

Supplementary Information

Preparative Separation and Structural Identification of Impurities of a New α_2 -Adrenoceptor Agonist Using Stacking Injection, LC-MSⁿ and LC-SPE-NMR

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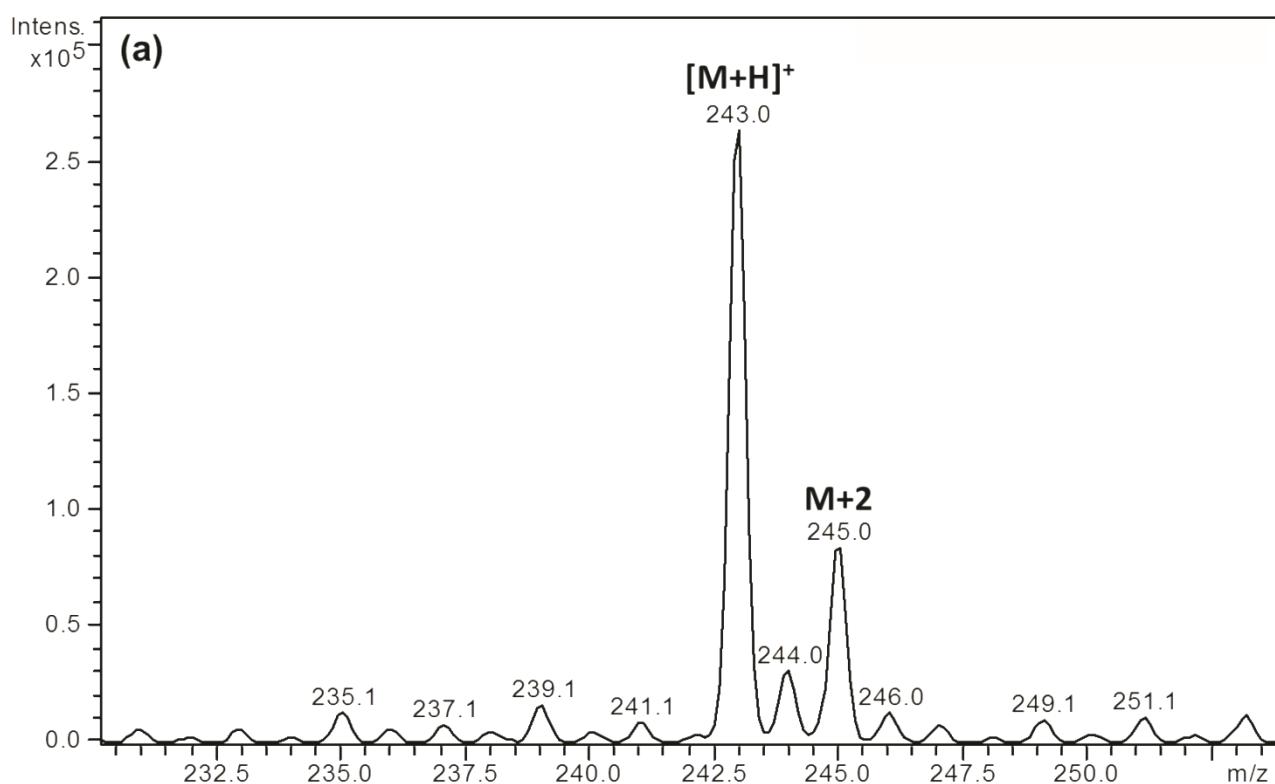


Figure S1. Mass spectra showing the presence of chlorine atoms. (a) LPSF-PT-31 and (b) its impurities.

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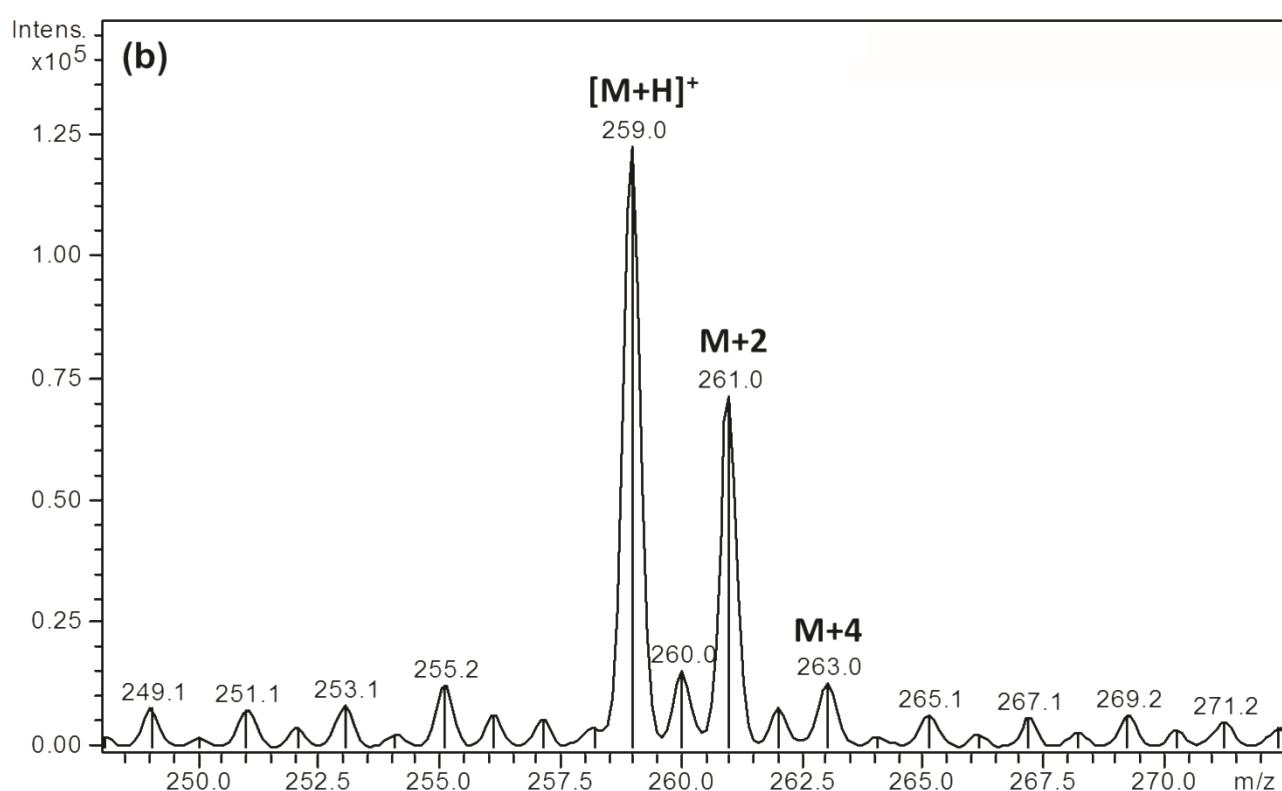


