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Aroma profiling and volatile fingerprinting of commercial caviar (*Acipenser baerii* and *Acipenser gueldenstaedtii*)

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ABSTRACT

The odorant composition and static headspace PTR-MS profiles of two commercial caviar samples (*Acipenser baerii* and *Acipenser gueldenstaedtii*) were investigated. A combined sensory-instrumental investigation applying solvent-assisted flavor evaporation (SAFE), gas chromatography-olfactometry (GC-O), gas chromatography-mass spectrometry (GC-MS), and two-dimensional gas chromatography-mass spectrometry/olfactometry (GC-GC-MS/O) revealed a total of 123 odorants in the undiluted extracts, of which 87 compounds were identified. Among these, 34 odorants showed a high flavor dilution (FD) factor, with notable differences observed between the two caviar types. Furthermore, sensory analysis described the aroma of both samples as "fishy", "fatty", "seawater-like/algae-like", "green/grassy", and "wheat-like". However, the intensities of these descriptors did not differ significantly between samples. Yet, the sensory attributes aligned well with the smells of the identified odorants. Finally, a fast headspace method using proton-transfer reaction mass spectrometry (PTR-MS) discriminated the investigated caviar samples based on Principal Component Analysis (PCA). This study provides valuable insights into the odorant profiles contributing to caviar flavor quality and highlights the benefits and limitations of PTR-MS as a rapid tool for volatile-based quality control in the caviar industry.

1. Introduction

Caviar is salted unfertilized sturgeon roe from the *Acipenseridae* family and a well-known culinary delicacy worldwide (Farag et al., 2021). It is appreciated for its flavor, texture and nutritional value, as it is rich in minerals, proteins, polyunsaturated fatty acids (PUFAs), and vitamins (Bledsoe et al., 2003; Tavakoli et al., 2021; Vilgis, 2020). In the past decades, due to the high market value of caviar and the poor management of fishing and trade, wild sturgeon populations have declined all over the world (Raymakers, 2006; Tavakoli et al., 2021). In response, many countries – such as those in Europe - have implemented widespread bans on wild sturgeon fishing (Raymakers, 2006). However, global demand has always been on the rise and is expected to grow further (Sicuro, 2019). Consequently, caviar from farmed sturgeon already today makes up almost the totality of the global supply (Bronzi et al., 2019; Sicuro, 2019). In a growing and dynamic industry such as sturgeon farming, which has expanded and delocalized to now more than 40 producing countries, ensuring product quality and consistency is therefore key (Sicuro, 2019).

From all the relevant caviar quality traits for consumers, namely roe size, color, texture, taste and aroma, the latter is rather unexplored (Vilgis, 2020). Caviar aroma quality can be influenced by pre-mortem variables, such as biological origin (Lopez et al., 2020; Rocchetti et al., 2025), diet (Baker et al., 2014; Caprino et al., 2008) and habitat conditions (Bledsoe et al., 2003; Caprino et al., 2008; Lu & Rasco, 2014), but also by post-mortem factors, such as maturation time, storage conditions, and conservation methods (Farag et al., 2021; Liu et al., 2024; Lopez et al., 2020b). In this regard, most of the scientific literature

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focuses on caviar quality and taste and their relation with customer acceptance (Baker et al., 2014; Cardinal et al., 2002; Sicuro, 2019). However, only a few studies have investigated sensory changes through olfactometry (Vilgis, 2020; X. Xu et al., 2022). Vilgis (2020) reported (*E*,*Z*)-2,4-decadienal, (*E*,*Z*)-2,6-nonadienal (*E*,*E*)-2,4-decadienal, 2-nonenal, nonanal, (*Z*)-4-heptanal, methional, octanal, hexanal, 1-octen-3-ol, 2-pentylfuran and phenylacetaldehyde amongst the most odor-active compounds in caviar and their origin was linked to fatty acid degradation (Vilgis, 2020). More recently, Xu et al. (2022) reported several relevant odor-active compounds, namely terpenes (humulene, caryophyllene, longifolene and D-limonene), aldehydes (heptanal, hexanal, pentanal and 3-methylbutanal) and alcohols (2-ethyl-1-hexanol) via gas chromatography-olfactometry (GC-O) in hybrid caviar (*Huso dauricus x Acipenser schrencki*) contributing to "fresh", "fatty" and "fishy" odor notes (X. Xu et al., 2022). Despite these reports on odorants in caviar, the relation between the sensory profile and potential aroma-active compounds has not been comprehensively studied.

Aroma compounds in foods are typically investigated through gas chromatography-olfactometry (GC-O), which enables the identification of odor-active substances among the broader set of volatile organic compounds (VOCs) and specifies the smell qualities of the respective substances (Song & Liu, 2018). Gas chromatography-mass spectrometry (GC-MS) is often used in parallel to provide structural information necessary for chemical identification (Song & Liu, 2018). For aroma compounds present at trace levels, more sophisticated analytical techniques, such as two-dimensional gas chromatography-mass spectrometry/olfactometry (GC-GC-MS/O) are required to avoid potential coelution effects with substances that are not of interest (Cordero et al., 2015). Nevertheless, GC-GC-MS/O and sensory studies are long and costly procedures and thus not feasible for routine quality control in the food industry. As a result, direct injection mass spectrometry (DIMS) techniques are gaining popularity for rapid, non-destructive, and automatic quality monitoring purposes (Biasioli et al., 2011; Ellis & Mayhew, 2014). These techniques have been successfully used in diverse applications concerning food quality, such as spoilage detection (Aprea et al., 2006), origin classification (Le Bechec et al., 2024), and product authentication (Erasmus et al., 2017). Among these, proton-transfer reaction mass spectrometry (PTR-MS) stands out as a highly sensitive DIMS technique that does not necessitate lengthy sample pretreatment procedures. The method uses H_3O^+ for ionization, which can selectively protonate those samples' VOCs exerting higher proton affinity than water. However, literature is scarce with respect to the applicability of these fast DIMS technologies for the monitoring of quality in aquatic food products, such as caviar.

Accordingly, the present study pursued two main objectives. First, to gain a deeper understanding of the odorants that may contribute to caviar flavor. For that, two commercial caviar samples, *Acipenser baerii* (BAE) *and Acipenser gueldenstaedtii* (GUE), were studied applying the above-mentioned olfactory-guided approaches (GC-(GC)-MS/O). In addition, a human-sensory characterization of the overall aroma of the samples was performed to assess whether differences in odorant composition translated into perceptible variations in their aroma profile. Second, proton transfer reaction time-of-flight mass spectrometry (PTR-MS) was explored – for the first time – as a rapid spectrometric approach for characterizing caviar samples based on their VOC profiles.

2. Materials and Methods

The analytical techniques used in the present study represent the current state-of-the-art for the identification of odor-active compounds. These included gas chromatography-olfactometry (GC-O), gas chromatography-mass spectrometry (GC-MS) and the dilution threshold method cAEDA (Brattoli et al., 2013). To identify trace-level odorants, two-dimensional gas chromatography-mass spectrometry/olfactometry (GC-GC-MS/O) was employed, as it enables the preconcentration of the analytes (collection cuts) over several injections of the sample extract to obtain clear mass spectra of otherwise-undetectable compounds (Cordero et al., 2015). Furthermore, PTR-MS was applied as a rapid method to investigate the volatile fingerprint of the samples (Mazzucotelli et al., 2022).

