Key Odorants of French Fries

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ABSTRACT: Twenty-one compounds, which had been screened in preceding experiments as potent odorants of french fries prepared in palm oil (PO), were quantified by stable isotope dilution assays. Nineteen odorants were dissolved in sunflower oil in concentrations equal to those in PO. The flavor profile of the model obtained was close to that of a real sample of PO. A comparison of the complete model with models lacking one or more compounds indicated the following key odorants of PO: 2-ethyl-3,5-dimethylpyrazine, 3-ethyl-2,5-dimethylpyrazine, 2,3-diethyl-5-methylpyrazine, 3-isobutyl-2-methoxypyrazine, (*E,Z*)- and (*E,E*)-2,4-decadienal, *trans*-4,5-epoxy-(*E*)-2-decenal, 4-hydroxy-2,5-dimethyl-3(2H)-furanone, methylpropanal, 2- and 3-methylbutanal, and methanethiol. Replacement of palm oil by coconut fat led to a coconut note in the profile of french fries. γ-Octalactone was identified as a major contributor to this note.

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Due to their pleasant flavor, french fries enjoy a great popularity among consumers. Several studies have been carried out to identify the compounds in the volatile fraction of french fries and in that of potato chips showing a similar aroma. According to a review published by Maga (1), more than 500 compounds have been elucidated.

The group of Buttery (2,3) made an attempt to differentiate between odor-active and odorless volatiles of potato chips. On the basis of high odor activity values (OAV, ratio of concentration to odor threshold), the authors (2,3) concluded that the most important odorant was methional followed by phenylacetaldehyde, 2- and 3-methylbutanal, 3-ethyl-2,5-dimethylpyrazine, (*E,E*)-2,4-decadienal, 1-penten-3-one, and hexanal.

Recently, the odorants which might cause the flavor of french fries prepared in palm oil (PO) were screened by aroma extract dilution analysis (AEDA) and by gas chromatography-olfactometry of headspace samples (GCOH) (4). The results indicated that compounds **1** to **21** in Table 1 have to be considered as possible contributors to the flavor of PO.

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The aim of the present study was to evaluate this suggestion. To this purpose, odorants **1** to **21** (Table 1) were quantified in PO and their OAV were calculated. Then those compounds having odor qualities which corresponded to the notes of the flavor profile of PO, and showing relatively high OAV, were dissolved in sunflower oil. The flavor profile of the model obtained (MPO) was compared to that of the real PO for similarity. Furthermore, changes in the overall flavor of MPO were evaluated after omission of one or more odorants to show their contributions to the flavor of PO.

The flavor profile of french fries prepared in coconut fat (CF) differed from that of PO by a coconut-like note which was additionally perceived. Identification of the odorants causing the flavor of CF by means of the analytical and sensorial methods reported for PO was included in the present study.

EXPERIMENTAL PROCEDURES

Materials. Potatoes, variety Agria, were from retail trade. The composition of the potatoes, which were stored at 8°C (relative humidity 70%) before use, and of the palm oil used for frying has been reported (4). In addition, coconut fat was used as frying medium; its fatty acid composition (g/100 g) was determined after transesterification (5): 8:0 (7.2), 10:0 (6.0), 12:0 (44.2), 14:0 (15.1), 16:0 (8.1), 18:0 (3.2), 18:1 (5.2), and 18:2 (1.9). French fries were prepared as reported (4) and then frozen in liquid nitrogen.

Reference substances for the odorants **1** to **13** (Table 1) were obtained from the sources detailed (4). Compounds **24** and **25** as well as (*E*)-2-decenal, 2-ethyl-3-methylpyrazine, and [$^2H_3$]-methyl lithium/lithium iodide complex in diethyl ether (0.5 mol/L, 100 mL) were from Aldrich (Steinheim, Germany). [$^2H_3$]-2-Ethenyl-3,5-dimethylpyrazine (**4a–d** in Table 1) was a gift of M. Czerny (Deutsche Forschungsanstalt für Lebensmittelchemie, Garching). Silica gel G 60 (70–230 mesh) from Merck (Darmstadt, Germany) was washed with concentrated HCl and water (6); the water content was adjusted to 5% w/w.

Instrumental analysis. High-resolution gas chromatography (HRGC) was performed with a Carlo Erba gas chromatograph (Type HRGC 5160; Carlo Erba, Hofheim, Germany) using the fused silica capillaries OV-1701 and DB-FFAP (30 m × 0.32 mm, 0.25 μm film thickness; J&W Scientific, Folsom, CA). The samples (0.5 μL each) were applied by the

TABLE 1
Mass Chromatography of the Flavor Compounds 1 to 25 and Their Labeled Standards: Selected Ions in the Mass Spectra, Calibration Factors, and Thin-Film Capillaries[a]

