Sample preparation

with DLLME and the

bilimex®-Assortment

from

LABC-Labortechnik

Brochure Nr.: 154_Vol1

DLLME with them **Bilimex®-Assortment**

Effective and automatable method for the concentration of liquid mixtures

In chemical analysis, dispersive liquid-liquid microextraction (DLLME) and its modifications are established sample preparation methods for the concentration of an analyte and the separation of interfering matrixes. The DLLME can be carried out quickly and with low material costs at high efficiency.

In the DLLME, small amounts of a water-immiscible solvent (extraction agent) and a water-miscible solvent (dispersion agent) are added to an aqueous sample. After mixing by emulsion formation, 2 phases are obtained by centrifugation. The organic phase (extracting agent) with the analyte concentrated and freed from interfering matrix is taken from a microlitre syringe via a cannula and analysed.

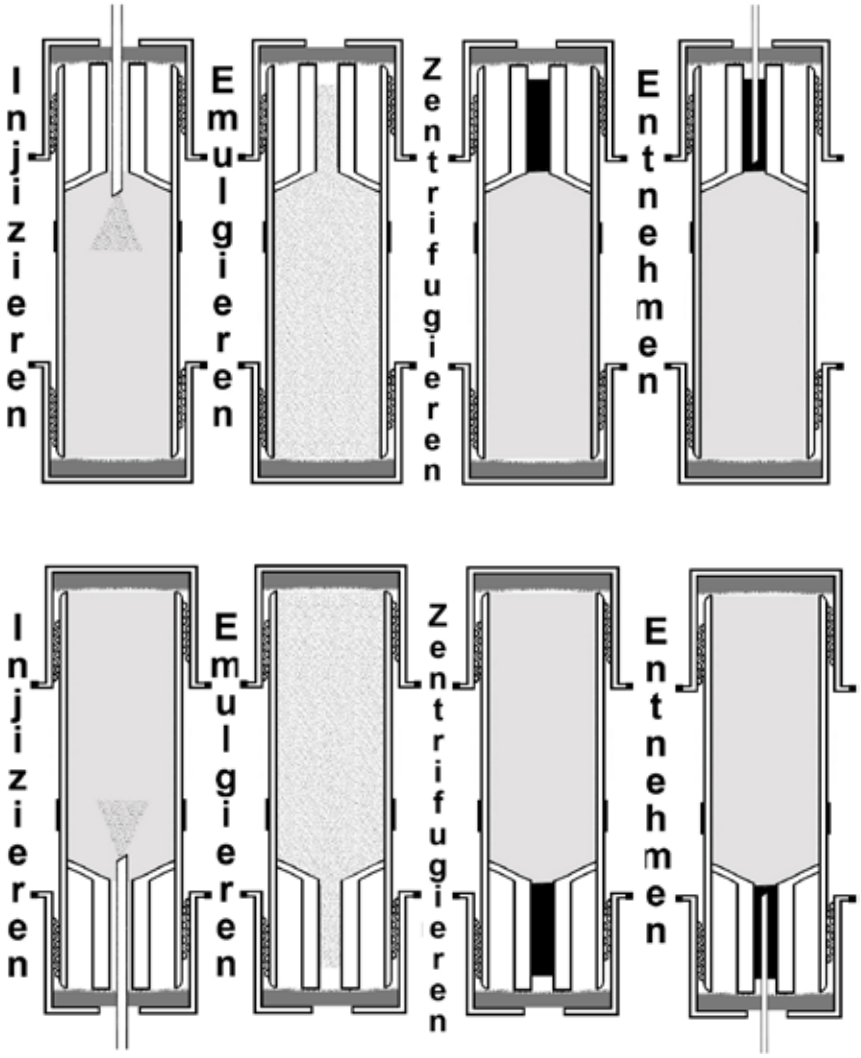
With the DLLME we differentiate between an extraction agent with a lower density (LD) and an extraction agent with a higher density (HD) than water.

LABC-Labortechnik offers sample vessels for the manual DLLME as well as for the automated DLLME. Each for samples with an extracting agent lighter (LD) and heavier (HD) than water.

