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# Assessment of labelled products with different radioanalytical methods: Study on <sup>18</sup>F-fluorination reaction of p-[<sup>18</sup>F]MPPF

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The crude reaction mixture consisted of p-[<sup>18</sup>F]MPPF and 2-4 labelled impurities eluting after the product fraction, and the reverse-phase HPLC method reported in the literature [1] failed sometimes to separate p-[<sup>18</sup>F]MPPF from its radioactive by-product with close retention time. Analytical HPLC was based on similar chromatographic conditions. By comparing it to preparative results it could be used in the estimation of radiochemical incorporation yield of p-[<sup>18</sup>F]MPPF. However, radio-TLC was not a reliable method for the radiochemical analysis of crude p-[<sup>18</sup>F]MPPF: the sample in DMSO was not well distinguished on silica plates and there was labelled impurity with same R<sub>f</sub>-value. Heating mechanism had no significant effect on the composition of labelled compounds.
Based on the molecular structures of the substituted benzamides, p-MPPF and its precursor p-MPPNO<sub>2</sub>, the reaction of basic nitrogen on piperazinyl moiety and/or amide hydrolysis could be reasonable explanation for the formation of side-products. Corresponding fractionated masses, molecule peaks with 192 and 120 smaller m/z ratios, were also measured in the ESI-MS studies of the inactive reference compounds. In the preliminary LC-(ESI)-MS tests of the radiolabelled product these peaks were not detected, suggesting different kind of decomposition of labelled product and/or precursor. However, p-[<sup>18</sup>F]MPPF was identified with m/z ratio of 435.
More than one radioanalytical method is needed to evaluate reliably the radiochemical incorporation of p-[<sup>18</sup>F]MPPF. Identification of various labelled impurities by radiochromatographic methods without broad reference material is difficult and characterisation by more complex method, MS, is needed. The ion trap MS seemed to be sufficient for the qualitative analysis of p-[<sup>18</sup>F]MPPF. Reliable identification of the labelled side-products, however, needs optimisation of the radioLC-method.

1) Le Bars, D. et al. Nucl Med Biol 1998; 25: 343-350

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