Flavor compound[a]	Ion (m/z)[b]	Labeled compound[c]	Ion (m/z)[b]	Calibration factor[d]	Capillary
2-Ethyl-3,5-dimethylpyrazine (**1**)	137	**1-d**	140	1.08	OV-1701
3-Ethyl-2,5-dimethylpyrazine (**2**)	137	**2-d**	140	1.07	OV-1701
2,3-Diethyl-5-methylpyrazine (**3**)	151	**3-d**	154	1.04	OV-1701
2-Ethenyl-3-ethyl-5-methylpyrazine (**4**)[e]	149	**4a–d**	138	1.29	OV-1701
3-Isobutyl-2-methoxypyrazine (**5**)	167	**5-d**	170	0.94	OV-1701
1-Octen-3-one (**6**)	127	**6-d**	129	0.77	OV-1701
(*Z*)-2-Nonenal (**7**)	123	**7-d**	125	0.77	OV-1701
(*E*)-2-Nonenal (**8**)	141	**8-d**	143	0.83	OV-1701
(*E,E*)-2,4-Nonadienal (**9**)	139	**9-d**	141	0.58	OV-1701
(*E,Z*)-2,4-Decadienal (**10**)	153	**10-d**	157	0.33	OV-1701
(*E,E*)-2,4-Decadienal (**11**)	153	**11-d**	157	0.41	OV-1701
trans-4,5-Epoxy-(*E*)-2-decenal (**12**)	169	**12-d**	173	0.45	DB-FFAP
4-Hydroxy-2,5-dimethyl-3(2H)-furanone (**13**)	129	**13-c**	131	1.00	DB-FFAP
3-Hydroxy-4,5-dimethyl-2(5H)-furanone (**14**)	129	**14-c**	131	1.00	DB-FFAP
Methylpropanal (**15**)	73	**15-d**	80	1.03	Rtx-5
2-Methylbutanal (**16**)	87	**16-d**	70–71[f]	1.25	Rtx-5
3-Methylbutanal (**17**)	69			1.15	Rtx-5
2,3-Butanedione (**18**)	87	**18-c**	91	1.00	Rtx-5
Methional (**19**)	105	**19-d**	108	1.08	OV-1071
Methanethiol (**20**)	49	**20-d**	52	1.00	Rtx-5
Dimethyltrisulfide (**21**)	127	**21-d**	133	1.15	OV-1701
γ-Octalactone (**22**)	143			0.50	OV-1701
γ-Nonalactone (**23**)	157	**25-d**	173–174[f]	0.61	OV-1701
γ-Decalactone (**24**)	171			0.73	OV-1701
δ-Decalactone (**25**)	171			0.73	OV-1701

[a]Compounds **1** to **11, 13, 14, 19,** and **21** to **25** were determined with their internal labeled standards by the ion trap detector ITD-800; compound **12** and its standard by the MS system MAT 95S; and compounds **15** to **18** and **20** and their standards by the MS system INCOS XL.
[b]Relative abundances of the ions in the MS(CI) were recorded (details in the Experimental Procedures section).
[c]d, deuterium; c, carbon-13.
[d]The calibration factor refers to the 1:1 (w/w) mixture of the labeled and unlabeled compounds (7,8).
[e]Odorant **4** was quantified using [$^2H_3$]-2-ethenyl-3,5-dimethylpyrazine (**4a–d**) as internal standard, odorant **16** using [$^2H_{1-2}$]-3-methylbutanal (**17-d**).
[f]Sum of the relative abundances of the ions was calculated.

on-column technique at 35°C. After the start, the temperature of the oven was increased to 40°C, held for 2 min isothermally, then raised at 40°C/min to 50°C (60°C for DB-FFAP), held again for 2 min and then raised at 4°C/min (6°C/min for DB-FFAP) to 250°C (230°C for DB-FFAP) and held for 10 min. The flow rate of the carrier gas helium was 2 mL/min.

HRGC-mass spectrometry (MS) of the odorants **1** to **14**, **19,** and **21** to **25** was carried out either with an MS MAT 95S or with an ion trap detector, ITD 800 (both Finnigan, Bremen, Germany), in tandem with the above-mentioned capillaries. Mass spectra in the chemical ionization mode [MS(CI)] were generated at 170 eV with NH_3 (MAT 95S) and at 70 eV with methanol (ITD) as reagent gas. Mass chromatograms were recorded at the ions selected in Table 1. Comparison of the integrated abundance of the selected ion of the odorant to that of the abundance of the selected ion of the labeled internal standard (Table 1) provided the data needed to carry out the quantitative calibration of the method (7,9). A calibration factor was determined for each of the 15 odorants as exemplified for (*Z*)-2-nonenal in (7). The factors calculated are listed in Table 1. Mass spectra in the electron impact mode [MS(EI)] were generated at 70 eV (4).

Static headspace analysis was carried out as reported earlier (4,10) by means of the purge and trap system TCT/PTI 4001 (Chrompack, Frankfurt, Germany). The fused silica capillary RTX-5 (30 m × 0. 52 mm, 1.5 µm film thickness; Amchro, Sulzbach, Germany) was coupled with the MS system INCOS XL (Finnigan, Bremen, Germany). MS(CI) were recorded at 115 eV with methane as reagent gas. The ions selected for mass chromatography are listed in Table 1.