2.1. Chemicals

The chemical references purchased are listed according to the nomenclature of the International Union of Pure and Applied Chemistry (IUPAC), with common names given in brackets, followed by the purity and supplier: dichloromethane (VWR Chemicals, Darmstadt, Germany), (*E*)-pent-2-enal 95 %, 2,6,6-trimethylbicyclo[3.1.1]hept-2-ene (α-pinene) 97 %, butan-1-ol 99 %, (1*S*,5*S*)-6,6-dimethyl-2-methylidenebicyclo[3.1.1]heptane (β-pinene) 99 %, hexanal 98 %, 1,4-dimethylbenzene (*p*-xylene) 99.5 %, 1,2-dimethylbenzene (*o*-xylene) 99.5 %, 3,7,7-trimethylbicyclo[4.1.0]hept-3-ene (3-carene) 90 %, pent-1-en-3-ol 99 % heptanal >92 %, (3*E*)-3,7-dimethylocta-1,3,6-triene (ocimene) 90 %, (*Z*)-hept-4-enal 98 %, 1-methyl-4-propan-2-ylbenzene (*p*-cymene) 99 %, 3-hydroxybutan-2-one (acetoin), (4*R*)-1-methyl-4-prop-1-en-2-ylcyclohexene ((*R*)-limonene) 97 %,(*E*)-pent-3-en-2-ol 96 %, oct-1-en-3-one 50 %, heptan-2-ol ≥ 97 %, (*E*)-pent-2-en-1-ol 95 %, octane-2,3-dione 97 %, nonan-2-one ≥99 %, 3-methylsulfanylpropanal (methional), decanal, (2*E*,4*E*)-hepta-2,4-dienal 88 %, benzaldehyde >99.5 %, undecanal 97 %, butane-2,3-diol 98 %, 5-methyl-2-propan-2-ylcyclohexan-1-ol (menthol) 99 %, nonan-1-ol >98 %, dodecanal 92 %, 2*H*-furan-5-one (γ-crotonolactone) 98 %, (4*S*,4a*S*,8a*R*)-4,8a-dimethyl-1,2,3,4,5,6,7,8-octahydronaphthalen-4a-ol (geosmin) ≥ 97 %, (2*E*,4*E*)-deca-2,4-dienal 85 %, phenylmethanol (benzyl alcohol) 99 %, hexanoic acid 99,5 %, 1,3-benzothiazole (benzothiazole) 96 %, 1,2,4,5-tetramethylbenzene ≥98 %, 4-methylphenol (*p*-cresol) 98 %, 1-methyl-4-propan-2-ylidenecyclohexene (terpinolene) 90 %, (*E*)-hex-2-en-1-ol 96 %, 3,7-dimethyloctan-3-ol (tetrahydrolinalool) ≥ 97 %, (*E*)-oct-2-enal 94 %, 1,2,4,5-tetramethylbenzene 98 %, heptan-1-ol 98 %, 6-methyl-2-(oxiran-2-yl)hept-5-en-2-ol (linalool oxide) 97 %, 2,6-dimethyloct-7-en-2-ol (dihydromyrcenol) ≥ 99 %, 1-methyl-4-prop-1-en-2-yl-7-oxabicyclo[4.1.0]heptane (limonene oxide) 97 %, 3,7-dimethylocta-1,6-dien-3-ol (linalool) 97 %, methyl benzoate 99 %, naphthalene >99.7 %, 2-methyl-5-prop-1-en-2-ylcyclohex-2-en-1-one (carvone) 97 %, decan-1-ol 99 %, (2*E*,4*E*)-undeca-2,4-dienal ≥90 %, 6-butyloxan-2-one (δ-nonalactone) 98 %, tridecan-1-ol 97 %, 2-phenoxyethanol 99 %, 5-hexyloxolan-2-one (γ-decalactone) 98 %, 5-heptyloxolan-2-one (γ-undecalactone) 98 %, hexadecan-1-ol 99 %, chromen-2-one (coumarin) 99 %, diphenylmethanone (benzophenone) ≥ 99 %, hexadecanoic acid 99 % (Sigma-Aldrich, Steinheim, Germany); oct-1-en-3-ol 98 %, hexan-1-ol ≥ 99 %, nonanal 95 %, undecan-1-ol 99.5 %, dodecan-1-ol 98 %, phenol 99 %, undec-1-ene ≥99.5 %, octan-1-ol > 99.5 %, undecan-2-ol 98 % (Fluka (Honeywell), Steinheim, Germany); 1-phenylethanone (acetophenone) 98 %, (*E*)-undec-2-enal (SAFC (Sigma-Aldrich), Steinheim, Germany); (3*R*)-3-hydroxy-4,4-dimethyloxolan-2-one (pantolactone), (1*R*,4*E*,9*S*)-4,11,11-trimethyl-8-methylidenebicyclo[7.2.0]undec-4-ene (β-caryophyllene) 80 %, 1-hydroxypropan-2-one (hydroxyacetone) 90 %, 2-ethylhexanal 99 %, 2,5-dimethylpyrazine > 98 %, 2,6-dimethylpyrazine > 98 %, 2-ethylhexan-1-ol > 99.5 %, 2-methylpentanal >95.0 %, 2,4,6-tribromoanisole >98.0 %, 2-(4-methyl-1,3-thiazol-5-yl)ethanol (4-methyl-5-thiazoleethanol) 98 % (TCI Deutschland GmbH, Eschborn, Germany); propanoic acid 99 % (Riedel-de Haen GmbH (Honeywell), Seelze, Germany) (Givaudan, Vernier, Switzerland); octanal 99 %, 5-ethyloxolan-2-one (γ-caprolactone) 98 % (Thermo Fischer Scientific GmbH, Kandel, Germany); 3-(3-pentyloxiran-2-yl)prop-2-enal (*tr*-4,5-epoxy-(E)-2-decenal) 97 %, (5*Z*)-octa-1,5-dien-3-one ((*Z*)-1,5-octadien-3-one) 99 % (aromaLab, Planegg, Germany), 1,3,5-trimethylbenzene (mesitylene) 98 % (VWR International GmbH, Pennsylvania, USA); 6-methylheptan-1-ol 95 % (Chemos GmbH & Co. KG, Regenstauf, Germany); 4-hydroxy-3-methoxybenzaldehyde (vanillin) 99 % (abcr GmbH, karlsruhe, Germany). A solution of n-alkanes (C6-C31) (Sigma-Aldrich, Steinheim,

Germany) in pentane was employed to calculate the retention indices.

2.2. Samples

Acipenser baerii (BAE) and *Acipenser gueldenstaedtii* (GUE) caviar samples were obtained from a caviar-producing company in France in December 2022 and January 2023. Approximately 3.3 g/100 g caviar of salt and 0.5 g/100 g caviar of sodium tetraborate (E285) were added to the samples, which were then stored in aluminum tins. After preparation, all caviar samples were delivered within two days to the Chair of Aroma and Smell Research (Erlangen, Germany). Samples were stored at −80 °C until further analysis.

2.3. Extraction methods of the volatile compounds

The isolate obtained by solvent-assisted flavor evaporation (SAFE) was used for all the GC-O/FID, GC-MS and GC-GC-MS/O analyses. The extraction was performed in triplicate for each caviar sample under the following conditions: (i) each caviar sample was thawed for 30 min at room temperature; (ii) then, 10g of sample (uncrushed) were extracted with 30 mL of dichloromethane by dynamic maceration at room temperature for 30 min (RO 10P, IKA, Germany); (iii) the dichloromethane extract was collected separately, and the volatiles in the extract solution were carefully isolated using solvent-assisted flavor evaporation (SAFE) at 50 °C (Engel et al., 1999); (iv) the distillate was dried with Na_2SO_4. Finally, the volume of each distillate was reduced to 100 μL via Vigreux distillation at 48 °C.

Static headspace analyses were performed for all the PTR-MS measurements. For these, 5 g of sample (uncrushed) were placed in 20-ml headspace vials. In order to reproduce the same sample conditions during volatile extraction as in the SAFE procedure, intact eggs were used for the PTR-MS measurements. It is also worth mentioning that a preliminary assessment of the impact of crushing on the volatile PTR-MS fingerprints of caviar samples was conducted. Major differences were not observed, so intact eggs were chosen for practicality (Fig. S4). Ten replicates were prepared for each of the samples. Before each measurement, each vial containing the caviar was incubated at 37 °C for 15 min under mild agitation conditions with a commercial MPS2 Autosampler (Gerstel GmbH & Co. KG, Mülheim an der Ruhr, Germany). After incubation, 1 mL of the vial headspace was taken and injected at 50 μL/s (injection time was therefore 20 s) into the heated transfer line (80 °C) of the PTR-MS (Fig. S5). Background or blank measurements were done with empty headspace vials pretreated the same way as the samples. The blank signal was subtracted from all masses identified for each of the injections performed.

2.4. Odorant compound identification

Odorants were identified by comparing their odor quality, retention indices on two capillaries (DB-FFAP and DB-5) and mass spectra with those of reference compounds (listed in Section 2.1).

2.4.1. Gas chromatography-olfactometry/flame ionization detector (GC-O/FID) and aroma extract dilution analysis (AEDA)

The separation of volatiles was performed following the protocol described by da Silva, Egiddi, Debeuf, Buettner, & Loos (2024). In short, a TRACE GC Ultra (Thermo Fischer Scientific, USA) was employed. A DB-5 (30 m × 0.32 mm x 0.25 μm; Agilent J&W, USA) or DB-FFAP (30 m × 0.32 mm x 0.25 μm; Agilent J&W, USA) capillary was used. For DB-5, an initial temperature of 40 °C was kept for 2 min, then increased to 250 °C at 8 °C/min and kept at 250 °C for 10 min. For FFAP, an initial temperature of 40 °C was kept for 2 min, then increased to 240 °C at 8 °C/min and kept at 240 °C for 8 min. Cold-on-column injections (2 μL) were performed, and the carrier gas (Helium) flow rate was set at 2.2 mL/min. A Y-splitter split the GC effluent 1:1 between the ODP and the flame ionization detector (FID) through two uncoated and deactivated fused silica capillaries (0.5 m × 0.2 mm). FID and ODP were held at a temperature of 250 °C and 270 °C, respectively. The GC-O analyses were performed by three trained experts selected from the sensory panel of the Chair of Aroma and Smell Research at the Friedrich-Alexander-University Erlangen-Nuremberg (FAU), Germany. The experts were asked to sniff the undiluted distillate of each sample.

Aroma extract dilution analyses (AEDA) (Grosch, 1993) were performed by one expert at the GC-O (DB-FFAP column). The flavor dilution factors (FD) were determined according to a range of dilutions: the undiluted distillate (FD = 1) was progressively diluted with DCM (1:3; v/v), with nine solutions for each sample, yielding aroma dilution factors (FD) that ranged from FD 3 to FD 19683. Each odorant's FD factor corresponds to the final solution where the compound was detected by GC-O.

2.4.2. Gas chromatography-mass spectrometry (GC-MS)

GC-MS analysis was performed as reported in da Silva, Egiddi, Debeuf, Buettner, & Loos (2024). Briefly, an Agilent 7890A GC system (Agilent Technologies, USA) equipped with either a DB-FFAP column (30 m × 0.25 mm x 0.25 μm; Agilent J&W) or a DB-5 column (30 m × 0.25 mm x 0.25 μm; Agilent J&W, USA) and a CIS4 injection system (Gerstel GmbH & Co. KG, Germany) was employed. The temperature program of the GC oven was as described in Section 2.4.1. Analyses were conducted in splitless mode and carrier gas at flow rate of 1.0 mL/min (Helium). Injections (volume of 1 μL) were performed with an MPS 2 XL autosampler (Gerstel GmbH & Co. KG, Germany). The GC system was coupled with an Agilent MSD 5975C and the EI (70 eV) mass spectra were recorded in scan mode (40–400 Da).

2.4.3. Two-dimensional gas chromatography-mass spectrometry/olfactometry (GC-GC-MS/O)

A GC-GC-MS/O system comprising two Agilent 7890B GC systems coupled to an Agilent 5977B MS (Agilent Technologies, USA) was used for the identification of trace odorant compounds. Specifications were as reported in da Silva, Egiddi, Debeuf, Buettner, & Loos (2024). Briefly, a DB-FFAP and DB-5 capillary columns were installed in the first and second GC ovens, respectively. Carrier gas flow rate was 2.5 mL/min (Helium). The sample was injected (1 or 2 μL) using a multipurpose sampler MPS 2XL (Gerstel GmbH & Co. KG, Germany) at 40 °C with the cold-on-column technique. The temperature program of the GC oven was the same as described in Section 2.4.1, except for the final temperature holding time (5 min for DB-FFAP and 1 min for DB-5). The effluent was split at the end of the first capillary; one part reached the Cryo-Trap System (CTS 1) (towards the second GC system) and the other reached the ODP (270 °C) and FID (250 °C) of the first GC system. A multi-column switching system MCS2 (Gerstel GmbH & Co. KG, Germany) was used to transfer the interested elution section to CTS 1 (Gerstel GmbH & Co. KG, Germany). The collected sections were subsequently transferred to the second GC system by thermal desorption (250 °C). MS and ODP were then connected to the second GC column. EI (70 eV) mass spectra were recorded in scan mode (40–400 Da) at the MS detector.