Figure S1. Mass spectra showing the presence of chlorine atoms. (a) LPSF-PT-31 and (b) its impurities (cont.).

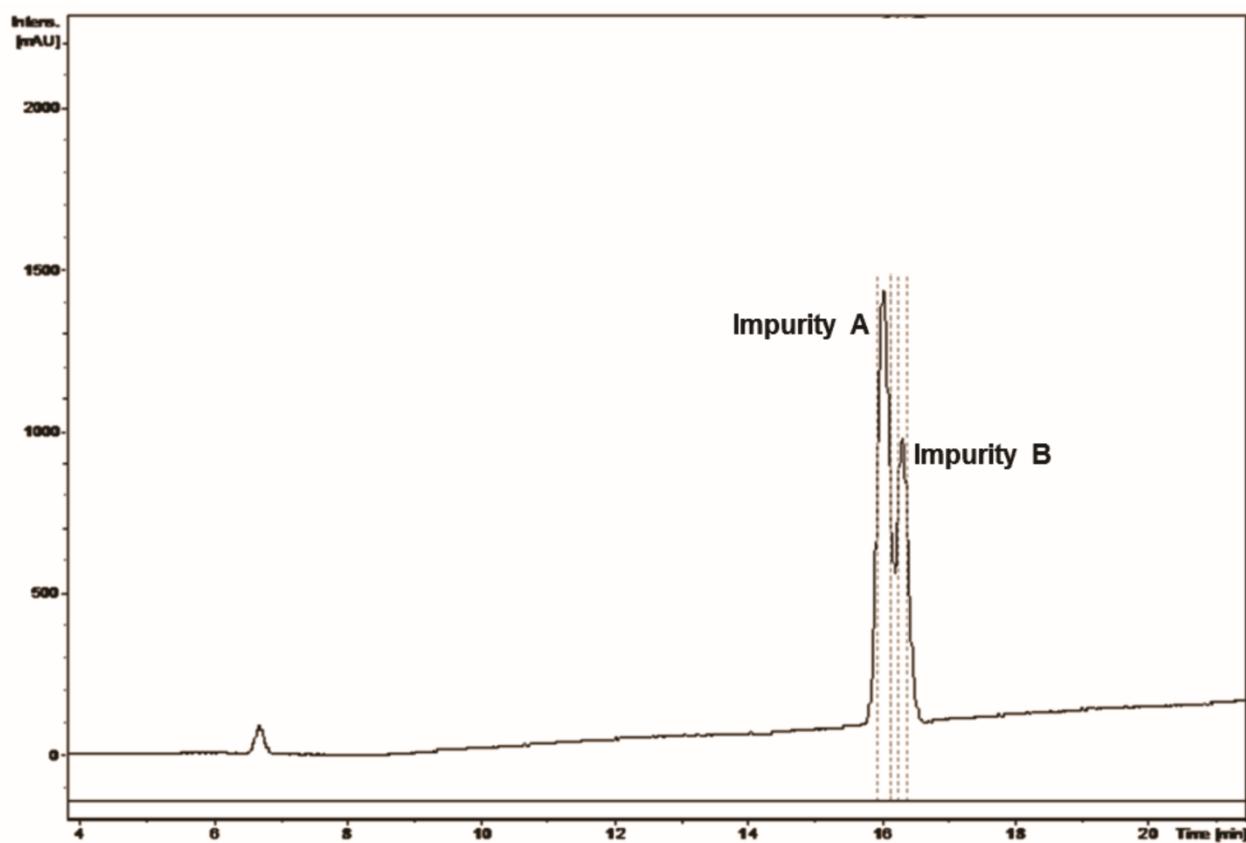


Figure S2. Chromatogram representative of the LC separation of impurities A and B and the trapping of reactions shown in the SPE cartridges.