Synthesis. The following labeled compounds (numbering refers to Table 1) were synthesized as reported earlier: [$^2H_3$]-3-isobutyl-2-methoxypyrazine [**5-d** (11)], [$^2H_2$]-1-octen-3-one (**6-d**), [$^2H_2$]-(*Z*)-2-nonenal (**7-d**), [$^2H_2$]-(*E*)-2-nonenal (**8-d**) and [$^2H_4$]- *trans*-4,5-epoxy-(*E*)-2-decenal [**12-d** (7)], [$^2H_2$]-(*E,E*)-2,4-nonadienal [**9-d** (12)], [$^2H_4$]-(*E,Z*)-2,4-decadienal (**10-d**) and [$^2H_4$]-(*E,E*)-2,4-decadienal [**11-d** (7,13)], [$^{13}C_2$]-4-hydroxy-2,5-dimethyl-3(2H)-furanone [**13-c** (14)], [$^{13}C_2$]-3-hydroxy-4,5-dimethyl-2(5H)-furanone [**14-c** (15)], [$^2H_7$]methylpropanal (**15-d**) and [$^2H_6$]dimethyltrisulfide [**21-d** (10)]; [$^2H_2$]-3-methylbutanal [**17-d** (16)], [$^{13}C_4$]-2,3-butanedione [**18-c** (17)], [$^2H_3$]methional [**19-d** (18)], [$^2H_3$]methanethiol [**20-d** (19)], and [$^2H_{2-4}$]-δ-decalactone [**25-d** (20)].

The mixture of [$^2H_3$]-2-ethyl-3,5-dimethylpyrazine (**1-d**) and [$^2H_3$]-3-ethyl-2,5-dimethylpyrazine (**2-d**) was prepared according to the procedure by Tas and Kleipool (21), using some modifications. During 30 min a solution of 2-ethyl-3-methylpyrazine (25 mmol) in 10 mL diethyl ether was added dropwise at room temperature to an ethereal solution (50 mL) of [$^2H_3$]methyllithium (25 mmol). After stirring the red suspension for a further 1 h at 40°C, the mixture was cooled to room temperature, diluted with water (20 mL), and then extracted with diethyl ether (3 × 50 mL). After drying over anhydrous Na_2SO_4, the concentrated extract was separated by TLC on silica gel 60 using pentane/diethyl ether (7:3, vol/vol) as solvent system. The zone which moved as far as unlabeled pyrazine **1** was rechromatographed using toluene/ethyl acetate (97:3, vol/vol) as developing solvent. The zone at R_f 0.45 consisting of a mixture of pyrazines **1-d** and **2-d** was extracted with diethyl ether. The two pyrazines were separated by HRGC on capillary OV-1701: (i) Pyrazine **1-d**, retention index (RI) (22) on capillary OV-1701: 1153; MS(CI) 140 (100%, $M^+ + 1$), MS(EI): 138 (100), 139 (82), 57 (26), 111 (15), 41 (14). (ii) Pyrazine **2-d**, RI on capillary OV-1701: 1146; MS(CI): 140 (100%, $M^+ + 1$); MS(EI): 138 (100), 139 (88), 43 (39), 111 (15), 57 (11), 41 (10).

Concentrations of labeled compounds. The concentrations of **1-d**, **2-d,** and **4a–d** were determined without a correction factor with 2,3-diethyl-5-methylpyrazine (**3**) as internal standard. The concentrations of compounds **3-d**, **5-d**, **6-d**, **7-d** to **11-d**, **15-d**, **17-d**, **19-d**, **21-d,** and **25-d** were determined by HRGC with methyl octanoate as the internal standard. HRGC was performed with the apparatus, the OV-1701 and DB-FFAP thin film capillaries, and the conditions reported above. The correction factors were determined by HRGC analysis of mixtures consisting of known amounts of methyl octanoate and of unlabeled compounds **3**, **5**, **6**, **7** to **11**, **15**, **17**, **19**, **21**, and **25**. The concentrations of compounds **7-d** and **12-d** were determined with (*E*)-2-nonenal and (*E*)-2-decenal, respectively, as internal standards. The concentrations of compounds **13-c**, **14-c**, **18-c,** and **20-d** were determined as reported earlier (8,19).

Analysis of french fries. Odorants **1** to **14**, **19,** and **21** to **25** were determined in extracts of french fries and odorants **15** to **18** and **20** by static headspace analysis.

Extraction. Frozen french fries were extracted by two different procedures. Procedure A: The sample was mixed with anhydrous Na_2SO_4 at a ratio of 1:1 (w/w). The weighed portions amounted to 20 g for compounds **2**, **9** to **11**, **12**, **19,** and **22** to **25**, and to 100 g for compounds **1**, **3** to **8,** and **14**. The sample was ground in a Waring blender, and the powder obtained was transferred to a Soxhlet apparatus and soaked with 0.9 L of dichloromethane which was spiked with the corresponding labeled internal standards (Table 1). The amounts of the standards varied between 0.5- and 2-fold the concentration of the odorant to be estimated. After 15 h, the solvent volume was increased to 1.4 L, and Soxhlet extraction was then carried out for 8 h. The extract obtained was concentrated to 150 mL by distilling off the solvent on a Vigreux column (50 × 1 cm) at 51°C. Procedure B was used for the determination of compounds **12** and **21**. The sample (100 g) was mixed with anhydrous Na_2SO_4 and then ground as described above. It was suspended in cold (−40°C) dichloromethane (200 mL) which was spiked with the corresponding labeled internal standards (Table 1). The suspension, which was cooled at −40°C using solid carbon dioxide, was homogenized for 3 min with an Ultra Turrax (Janke & Kunkel, Oberstaufen, Germany). After filtration, the residue was extracted twice with dichloromethane (200 mL each). The combined filtrates were concentrated to 150 mL by distilling off the solvent on a Vigreux column (50 × 1 cm) at 51°C.

