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Method development and validation of a quantitative LCMS method for a panel of food intake biomarkers

Background

•Food intake biomarkers (BFIs) are used as objective tools to estimate food intake 1,2. However only few BFIs are validated for this purpose and for many important foods, biomarkers are currently lacking.

• We have identified 36 potential BFIs, that have been shown in previous studies to reflect the intake of foods or food groups commonly consumed in western diets. A controlled dietary intervention in humans was performed where combination of the specific foods were provided in single and repeated doses to establish pharmacokinetic parameters inlcuding half-life and dose-response. To validate the identified candidate biomarkers as specific biomarkers of the foods tested, a rapid multi-method is needed. The aim of this project is therefore to establish a method and apply it to samples from the intervention study.

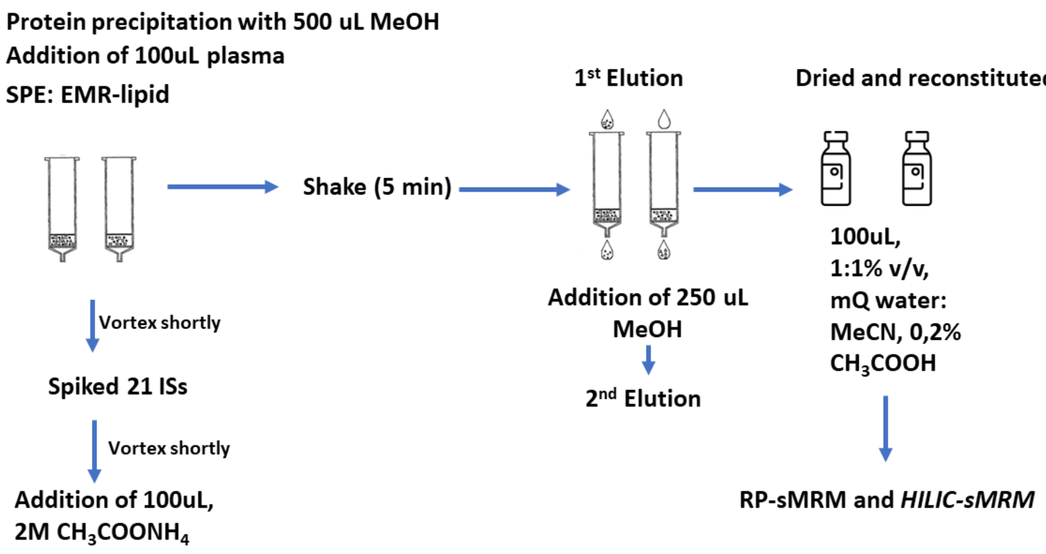
Aim

•Quantification and validation of 36 BFIs in blood plasma with a high-throughput LCMS assay, including separation in HILIC and RP chromatography and scheduled MRMs in positive and negative ionization mode.

•Method to be applied and validated for human blood plasma samples from intervention study.

•To fill the gap between academic research and the methodological requirements necessary for the developments of clinical and commercial applications for diet assessments.

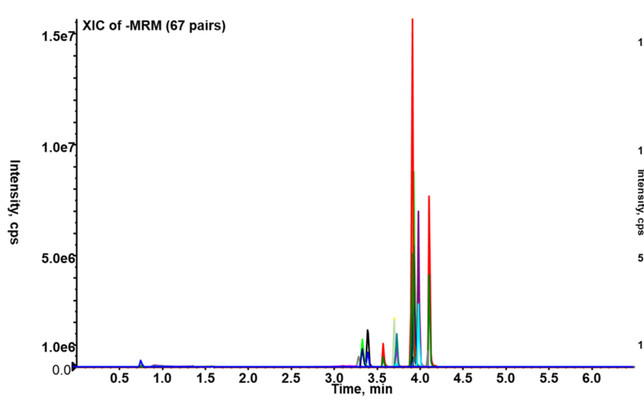
Sample purification



Intervention study

Figure 1: Intervention study in which the current method will be emplyed. During the intervention weeks 1,3 and 5 the participants consume 3 meals daily for 4 days and the proportion of food changes weekly. Weeks 2 and 4 are the wash-out periods.

Chromatographic separation



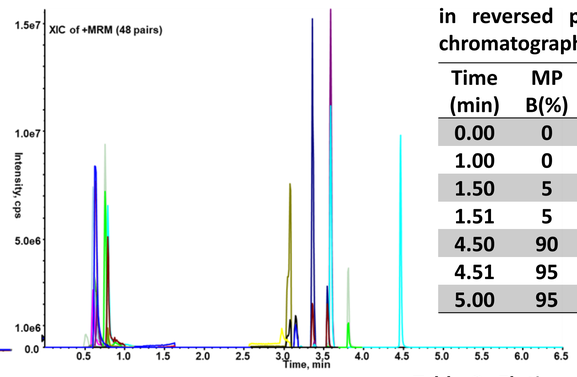
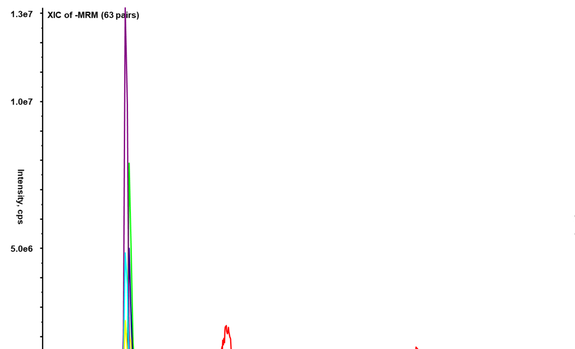


