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Fatty Acid Methyl Esters with BF_3

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Chemicals

dd H_2O	in-house distillery
C17:0 fatty acid	Sigma H3500
TolueneI	Merck 1.08325.2500
BF_3 -complex	Sigma B1252
CHCl_3	Sigma 34854
Hexane	Roth T.861.1
Methanol	Roth P.717.1

solutions

internal standard	C17:0 fatty acid 250 mM in methanol
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Procedure

General remarks

The glas tubes have to be absolutely clean, especially the cap and the sealing. Pyrex-tubes with screw-caps and grey Teflon-sealing turned out to be most suitable.

Procedure

- add 100 μl standard solution to solvent solution in Pyrex-glas-tubes
- dry under stream of nitrogen
- add 0.5 ml toluene
- add 2 ml BF_3 -complex
- vortex briefly and close glas tube
- incubate 1 h at 100 $^\circ\text{C}$ in drying oven
- cool samples in ice-bath
- add 1ml ice-cold water
- add 2 ml hexane/ CHCl_3 (4/1, v/v)
- shake for 15 min
- centrifuge (2000 rpm, 5min)
- remove and collect (!) upper phase
- add 1ml ice-cold water
- add 2 ml hexane/ CHCl_3 (4/1, v/v)
- shake for 15 min
- centrifuge (2000 rpm, 5min)
- remove upper phase, pool with first upper phase
- dry under stream of nitrogen
- add 500 μl hexane
- vortex briefly
- dry under stream of nitrogen
- resolve in 100 μl hexane
- vortex briefly
- transfer into autosampler vials