2.5. Proton-transfer reaction mass spectrometry (PTR-MS)

Instrumental pieces (injection syringe and transfer line) in contact with the extracted volatiles from the headspace until reaching the drift tube were kept at 80 °C during analysis. Measurements were conducted using a commercial PTR-ToF-MS model 6000 X2 instrument (IONICON Analytik, Innsbruck, Austria). The drift tube was operated at a pressure of 2.6 mbar, a temperature of 80 °C, and with a drift voltage (U_{drift}) of 450 V and an extraction voltage (U_{dx}) of 4.25 V that resulted in a E/N ratio of 125 Td. The transmission efficiency of the ions through the ToF was previously assessed using a gas calibration standard mixture (SIAD, Italy) and the resulting transmission calibration curve was applied to all the recorded data.

Spectra were recorded every 0.5 s for the mass range of 10–340 Da (Fig. S5). Sample and blank injection spectra were averaged over 15 cycles (injection plateau). The final concentrations reported correspond to the average of ten replicates per sample. Signal integration and mass selection were done by fitting Gaussian curves to the averaged spectral peaks and by automatically generating peak systems that could discriminate the exact masses under investigation using the IONICON Data Analyzer (IDA) (IONICON Analytik, Innsbruck, Austria). The ions used for the calibration of the masses across spectra were m/z 21.022 ($H_3^{18}O^+$), m/z 203.943 ($C_6H_5I^+$) and m/z 330.848 ($C_6H_5I_2^+$)). The deconvolution tool of the IDA software was used to analyze all mass peaks recorded across injections. In addition, the IDA's automatic chemical formula assignment tool (set at 100 % isotope matching and including the elements C, H, N, and S) was used. The mass accuracy cut-off value of the detected experimental masses was set at 100 ppm. Finally, concentration values were converted to parts-per-billion-per-volume (ppbV) by accounting for the specific ion/mass calibration curve previously calculated, the relative abundance of H_3O^+ and the advanced reaction rate constants calculated by the software. Post-processing data analyses were necessary to extract more relevant information out of the spectral data. Hence, a *t*-test ($p < 0.01$) and an artifact and concentration (0.01 ppbV) filter corrections were performed, which resulted in a final dataset containing 51 masses (Fig. S1).

2.6. Sensory evaluation

The sensory evaluations were performed by eight trained panelists (six women, two men) from the Chair of Aroma and Smell Research at FAU. The panelists were trained weekly in the recognition and description of approximately 150 different odorants based on an internal odor language that included odorants from the food and non-food sectors. The panelists had no known disease or odor disorder at the time of the study.

Following the ISO 13299:2016–09 (General guidance for establishing a sensory profile), sensory analyses were performed in two sessions in a ventilated sensory panel room (21 °C). Briefly, approximately 5 g of each caviar sample was added to 80 mL Weck glasses (WECK GmbH u. CO KG, Germany) and covered with aluminum foil to avoid any perception influence by the sample's appearance as well as light-induced oxidation processes. A random three-digit code was used to code the samples. First, panelists were asked to name their individual odor impressions of the caviar samples. Then, the odor attributes list was discussed, and only descriptors with >50 % agreement were selected. Second, the panelists evaluated the intensity of each attribute of the samples individually using a scale from 0 (no perception) to 10 (strong perception). The same scale was used to rate the overall odor intensity, whereby the intensity of each attribute was not allowed to exceed the overall intensity rating. Odor pens containing hexanal and commercial odor test pen 16 (Burghart Messtechnik GmbH, Germany) were used as references for the ratings of the odor attributes green/grassy and fishy, respectively. For the fatty attribute, two aqueous solutions of acetoin (0.2–0.4 μL/mL) were added to 120 mL Weck glasses and used as a reference. In addition, wheat grains were used for the wheat odor attribute. No reference was used for the seawater-like/algae-like attribute, and this was classified only based on the knowledge of each panelist.

The sensory evaluations conducted in this study with trained panelists at the Chair of Aroma and Smell Research (FAU), were not subject to formal ethical approval but are compliant with institutional work safety regulations. Prior to the sensory evaluation, each panelist received information concerning the nature of the sample (caviar). The participation was entirely voluntary and participants retained the right to withdraw at any time.

2.7. Data analysis

The average values of the sensory analyses data were plotted in spider web diagrams using Excel 2016. Statistical evaluation of the ratings of the single odor attributes and overall odor intensities was done by applying the Kruskal-Wallis test using IBM SPSS Statistics 29.0 (IBM SPSS, Armonk, NY, United States). For the PTR-MS data, *t*-test and Principal Component Analysis (PCA) of the different datasets were performed using MATLAB R2023b (Mathworks, Massachusetts, USA) and the PLS Toolbox (Eigenvector Technologies, Manson, Washington, USA).

3. Results and discussion

3.1. Sensory evaluation

The panel agreed on the odor qualities "fishy", "fatty", "seawater-like/algae-like", "green/grassy" and "wheat-like" to describe the aroma profile of the caviar samples. The average intensity ratings of the individual odor attributes and the overall intensities are shown in Fig. 1A and B, respectively. For both samples, the average intensities of the odor descriptors were scored between 1.5 and 4. "Fatty" and "sea water-like/algae-like" were the most dominant odor notes in the caviar samples. With respect to the overall odor intensity evaluation, the average ratings were 6.3 and 5.6 for BAE and GUE, respectively.

Small differences in the aroma attributes used to describe the caviar samples were observed for the "sea water-like/algae-like impression", while the other attributes ("fishy", "fatty", "green/grassy" and "wheat") were rated quite similarly. For the "seawater-like/algae-like" odor impression, the highest average intensity was perceived in BAE. Nevertheless, no statistically significant differences were observed between the samples for any descriptors ($p < 0.05$). With respect to their overall odor intensity, BAE was rated on average slightly higher than GUE, but no statistically significant difference was found between them (chi-square (^{2}) = 1.714, $p = 0.230$). The raw data for the aroma profile and overall odor intensity can be found in Table S1 in the supplementary material. All odor descriptors used to describe the odor of the caviar samples in this study were in accordance with the predominant odor qualities of the odorant compounds perceived by means of GC-O/MS described in the next section. Furthermore, the present results are in line with odor attributes previously described for caviar (Baker et al., 2014; Cardinal et al., 2002).

3.2. Odorant identification through cAEDA-GC-(GC)-O/MS

A total of 123 odor-active compounds were detected in cAEDA with FD factors ranging from 1 to 19783, as indicated in Table 1. Among these compounds, 87 were identified by comparing their retention indices, mass spectra, and odor qualities with their corresponding standard compounds. On the other hand, 32 compounds (FD ≤ 81) were not determined due to their low concentration. Two compounds were tentatively identified through retention indices, odor qualities, and mass spectra comparison with the NIST library, namely chloroiodomethane and 4-cyanocyclohexene. Furthermore, the compound geosmin was tentatively identified based on the RIs on two different columns and the odor quality of its corresponding reference compound. Despite no visible peak signal in the GC-(GC)-O/MS chromatogram, geosmin was detectable by human assessors using GC-O due to the very low odor threshold (0.25–1.5 μg/kg depending on the species) (Lindholm-Lehto & Vielma, 2019).

Both caviar samples (BAE and GUE) exhibited highly similar odor profiles, with alcohols being the predominant chemical group, followed by aldehydes and terpenes, as shown in Table 1 and Fig. S2. These results are in accordance with earlier findings in caviar and fish roe matrices. For instance, Vilgis (2020) reported ten aldehydes that might derived from fatty acids amongst the most odor-active compounds in caviar

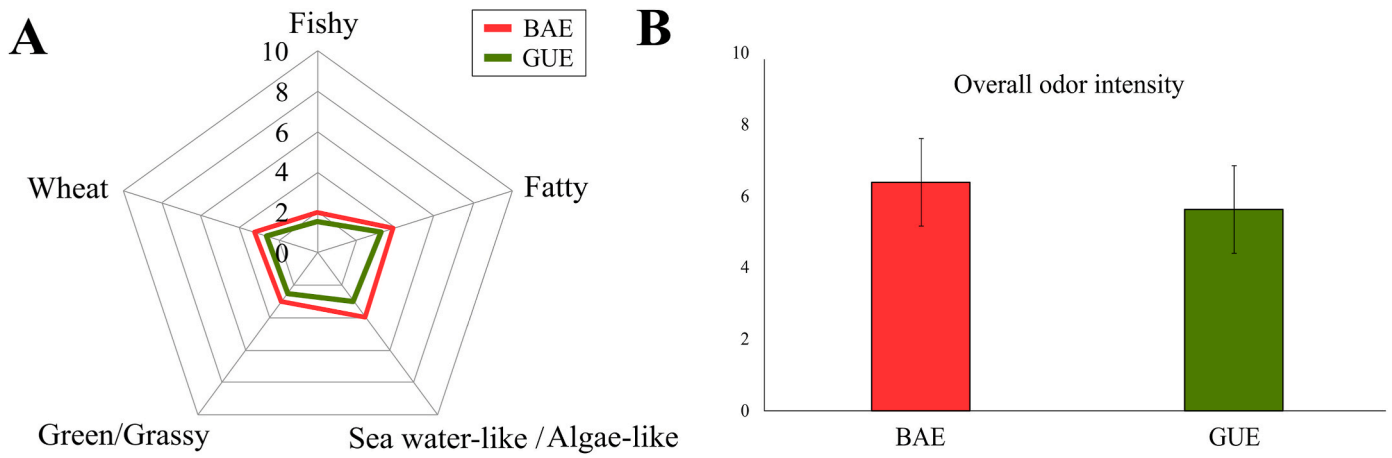


Fig. 1. Sensory evaluation results of the aroma profile of the commercial caviar samples *Acipenser baerii* (BAE−red) and *Acipenser gueldenstaedtii* (GUE−green). A: The *spiderweb* plot shows the main aroma attributes used for the description of the samples' aroma ("fishy", "fatty", "wheat", "green/grassy" and "sea water-like/algae-like") and their average intensity (and standard deviation) rated by all panelists ($n = 8$). B: Overall odor intensity average (and standard deviation) of both caviar samples, BAE (red) and GUE (green).