Table 1: Elution program in reversed phase (RP) chromatography

Time (min)	MP B(%)	Flow (mL/min)
0.00	0	0.4
1.00	0	0.4
1.50	5	0.4
1.51	5	0.6
4.50	90	0.6
4.51	95	0.8
5.00	95	0.8



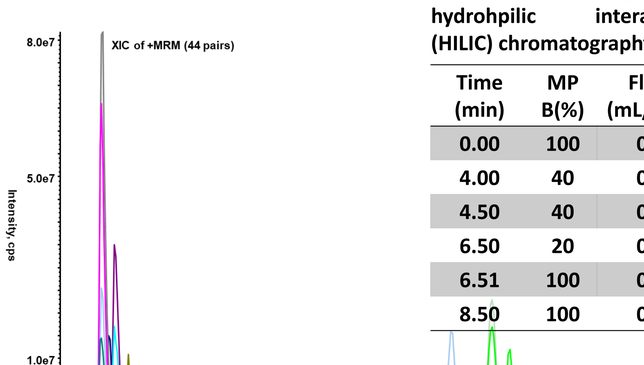


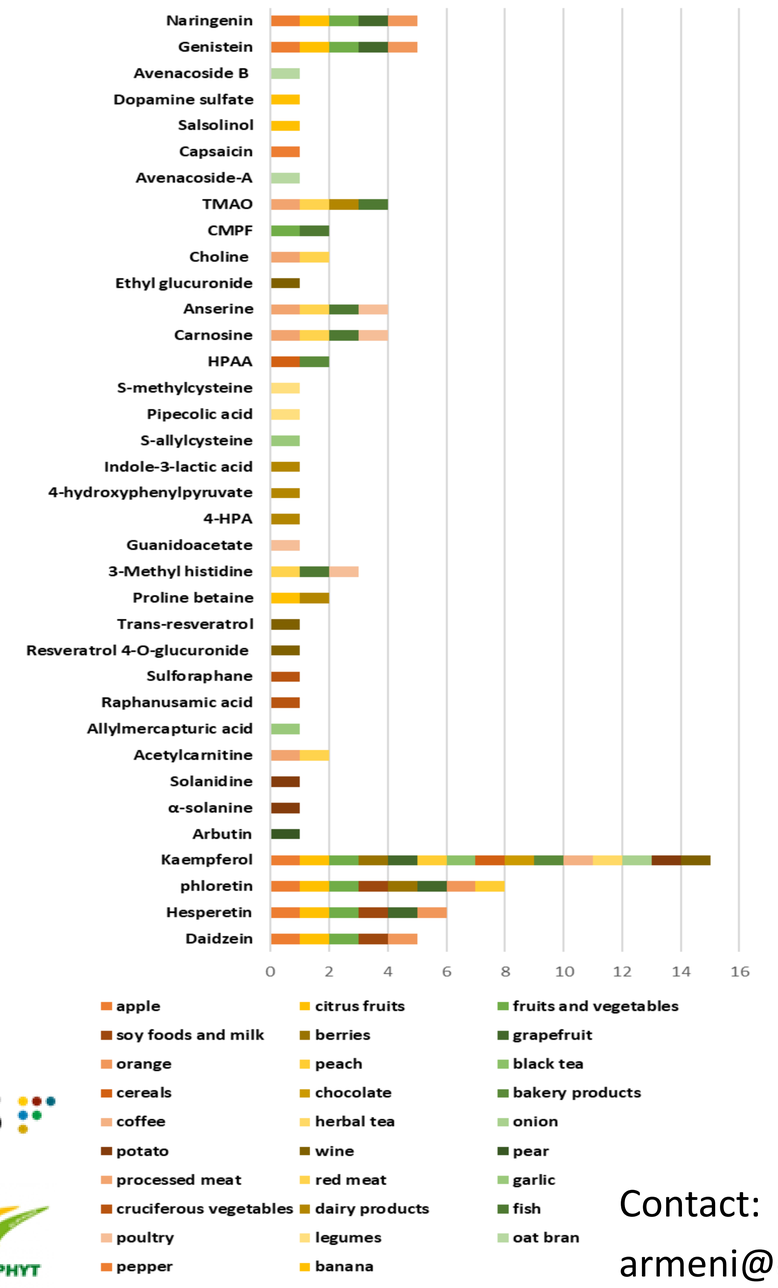
Table 2: Elution program in hydrohpilic interaction (HILIC) chromatography

Time (min)	MP B(%)	Flow (mL/min)
0.00	100	0.5
4.00	40	0.5
4.50	40	0.5
6.50	20	0.5
6.51	100	0.5
8.50	100	0.5

- **Instrument:** UPLC-MMR method; ExionLC™ coupled with QTRAP 6500+
- **Column 1**: ACQUITY UPLC BEH C18 1.7um, 2.1x 100 mm
- **Mobile Phase for RP**: A; water, 0.2% acetic acid , B; acetonitrile, 0.2% acetic acid
- **Column 2**: ACQUITY UPLC BEH Amide 1.7um, 2.1x 100 mm
- **Mobile Phase for HILIC**: A; water, 10 mM ammonium acetate, B; acetonitrile, 10 mM amm. acetate

❖ **Total time of analysis: 14.5 min**

Food Intake Biomarkers and food groups



References
1. Landberg, R., et al., Biomarkers of cereal food intake. Genes & Nutrition, 2019. 14(1): p. 28.
2.Brennan, L. and F.B. Hu, Metabolomics-Based Dietary Biomarkers in Nutritional Epidemiology-Current Status and Future Opportunities. Mol Nutr Food Res, 2019. 63(1): p. e1701064.

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