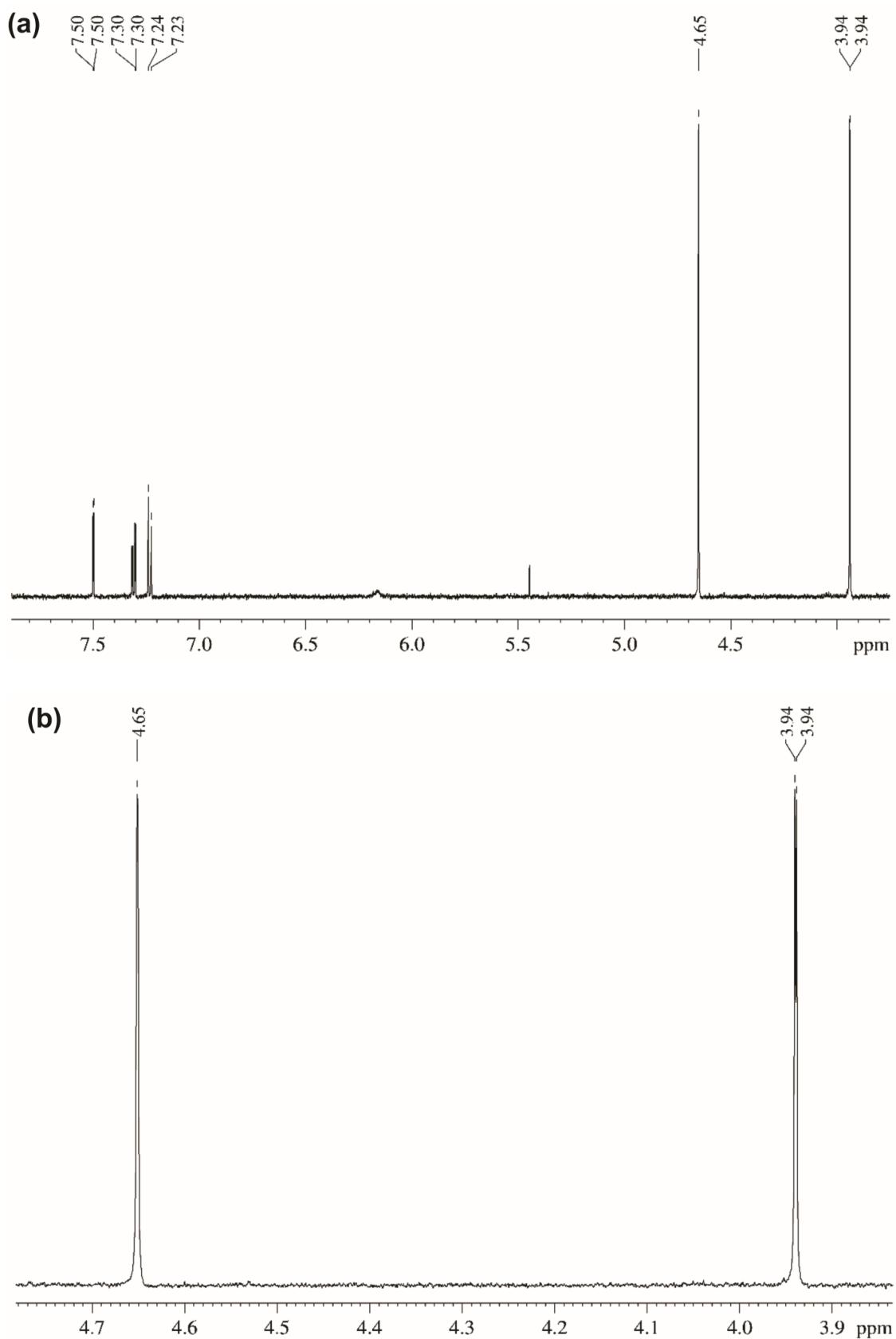


Figure S3. ^1H NMR spectra (600 MHz, $\text{MeCN}-d_3$) of LPSF-PT-31 impurity A (a) and the expanded aliphatic and aromatic regions (b and c).

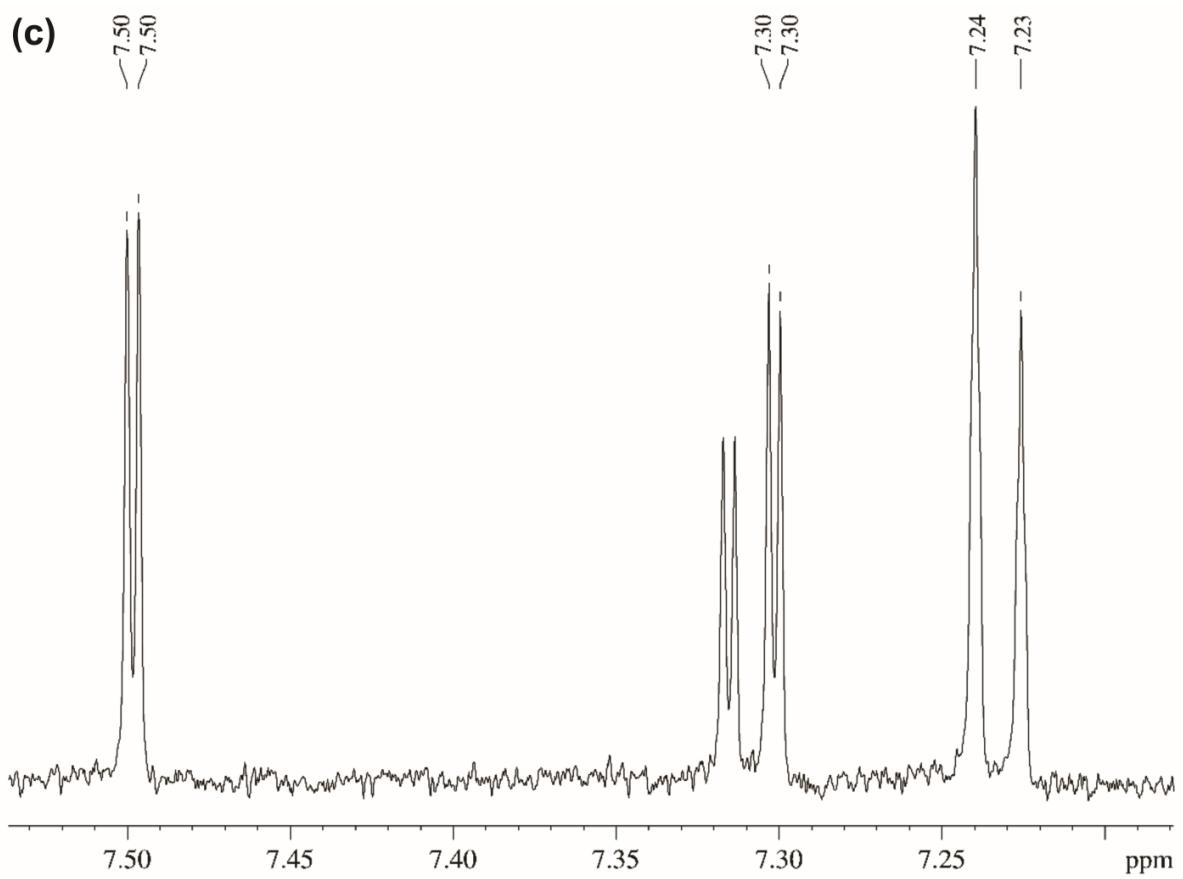


Figure S3. ^1H NMR spectra (600 MHz, MeCN- d_3) of LPSF-PT-31 impurity A (a) and the expanded aliphatic and aromatic regions (b and c) (cont.).