(Vilgis, 2020), five of which were also identified in the present study (hexanal, (*Z*)-4-heptenal, octanal, nonanal, (*2E,4E*)-deca-2,4-dienal). Furthermore, 34 odorants were perceived with FD factors higher than 243 (Fig. 2). The substances with the highest FD factors in BAE were octane-2,3-dione (27), (*Z*)-1,5-octadien-3-one (28), nonanal (7), nonan-2-one (29), benzaldehyde (11), benzyl alcohol (49), *tr*-4,5-epoxy-(*E*)-2-decenal (18) and tridecan-1-ol (51) (FD = 19683); while in GUE the highest FD factor was detected for benzyl alcohol (51) (FD = 2181). These results show that, even though the chemical profile in general was similar in the investigated samples, each odorant compound might have been present at different concentrations. These differences might be due to different fish species, which could explain the perceptual differences in taste, despite the similar odor descriptors found in the sensory evaluation (Farag et al., 2021; Lopez et al., 2020). However, further studies are necessary to evaluate the differences between species at the same maturation time and production process, such as target quantification of the relevant odorant compounds, aroma omission and recombination experiments. Overall, the odorant compounds identified are in accordance with findings of previous studies in caviar flavor analysis, in which SAFE and GC-O techniques led to the identification of 37 volatile compounds in caviar from hybrid sturgeon (*Huso dauricus* × *Acipenser schrencki*) (X. Xu et al., 2022). Nevertheless, the present study provides a more thorough odorant characterization of *Acipenser baerii* and *Acipenser gueldenstaedtii* caviar. The sensory and analytical approach applied here allowed for the detection of trace-level odorants with high FD factors and improved chemical resolution. This demonstrates the benefit of using multidimensional GC-O/MS to elucidate specific odorants in complex food matrices like caviar.

It is important to note that cAEDA is a screening method that provides evidence of potent odorants and allows a preliminary classification of the most significant odor compounds in caviar for further targeted identification experiments. Nevertheless, AEDA-GC-(GC)-O/MS analysis revealed a complex aroma profile in caviar, with several odor-active compounds exhibiting different FD factors. These results highlight the importance of the sensory analytical approach and add valuable information to the current understanding of caviar aroma.

3.3. Volatile fingerprints through static headspace PTR-MS

The mass peaks in the reduced dataset were chosen as variables, and Principal Component Analysis (PCA) was utilized as an unsupervised method to explore the differences between sample types. The biplot of PC1 vs PC2 is shown in Fig. 3.

Accordingly, PC1 captured the majority of the variance of the data (78.42 %), while PC2 accounted for 9.54 %. The samples (BAE marked by red circles and GUE by green circles) distinctly clustered along PC1 and both clusters were associated with two separated sets of loadings (detected masses). This separation, however, was not observed when comparing PC2 vs PC3 (Fig. S3), which means that the information contained in PC2 and PC3 is not relevant to discriminate the samples based on the static headspace VOC composition analyzed by PTR-MS. Given the limited time required for each measurement (total measurement time 15 min) and the clear clustering generated, this PTR-MS-based method appears to be a promising approach in discriminating two commercial caviar types with different biological origins, that would, however, need to be substantiated by larger samples cohorts. Nevertheless, PTR-MS has previously been used as a fingerprinting/classification technique in other food products and processes (Araghipour et al., 2008; Erasmus et al., 2017; Pustjens A. et al., 2018), and might be thus a suitable tool to help caviar producers in speeding up and automating quality control processes. Developing such classification tools further to be able to discriminate samples based on aspects such as geographical origin, authenticity or fraud, seasonality, and specific off-flavors could be interesting avenues for future research.

Besides the use of PCA to explore sample clustering, one can easily dig deeper into the variables that are driving the separation along PC1. The concentration of the detected masses and the corresponding tentative identification of potential chemical substances associated with these are provided in Table 2. Of the 51 mass peaks listed, 24 structural substance suggestions are given on the basis of their predicted chemical formula, experimental mass (mass accuracy <100 ppm), isotopic pattern, and matching PTR-MS mass spectral data from different food products reported in the literature (Bianchi et al., 2017; Boscaini et al., 2003; Pedrotti et al., 2018, 2020; Phillips et al., 2010; Yener et al., 2016). The most abundant mass peaks detected in the headspace of the samples were m/z 31.0164 (2), m/z 43.0152 (7), m/z 45.005 (12), m/z 45.034 (13) and m/z 47.0463 (17), m/z 59.04604 (24), m/z 75.07756 (34). These may correspond to highly volatile compounds such as formaldehyde (2), acetaldehyde (13), ethanol (17), propanal/acetone (24) and butanol (34), as reported in Table 2 (Bianchi et al., 2017; Boscaini et al., 2003; Pedrotti et al., 2018, 2020; Phillips et al., 2010; Yener et al., 2016), but also likely be the breakdown products of larger volatile substances. In fact, it is noteworthy to realize that, excluding the non-identified mass (m/z 45.005) and butanol (detected by GC-O/MS), none of these tentatively identified compounds at higher concentrations are identified as odor-active in the GC-O/MS analysis described before (Table 2). We argue that these masses could be, besides fragmentation products, associated with compounds not trapped by the volatile

Table 1
Odor-active compounds identified in two commercial caviars: BAE (*Acipenser baerii*) and GUE (*Acipenser gueldenstaedtii*). Displayed are the FD factors of the odorants obtained by cAEDA, together with the respective retention indices (RI) and identification criteria.

			FD factors[c]		RI values on[d]		
No.[a]	Odorant	Odor quality[b]	BAE	GUE	DB-FFAP	DB-5	Identification criteria[e]
Aldehydes (18)							
1	(*E*)-Pent-2-enal	Green apple-like, almond-like	n.d.	1	–	742	RI, O, MS, RC
2	Hexanal	Grassy	9	27	1092	812	RI, O, MS, RC
3	Heptanal	Soapy, fatty	9	81	1188	906	RI, O, MS, RC
4	(*Z*)-4-Heptenal	Fishy, fatty	243	81	1244	905	RI, O, MS, RC
5	Octanal	Citrus-like, soapy	27	81	1313	1011	RI, O, MS, RC
6	2-Ethyl hexanal	Green, fruity[f]	27	n.d.	1325	957	RI, O, MS, RC
7	Nonanal	Soapy, citrus-like	19683	81	1388	1106	RI, O, MS, RC
8	(*E*)-Oct-2-enal	Fatty, soapy, grassy	3	n.d.	1427	1056	RI, O, MS, RC
9	(2*E*,4*E*)-Hepta-2,4-dienal	Fatty, fruity	27	n.d.	1487	997	RI, O, MS, RC
10	Decanal	Soapy	729	81	1503	1206	RI, O, MS, RC
11	Benzaldehyde	Bitter almond-like, almond-like	19683	729	1520	950	RI, O, MS, RC
12	Undecanal	Coriander-like, soapy, citrus-like	27	9	1567	1305	RI, O, MS, RC
13	Dodecanal	Coriander-like, soapy	243	n.d.	1715	1408	RI, O, MS, RC
14	2-Methylpentanal	Malty, fruity	n.d.	27	1731	767	RI, O, MS, RC
15	(*E*)-Undec-2-enal	Coriander-like	81	81	1769	1356	RI, O, MS, RC
16	(2*E*,4*E*)-Deca-2,4-dienal	Fatty, deep-fried	n.d.	81	1801	1311	RI, O, MS, RC
17	(2*E*,4*E*)-Undeca-2,4-dienal	Fatty, deep-fried, coriander-like	9	n.d.	1908	1407	RI, O, MS, RC
18	*Tr*-4,5-Epoxy-(*E*)-2-decenal	Metallic	19683	81	2001	1369	RI, O, MS, RC
Sulphur compounds (3)							
19	Methional	Cooked potato-like	243	243	1467	926	RI, O, MS, RC
20	Benzothiazole	Rubber-like, car tire-like	243	n.d.	1925	1229	RI, O, MS, RC
21	4-Methyl-5-thiazoleethanol	Meat broth, savoury[f]	3	81	2291	1277	RI, O, MS, RC
Pyrazines (2)							
22	2,5-Dimethylpyrazine	Popcorn, fatty, green	243	27	1338	911	RI, O, MS, RC
23	2,6-Dimethylpyrazine	Nutty, malty, solvent-like	243	27	1350	932	RI, O, MS, RC
Ketones (7)							
24	Acetoin	Butter-like	243	729	1275	730	RI, O, MS, RC
25	Oct-1-en-3-one	Mushroom-like	19683	729	1294	958	RI, O, MS, RC
26	Hydroxyacetone	Fuel like, fruity, solvent like	3	3	1299	–	RI*, O, MS, RC
27	Octane-2,3-dione	Strawberry-like, licorice-like, mint-like	19683	729	1369	974	RI, O, MS, RC
28	(*Z*)-1,5-Octadien-3-one	Geranium leaf-like, green, metallic	19683	729	1375	976	RI, O, MS, RC
29	Nonan-2-one	Fruity, soapy	19683	81	1394	1067	RI, O, MS, RC
30	Acetophenone	Bitter almond-like, flowery	243	9	1657	1068	RI, O, MS
Alcohols (23)							
31	Butan-1-ol	Malty	27	81	1131	–	RI, O, MS, RC
32	Pent-1-en-3-ol	Plastic-like	27	27	1163	–	RI, O, MS, RC
33	(*E*)-Pent-3-en-2-ol	Nutty	9	1	1181	708	RI, O, MS, RC
34	Heptan-2-ol	Soapy, citrus-like	3	n.d.	1306	982	RI, O, MS, RC
35	(*E*)-Pent-2-en-1-ol	Fruity, green apple-like, cheesy	3	n.d.	1309	746	RI, O, MS, RC
36	Hexan-1-ol	Green apple-like, green, almond-like	27	n.d.	1356	859	RI, O, MS, RC
37	(*E*)-Hex-2-en-1-ol	Bitter almond-like, grassy	243	n.d.	1407	871	RI, O, MS, RC
38	Oct-1-en-3-ol	Mushroom-like	243	243	1423	968	RI, O, MS, RC
39	Heptan-1-ol	Citrus-like, soapy, fatty	3	n.d.	1440	979	RI, O, MS, RC
40	2-Ethylhexan-1-ol	Eucalyptus-like, band aid-like	81	27	1480	1028	RI, O, MS, RC
41	6-Methylheptan-1-ol	Citrus-like, flowery, soapy	27	n.d.	1501	829	RI, O, MS, RC
42	Octan-1-ol	Soapy, citrus-like, fatty	n.d.	27	1560	1069	RI, O, MS, RC
43	Butane-2,3-diol	Fruity, solvent-like	243	81	1563	792	RI, O, MS, RC
44	Nonan-1-ol	Soapy, citrus-like	27	9	1643	1170	RI, O, MS, RC
45	Undecan-2-ol	Green, geranium	3	9	1708	1305	RI, O, MS, RC
46	Decan-1-ol	Soapy, coriander-like	729	27	1746	1271	RI, O, MS, RC
47	Geosmin	Earthy, moldy	9	n.d.	1801	1420	RI, O, RC
48	Undecan-1-ol	Soapy, coriander-like	n.d.	81	1831	1373	RI, O, MS, RC
49	Benzyl alcohol	Bitter almond-like	19683	2181	1862	1039	RI, O, MS, RC
50	Dodecan-1-ol	Soapy	9	9	1983	1480	RI, O, MS, RC
51	Tridecan-1-ol	Medicinal, metallic, smoky	19683	27	2058	1586	RI, O, MS, RC
52	2-Phenoxyethanol	Flowery, soapy	27	27	2136	1230	RI, O, MS, RC
53	Hexadecan-1-ol	Waxy	3	n.d.	2382	1880	RI, O, MS, RC
Lactones (6)							
54	γ-Caprolactone	Coconut-like	81	n.d.	1686	1052	RI, O, MS, RC
55	γ-Crotonolactone	Fruity, soapy, greenf	27	n.d.	1731	888	RI, O, MS, RC
56	Pantolactone	Smoked-ham	3	27	2025	1017	RI, O, MS, RC
57	δ-Nonalactone	Coconut-like	81	9	2042	1338	RI, O, MS, RC
58	γ-Decalactone	Peach-like, fruity	81	243	2155	1478	RI, O, MS, RC
59	γ-Undecalactone	Peach-like, soapy	81	n.d.	2236	1557	RI, O, MS, RC
Terpenes/terpenoids (15)							
60	α-Pinene	Resin-like, conifer-like	9	9	1015	941	RI, O, MS, RC
61	β-Pinene	Eucalyptus-like	9	27	1106	989	RI, O, MS, RC
62	3-Carene	Citrus-like, eucalyptus-like	81	81	1144	847	RI, O, MS, RC
63	Limonene	Citrus-like, orange-like	9	243	1194	1033	RI, O, MS, RC
64	Ocimene	Celery-like	3	27	1231	1022	RI, O, MS, RC
65	*p*-Cymene	Oregano-like	9	n.d.	1250	1031	RI, O, MS, RC