Purification of the extracts. Each extract was distilled under high vacuum (temperature 50°C, pressure 5 mPa) with the apparatus reported earlier (9,23). The condensate was treated with aqueous $NaHCO_3$ (0.5 mL/L, 3 × 100 mL). Compounds **1** to **12**, **19**, **21,** and **22** to **25** were determined in the organic layer, and **13** and **14** in the aqueous layer.

The organic layer was concentrated by distillation and microdistillation (24) to 0.5 mL and then subjected to chromatography on a column (30 × 1.6 cm) packed with silica gel G60 in pentane/diethyl ether 85:15 (vol/vol). The column was maintained at 10°C by a cooling jacket. Stepwise elution (flow rate 1.5–2 mL/min) was performed with the pentane/diethyl ether mixtures 85:15 (vol/vol, 100 mL, fraction A) and 1:1 (vol/vol, 130 mL, fraction B) as well as with diethyl ether (200 mL, fraction C). Table 2 indicates the distribution of the odorants in the fractions A to C. Each fraction was concentrated by distillation and microdistillation (24) and then analyzed by HRGC-MS. The pH of the aqueous layer was adjusted to 3 by addition of aqueous HCl (1 mol/L) and then extracted with dichloromethane (3 × 100 mL). The extract was washed with saturated aqueous NaCl (100 mL), dried over anhydrous Na_2SO_4, and concentrated to 100 µL for HRGC-MS analysis.

Static headspace analysis. Frozen french fries (2 to 6 g) were ground and then put into a vessel (volume 250 mL) which was sealed by a septum. Known amounts of the internal standards **15-d**, **17-d,** and **18-c** were injected through the septum. The standard **20-d** was liberated by alkaline hydroly-

TABLE 2
Result of Column Chromatography

Fraction	Odorants[a]
A	1-Octen-3-one (**6**), (*Z*)-2-nonenal (**7**), (*E*)-2-nonenal (**8**), (*E,E*)-2,4-nonadienal (**9**), (*E,Z*)-2,4-decadienal (**10**), (*E,E*)-2,4-decadienal (**11**), dimethyltrisulfide (**21**)
B	2,3-Diethyl-5-methylpyrazine (**3**), 2-ethenyl-3-ethyl-5-methylpyrazine (**4**), 3-isobutyl-2-methoxypyrazine (**5**), methional (**19**)
C	2-Ethyl-3,5-dimethylpyrazine (**1**), 3-ethyl-2,5-dimethylpyrazine (**2**), *trans*-4,5-epoxy-(*E*)-2-decenal (**12**), γ-octalactone (**22**), γ-nonalactone (**23**), γ-decalactone (**24**), δ-decalactone (**25**)

[a]Numbers refer to Table 1.

sis of [$^2H_3$]methylisothiouronium iodide (19) and then injected by a gas-tight syringe into the vessel. The amounts of labeled standards varied between 0.5- and 2-fold the concentration of the odorant to be estimated. The sample in the vessel was equilibrated for 60 min at 40°C. Then headspace volumes of 1 to 5 mL were analyzed for **15** to **18** and **20**.

Flavor profile analysis. Aliquots from stock solutions of the odorants in ethanol were pipetted into odorless sunflower oil (0.5 L) at 21°C for the preparation of aroma models (Table 3). After dilution with sunflower oil to 1 L, the models MPO and MCF were stirred at room temperature for 30 min. In each session the models (15 mL each) were presented in covered glass beakers (diameter, 40 mm; capacity, 45 mL) at 21 ± 1°C. The beaker was swirled and, after the cover was removed, the sample was sniffed by the panelist (nasal evaluation) and was then rinsed into the mouth (retronasal evaluation). In comparative tests the flavor profile of freshly prepared french fries was evaluated as reported in (4). The panel consisted of five experienced assessors, aged 28 to 33 years, two women and three men. The panelists were familiar with the flavor profile of french fries due to the preceding experiments (4). Furthermore, they were trained by using both the reference stimuli listed in (4) and a solution of γ-octalactone (2 mg/L) in sunflower oil smelling coconut-like. In flavor profile analyses of the french fries and their models, the intensities of the attributes were scored on a category scale of 0 (absent) to 3.0 (strong) in increments of 0.5. The results obtained in duplicates were averaged.

Comparison of models. Models were prepared as reported for MPO, each missing one or more compounds. In triangle tests the overall odor of the reduced model was compared with that of the complete model MPO. The number of assessors who were able to distinguish the two models was ascertained. Furthermore, if a difference was found, the assessor was asked to estimate its intensity using the category scale mentioned above. The results obtained in duplicates were averaged and rounded to the nearest 0.5 points.

Odor threshold values. Threshold values of odorants dissolved in sunflower oil were nasally and retronasally estimated (25).