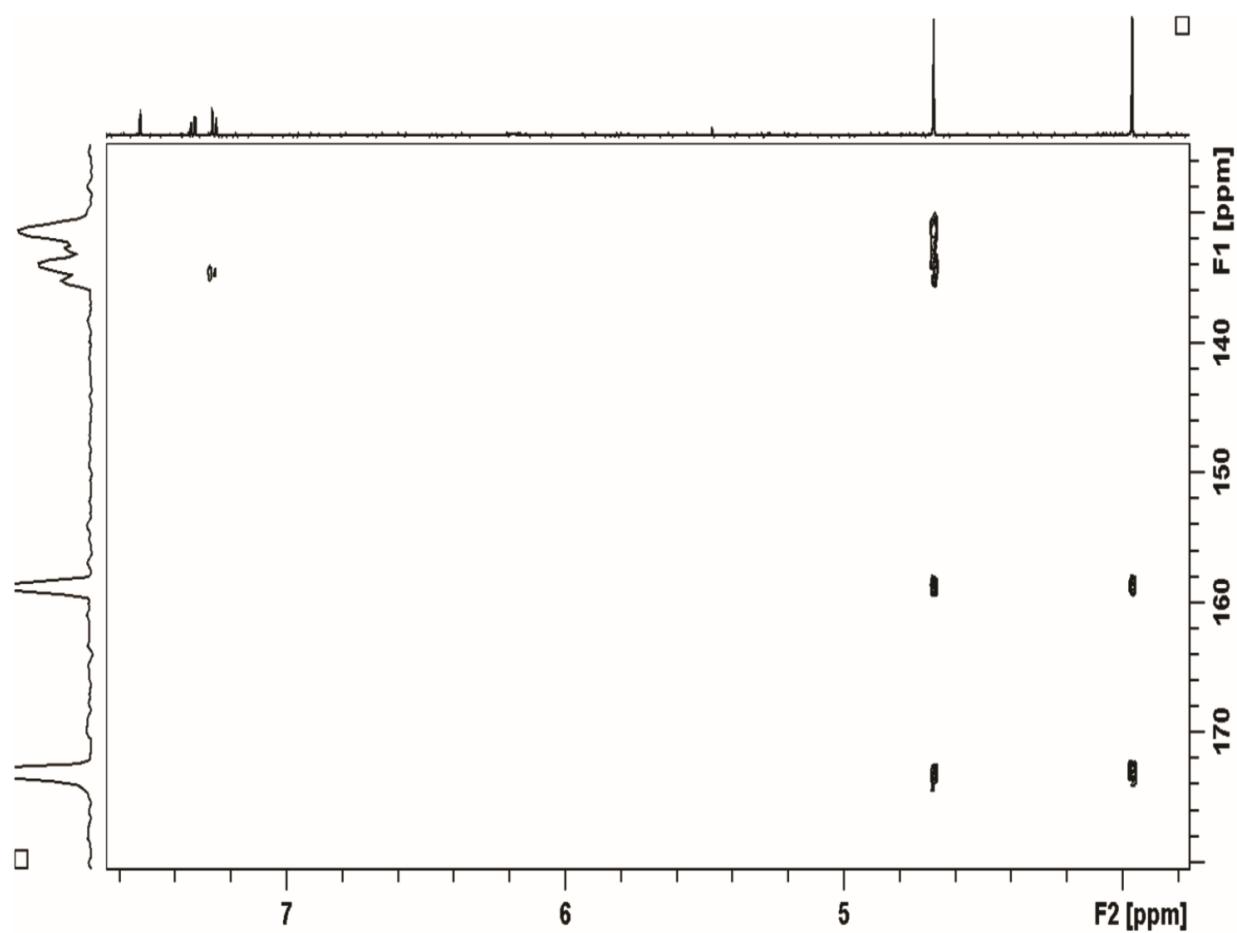


Figure S4. ¹H-¹³C g-HMBC correlation map (600 MHz, MeCN-*d*₃) of LPSF-PT-31 impurity A.