The advantages of the DLLME sample preparation method:

- More universal than SPME
- less costly than solid phase extraction
- low solvent consumption
- simple concentration
- high recovery rate and reproducibility

In the basic structure of the DLLME, an aqueous sample is mixed with a water-immiscible solvent (extraction agent) and a water-miscible solvent (dispersion agent). After mixing and emulsion formation 2 phases are obtained by centrifugation. The organic phase (extracting agent) with the analyte concentrated and freed from interfering matrix is analysed with a microlitre syringe after sampling.



Examples of use:
For an LD-DLLME, 11.2ml of aqueous solution is provided and 300µl of solvent mixture is injected.

Example 1
For a complete extraction, this corresponds to an enrichment by a factor of 37.3.
Using a microlitre syringe, approximately 250µl can be extracted above the phase boundary for analysis.

Example 2
For a HD-DLLME, 11.5 ml of aqueous solution is prepared and 30 µl of solvent mixture is injected.
With a complete extraction this corresponds to an enrichment by a factor of 383. With a microlitre syringe approx. 25µl below the phase boundary can be extracted for analysis.

bilimex®-MAN

classic sample container for the manual DLLME. The glass container is designed for method development.

bilimex®-HD-MAN
Centrifuge tubes of borosilicate 3.3
Tip bottom 60° and glass thread (GL).
Centrifugeable up to 1800g.
Scope of delivery: without screw cap

Article n°	Volume	Ø	Height	GL-Thread
120-0644198	9 ml	16 mm	100 mm	GL14
120-0644211	9 ml	16 mm	100 mm	GL18
120-0591805	23 ml	24 mm	100 mm	GL25
120-0644228	37 ml	28 mm	100 mm	GL25
120-0644242	45 ml	34 mm	100 mm	GL25
120-0019507	75 ml	44 mm	100 mm	GL32
120-0644259	75 ml	40 mm	115 mm	GL32
120-0644266	240 ml	56 mm	147 mm	GL45

bilimex®LD/HD-MAN is a liquid-liquid micro-extractor for the sample preparation method DLLME (dispersive liquid-liquid microextraction) with an extraction agent that is lighter (LD =low density) or heavier (HD = high density) than water. bilimex®-LD/HD-Manual is an extractor for manual injection and removal via microlitre syringe. bilimex®-LD/HD-MAN can be cleaned and used several times.

bilimex®LD/HD-MAN
Centrifuge tubes made of borosilicate 3.3 with fused-in capillaries and glass threads on both sides (GL 25).
With PTB-red screw cap and PTFE-coated 3mm silicone seals.
Centrifugable up to 1800g.

Optional: silanised version (IS-2 inerting)

Article n°	Volume	Capillary volume	Length
280-0396615	17 ml	35 µl	100 mm
280-0396592	17 ml	100 µl	100 mm
280-0400701	12 ml	35 µl	80 mm
280-0400725	12 ml	100 µl	80 mm
280-0400695	7 ml	35 µl	60 mm
280-0400718	7 ml	100 µl	60 mm

Spare parts bilimex®-MAN

bilimex®-MAN can be used several times, only the septa have to be exchanged.

Schraubverschlusskappe PBT, red, with PTFE-laminated 3mm Silikon-seal

GL-Thread	Hole	Article n°.	Hole	Article n°.
GL 14	no	120-0019217	9,5 mm	GL14
GL 18	no	120-0159920	11 mm	GL18
GL 25	no	120-0190831	15 mm	GL25
GL 32	no	120-0190671	20 mm	GL32
GL 45	no	120-0019231	34 mm	GL45

GL-Srewcap with hole made by PBT, red, Schott with 4mm Bore to prevent bulging of the septum during centrifugation

