**- Supplementary Material –**

# Comparison of Sample Preparation and Detection Methods for the Quantification of Synthetic Musk Compounds (SMCs) in Carp Fish Samples

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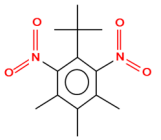
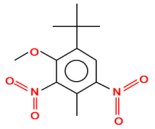
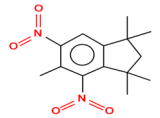
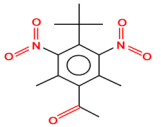
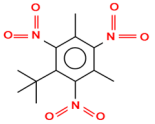
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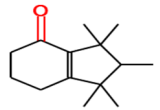
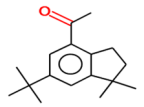
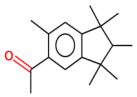
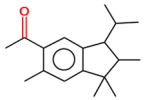
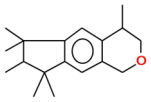
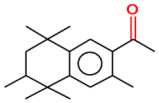
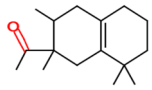
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**Table S1.** Comparison of MDL levels of the proposed method with previously reported literature on SMC analysis in fish

| Sample | Sample weight (g) | Method | Target compound number | MDL (ng/g) | Ref. |
|---|---|---|---|---|---|
| crucian carp (Carassius auratus) | 2 | Ultrasonication (Extraction)<br>Florisil SPE (clean up)<br>GC-QS/MS and GC-QqQ-MS/MS | 12 | 1.03-6.02 (GC-QS/MS)<br>0.087-1.02 (GC-QqQ MS/MS) | This study |
| Bluegill (muscle tissue only ~0.4% lipid) | 1 | Silicagel columm<br>GC–SIM–MS | 5 | 4.0-17 | [1] |
| Sonora sucker (muscle, skin and belly flap tissue ~4.9% lipid) | 1 | Silicagel columm<br>GPC (gel-permeation chromatography)<br>GC–MS/MS | 5 | 12-397 | [1] |
| Red mullet (Mullus surmuletus) | 0.5 | PLE (Pressurised liquid extraction)<br>QuEChers<br>GC-IT-MS/MS | 11 | 0.25-5 (PLE)<br>0.25-10 (QuEChers) | [2] |
| codfish (Gadus morhua), sole (Solea, solea) and hake (Merluccius merluccius) | 0.25 | GC-MS/MS - SPME Arrow | 8 | 0.5-2.5 | [3] |
| hake and salmon | 0.5 | QuEChERS<br>Florisil (dSPE)<br>GC-IT-MS/MS | 10 | 1-5 (hake)<br>1-5 (salmon) | [4] |
| Tilapia, Smallmouth bass and Bream | ~ 2.5 | PLE (Pressurised liquid extraction)<br>Silica<br>In-cellclean-up<br>GPC (gel-permeation chromatography)<br>GC-MS/MS | 5 | 8-360 (Tilapia)<br>1.6-38 (Bream) | [5] |

**Table S2**. Chemical names, abbreviations, CAS numbers, molecular formulas, molecular weights, log $K_{ow}$ and chemical structures of the 12 SMCs classified into two groups