(*continued on next page*)

Table 1 (*continued*)

No.[a]	Odorant	Odor quality[b]	FD factors[c]		RI values on[d]		Identification criteria[e]
			BAE	GUE	DB-FFAP	DB-5	
66	Terpinolene	Fruity, citrus-like, mint-like	9	27	1263	1097	RI, O, MS, RC
67	Tetrahydrolinalool	Flowery, soapy	243	27	1413	1099	RI, O, MS, RC
68	Linalool oxide	Eucalyptus-like	3	n.d.	1447	1083	RI, O, MS, RC
69	Dihydromyrcenol	Soapy, citrus-like, flowery	243	729	1453	1078	RI, O, MS, RC
70	Limonene oxide	Fruity, citrus-like, solvent-like	243	729	1460	1128	RI, O, MS, RC
71	Linalool	Flowery	81	n.d.	1533	1050	RI, O, MS, RC
72	β-Caryophyllene	Carrot-like, green	243	n.d.	1614	1434	RI, O, MS, RC
73	Menthol	Eucalyptus-like	81	3	1629	1176	RI, O, MS, RC
74	Carvone	Spearmint-like	729	243	1723	1241	RI, O, MS, RC
Carboxylic acids (3)							
75	Propanoic acid	Vinegar-like, fruity, cheesy	27	n.d.	1540	921	RI, O, MS, RC
76	Hexanoic acid	Fruity, musty	27	n.d.	1823	984	RI, O, MS, RC
77	Hexadecanoic acid	Plastic[f]	3	1	2913	1975	RI, O, MS, RC
Aromatic compounds (11)							
78	*p*-Xylene	Fuel like, glue stick-like	1	1	1119	878	RI, O, MS, RC
79	*o*-Xylene	Fatty, green[f]	3	1	1138	995	RI, O, MS, RC
80	1,2,4,5-Tetramethylbenzene	Rubber-like, petroleum-like	3	n.d.	1433	1111	RI, O, MS, RC
81	Methyl benzoate	Almond-like, fruity	27	9	1607	1094	RI, O, MS, RC
82	Naphthalene	Gasoline-like, faecal	1	n.d.	1719	1194	RI, O, MS, RC
83	Phenol	Ink-like	729	81	1975	963	RI, O, MS, RC
84	*p*-Cresol	Horse stable-like, faecal	3	27	2083	1092	RI, O, MS, RC
85	2,4,6-Tribromoanisole	Cork-like	729	243	2273	1640	RI, O, MS, RC
86	Coumarin	Coconut-like, cinnamon-like	6561	n.d.	2430	1447	RI, O, MS, RC
87	Benzophenone	Solvent, rubber[f]	243	1	2470	1647	RI, O, MS, RC
88	Vanillin	Vanilla-like	243	n.d.	2550	1394	RI, O, MS, RC
Others (3)							
89	Undec-1-ene	Soapy, fruity	3	n.d.	1150	1094	RI, O, MS, RC
90	Chloroiodomethane	Solvent-like	n.d.	1	1204	748	RI, O, MS
91	4-Cyanocyclohexene	Bitter almond-like	27	27	1553	1017	RI, O, MS
Unknowns (32)							
92	Unknown	Butter[f]	9	3	1046	–	
93	Unknown	Butter, Fruity[f]	3	27	1062	–	
94	Unknown	Fruity$_f$	n.d.	9	1212	–	
95	Unknown	Geranium, metallic	n.d.	27	1381	–	
96	Unknown	Fish, green, plastic[f]	3	9	1513	–	
97	Unknown	Cheesy, soapy, nutty[f]	n.d.	3	1621	–	
98	Unknown	Soapy, solvent[f]	27	27	1671	–	
99	Unknown	Popcorn-like[f]	27	n.d.	1762	–	
100	Unknown	Butter, green, soapy[f]	27	n.d.	1792	–	
101	Unknown	Fishy[f]	n.d.	27	1815	–	
102	Unknown	Soapy, green, cucumber[f]	n.d.	27	1869		
103	Unknown	Nutty, soapy[f]	n.d.	27	1917	–	
104	Unknown	Faecal, musty[f]	27	27	2109		
105	Unknown	Flower, plastic, soapy[f]	27	27	2182	–	
106	Unknown	Fruity, almond[f]	9	27	2209	–	
107	Unknown	Malty[f]	3	9	2227	–	
108	Unknown	Almond, rubber[f]	3	3	2255	–	
109	Unknown	Fruity[f]	3	1	2309	–	
110	Unknown	Rubber[f]	n.d.	27	2327	–	
111	Unknown	Rubber, oil[f]	3	27	2345	–	
112	Unknown	Cheesy, rubber[f]	9	9	2364	–	
113	Unknown	Car tire, rubber[f]	3	9	2401	–	
114	Unknown	Solvent, rubber[f]	n.d.	1	2450	–	
115	Unknown	Rubber, green[f]	27	1	2501	–	
116	Unknown	Rubber[f]	3	3	2520	–	
117	Unknown	Vanilla, fruity[f]	81	3	2530	–	
118	Unknown	Sweaty, rubber[f]	27	3	2570	–	
119	Unknown	Plastic, oily[f]	81	9	2656	–	
120	Unknown	Green[f]	3	3	2744	–	
121	Unknown	Plastic, sweaty, pepper[f]	9	3	2856	–	
122	Unknown	Plastic[f]	3	1	2988	–	
123	Unknown	Sweaty, plastic[f]	1	1	3056	–	

n.d.: not detected.

[a] Ordered according to retention times on DB-FFAP.

[b] Odor quality described by the Chair of Aroma and Smell Research at FAU database.

[c] Flavor dilution (FD) factor on DB-FFAP from 1 to 19683. Therefore, 1corresponds to the non-diluted sample.

[d] RIs on DB-FFAP and DB-5.

[e] Identification criteria: retention indices (RI), odor quality (O), mass spectrum (MS), and reference compound (RC).

[f] Odor quality determined by one trained panelist at the sniffing port.

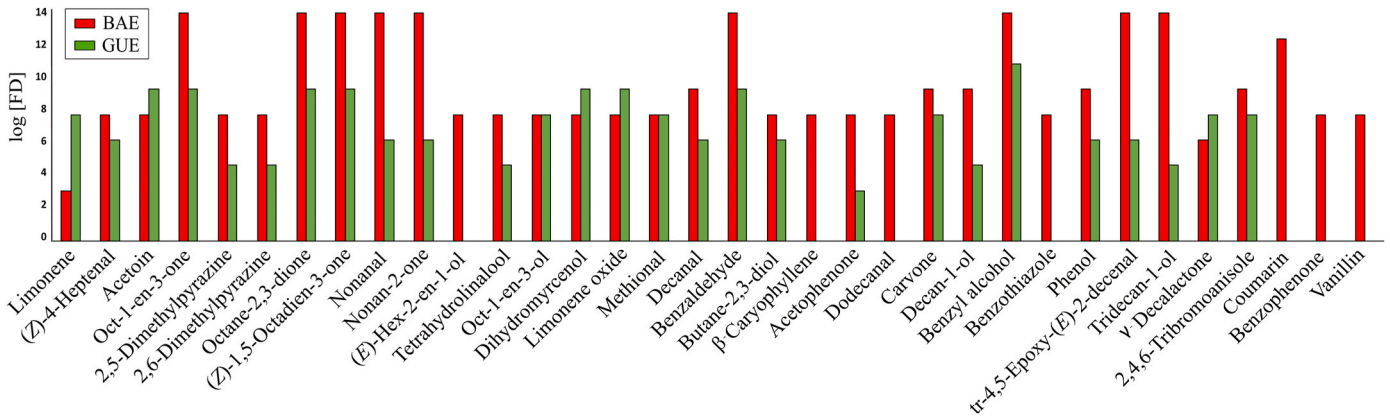


Fig. 2. Odorants with FD factors >243 as displayed in Table 1, presented on a logarithmic scale (base 2).