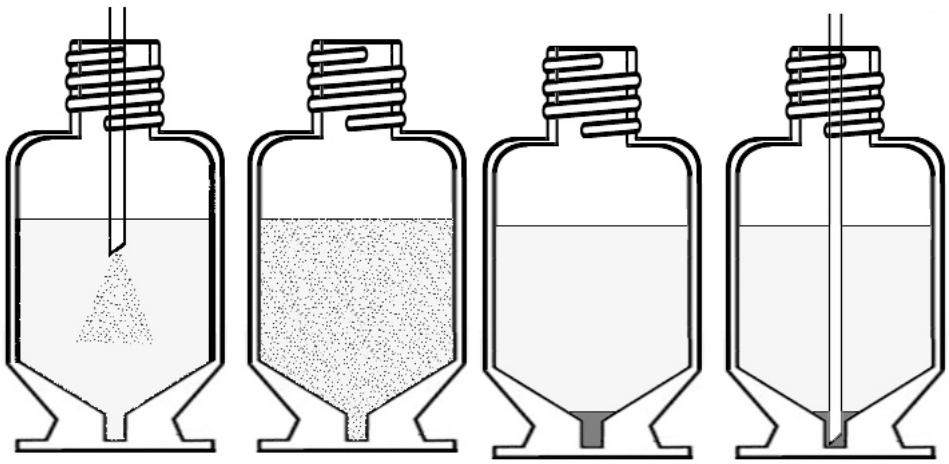
Article n°.	GL-Thread	Bore	VPE
120-0940351	GL14	4 mm	1 Stk.
120-0940368	GL18	4 mm	1 Stk.
120-0940375	GL25	4 mm	1 Stk.
120-0940399	GL32	4 mm	1 Stk.
120-0940412	GL45	4 mm	1 Stk.

Optional septa to GL - screw cap GL 25

Article n°	Material	GL-Thread
120-0400626	Butyl rubber red / PTFE grey, 2.4mm thick, 55 shore A	GL25

If a silicone seal is not practical for an analysis, a sealing washer made of BK/PTFE could be an alternative. We would be happy to manufacture other sizes and alternative septum materials for GL screw caps on request.

bilimex®HD-AS is a liquid-liquid micro-extractor for the sample preparation method DLLME (dispersive liquid-liquid microextraction) with an extractant that is heavier (HD = high density) than water. bilimex®-HD-AS is an extractor for a automated injection and withdrawal via microlitre syringe.

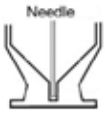


In the outlined DLLME, an aqueous sample is mixed with a water-immiscible extraction agent and a water-miscible dispersion agent.
After mixing and emulsion formation, 2 phases are separated by centrifugation. The extraction agent - HD - with the concentrated analyte freed from interfering matrix is located in the conical base. It is extracted by a microlitre syringe and indexed into the analyser.

The extraction medium can be easily removed by using a conical bilimex tube. Bilimex®HD-AS can be used in centrifuges up to 1800g and is designed for DLLME with extraction agents in high desity (HD).

The advantages of bilimex®HD-AS for automating the DLLME-HD

bilimex®HD-AS with a high-quality PTFE-coated silicone septum and a magnetic barrel screw cap for automated operation by autosamplers.



bilimex®HD-AS with an optimised residual drainage through a cylindrical instead of a conical design in the interior of the vial is the ideal separating funnel in the DLLME-HD for extraction agents that have a higher density (high density) than water.

bilimex®HD-AS with stand for convenient handling during sample preparation with the DLLME-HD method. bilimex®HD can be used in centrifuges with swing-out rotors up to 3500 rpm.

bilimex®HD-AS
made of brown / clear glass of the first hydrolytic class
23 x 72mm
bottom conical with stand
magnetic headspace holescrew cap green
silicone/white PTFE septum
can be used up to 150°C
CTC autosampler suitable
Centrifugable up to 1800g

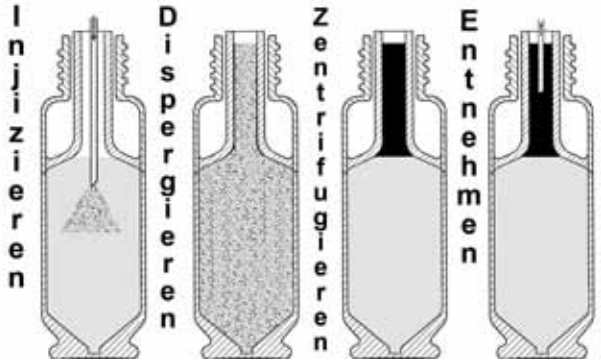
Optional: silanised version (IS-2 inerting)

Article n°.	Sample volume	KG/BG	IS-2 Inerting	Size
280-0397322	8 ml	Clear glass	no	42 x 23 mm
280-0400930	8 ml	Clear glass	yes	42 x 23 mm
280-0397414	8 ml	Amber glass	no	42 x 23 mm
280-0400602	8 ml	Amber glass	yes	42 x 23 mm
280-0401968	17 ml	Clear glass	no	72 x 23 mm
280-0404877	17 ml	Clear glass	yes	72 x 23 mm
280-0404907	17 ml	Amber glass	no	72 x 23 mm
280-0404921	17 ml	Amber glass	yes	72 x 23 mm