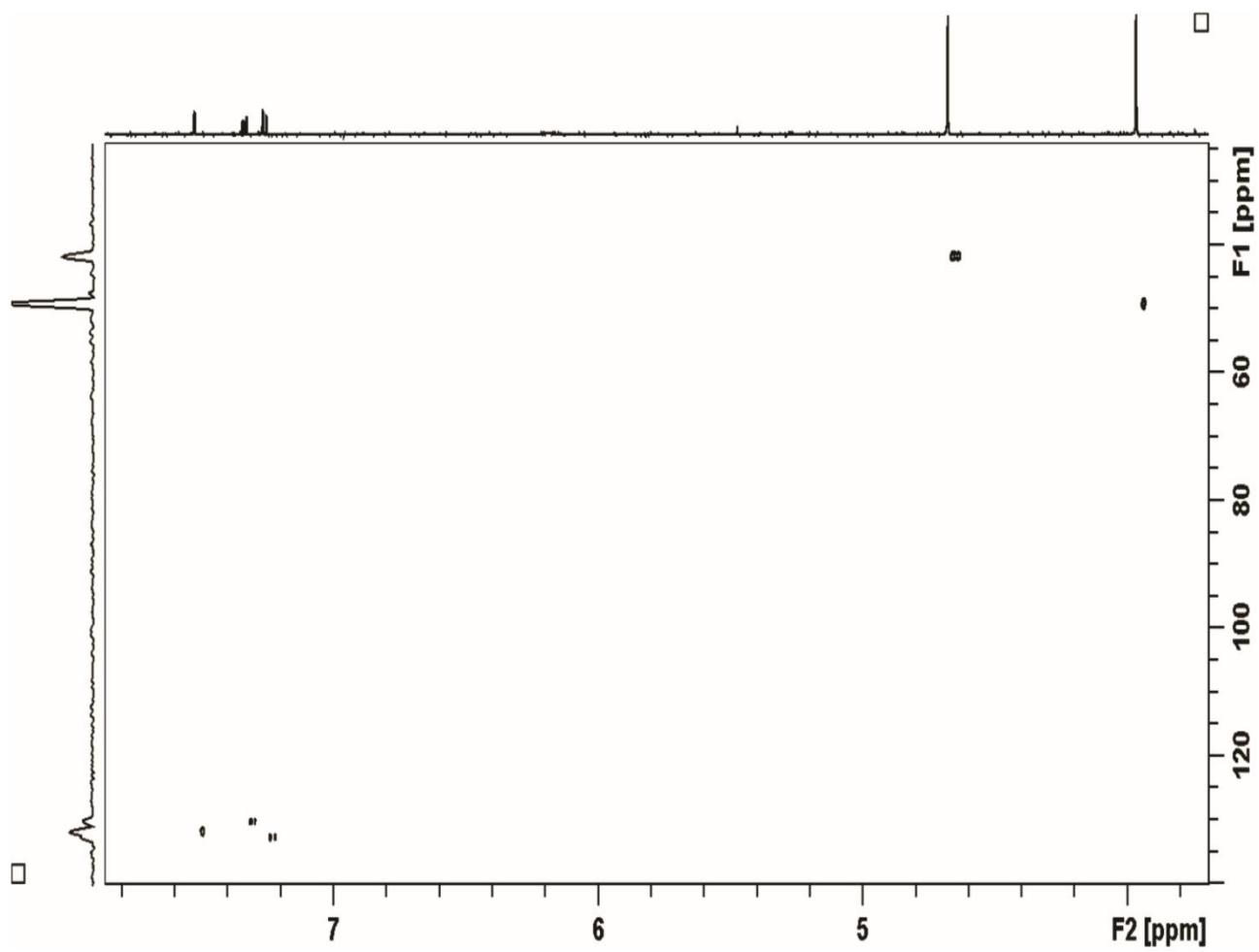


Figure S5. ^1H - ^{13}C g-HSQC spectra (600 MHz, MeCN- d_3) of LPSF-PT-31 impurity A.

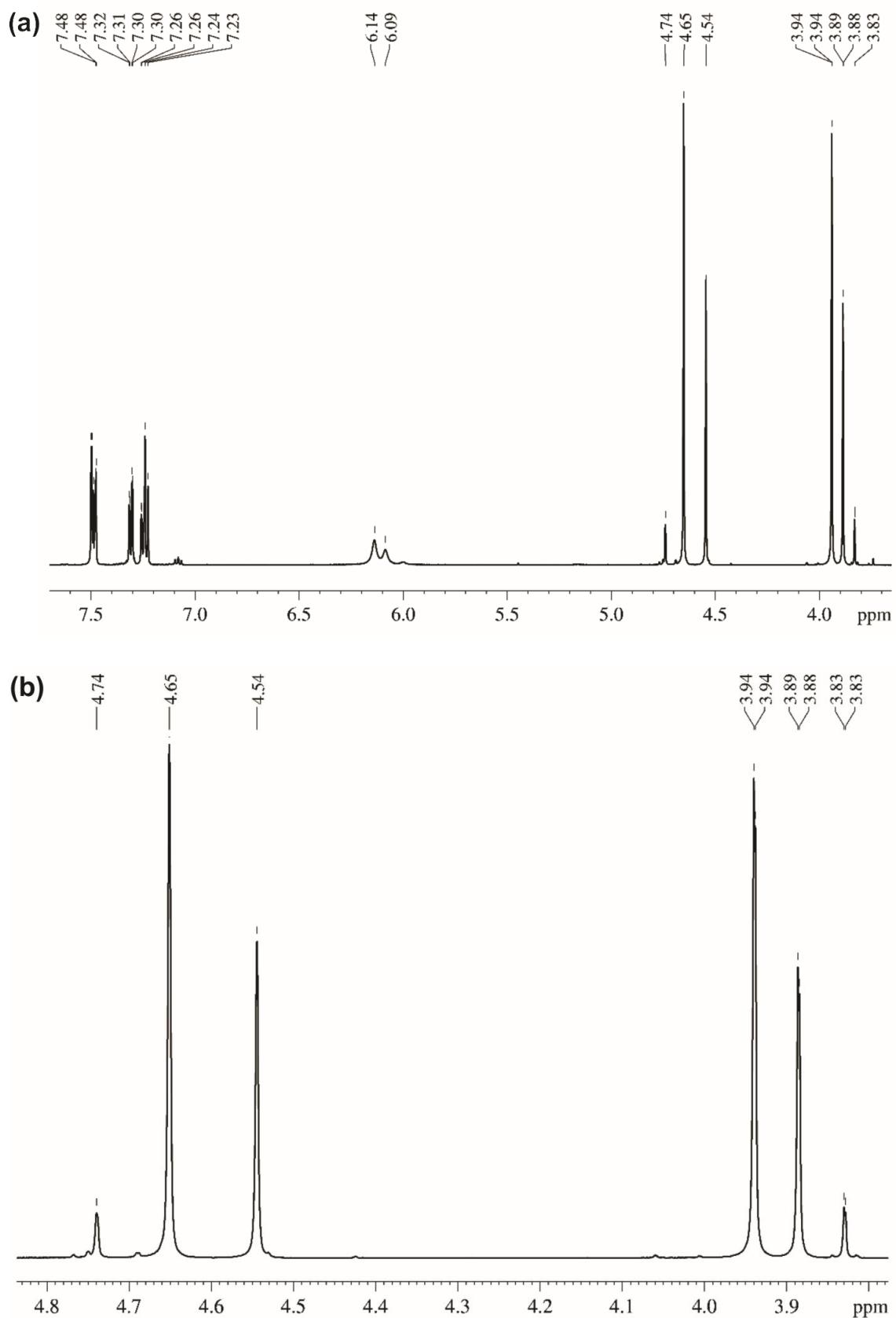


Figure S6. ^1H NMR spectra (600 MHz, MeCN- d_3) of the mixture of impurities of LPSF-PT-31 (a) and the expanded aliphatic and aromatic regions (b and c).

