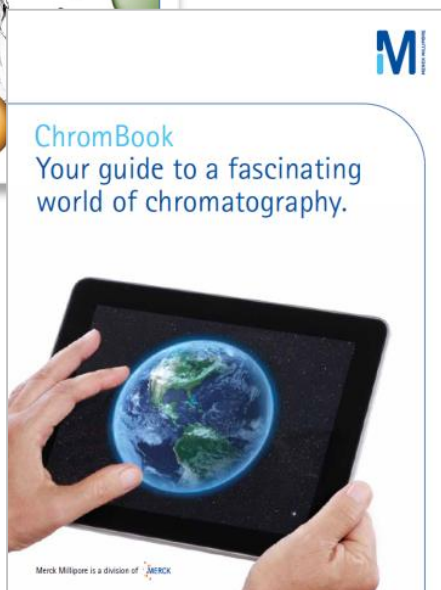
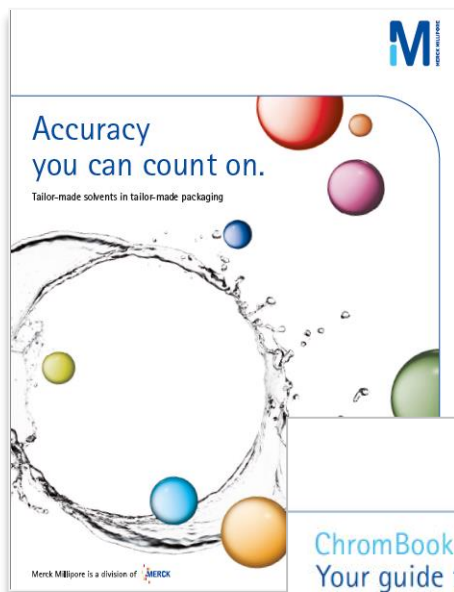
Ordering information - Lichrosolv® A-C

Product	Ordering No.	Content / Packaging	Purity (%)	Evap. residue max. (mg/L)	Water max. (%)	Acidity max. (mg/g)	Alkalinity max. (mg/g)	UV-trans. at 254 nm
Acetone	1.00020.1000	1 L GL	99.8	2	0.06	0.0002	0.0002	235 (20%), 240 (20%), 260 (20%)
	1.00020.2500	2.5 L GL						
	1.00020.4000	4 L GL						
	1.00020.5000	5 L AL						
	1.00020.9010	10 L ST						
Acetonitrile	1.00020.1000	1 L GL	99.9	1	0.01	0.0001	0.0002	191 (20%), 195 (20%), 200 (20%), 215 (20%), 230 (20%)
	1.00020.2500	2.5 L GL						
	1.00020.4000	4 L GL						
	1.00020.5000	5 L AL						
	1.00020.9010	10 L ST						
Acetonitrile gradient grade, suitable for UPLC / MS/MS, HPLC, ACS*	1.00030.1000	1 L GL	99.9	2	0.02	0.0002	0.0002	191 (20%), 195 (20%), 200 (20%)
	1.00030.2500	2.5 L GL						
	1.00030.4000	4 L GL						
	1.00030.5000	5 L AL						
	1.00030.9010	10 L ST						
Acetonitrile isocratic grade	1.14291.1000	1 L GL	99.8	4	0.06	0.0006	0.0002	196 (20%), 200 (20%), 240 (20%)
	1.14291.2500	2.5 L GL						
	1.14291.4000	4 L GL						
	1.14291.5000	5 L AL						
	1.14291.9010	10 L ST						
Benzene	1.01788.1000	1 L GL	99.8	2	0.03	0.0002	0.0002	285 (20%), 290 (20%), 340 (20%)
	1.01788.2500	2.5 L GL						
	1.01788.4000	4 L GL						
	1.01788.5000	5 L AL						
	1.01788.9010	10 L ST						
tert-Butyl methyl ether	1.01845.1000	1 L GL	99.8	2	0.02	0.0002	0.0002	240 (20%), 255 (20%), 260 (20%)
	1.01845.2500	2.5 L GL						
	1.01845.4000	4 L GL						
	1.01845.5000	5 L AL						
	1.01845.9010	10 L ST						
1-Chlorobutane	1.02444.1000	1 L GL	99.8	2	0.01	0.0002	0.0002	227 (20%), 232 (20%), 260 (20%)
	1.02444.2500	2.5 L GL						
	1.02444.4000	4 L GL						
	1.02444.5000	5 L AL						
	1.02444.9010	10 L ST						

* All solvents are filtered through 0.2 µm (GL - glass bottles) / 0.1 µm (AL - aluminum bottles) / 0.1 µm (ST - specially treated amber glass) filters. Some solvents are available in returnable stainless steel barrels. For information on safe and contamination-free solvent withdrawal from bottles and barrels, please refer to the action "Solvent Management System".

www.merck-chemicals.com/mobile-phases-for-chromatography

Information Variety



LiChrosolv® marks the difference

- Safety through reliable quality **avoids misinterpretation** of analytical results; saves cost- & time-intensive repetition of analysis
- Reduced baseline drift for **better separation performance**
- Interference free baseline for **better reproducibility**
- **Enhanced sensitivity** due to lower basic absorbance
- **Convenience**
 - No need for filtration, already done through 0.2 µm stainless steel filters (CoA)
 - No blank run necessary due to high batch-to-batch consistency (less solvent consumption, winning time & money)
 - “Audit resistant”

Ačīū!