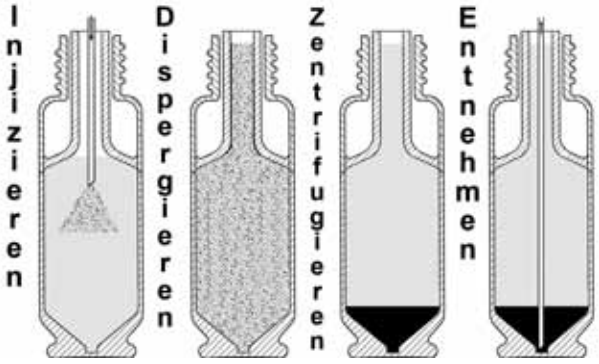
IS-2 inertisation is a new deactivation process. It ensures maximum inertness of the glass surface and prevents adsorption even of sensitive substances such as pesticides, amines, steroids and phenols.

The centrifugability according to EN 10204 is tested per batch. The test is carried out with 1800g in a fixed-angle rotor at room temperature and a substance mixture with a density of max. 1.2 g/ml.
The batch certificate is supplied.

bilimex®-LD/HD-AS is a liquid-liquid micro-extractor for the sample preparation method DLLME (dispersive liquid-liquid microextraction). The extractant can be heavier (high desity HD) and lighter (low density LD) than water. Bilimex®-LD/HD-AS is an extractor for a automated injection and withdrawal via microlitre syringe.



DLLME (LD =low density)



DLLME (HD =high density)

In the DLLME outlined on the left, an aqueous sample is mixed with a water-immiscible extractant and a water-miscible dispersant. The extraction agent is low density LD.
After mixing and emulsion formation, the two phases are separated by centrifugation. The extracting agent - LD - with the concentrated analyte freed from interfering matrix is located in the inner funnel. It is extracted through a microlitre syringe and injected into the analyser. With bilimex®LD/HD-AS a DLLME with LD or HD extraction agent is possible. For this purpose, see the procedure sketches above.

The extraction agent can be easily removed by using bilimex® vials with internal funnels. Bilimex®LD/HD-AS can be used in centrifuges up to 1800g. and is designed for DLLME with extraction media in low density (LD) or high density (HD). The centrifugability is documented with a batch test certificate. The vial can be loaded with a liquid of 1.2g/ml density up to at least 1800g.

The vial is manufactured in Germany in a complex production process. The bilimex®-LD/HD-AS vials are cleaned in a laboratory dishwasher or (automatically e.g. with an MHE tool for CTC autosampler. In this case, a liquid microlitre syringe is used instead of a headspace microlitre syringe to remove the solvents. Spare septa are also available as accessories made of other materials.

Advantage of the DLLME LD in bilimex®LD/HD-AS:

Low density (LD) solvents suitable as extraction agents are more environmentally friendly.

bilimex®LD/HD-AS Headspace Screw Vial
made of amber / clear glass of the first hydrolytic class
23 x 72 mm
conically narrowed at the top and bottom with stand
Centrifugable up to 1800g
Suitable for CTC autosampler

Optional: silanised version (IS-2 inerting)

Article n°.	Sample volume	KG/BG	Size	Thread
120-0934206	10 ml	Clear glass	72x23 mm	HS6-19
120-0750035	10 ml	Amber glass	72x23 mm	HS6-19

bilimex®LD/HD-AS Crimp
made of brown / clear glass of the first hydrolytic class
23 x 75 mm
top and bottom conical with base
ND20 flared rim
loadable up to 150°C
Centrifugable up to 1800g
Suitable for CTC autosampler

Optional: silanised version (IS-2 inerting)

Article n°.	Sample volume	KG/BG	Size
120-0941730	10 ml	Clear glass	75x23 mm
	10 ml	Amber glass	75x23 mm

The centrifugability according to EN 10204 is tested per batch. The test is carried out with 1800g in a fixed-angle rotor at room temperature and a substance mixture with a density of max. 1.2 g/ml.
The batch certificate is supplied.

bilimex®LD PTFE165/Glass is a liquid-liquid micro-extractor for the sample preparation method DLLME (dispersive liquid-liquid microextraction). The extraction agent is lighter (low density LD) than water. bilimex®-LD PTFE165/Glass is an extractor for automated injection and withdrawal via microlitre syringe in routine analysis.

bilimex®LD PTFE165/glass is inexpensive to produce and also allows DLLME with a solvent lighter than water. It is particularly suitable for routine applications with very high throughput, where cleaning of the bilimex® extractors is not desired.

