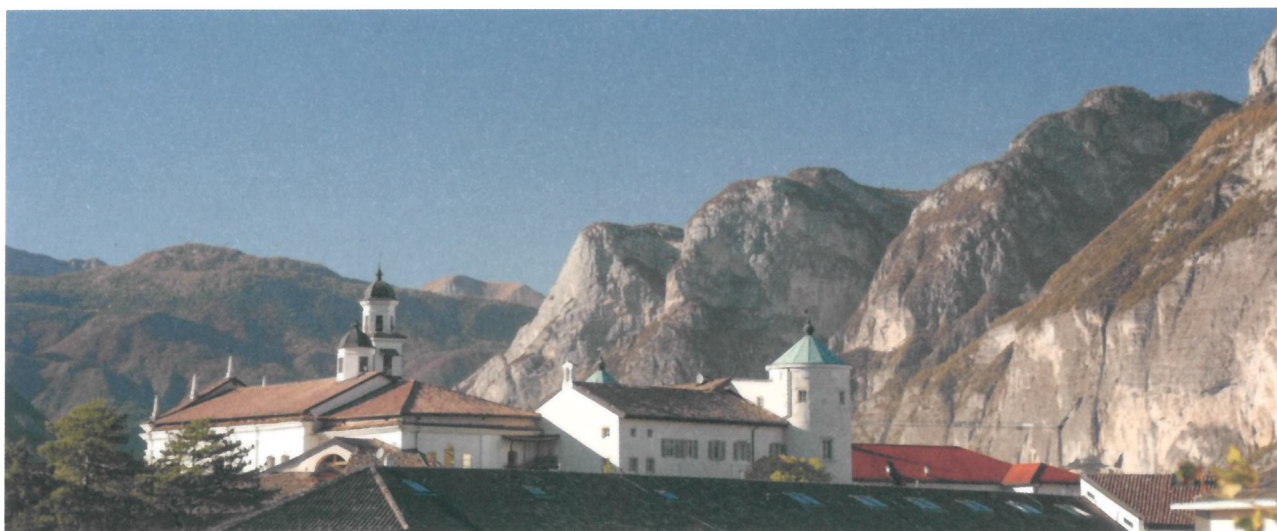


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## Untargeted metabolomics strategy based on LC-MS-Orbitrap for discovering new polyphenol metabolites in humans after acute ingestion of *Vaccinium myrtillus* berry supplement

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In this work, liquid chromatography coupled with an electrospray ionization hybrid linear ion trap quadrupole/Orbitrap mass spectrometry, has been used to accurately identify polyphenol metabolites in human serum and urine after acute ingestion of a *Vaccinium myrtillus* berry supplement. The supplement was obtained by cryo-milling of bilberries, which were freeze-dried within one week after their harvesting, so as to maintain the berry native composition. Thirty-six derivatives of benzoic acids, hydroxyhippuric acids, cinnamic acids, phenylpropionic acids, phenylvaleric acids, phenylpentenoic acids and abscisic acid, together with two berry-native anthocyanins, one flavanol metabolite and two catechol derivatives, were putatively identified in the investigated biofluids. The annotated compounds included thirteen metabolites, among glucuronides and sulphates of phenylvaleric and phenylpentenoic acids, which have been identified for the first time in human biofluids after ingestion of *V. myrtillus* berries. It should be emphasized that the presence of phenylvaleric and phenylpentenoic acid derivatives is in agreement with their origin from fruit native flavanol monomers and oligomers, which are widely distributed in *Vaccinium* berries, but usually overlooked in metabolomics studies regarding bilberry. The identification of these compounds confirmed the key-role of untargeted metabolomics approach in the discovery of new metabolites which could result biologically active

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