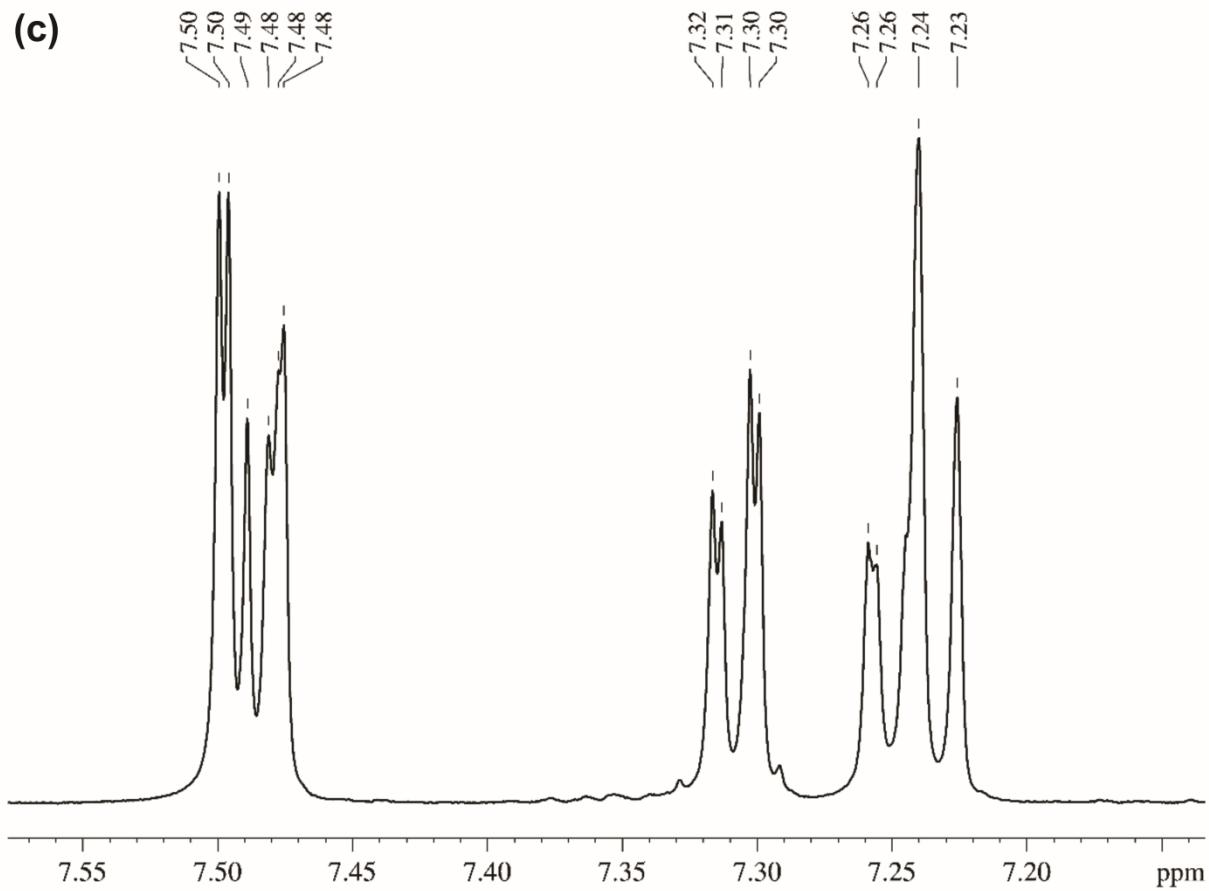


Figure S6. ^1H NMR spectra (600 MHz, MeCN- d_3) of the mixture of impurities of LPSF-PT-31 (a) and the expanded aliphatic and aromatic regions (b and c) (cont.).