TABLE 3
Amounts of Odorants Used for the Preparation of the Aroma Models for French Fries Prepared in Palm Oil (MPO) and in Coconut Fat (MCF)

Odorant[a]	Stock solution[b]	Aliquot of stock solution[c,d]	
		MPO	MCF
2-Ethyl-3,5-dimethylpyrazine (**1**)	1.3	33	35
3-Ethyl-2,5-dimethylpyrazine (**2**)	21.6	27	27
2,3-Diethyl-5-methylpyrazine (**3**)	4.1	10	11
2-Ethenyl-3-ethyl-5-methylpyrazine (**4**)	0.5	10	10
3-Isobutyl-2-methoxypyrazine (**5**)	0.9	10	10
1-Octen-3-one (**6**)	0.4	10	18
(*Z*)-2-Nonenal (**7**)	0.1	119	158
(*E*)-2-Nonenal (**8**)	13.8	10	17
(*E,Z*)-2,4-Decadienal (**10**)	7.4	209	61
(*E,E*)-2,4-Decadienal (**11**)	51.7	123	50
trans-4,5-Epoxy-(*E*)-2-decenal (**12**)	1.5	512	185
4-Hydroxy-2,5-dimethyl-3(2H)-furanone (**13**)	138.9	20	19
3-Hydroxy-4,5-dimethyl-2(5H)-furanone (**14**)	0.5	10	10
Methylpropanal (**15**)	591	10	10
2-Methylbutanal (**16**)	1060	10	10
3-Methylbutanal (**17**)	272	10	10
2,3-Butanedione (**18**)	30.6	10	10
Methional (**19**)	78.3	10	18
Methanethiol (**20**)	48[e]	579	579
γ-Octalactone (**22**)	276	—	20
γ-Nonalactone (**23**)	179	—	10
γ-Decalactone (**24**)	142	—	10
δ-Decalactone (**25**)	80.8	—	10

[a]The numbering of the compounds refers to Table 1.
[b]Concentration (mg/mL) of the odorant in the ethanolic stock solution.
[c]Aliquot (μL) of the stock solution used for the preparation of 1 L of the model.
[d]The amounts of **4, 5, 14–18,** and **20** which were not analyzed in coconut fat were equal in MCF to those in MPO.
[e]The mass of 48 mg was equal to 22.4 mL gaseous methanethiol (**20**) at room temperature.

RESULTS

A set of PO and CF samples was prepared and then analyzed. The concentrations of the odorants found in these samples are listed in Table 4.

PO contained relatively high levels of the *Strecker* aldehydes methylpropanal (**15**), 2- and 3-methylbutanal (**16**, **17**), (*E,Z*)- and (*E,E*)-2,4-decadienal (**10**, **11**), furanone (**13**), and methanethiol (**20**). The concentrations of these odorants in PO surpassed a level of 1 mg/kg. On the other hand, pyrazines **4** and **5**, 1-octen-3-one (**6**), furanone **14**, and dimethyltrisulfide (**21**) were only minor components with concentrations below 10 μg/kg. Only a part of the odorants quantified in PO was also determined in CF (Table 4). A comparison with the corresponding data obtained for PO indicated that the two sorts of fat had affected the formation of 1-octen-3-one (**6**), (*Z*)- and (*E*)-2-nonenal (**7**, **8**), (*E,E*)-2,4-nonadienal (**9**), (*E,Z*)- and (*E,E*)-2,4-decadienal (**10**, **11**), and *trans*-4,5-epoxy-(*E*)-2-decenal (**12**). The amounts of carbonyl compounds **6** to **9** were higher in CF than in PO and those of **10** to **12** were lower.

To confirm the compounds responsible for the coconut-like note in the flavor of CF, lactones **22** to **25** were quantified because they appeared among the coconut-smelling compounds with the highest flavor dilution (FD) factors in AEDA of CF (data not shown). The results in Table 4 indicated that γ-octalactone (**22**) was the major lactone followed by γ-nonalactone.

The odor threshold values of compounds **1** to **5**, **10**, **14** to **16**, and **22** to **24** dissolved in sunflower oil were estimated. The results listed in Table 5 indicated that the nasal and retronasal odor threshold values of these odorants differed at the most by a factor 2.3 which was found for 2-methylbutanal (**16**).

The OAV of the 21 and the 16 odorants, respectively, quantified in PO and CF were calculated on the basis of the

TABLE 4
Concentration and Odor Activity Values (OAV) of Potent Odorants of French Fries Produced in Palm Oil (PO) and Coconut Fat (CF)

	Concentration (μg/kg) in PO				Concentration (μg/kg) in CF			
Odorant	Mean[a]	S.D.[b]	*n*[c]	OAV[d]	Mean[a]	S.D.[b]	*n*[c]	OAV[d]
2-Ethyl-3,5-dimethylpyrazine (**1**)	41.9	5.9	8	19	57	7.9	4	26
3-Ethyl-2,5-dimethylpyrazine (**2**)	592	70	8	10	579	38	4	10
2,3-Diethyl-5-methylpyrazine (**3**)	41.4	5.8	7	83	43	4.4	4	86
2-Ethenyl-3-ethyl-5-methylpyrazine (**4**)[e]	5.4	0.9	7	11	n.a.			
3-Isobutyl-2-methoxypyrazine (**5**)	8.6	0.7	4	11	n.a.			
1-Octen-3-one (**6**)	3.9	0.5	7	<1	6.9	0.3	4	<1
(*Z*)-2-Nonenal (**7**)	15.7	1.8	8	3	21	0.4	4	5
(*E*)-2-Nonenal (**8**)	138	13	7	<1	237	16	4	<1
(*E,E*)-2,4-Nonadienal (**9**)	111	17	5	<1	178	27	4	<1
(*E,Z*)-2,4-Decadienal (**10**)	1533	208	4	383	449	27	4	112
(*E,E*)-2,4-Decadienal (**11**)	6340	344	4	35	4115	581	4	23
trans-4,5-Epoxy-(*E*)-2-decenal (**12**)	771	29	8	592	278	11	4	214
4-Hydroxy-2,5-dimethyl-3(2H)-furanone (**13**)	2778	458	4	111	2591	229	4	104
3-Hydroxy-4,5-dimethyl-2(5H)-furanone (**14**)	5.2	0.9	6	26	n.a.			
Methylpropanal (**15**)	5912	382	4	1739	n.a.			
2-Methylbutanal (**16**)	10599	840	4	1059	n.a.			
3-Methylbutanal (**17**)	2716	137	4	503	n.a.			
2,3-Butanedione (**18**)	306	8	4	31	n.a.			
Methional (**19**)	783	91	9	3915	1370	82	4	6850
Methanethiol (**20**)	1240	75	5	20667	n.a.			
Dimethyltrisulfide (**21**)	0.33	0.06	4	<1	n.a.			
γ-Octalactone (**22**)	n.d.				5533	404	4	46
γ-Nonalactone (**23**)	n.d.				1785	281	4	12
γ-Decalactone (**24**)	n.d.				420	155	4	4
δ-Decalactone (**25**)	n.d.				808	44	4	4