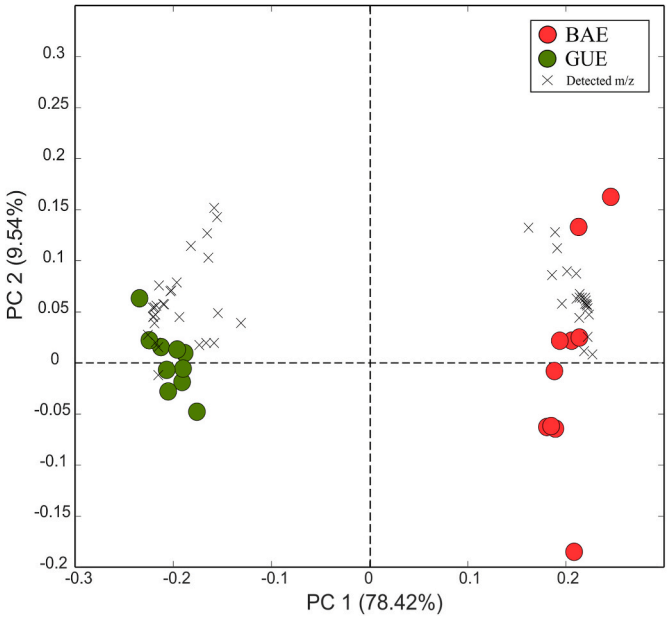


Fig. 3. PCA biplot (PC1 vs PC2) of the different caviar samples investigated, based on the m/z ions (displayed with an X symbol) quantified by PTR-MS at concentrations higher than 0.01 ppbV. BAE and GUE sample replicates are marked with red and green circles, respectively.

extraction method detailed in Materials and Methods 2.3. Highly volatile compounds, such as acetaldehyde and formaldehyde, might not be easily trapped by DCM-based distillation methods because their boiling point is lower than that of the solvent (Linstrom & Mallard, 2023). Hence, alternative methods ought to be used for the analysis of these compounds (Buettner & Schieberle, 2001; Y. Xu et al., 2007). However, these can still be important aroma contributors in many different food products, especially when they are present at concentrations above their odor threshold. For instance, m/z 45.034 (tentatively acetaldehyde), described as "fruity", was reported with an odor threshold in air of 41 ng/L (Rychlik et al., 1998), an order of magnitude lower than the concentration of the corresponding mass in the present study. Since it is presently unverified and speculative, this would, therefore, need to be substantiated by additional headspace GC-MS techniques. However, static headspace PTR-MS may thus potentially serve as a complementary technique to traditional extraction methods for GC-MS analyses to obtain hints on the presence and quantities of highly volatile substances. In line with the results presented in this study, Le Bechec et al. (2024) also reported the relevance of ions H_3O^+ 45, 75 and 111 in discriminating caviar samples from Aquitaine by their maturity level, but in this case using SIFT-MS.

3.4. Comparison of the sensory and analytical results

The odor descriptors described by the panelists in the sensory evaluation aligned well with the odor qualities of the identified odor-active compounds. For example, "fishy" and "seawater-like/algae-like" could be related to the compound (*Z*)-4-heptenal (4), which was detected with FD = 243 and 81 in BAE and GUE, respectively. Moreover, other compounds identified in this study, namely hexanal (2), heptanal (3), nonanal (7), (*E*)-oct-2-enal (8), (2*E*,4*E*)-hepta-2,4-dienal (9), decanal (10), oct-1-en-3-one (25), pent-1-en-3-ol (32) and oct-1-en-3-ol (38) have also been reported in the literature as contributors to "fishy" odor (Dai et al., 2024). It should be mentioned that there are some studies that report synergistic effects between odorants for the formation of "fishy" off-odor in algal oils, namely, between heptanal and (*E*,*Z*)-3, 5-octadien-2-one(Marsili & Laskonis, 2014a, 2014b). The attribute "green" can be related to the odorant compounds hexanal (2), 2-ethylhexanal (6), 2,5-dimethylpyrazine (22), hexan-1-ol (36), (*Z*)-1,5-octadien-3-one (28), undecan-2-ol (45), and β-caryophyllene (72).

The odor attribute "fatty" aligned well with several aldehydes, alcohols and other substances with fatty smell characteristics, notably heptanal (3), (*Z*)-hept-4-enal (4), (*E*)-oct-2-enal (8), (2*E*,4*E*)-hepta-2,4-dienal (9), (2*E*,4*E*)-deca-2,4-dienal (16), (2*E*,4*E*)-undeca-2,4-dienal (17), 2,5-dimethylpyrazine (22), heptan-1-ol (39) and octan-1-ol (42). It is noteworthy that the panelists selected acetoin ("butter-like") as the reference for the fatty descriptor. The FD factor for this compound was high in both samples, BAE (FD = 243) and GUE (FD = 729). This molecule is both volatile and reactive, so losses can occur during sample workup and affect the FD factor representation. Nonetheless, it is worth pointing out again that cAEDA is only a screening approach, revealing odorants with that are likely to be important contributors to a given flavor and that divergent FD factors may indicate flavor differences in foods (Grosch, 1994). In the present study, acetoin was detected with a high FD factor through AEDA-GC-(GC)-O/MS and acetaldehyde was tentatively proposed from the masses detected at higher concentrations through PTR-MS (Tables 1 and 2). Previous studies reported synergistic effects between acetoin and acetaldehyde (tentatively proposed in the PTR-MS analysis) compounds, leading to enhanced overall aroma intensity in yogurt (Tian et al., 2020). In this respect, future investigations may need to consider such potential perceptual interaction effects between odor compounds. The attribute "wheat" did not find any odorant counterpart in the present study. However, wheat aroma is likewise a percept generated from a complex mixture of odorants (Czerny & Schieberle, 2002).

Table 2
Mass peaks from the static headspace PTR-MS analysis of the caviar samples and proposed chemical structures. Experimental mass detected (m/z ion), predicted formula, theoretical mass, structure proposition, and concentrations in ppbV in both samples (average and standard deviation of ten injections) are specified.

n	m/z ion	Predicted formula	Theoretical mass	Structure proposition	BAE Concentration (ppbV)	GUE Concentration (ppbV)	References
1	m/z 26.01453	–	–	Unknown	0.12 ± 0.02	0.15 ± 0.01	–
2	m/z 31.01637	CH_3O^+	31.0183	Formaldehyde, Aldehyde fragment	6.68 ± 1.12	3.32 ± 0.13	d,e
3	m/z 33.03127	CH_5O^+	33.0339	Methanol	3.12 ± 0.15	0.75 ± 0.11	b,d,e,f,g
4	m/z 39.02025	$C_3H_3^+$	39.0234	Unknown	0.97 ± 0.10	1.70 ± 0.09	–
5	m/z 40.02457	–	–	Unknown	0.03 ± 0.01	0.05 ± 0.01	–
6	m/z 41.03579	$C_3H_5^+$	41.0391	Alkyl fragment (alcohol, ester)	2.48 ± 0.30	3.90 ± 0.18	a,b,d,e
7	m/z 43.01516	$C_2H_3O^+$	43.0184	Fragments/Acetic acid fragment	4.29 ± 0.40	6.86 ± 0.44	a,b,d,e
8	m/z 43.05161	$C_3H_7^+$	43.0548	Alkyl fragment (alcohol, ester, acetate)	0.68 ± 0.08	1.99 ± 0.12	a,b,d,f
9	m/z 44.01195	CNH_2O^+	44.0136	Unknown	0.11 ± 0.02	0.18 ± 0.01	–
10	m/z 44.05318	$C_2H_6N^+$	44.0500	Unknown	0.00 ± 0.02	0.05 ± 0.01	–
11	m/z 44.97698	CSH^+	44.9798	Unknown	0.17 ± 0.08	0.65 ± 0.13	–
12	m/z 45.00499	–	44.9894	Unknown	3.09 ± 0.83	6.92 ± 1.17	–
13	m/z 45.03064	$C_2H_5O^+$	45.0340	Acetaldehyde	84.35 ± 8.09	144.16 ± 9.25	a,b,c,d,f,g
14	m/z 45.05785	–	–	Unknown	0.88 ± 0.22	1.99 ± 0.47	–
15	m/z 46.03205	CH_4NO^+	46.0287	Unknown	1.78 ± 0.16	3.01 ± 0.19	–
16	m/z 47.02355	–	47.0128	Unknown	0.11 ± 0.02	0.17 ± 0.04	–
17	m/z 47.0463	$C_2H_7O^+$	47.0497	Ethanol	3.90 ± 0.65	5.60 ± 0.58	a,b,c,d,e
18	m/z 48.0497	CH_6NO^+	48.0449	Unknown	0.09 ± 0.02	0.12 ± 0.02	–
19	m/z 49.97817	–	–	Unknown	0.01 ± 0.00	0.00 ± 0.00	–
20	m/z 55.05072	$C_4H_7^+$	55.0548	Fragment (aldehyde)	0.29 ± 0.04	0.36 ± 0.02	a,b,c,d,e,f
21	m/z 57.03046	$C_3H_5O^+$	57.0340	2-Propenal/Acetol fragment	0.05 ± 0.01	0.04 ± 0.00	b
22	m/z 57.06636	$C_4H_9^+$	57.0699	Fragment (alcohol, ester, aldehyde)	0.05 ± 0.05	0.00 ± 0.02	a,b,c,d,e
23	m/z 58.89333	–	–	Unknown	0.02 ± 0.01	0.04 ± 0.00	–
24	m/z 59.04604	$C_3H_7O^+$	59.0496	Propanal/Acetone	3.17 ± 0.45	2.28 ± 0.10	e,f,g,h
25	m/z 60.04573	$C_2H_6NO^+$	60.0449	Unknown	0.13 ± 0.02	0.09 ± 0.01	–
26	m/z 61.06636	$C_3H_9O^+$	61.0653	Unknown	0.03 ± 0.00	0.07 ± 0.01	–
27	m/z 63.0405	$C_2H_7O_2^+$	63.0446	Unknown	0.36 ± 0.03	0.60 ± 0.04	–
28	m/z 64.04414	$CH_6NO_2^+$	64.0398	Unknown	0.01 ± 0.00	0.02 ± 0.00	–
29	m/z 65.05637	$C_2H_9O_2^+$	65.0602	Ethanol hydrate	0.08 ± 0.02	0.11 ± 0.01	f
30	m/z 67.0509	$C_5H_7^+$	67.0547	Monoterpene fragments/Butanol, 2-Pentanal fragment	0.02 ± 0.00	0.00 ± 0.00	a,b
31	m/z 69.06628	$C_5H_9^+$	69.0704	Myrcene fragment/Other fragments	0.17 ± 0.02	0.14 ± 0.01	a,b,c,d,e
32	m/z 73.06161	$C_4H_9O^+$	73.0653	2-Butanone/Butanal/Methylpropanal	0.46 ± 0.06	0.92 ± 0.07	b,c,d,e
33	m/z 75.02286	$C_3H_7S^+$	75.0263	Unknown	0.01 ± 0.02	0.05 ± 0.04	–
34	m/z 75.07756	$C_4H_{11}O^+$	75.0809	1-/2-Butanol/2-Methylpropanol	2.92 ± 0.68	0.08 ± 0.03	d
35	m/z 76.08224	$C_3H_{10}NO^+$	76.0762	Unknown	0.21 ± 0.05	0.01 ± 0.00	–
36	m/z 77.05534	$C_3H_9O_2^+$	77.0602	Propylene glycol/1,3-Propanediol	0.06 ± 0.01	0.04 ± 0.00	a
37	m/z 77.08682	–	–	Unknown	0.01 ± 0.00	0.00 ± 0.00	–