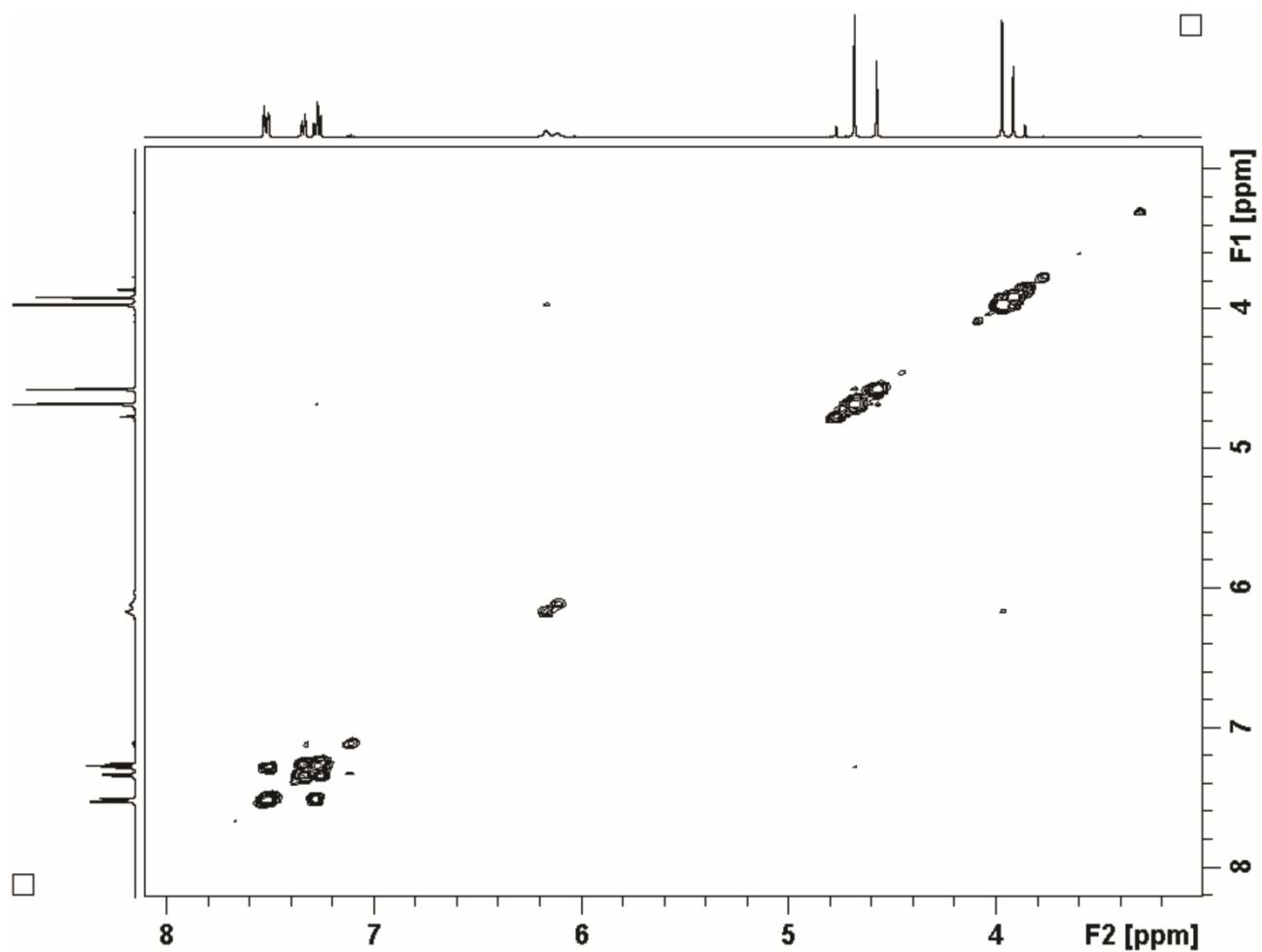


Figure S7. ^1H - ^1H g-COSY spectrum (600 MHz, MeCN- d_3) of the mixture of impurities of LPSF-PT-31.

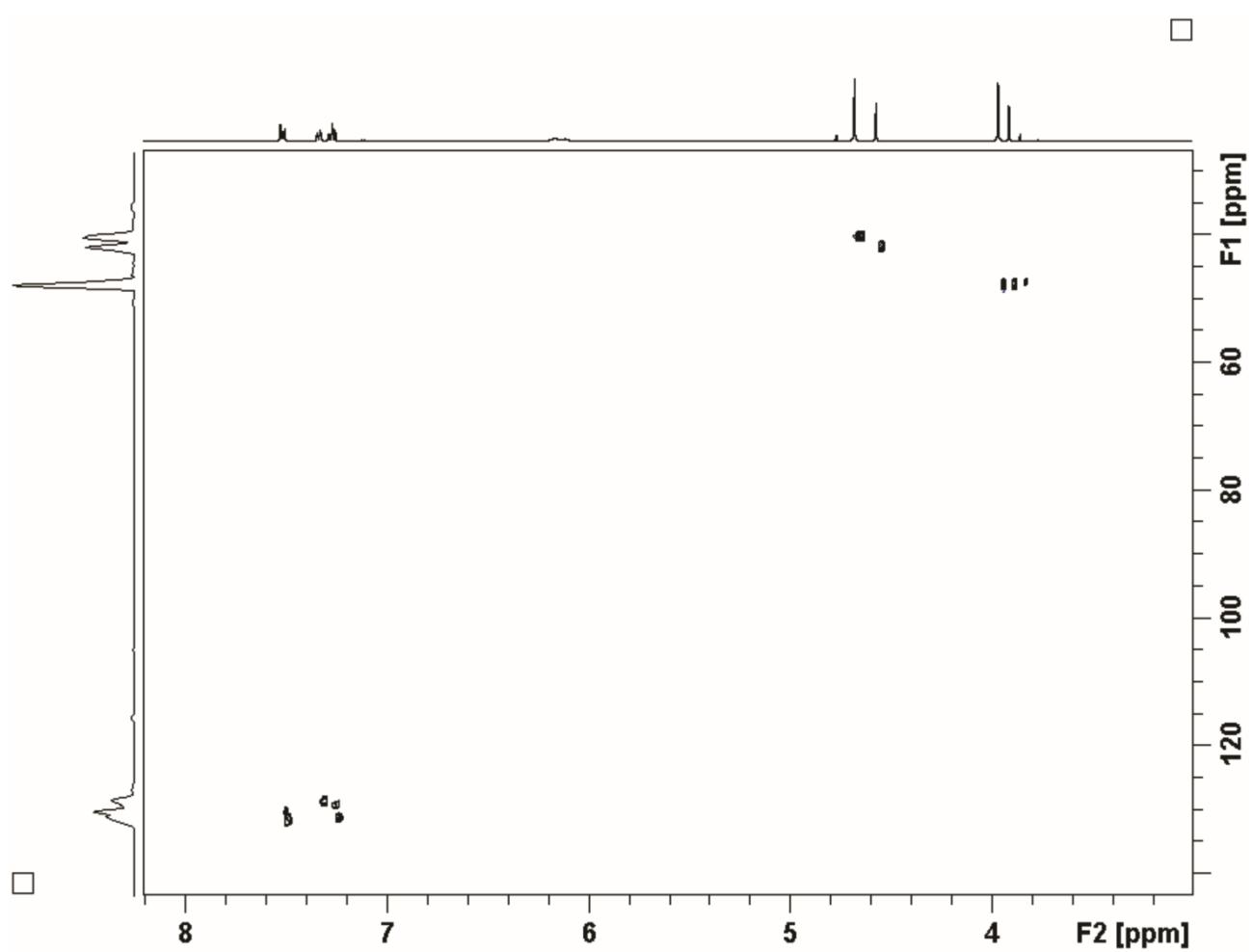


Figure S8. ^1H - ^{13}C g-HSQC spectra (600 MHz, $\text{MeCN}-d_3$) of the mixture of impurities of LPSF-PT-31.