The vial closure is magnetic and fits the common autosampler systems.

Bilimex®LD PTFE165/Glass consist of ND18 fine thread bottles with rounded bottom and 1st hydrolytic class glass, as well as a PTFE insert with a capillary volume of 165 µl. Bilimex®LD is suitable for use in centrifuges up to 1800g. A factory certificate according to EN 10204 is provided for each batch.
VPE = 10 pieces

Volume	Size	KG/BG	Article n°
10ml	46 x 22,5mm	Clear glass	120-0933513
10ml	46 x 22,5mm	Amber glass	120-0933544
20ml	75,5 x 22,5mm	Clear glass	120-0933537
20ml	75,5 x 22,5mm	Amber glass	120-0933551

Magnetischer Schraubverschlüsse ND18 für Bilimex®LD PTFE165/Glas finden Sie in unserer Broschüre 152 oder auf www.LABC.de

Dispersive Liquid-Liquid-Microextraction with the bilimex®-assortment

In the alternative sample preparation method Dispersive Liquid-Liquid-Microextraction (DLLME), the desired analyte is extracted from aqueous solutions with a small amount of organic solvent and simultaneously concentrated.

Dispersive liquid-liquid microextraction (DLLME) is fast, automated, requires little material and can be performed with high efficiency. In this method, the analyte separation is carried out by extracting aqueous samples with 10 µl to approx. 300 µl of a water-immiscible organic solvent (extractant), which has a higher or lower density than water, and a water-miscible solvent (dispersant). The organic solvent (with higher density) used is halogenated hydrocarbons or ionic liquids. The miscible solvent is, for example, acetone or methanol. Hydrocarbons are used as the organic solvent (with a lower density) and acetone or ethanol, for example, as the miscible solvent.

The DLLME method achieves high recoveries and enrichment factors.

After adding the extraction and dispersion agents, a dispersion forms either spontaneously or after a short shaking - possibly also with ultrasound irradiation - in which the non-polar analytes dissolve abruptly into the finely dispersed organic extraction agent droplets. The formation of the emulsion increases the surface area of the non-polar phase and an equilibrium is immediately established. In the final centrifugation step, the dispersed droplets with the enriched analytes are centrifuged off and the analytes freed from the interfering matrix are removed by sampling with a microlitre syringe and analysed.

For the requirement of LD and HD DLLME, LABC-Labortechnik developed four products of the bilimex® range.

The bilimex®-HD-MAN and the bilimex®-LD/HD-MAN were developed for manual DLLME. In LD-DLLME, the organic phase (extraction agent), which is lighter than water (LD, low density), separates at the top of the capillary. In HD-DLLME, the organic phase (extracting agent), which is heavier than water (HD, high density), is deposited at the bottom of the capillary by rotating the bilimex® by 180°. By piercing the septum with the needle of a microlitre syringe, the separated organic phase (extractant) can be removed and analysed.

The bilimex®-HD-AS, bilimex®-LD/HD-AS and Bilimex®LD PTFE165/Glass were developed for DLLME in the autosampler.

The bilimex®-HD-AS extractor made of glass in vial form with magnetic cap enables automatic injection and withdrawal via microlitre syringe and autosampler. Its conical bottom is suitable for the DLLME-HD. It is used as a disposable item.

The bilimex®-LD/HD-AS extractor made of glass in vial form with magnetic cap enables automatic injection and withdrawal via microlitre syringe and autosampler. Its conical bottom and the fused funnel on top are suitable for DLLME-HD and LD. It can be used and cleaned several times.

Bilimex®LD PTFE165/Glass is for extractor made of glass in vial form with magnetic cap allows automatic injection and withdrawal via microlitre syringe and autosampler. The PTFE insert in the standard vial for the DLLME-LD. it is used as a disposable item and is compatible with all ND18 caps.

Contact:

For additional information and/or your orders, please contact:

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