(*continued on next page*)

Table 2 (*continued*)

n	m/z ion	Predicted formula	Theoretical mass	Structure proposition	BAE Concentration (ppbV)	GUE Concentration (ppbV)	References
38	m/z 82.94186	–	82.9955	Unknown	1.11 ± 0.23	0.03 ± 0.01	
39	m/z 82.98473	–	82.9955	Unknown	0.01 ± 0.01	0.00 ± 0.00	–
40	m/z 83.08249	$C_6H_{11}^+$	83.0860	Cyclohexene/Fragments	0.05 ± 0.01	0.00 ± 0.00	a,b,c,d,e,f
41	m/z 83.9525	–	–	Unknown	0.03 ± 0.00	0.00 ± 0.00	–
42	m/z 84.93936	C_2HN_2S	84.9860	Unknown	0.68 ± 0.15	0.02 ± 0.00	–
43	m/z 85.06163	$C_5H_9O^+$	85.0653	trans-2-Methyl-2-butenal/2-Pentenal (E)/1-penten-3-one	0.02 ± 0.00	0.00 ± 0.00	b,c,e,f
44	m/z 85.94281	–	–	Unknown	0.02 ± 0.00	0.00 ± 0.00	–
45	m/z 86.94144	–	86.9813	Unknown	0.11 ± 0.02	0.00 ± 0.00	–
46	m/z 91.07049	$C_4H_{11}O_2^+$	91.0759	2,3/1,4-Butanediol	0.01 ± 0.00	0.02 ± 0.00	d
47	m/z 93.03413	$C_3H_9OS^+$	93.0374	2-Methylmercaptoethanol	0.02 ± 0.02	0.09 ± 0.04	c
48	m/z 93.06739	$C_7H_9^+$	93.0704	Toluene/p-Cymene fragment	0.02 ± 0.01	0.00 ± 0.00	b,c,e,f
49	m/z 93.09557	$C_4H_{13}O_2^+$	93.0915	Unknown	0.03 ± 0.01	0.00 ± 0.00	–
50	m/z 95.01373	$C_2H_7SO_2^+$	95.0166	Dimethyl sulfone	0.00 ± 0.00	0.02 ± 0.01	c
51	m/z 111.044	$C_6H_7O_2^+$	111.0446	2-Acetylfuran/5-Methylfurfural	0.00 ± 0.00	0.01 ± 0.00	c

rrReferences: [a] Pedrotti et al., 2018; [b] Pedrotti et al., 2020; [c] Yener et al., 2016; [d] Bianchi et al., 2017; [e] Boscaini et al., 2003; [f] Phillips et al., 2010.
Unknown: masses not associated to any compound in the reviewed studies.

4. Conclusion

Differences in the relative potency of the aroma compounds identified by cAEDA-GC-(GC)-O/MS, as well as in the static headspace PTR-MS fingerprints of commercial caviar samples (*Acipenser baerii* and *Acipenser gueldenstaedtii*) were investigated. Among the 123 detected odorants, 34 were reported with FD factors higher than 243, which indicates that these compounds may contribute to caviar aroma. The odor qualities of the identified odorants aligned well with the sensory descriptors provided by trained panelists. However, no statistical differences were observed between the rated aroma attributes of the two caviar types. The PTR-MS results revealed differences in the volatile composition of the samples, allowing for a differentiation of the caviar types. Additionally, a tentative identification was proposed for the masses detected at higher concentrations, potentially indicating (highly) volatile compounds that would elapse detection via cAEDA-GC-(GC)-O/MS. Nevertheless, further validation experiments are needed, e.g. by means of headspace GC-MS techniques.

This study highlights the need for a combination of sensory and instrumental analyses for a comprehensive characterization of the odorant compounds in caviar. In the aquaculture sector, this information is essential for future development of production methods for caviar that meet sensory quality standards. The novel insights on the odorant composition, sensory characteristics of caviar, and potential of quick fingerprinting methods for use in industrial application will help improve flavor quality assessments in the caviar industry.

CRediT authorship contribution statement

Mariana Rodrigues da Silva: Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Conceptualization. **Pedro Martínez Noguera:** Writing – original draft, Validation, Methodology, Investigation, Formal analysis, Conceptualization. **Bastien Debeuf:** Resources, Conceptualization. **Mikael A. Petersen:** Writing – review & editing, Supervision, Funding acquisition, Conceptualization. **Helene M. Loos:** Writing – review & editing, Supervision, Funding acquisition, Conceptualization. **Andrea Buettner:** Writing – review & editing, Supervision, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Andrea Buettner reports financial support was provided by Horizon Europe. Andrea Buettner reports financial support was provided by Deutsche Forschungsgemeinschaft. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.lwt.2025.118261.

Data availability

Data will be made available on request.

References

Aprea, E., Biasioli, F., Sani, G., Cantini, C., Mark, T., & Gasperi, F. (2006). Proton transfer reaction-mass spectrometry (PTR-MS) headspace analysis for rapid detection of oxidative alteration of olive oil. *Journal of Agricultural and Food Chemistry, 54*, 7635–7640.

Araghipour, N., Colineau, J., Koot, A., Akkermans, W., Rojas, J. M. M., Beauchamp, J., Wisthaler, A., Märk, T. D., Downey, G., Guillou, C., Mannina, L., & Ruth, S. van (2008). Geographical origin classification of olive oils by PTR-MS. *Food Chemistry, 108*(1), 374–383. https://doi.org/10.1016/j.foodchem.2007.10.056

Baker, A. K., Vixie, B., Rasco, B. A., Ovissipour, M., & Ross, C. F. (2014). Development of a lexicon for caviar and its usefulness for determining consumer preference. *Journal of Food Science, 79*(12), S2533–S2541. https://doi.org/10.1111/1750-3841.12703

Bianchi, T., Weesepoel, Y., Koot, A., Iglesias, I., Eduardo, I., Gratacós-Cubarsí, M., Guerrero, L., Hortós, M., & van Ruth, S. (2017). Investigation of the aroma of commercial peach (Prunus persica L. batsch) types by Proton Transfer Reaction–Mass Spectrometry (PTR-MS) and sensory analysis. *Food Research International, 99*(January), 133–146. https://doi.org/10.1016/j.foodres.2017.05.007

Biasioli, F., Yeretzian, C., Märk, T. D., Dewulf, J., & Van Langenhove, H. (2011). Direct-injection mass spectrometry adds the time dimension to (B)VOC analysis. *TrAC, Trends in Analytical Chemistry, 30*(7), 1003–1017. https://doi.org/10.1016/j.trac.2011.04.005

Bledsoe, G. E., Bledsoe, C. D., & Rasco, B. (2003). Caviars and fish roe products. *Critical Reviews in Food Science and Nutrition, 43*(3), 317–356. https://doi.org/10.1080/10408690390826545

Boscaini, E., Van Ruth, S., Biasioli, F., Gasperi, F., & Märk, T. D. (2003). Gas chromatography-olfactometry (GC-O) and proton transfer reaction-mass spectrometry (PTR-MS) analysis of the flavor profile of grana padano, parmigiano reggiano, and grana trentino cheeses. *Journal of Agricultural and Food Chemistry, 51*(7), 1782–1790. https://doi.org/10.1021/jf020922g

Brattoli, M., Cisternino, E., Rosario Dambruoso, P., de Gennaro, G., Giungato, P., Mazzone, A., Palmisani, J., & Tutino, M. (2013). Gas chromatography analysis with olfactometric detection (GC-O) as a useful methodology for chemical characterization of odorous compounds. *Sensors, 13*(12), 16759–16800. https://doi.org/10.3390/s131216759

Bronzi, P., Chebanov, M., Michaels, J. T., Wei, Q., Rosenthal, H., & Gessner, J. (2019). Sturgeon meat and caviar production: Global update 2017. *Journal of Applied Ichthyology, 35*(1), 257–266. https://doi.org/10.1111/jai.13870

Buettner, A., & Schieberle, P. (2001). Evaluation of aroma differences between hand-squeezed juices from Valencia late and navel oranges by quantitation of key odorants and flavor reconstitution experiments. *Journal of Agricultural and Food Chemistry, 49*(5), 2387–2394. https://doi.org/10.1021/jf001363l

Caprino, F., Moretti, V. M., Bellagamba, F., Turchini, G. M., Busetto, M. L., Giani, I., Paleari, M. A., & Pazzaglia, M. (2008). Fatty acid composition and volatile compounds of caviar from farmed white sturgeon (Acipenser transmontanus). *Analytica Chimica Acta, 617*(1–2), 139–147. https://doi.org/10.1016/j.aca.2008.02.005

Cardinal, M., Cornet, J., & Vallet, L. J. (2002). Sensory characteristics of caviar from wild and farmed sturgeon. *International Review of Hydrobiology, 87*(5–6), 651–659. https://doi.org/10.1002/1522-2632(200211)87:5/6<651::AID-IROH651>3.0.CO;2-W

Cordero, C., Kiefl, J., Schieberle, P., Reichenbach, S. E., & Bicchi, C. (2015). Comprehensive two-dimensional gas chromatography and food sensory properties: Potential and challenges. *Analytical and Bioanalytical Chemistry, 407*(1), 169–191. https://doi.org/10.1007/s00216-014-8248-z

Czerny, M., & Schieberle, P. (2002). Important aroma compounds in freshly ground wholemeal and white wheat flour - Identification and quantitative changes during sourdough fermentation. *Journal of Agricultural and Food Chemistry, 50*(23), 6835–6840. https://doi.org/10.1021/jf020638p

da Silva, M. R., Egiddi, M., Debeuf, B., Buettner, A., & Loos, H. M. (2024). Aroma-active compounds in Russian sturgeon (Acipenser gueldenstaedtii) raised in recirculating aquaculture systems. *Journal of Agriculture and Food Research, 18*. https://doi.org/10.1016/j.jafr.2024.101443

Dai, W., He, S., Huang, L., Lin, S., Zhang, M., Chi, C., & Chen, H. (2024). Strategies to reduce fishy odor in aquatic products: Focusing on formation mechanism and mitigation means. *Food Chemistry, 444*(October 2023), Article 138625. https://doi.org/10.1016/j.foodchem.2024.138625

Ellis, A. M., & Mayhew, C. A. (2014). *Proton transfer reaction mass spectrometry*.