[a]Mean value corrected for runaways with the test according to Nalimov (26).
[b]S.D., standard deviation.
[c]*n*, number of freshly prepared french fry samples which were analyzed.
[d]The odor activity values were calculated by dividing the concentration by the nasal odor threshold values in oil which were obtained for **1** to **5, 10, 14** to **16,** and **22** to **24** from Table 5, for **6** to **8, 11,** and **12** from (7), for **9** from (27), for **13, 17** to **19,** and **25** from (28) and **20** and **21** from J. Kubickova (personal communication).
[e]Odorant **4** was eluted together with its isomer 3-ethenyl-2-ethyl-5-methylpyrazine. The concentration of **4** was calculated on the basis that, as found earlier (4), its portion amounted to 45%. n.a., not analyzed; n.d., not detected.

nasal odor threshold values given in Table 5 and by using corresponding data from the literature. The results are included in Table 4.

Among the odorants, those compounds having odor qualities which corresponded to the notes in the odor profile of PO (Table 6) were classified according to odor notes. It was assumed that in each class the odorant showing the highest OAV was the most important contributor to this note in the flavor profile of PO.

Of the substances **1** to **5** with an earthy odor, the higher OAV of pyrazine **3** suggested that **3** contributed the most to that character in the odor profile of PO. In the group consisting of carbonyl compounds **6** to **12** with fatty odor qualities, the aldehydes **10** to **12** showing higher OAV might stimulate the deep-fried, fatty impression in the flavor of french fries.

As the odor of methional (**19**) reminded us of boiled potatoes (4), **19** might be responsible for this note on the basis of its high OAV. Furthermore, higher OAV indicated that the *Strecker* aldehydes methylpropanal (**15**) and 2-methylbutanal (**16**) were contributors to the malty and furanone **13** to the caramel-like notes in the flavor profile of PO. Methanethiol (**20**), which has a sulfurous, cabbage-like odor, showed by far the highest OAV of all compounds. For this reason, its contribution to the flavor of PO was assumed, but the note which was caused or intensified by **20** remained unresolved. The coconut-like note in the flavor profile of CF was mainly stimulated by γ-octalactone (**22**).

In order to check whether the assumptions about the contributions of the compounds to the different notes in the flavor profiles of PO and CF were correct, a sensory study was undertaken.

At first, flavor profiles of the models MPO and MCF were nasally and retronasally compared with those of the corresponding real samples. MPO contained all of the odorants quantified in PO with exception of (*E,E*)-2,4-nonadienal (**9**) and dimethyltrisulfide (**21**), the OAV of which were lower than 1 (Table 4). This was also found for 1-octen-3-one (**6**) and (*E*)-2-nonenal (**8**), but in contrast to **9** and **21**, the OAV calculated on their retronasal odor threshold values of 0.3 and 66 μg/kg (7), respectively, was greater than 1. Therefore, **6** and **8** were included in the models.

The results in Table 6 indicate that the flavor profiles of the models were close to those of the corresponding french fries. In the case of PO and its model, the greatest intensity

TABLE 5
Nasal (N) and Retronasal (RN) Odor Threshold Values of the Odorants 1 to 5, 10, 14 to 16, and 22 to 24 Dissolved in Freshly Refined Sunflower Oil

	Mean odor threshold value	
Compound[a]	N (μg/kg)	RN (μg/kg)
2-Ethyl-3,5-dimethylpyrazine (**1**)	2.2	2.2
3-Ethyl-2,4-dimethylpyrazine (**2**)	57	79
2,3-Diethyl-5-methylpyrazine (**3**)	0.5	0.9
2-Ethenyl-3-ethyl-5-methylpyrazine (**4**)	0.5	0.9
3-Isobutyl-2-methoxypyrazine (**5**)	0.8	0.6
(*E,Z*)-2,4-Decadienal (**10**)	4	4
3-Hydroxy-4,5-dimethyl-2(5H)-furanone (**14**)	0.2	0.2
Methylpropanal (**15**)	3.4	3.4
2-Methylbutanal (**16**)	10	23
γ-Octalactone (**22**)	120	197
γ-Nonalactone (**23**)	148	219
γ-Decalactone (**24**)	320	385

[a]The numbering of the compounds refers to Table 1.

differences were found for the boiled potato-like, caramel, and malty notes in particular when the flavor profile was retronasally evaluated. Also the agreement of CF with its model was better in the nasal than in the retronasal test.