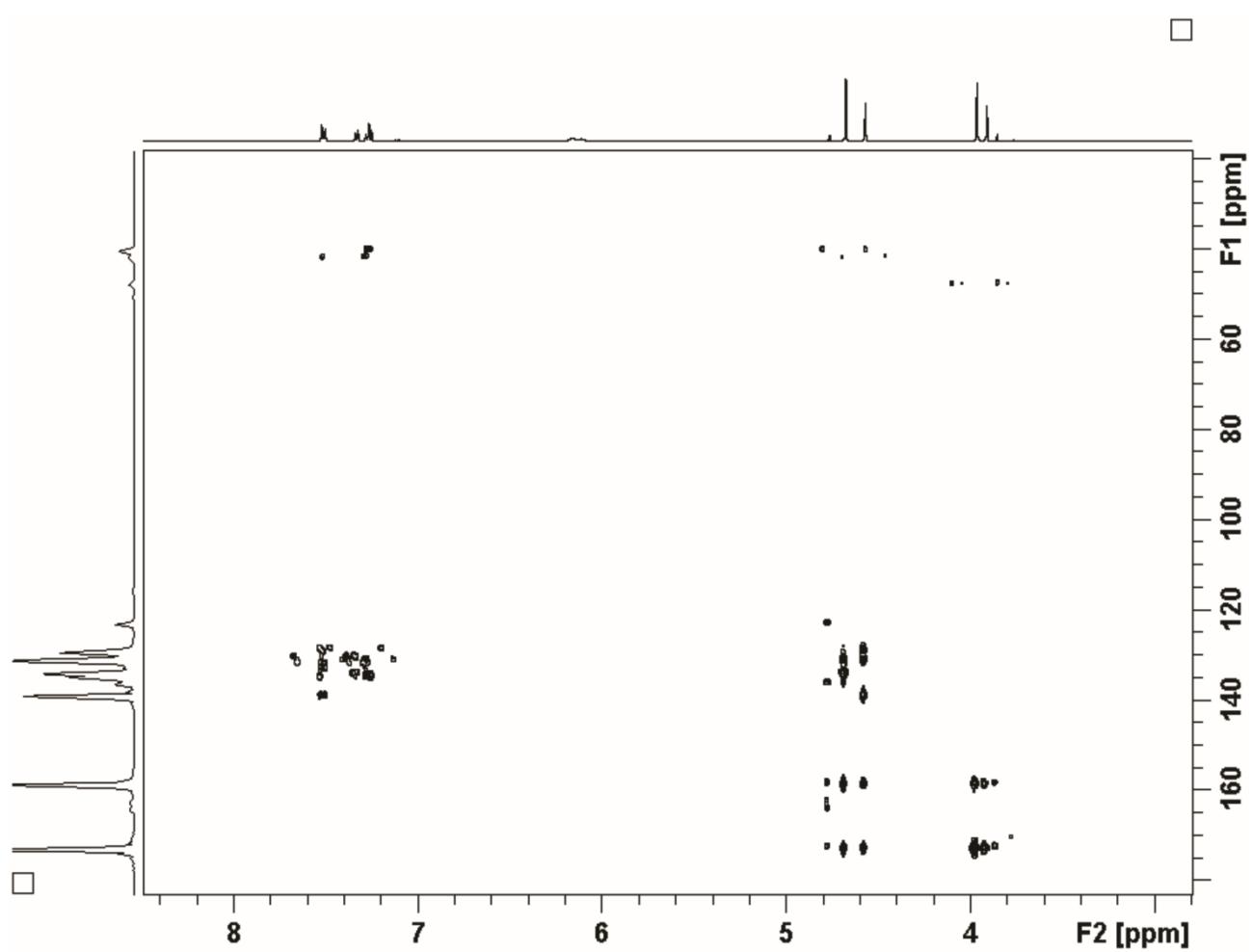


Figure S9. ^1H - ^{13}C g-HMBC correlation map (600 MHz, MeCN- d_3) of the mixture of impurities of LPSF-PT-31.

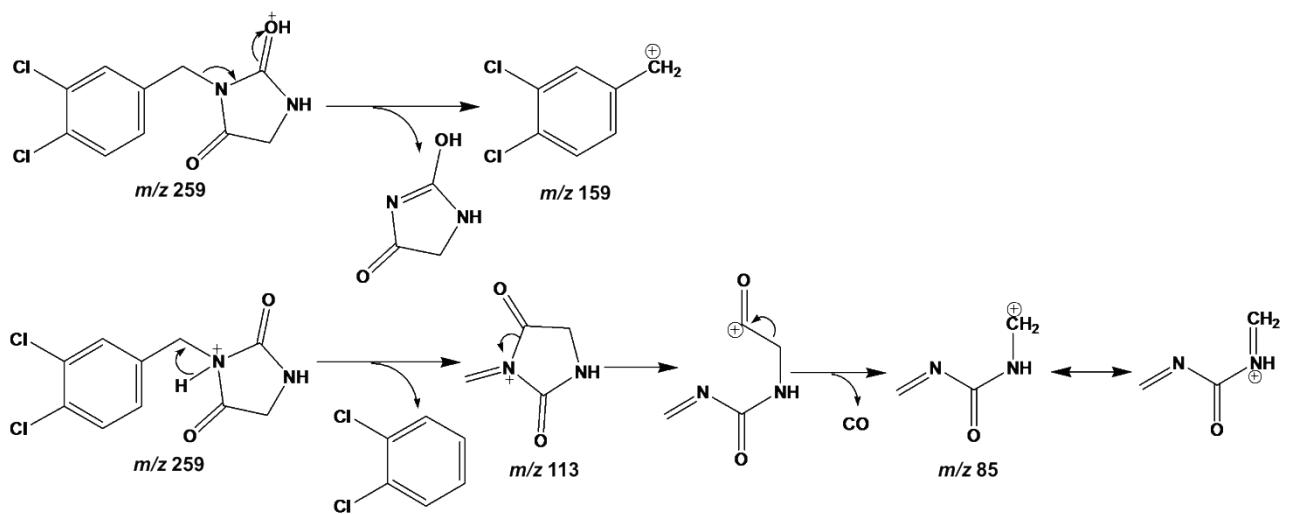


Figure S10. Proposed fragmentation pathway of impurity B.