Engel, W., Bahr, W., & Schieberle, P. (1999). Solvent assisted flavour evaporation - A new and versatile technique for the careful and direct isolation of aroma compounds from complex food matrices. *European Food Research and Technology, 209*(3–4), 237–241. https://doi.org/10.1007/s002170050486

Erasmus, S. W., Muller, M., Alewijn, M., Koot, A. H., van Ruth, S. M., & Hoffman, L. C. (2017). Proton-transfer reaction mass spectrometry (PTR-MS) for the authentication of regionally unique South African lamb. *Food Chemistry, 233*, 331–342. https://doi.org/10.1016/j.foodchem.2017.04.131

Farag, M. A., Abib, B., Tawfik, S., Shafik, N., & Khattab, A. R. (2021). Caviar and fish roe substitutes: Current status of their nutritive value, bio-chemical diversity, authenticity and quality control methods with future perspectives. *Trends in Food Science and Technology, 110*(October 2020), 405–417. https://doi.org/10.1016/j.tifs.2021.02.015

Grosch, W. (1993). Detection of potent odorants in foods by aroma extract dilution analysis. *Trends in Food Science and Technology, 4*(3), 68–73. https://doi.org/10.1016/0924-2244(93)90187-F

Grosch, W. (1994). Determination of potent odourants in foods by aroma extract dilution analysis (AEDA) and calculation of odour activity values (OAVs). *Flavour and Fragrance Journal, 9*, 147–158. https://doi.org/10.1002/ffj.2730090403

Le Bechec, M., Reyrolle, M., Chesneau, V., Breuil, M., & Desauziers, V. (2024). Rapid analysis of VOCs with SIFT-MS as a decision-making support tool for caviar producers. *Food Control, 155*(July 2023). https://doi.org/10.1016/j.foodcont.2023.110094

Lindholm-Lehto, P. C., & Vielma, J. (2019). Controlling of geosmin and 2-methylisoborneol induced off-flavours in recirculating aquaculture system farmed fish—A review. *Aquaculture Research, 50*(1), 9–28. https://doi.org/10.1111/are.13881

Linstrom, P., & Mallard, W. (2023). *No title*. National Institute of Standards and Technology. https://webbook.nist.gov.

Liu, L., Lu, S., Zhang, W., Bai, F., Wang, J., Zhang, X., Xu, H., Jiang, X., An, S., Li, W., Zhao, Y., & Xu, X. (2024). Correlation investigation between core microbe inoculation and the evolution of flavor characteristics during the storage of sturgeon caviar (Acipenser gueldenstaedtii). *Food Research International, 178*(1299). https://doi.org/10.1016/j.foodres.2023.113903

Lopez, A., Vasconi, M., Bellagamba, F., Mentasti, T., & Moretti, V. M. (2020). Sturgeon meat and caviar quality from different cultured species. *Fishes, 5*(1). https://doi.org/10.3390/fishes5010009

Lopez, A., Vasconi, M., Bellagamba, F., Mentasti, T., Pazzaglia, M., & Moretti, V. M. (2020). Volatile organic compounds profile in white sturgeon (Acipenser transmontanus) caviar at different stages of ripening by multiple headspace solid phase microextraction. *Molecules, 25*(5). https://doi.org/10.3390/molecules25051074

Lu, X., & Rasco, B. A. (2014). Sturgeon (Acipenser transmontanus) sexual maturation and caviar quality. *Reviews in Aquaculture, 6*(2), 89–99. https://doi.org/10.1111/raq.12029

Marsili, R., & Laskonis, C. (2014a). Odorant synergy effects as the cause of fishy malodors in algal marine oils. *Journal of Agricultural and Food Chemistry, 62*(40), 9676–9682. https://doi.org/10.1021/jf502252q

Marsili, R., & Laskonis, C. (2014b). The importance of odorant synergy effects in understanding malodour problems in DHA and EPA products. *Lipid Technology, 26*(2), 31–34. https://doi.org/10.1002/lite.201400009

Mazzucotelli, M., Farneti, B., Khomenko, I., Gonzalez-Estanol, K., Pedrotti, M., Fragasso, M., Capozzi, V., & Biasioli, F. (2022). Proton transfer reaction mass spectrometry: A green alternative for food volatilome profiling. *Green Analytical Chemistry, 3*(October), Article 100041. https://doi.org/10.1016/j.greeac.2022.100041

Pedrotti, M., Khomenko, I., Cappellin, L., Fontana, M., Somenzi, M., Falchero, L., Arveda, M., Fogliano, V., & Biasioli, F. (2018). Rapid and noninvasive quality control of anhydrous milk fat by PTR-MS: The effect of storage time and packaging. *Journal of Mass Spectrometry, 53*(9), 753–762. https://doi.org/10.1002/jms.4204

Pedrotti, M., Khomenko, I., Fontana, M., Somenzi, M., Falchero, L., Arveda, M., Cappellin, L., Fogliano, V., & Biasioli, F. (2020). The good, the bad and the aged: Predicting sensory quality of anhydrous milk fat by PTR/SRI-Tof-MS analysis and data mining. *International Dairy Journal, 109*, Article 104729. https://doi.org/10.1016/j.idairyj.2020.104729

Phillips, K., Niimi, J., Hamid, N., Silcock, P., Delahunty, C., Barker, M., Sewell, M., & Bremer, P. (2010). Sensory and volatile analysis of sea urchin roe from different geographical regions in New Zealand. *Lwt, 43*(2), 202–213. https://doi.org/10.1016/j.lwt.2009.08.008

Pustjens A, M., Boerrigter-Eenling, R., Koot A, H., & M van Ruth, S. (2018). Food authenticity: Provenancing. A case study of fish. *Descriptive Food Science*. https://doi.org/10.5772/intechopen.80470

Raymakers, C. (2006). CITES, the convention on international trade in endangered species of wild fauna and flora: Its role in the conservation of Acipenseriformes. *Journal of Applied Ichthyology, 22*(SUPPL. 1), 53–65. https://doi.org/10.1111/j.1439-0426.2007.00929.x

Rocchetti, G., Vezzulli, F., Senizza, B., Betti, A., Dordoni, R., Lambri, M., & Lucini, L. (2025). The contribution of sturgeon species and processing conditions on caviar texture, metabolomic profile and sensory traits. *Food Chemistry, 463*(P4), Article 141516. https://doi.org/10.1016/j.foodchem.2024.141516

Rychlik, M., Schieberle, P., & Grosch, W. (1998). Compilation of odor thresholds, odor qualities and retention indices of key food odorants. *Deutsche Forschungsanstalt fuer Lebensmittelchemie: Sicuro, B, 11*(1), 204–219. https://doi.org/10.1111/raq.12235 (2019). The future of caviar production on the light of social changes: a new dawn for caviar? *Reviews in Aquaculture*.

Sicuro, B. (2019). The future of caviar production on the light of social changes: a new dawn for caviar? *Reviews in Aquaculture, 11*(1), 204–219. https://doi.org/10.1111/raq.12235

Song, H., & Liu, J. (2018). GC-O-MS technique and its applications in food flavor analysis. *Food Research International, 114*(August), 187–198. https://doi.org/10.1016/j.foodres.2018.07.037

Tavakoli, S., Luo, Y., Regenstein, J. M., Daneshvar, E., Bhatnagar, A., Tan, Y., & Hong, H. (2021). Sturgeon, caviar, and caviar substitutes: From production, gastronomy, nutrition, and quality change to trade and commercial mimicry. *Reviews in Fisheries Science and Aquaculture, 29*(4), 753–768. https://doi.org/10.1080/23308249.2021.1873244

Tian, H., Yu, B., Yu, H., & Chen, C. (2020). Evaluation of the synergistic olfactory effects of diacetyl, acetaldehyde, and acetoin in a yogurt matrix using odor threshold, aroma intensity, and electronic nose analyses. *Journal of Dairy Science, 103*(9), 7957–7967. https://doi.org/10.3168/jds.2019-17495

Vilgis, T. A. (2020). The physics of the mouthfeel of caviar and other fish roe. *International Journal of Gastronomy and Food Science, 19*(November 2019), Article 100192. https://doi.org/10.1016/j.ijgfs.2019.100192

Xu, Y., Fan, W., & Qian, M. C. (2007). Characterization of aroma compounds in apple cider using solvent-assisted flavor evaporation and headspace solid-phase microextraction. *Journal of Agricultural and Food Chemistry, 55*(8), 3051–3057. https://doi.org/10.1021/jf0631732

Xu, X., Lu, S., Li, X., Bai, F., Wang, J., Zhou, X., Gao, R., Zeng, M., & Zhao, Y. (2022). Effects of microbial diversity and phospholipids on flavor profile of caviar from hybrid sturgeon (Huso dauricus × Acipenser schrencki). *Food Chemistry, 377*. https://doi.org/10.1016/j.foodchem.2021.131969

Yener, S., Sánchez-López, J. A., Granitto, P. M., Cappellin, L., Märk, T. D., Zimmermann, R., Bonn, G. K., Yeretzian, C., & Biasioli, F. (2016). Rapid and direct volatile compound profiling of black and green teas (Camellia sinensis) from different countries with PTR-ToF-MS. *Talanta, 152*, 45–53. https://doi.org/10.1016/j.talanta.2016.01.050