The composition of MPO was varied to identify the character impact flavor compounds of potatoes deep-fried in palm oil. In the experiments (exp.) listed in Table 7, the complete model MPO was compared with models in which one or more odorants were omitted.

Methanethiol (**20**) in exp. 1 was missed by each member of the panel. The odor impression accorded to boiled potatoes was lacking, and therefore the odor difference between the complete and the reduced model was scored at 2.5. This was in contrast to the absence of 2,3-butanedione (**18**) which was only weakly perceived by one panelist (exp. 2).

The *Strecker* aldehydes **15** to **17** played a role in the flavor (exp. 3). Their absence and that of 2,3-butanedione (**18**) led to a loss of malty, sweet odor notes in the flavor profile of MPO and to a breakthrough of a cabbage-like note which was most likely caused by methanethiol (**20**). It was surprising that the absence of methional (**19**) in exp. 5 was not perceived. This indicated that the odor of **19** was masked in the model by other odorants. Pyrazines **3** and **4** alone (exp. 6) and in combination with methional (**19**, exp. 7) did not significantly affect the flavor of MPO, as only one of five panelists detected a deviation of the odor when these three odorants were lacking in exp. 7. Absence of furanone **13** was perceived by 3 panelists (exp. 8), who agreed that the flavor intensity of the reduced model was weaker, but the caramel-like note was not affected. The characteristic flavor of french fries including the caramel-like note was still perceived when pyrazines **3** and **4** as well as furanone **13** and methional (**19**) were omitted in MPO (exp. 9), but the intensity of this flavor was even weaker than in exp. 8. When pyrazines **1** and **2** were missing (exp. 10), the roasty note was abolished and a note reminiscent of raw potatoes appeared. The absence of methoxypyrazine **5** somewhat affected the flavor (exp. 11). Surprisingly, the effect caused by the lack of pyrazines **1** and **2** (exp. 10) was reduced when methoxypyrazine **5** was also omitted (exp. 12). To explain this difference, we assume that methoxypyrazine **5** had inhibited, to a certain extent, the contribution of pyrazines **3** and **4** to the flavor which was perceived in exp. 10. In exp. 12 this inhibition was abolished by the lack of **5**.

Exp. 13 to 19 were performed to show the contribution of carbonyl compounds derived from linoleic acid to the flavor. All five judges agreed that of these compounds the mixture of (*E,Z*)- and (*E,E*)-2,4-decadienal (**10**, **11**) in exp. 16 had the greatest impact, because the deep fried, fatty odor quality was lost in the model in which these aldehydes were lacking and instead it smelled roasty, malty, and cabbage-like. Although the OAV of (*E,E*)-2,4-decadienal (**11**) was much lower than the OAV of the (*E,Z*)-isomer (**10**, Table 4), a comparison of exp. 14 and 15 (Table 7) indicated that the flavor of MPO was more affected by the absence of **11** than of **10**. Lack of epoxide **12** in exp. 13 was recognized as loss of freshness by four judges. Exp. 17 to 19 revealed that the contribution of the carbonyl compounds **6** to **8** to the flavor of MPO was very small.

TABLE 6
Flavor Profiles of the French Fries PO and CF and the Corresponding Aroma Models MPO and MCF

	Intensity[a]							
	Nasal				Retronasal			
Attribute	PO	MPO	CF	MCF	PO	MPO	CF	MCF
Earthy	1.8	1.9	1.5	1.4	1.8	1.7	1.6	1.5
Deep fried, fatty	2.7	2.5	2.3	2.2	2.4	2.6	1.6	1.8
Boiled potato	1.6	1.7	1.8	1.6	2.3	2.0	2.3	2.0
Caramel	1.5	1.3	1.3	1.4	0.8	1.2	0.9	1.2
Malty	1.6	1.4	1.3	1.3	1.6	1.2	1.4	1.1
Coconut-like	—	—	0.9	1.2	—	—	0.5	0.8

[a]The intensity of the attributes was nasally and retronasally scored on the scale 0 (absent) to 3 (strong). See Tables 3 and 4 for abbreviations.

DISCUSSION

2-Ethyl-3,5-dimethylpyrazine (**1**), 3-ethyl-2,5-dimethylpyrazine (**2**), 2,3-diethyl-5-methylpyrazine (**3**), 3-isobutyl-2-methoxypyrazine (**5**), (*E,Z*)- and (*E,E*)-2,4-decadienal (**10**, **11**), *trans*-4,5-epoxy-(*E*)-2-decenal (**12**), 4-hydroxy-2,5-dimethyl-3(2H)-furanone (**13**), methylpropanal (**15**), and 2- and 3-methylbutanal (**16**, **17**) as well as methanethiol (**20**) were identified in the present study as character impact odorants of PO. γ-Lactones with 8 to 10 carbon atoms causing the coconut-like note were additionally involved in the CF flavor.

As detailed in (4), the odorants found in PO have been earlier reported as constituents of french fries and/or potato chips with the exception of compounds **5**, **12,** and **13**. Potato chips were included in the discussion, as their flavor is very similar to that of french fries.