| Category | IUPAC name | Trade name | Abbreviation | CAS No. | Molecular formular | Molecular weight (g/mol) | log $K_{ow}$ | Chemical structures |
|---|---|---|---|---|---|---|---|---|
| Nitro musks | 1-tert-butyl-3,4,5-trimethyl-2,6-dinitrobenzene | Musk tibetene | MT | 145-39-1 | $C_{13}H_{18}N_2O_4$ | 266.3 | 5.9[a] |  |
| | 1-tert-butyl-2-methoxy-4-methyl-3,5-dinitrobenzene | Musk ambrette | MA | 83-66-9 | $C_{12}H_{16}N_2O_5$ | 268.3 | 4.7[a] |  |
| | 1,1,3,3,5-pentamethyl-4,6-dinitro-2H-indene | Musk moskene | MM | 116-66-5 | $C_{14}H_{18}N_2O_4$ | 278.3 | 6.1[a] |  |
| | 1-(4-tert-butyl-2,6-dimethyl-3,5-dinitrophenyl)ethanone | Musk ketone | MK | 81-14-1 | $C_{14}H_{18}N_2O_5$ | 294.3 | 4.24[b] |  |
| | 1-tert-butyl-3,5-dimethyl-2,4,6-trinitrobenzene | Musk xylene | MX | 81-15-2 | $C_{12}H_{15}N_3O_6$ | 297.3 | 5[a] |  |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | 1,1,2,3,3-pentamethyl-2,5,6,7-tetrahydroinden-4-one | Cashmeran | DPMI | 33704-61-9 | $C_{14}H_{22}O$ | 206.3 | 4.2[b] |  |
| | 1-(6-tert-butyl-1,1-dimethyl-2,3-dihydroinden-4-yl)ethanone | Celestolide | ADBI | 13171-00-1 | $C_{17}H_{24}O$ | 244.4 | 5.7[b] |  |
| | 1-(1,1,2,3,3,6-hexamethyl-2H-inden-5-yl)ethanone | Phantolide | AHDI | 15323-35-0 | $C_{17}H_{24}O$ | 244.4 | 5.59[be] |  |
| Polycyclic musks | 1-(1,1,2,6-tetramethyl-3-propan-2-yl-2,3-dihydroinden-5-yl)ethanone | Traesolide | ATII | 68140-48-7 | $C_{18}H_{26}O$ | 258.4 | 6.3[a] |  |
| | 4,6,6,7,8,8-hexamethyl-1,3,4,7-tetrahydrocyclopenta[g]isochromene | Galaxolide | HHCB | 1222-05-5 | $C_{18}H_{26}O$ | 258.4 | 5.3[b] |  |
| | 1-(3,5,5,6,8,8-hexamethyl-6,7-dihydronaphthalen-2-yl)ethanone | Tonalid | AHTN | 1506-02-1 | $C_{18}H_{26}O$ | 258.4 | 5.7[b] |  |
| | 1-(2,3,8,8-tetramethyl-1,3,4,5,6,7-hexahydronaphthalen-2-yl)ethanone | Iso E Super | OTNE | 54464-57-2 | $C_{16}H_{26}O$ | 234.4 | 5.65[b] |  |

[a][6] , [b] https://echa.europa.eu/information-on-chemicals

**Table S3.** List and the properties of the four SPE sorbents used in this study

| Sorbent phase type | Normal-Phase | | | Ion-Exchange |
|---|---|---|---|---|
| Bed sorbent | Aminopropyl (-$NH_2$) | Florisil | Alumina-N (neutral) | Primary Secondary Amine (PSA) |
| Structure of bonded phase | -($CH_2$)$NH_2$ | $Mg_2SiO_3$ | $Al_2O_3$ | ethylenediamine-N-propyl |
| Sorbent amount/cartridge volume | 500mg / 6cc | 1g / 6cc | 1g / 6cc | 500mg /6cc |
| Particle Size | 55 - 105 μm | 50 - 200 μm | 50 - 300 μm | 50 μm |
| Pore Size | 125 Å | 60 Å | 120 Å | 70 Å |
| Distributor | Waters (Milford, MA, USA) | Waters (Milford, MA, USA) | Waters (Milford, MA, USA) | Sigma-Aldrich (St. Louis, MO, USA) |

