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**New supported liquid membrane for electromembrane extraction of polar basic endogenous metabolites.**

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**Highlights:**

- 22 organic solvents were screened for the electromembrane extraction of 45 highly polar basic metabolites
- A new multi-segment injection CE-MS/MS method was developed, with electrokinetically injected plugs between the samples
- Nitrophenyl pentyl ether was identified the most efficient solvent to extract polar basic compounds
- High recovery (up to 90%) and low variability (down to 2%) were achieved on neat standards
- A large panel of endogenous polar metabolites was extracted from biological samples with the new approach

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