The results of our study confirmed some suggestions re-

TABLE 7
Odor of the Model MPO Affected by the Absence of One or More Components[a]

Experiment	Odorant omitted in the model	Number of five panelists detecting an odor difference	Intensity of the odor difference[b]
1	Methanethiol (**20**)	5	2.5
2	2,3-Butanedione (**18**)	1	0.5
3	Methylpropanal (**15**), 2-methylbutanal (**16**), 3-methylbutanal (**17**)	4	0.5
4	2,3-Butanedione (**18**), methylpropanal (**15**), 2-methylbutanal (**16**), 3-methylbutanal (**17**)	5	1.0
5	Methional (**19**)	0	
6	2,3-Diethyl-5-methylpyrazine (**3**), 2-ethenyl-3-ethyl-5-methylpyrazine (**4**)	0	
7	2,3-Diethyl-5-methylpyrazine (**3**), 2-ethenyl-3-ethyl-5-methylpyrazine (**4**), methional (**19**)	1	0.5
8	4-Hydroxy-2,5-dimethyl-3(2H)-furanone (**13**)	3	1.0
9	2,3-Diethyl-5-methylpyrazine (**3**), 2-ethenyl-3-ethyl-5-methylpyrazine (**4**), 4-hydroxy-2,5-dimethyl-3(2H)-furanone (**13**), methional (**19**)	4	1.5
10	2-Ethyl-3,5-dimethylpyrazine (**1**), 3-ethyl-2,5-dimethylpyrazine (**2**)	4	1.5
11	3-Isobutyl-2-methoxypyrazine (**5**)	3	1.0
12	2-Ethyl-3,5-dimethylpyrazine (**1**), 3-ethyl-2,5-dimethylpyrazine (**2**), 3-isobutyl-2-methoxypyrazine (**5**)	3	1.0
13	*trans*-4,5-Epoxy-(*E*)-2-decenal (**12**)	4	0.5
14	(*E,Z*)-2,4-Decadienal (**10**)	4	0.5
15	(*E,E*)-2,4-Decadienal (**11**)	4	1.5
16	(*E,Z*)-2,4-Decadienal (**10**), (E,*E*)-2,4-decadienal (**11**)	5	2.5
17	(*Z*)-2-Nonenal (**8**)	0	
18	1-Octen-3-one (**6**), (*E*)-2-nonenal (**8**)	1	0.5
19	1-Octen-3-one (**6**), (*Z*)-2-nonenal (**7**), (*E*)-2-nonenal (**8**)	1	0.5

[a]Models lacking in one or more components were singly compared to the model MPO containing the complete set of 19 odorants (*cf.* Comparison of models).
[b]Rating scale: 0 (no deviation) to 3 (strong). See Table 3 for abbreviation.

ported in the literature about important odorants of potato products. The contribution of (*E,Z*)- and (*E,E*)-2,4-decadienal (**10**, **11**) to the flavor of potato chips was proposed by Mookherjee *et al*. (29) due to the deep-fried odor quality of **10** and **11** (30). This proposal was further supported by the observation that a stale off-flavor, which was formed during the storage of potato chips, was accompanied by a decrease in 2,4-decadienal (29). As 2,4-decadienal is formed by autoxidation of linoleic acid (31), the authors (29) concluded that oils used for the frying process should contain a certain amount of this unsaturated fatty acid.

Our results indicated clearly that the dienals **10** and **11** cause the deep-fried note in the odor profile of french fries. As the linoleic acid content of the palm oil sample [10.5%, (1)] was higher than that of the coconut fat (1.9%), the higher concentrations of decadienal isomers **10** and **11** and, in consequence, the more intense deep-fried note in the flavor of PO compared to that of CF can be explained. However, the concentration differences of the dienals **10** and **11** were relatively small in the two samples of french fries with regard to the difference in the linoleic acid contents. However, a clear relation between the concentrations of dienals **10** to **11** and the linoleic content was not found for puff pastries prepared with butter and margarine (32).

3-Ethyl-2,5-dimethylpyrazine (**2**) was suggested as the major contributor to the flavor of potato chips (2) and baked potatoes (33). Pareles and Chang (34) confirmed the importance of pyrazine **2** for the latter and added that this pyrazine in combination with 2,3-diethyl-5-methylpyrazine (**3**) and 2-isobutyl-3-methylpyrazine had a very characteristic baked potato aroma. Our study shows that pyrazines **2** and **3** are involved in the flavor of processed potatoes but in combination with 3-isobutyl-2-methoxypyrazine (**5**) smell like raw potatoes (4).

On the basis of a high OAV, Guadagni *et al*. (2) concluded that the most important odorant of potato chips was methional (**19**), followed by phenylacetaldehyde, 3-methylbutanal (**17**), and pyrazine **2**. Also in odor similarity tests, methional (**19**) was rated the most similar to chip aroma (2). However, these authors had found in a preceding study (35) that additions of 3 to 10 mg/kg of **19** to dehydrated mashed potato products failed to significantly increase potato flavor.

We calculated also high OAV for methional (**19**) in PO and CF. Nevertheless, the absence of this aldehyde in the MPO imitating the flavor of french fries was not perceived by the sensory panel. The result suggests that **19** does not contribute to the flavor of french fries and most likely not to that of potato chips. This conclusion is not in contrast to a patent of Chang and Reddy (36,37) for improving potato-chip flavor by frying the chips in vegetable oil containing methionine, since the products of the degradation of methionine are not only methional (**19**) but also methanethiol (**20**) (38) which, according to our results, contributes with great impact to the flavor of french fries.

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