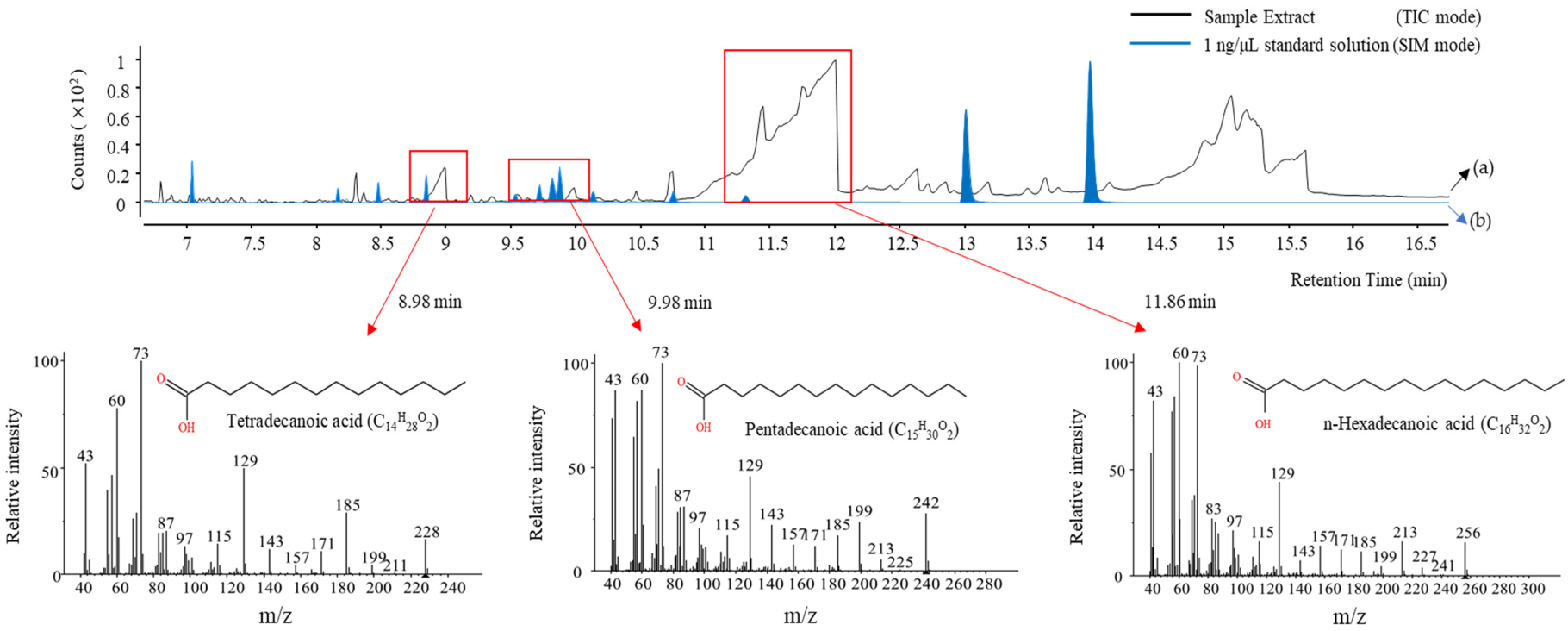



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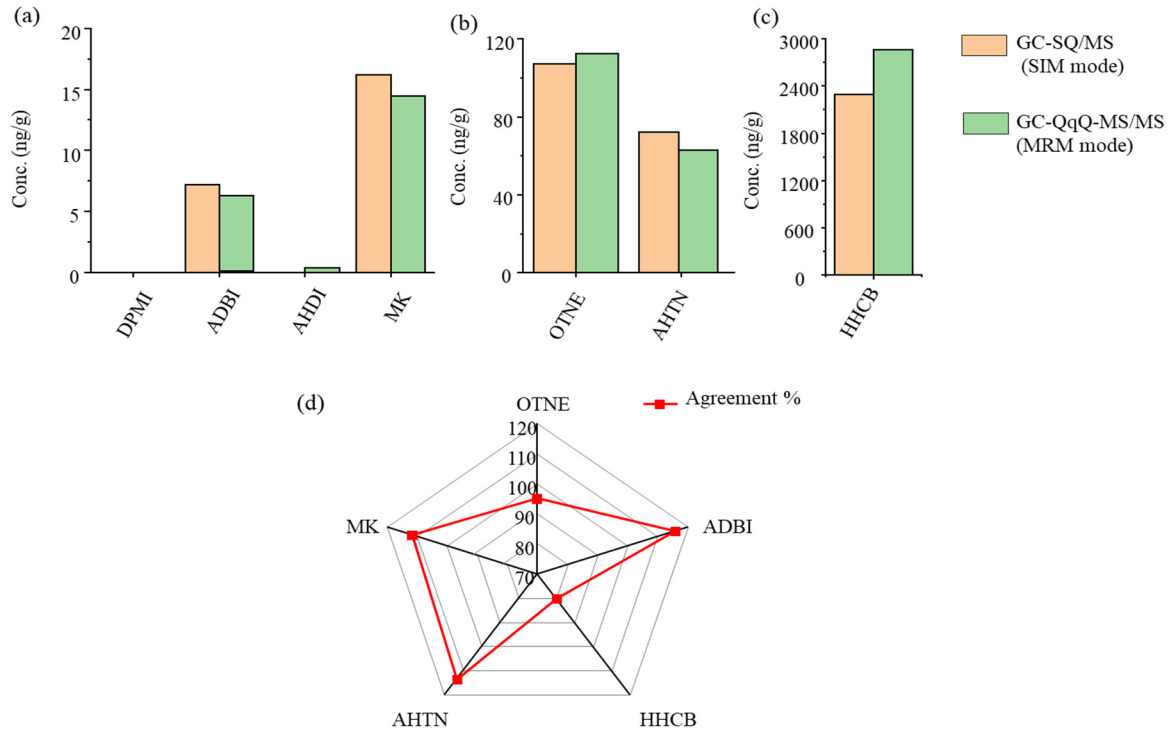



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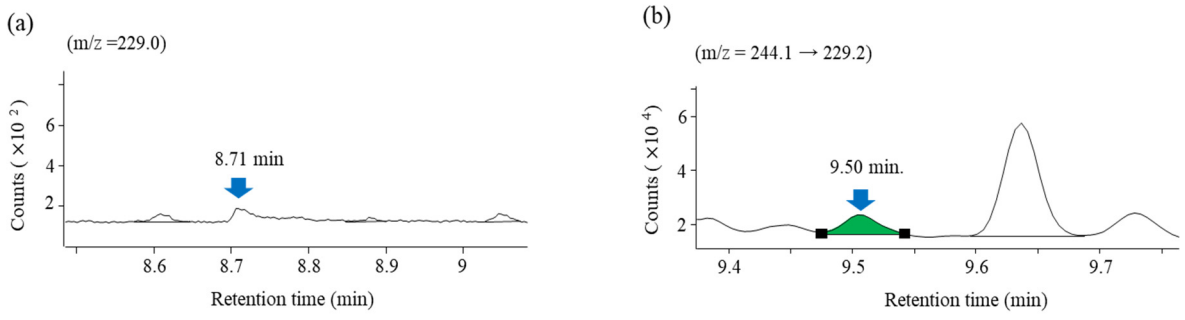
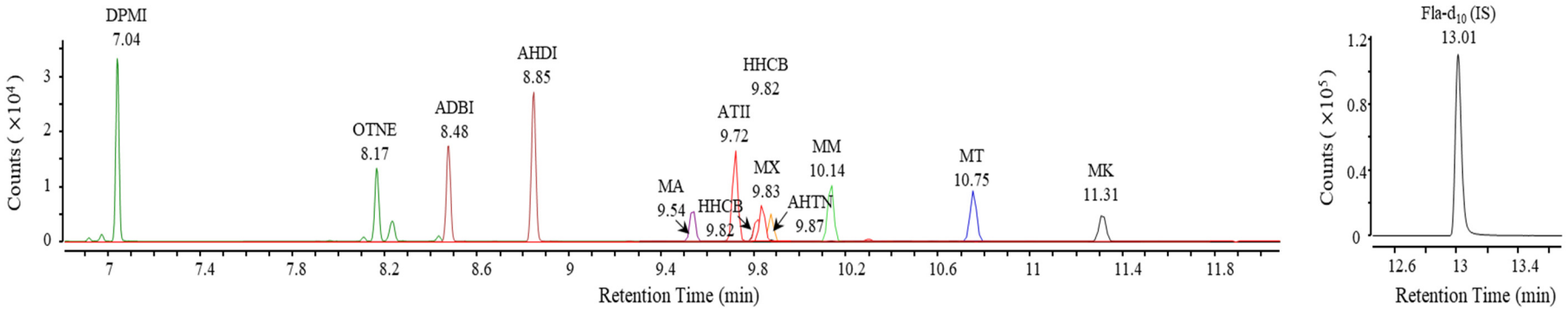



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(a) SIM mode (1 ng/μL)




(b) MRM mode (1 ng/μL)

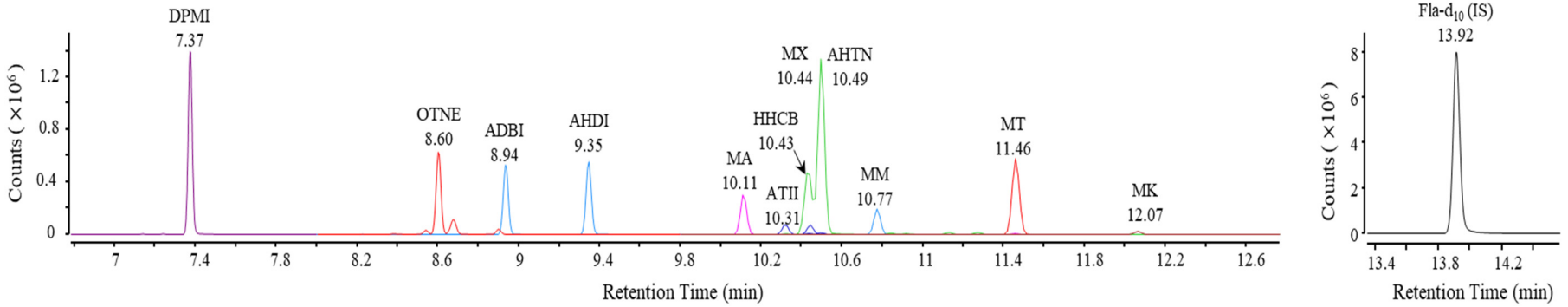



**Figure S4**. Typical chromatograms of the standard solutions with 12 SMCs and Fla-$d_{10}$ (IS) obtained by GC-SQ/MS with SIM mode and GC-QqQ-MS/MS with MRM mode

## Reference


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