

Sensory Study on the Character Impact Odorants of Roasted Arabica Coffee

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The potent odorants were quantified in a sample of roasted Arabica coffee. On the basis of the results, 27 odorants were dissolved in an oil/water mixture. The flavor profile of the model obtained was very close to that of the real sample. In duo and triangle tests, the model was compared with models missing one or more odorants. These experiments indicated that 2-furfurylthiol, 4-vinylguaiacol, several alkyl pyrazines, furanones, acetaldehyde, propanal, methylpropanal, and 2- and 3-methylbutanal had the greatest impact on the coffee flavor.

Keywords: *Aroma model; coffee; flavor; flavor profile; key odorants; omission experiments*

INTRODUCTION

Analytical studies on the substances causing the pleasant aroma of roasted coffee have been carried out for more than 100 years. In numerous papers reviewed by Flament (1989, 1991) as well as by Nijssen et al. (1996), more than 800 volatile compounds with a wide variety of functional groups have been reported.

To differentiate the volatiles contributing to the aroma of roasted coffee from those which did not, aroma extract dilution analysis (AEDA) (Holscher et al., 1990; Blank et al., 1992a; Czerny et al., 1996) and gas chromatography–olfactometry of headspace samples (GCOH) (Holscher and Steinhart, 1992; Semmelroch and Grosch, 1995) were carried out. Altogether, 28 odorants (Table 1) showing a high odor potency were identified.

In AEDA and GCOH, the odor impact of the volatiles is separately evaluated. Interactions of the odorants, which in most cases are characterized by inhibition and suppression (Acree, 1993), are abolished. Therefore, the question of which compound among the potent odorants actually contributed to the flavor remains open.

To answer this question, the following approach was adapted that had been developed in studies on the character impact odorants of dill herb (Blank et al., 1992b), sour cream butter (Schieberle et al., 1993), stewed beef juice (Guth and Grosch, 1994), Swiss cheese (Preininger et al., 1996), strawberry juice (Schieberle and Hofmann, 1997), Camembert cheese (Kubickova and Grosch, 1998), olive oil (Reiners and Grosch, 1998), and french fries (Wagner and Grosch, 1998).

The potent odorants were quantified, and on the basis of the results, a synthetic blend of the odorants (aroma model) duplicating the odor profile of the food sample was prepared. Changes in the overall flavor of the aroma model were evaluated by a sensory panel after omission of one or more odorants. In the present study, this methodology was applied on a sample of medium-roasted Arabica coffee to reveal which of the odorants listed in Table 1 actually contributed to the flavor.

MATERIALS AND METHODS

Materials. Coffee beans (*Coffea arabica*) that originated in Colombia were roasted with a Neotec RFBS fluidized bed roaster, packed in 300 g portions, and stored at -35°C . The roast degree of the beans was characterized by a color value of 12.2 (Color Tester LK 100, Dr. Lange, Berlin, Germany). At the beginning of the analytical procedure, the beans were frozen in liquid nitrogen, ground, and sieved (diameter of the pores: 2 mm) with an ultracentrifugation mill (type ZM1; Retsch, Haan, Germany). Particle size (μm) of the ground material: <200 (16–18%), 200–500 (59–60%), 500–800 (17–18%), and >800 (6–7%).

Sunflower oil (Thomy, Karlsruhe, Germany) was purchased from a local market, and cellulose (powder from spruce) was obtained from Fluka (Neu-Ulm, Germany). For purification, the cellulose (400 g) was filled into a column (100×5 cm) and rinsed with methanol (4 L). The cellulose was then spread on glass dishes, and the solvent was removed in a stream of air within 24 h.

Chemicals. Pure samples of the odorants **1–7**, **10**, **12–16**, **18–25**, and **28** (Table 1) were obtained commercially from the sources reported earlier (Semmelroch and Grosch, 1995, 1996; Semmelroch et al., 1995; Kerscher and Grosch, 1998). Odorant **22** was purified according to Kerscher and Grosch (1998). Odorant **17** was a gift of Haarmann & Reimer (Holzminden, Germany). The following odorants were synthesized according to the literature cited: **8** (Cerny and Grosch, 1993), **9**, **11** (Czerny et al., 1996), and **27** (Semmelroch and Grosch, 1996).

Ethyl 3,3-dimethylacrylate, *n*-butyl thioglycolate, and sodium hydrogen sulfide were purchased from Aldrich (Steinheim, Germany); formic acid from Merck (Darmstadt, Germany); and *p*-toluenesulfonic acid monohydrate from Sigma (Munich, Germany).

Synthesis of 3-Mercapto-3-methylbutylformate (26). Ester **26** was synthesized by addition of hydrogen sulfide to ethyl 3,3-dimethylacrylate, reduction with lithium aluminum hydride, and esterification with formic acid.

Ethyl 3,3-dimethylacrylate (25 mmol, 3.2 g) dissolved in ethanol (5 mL) was added to a solution of sodium hydrogen sulfide monohydrate (100 mmol, 5.6 g) in water (5 mL). The mixture was heated in a closed vessel for 30 h at 80°C , acidified with sulfuric acid to pH 2, and extracted with diethyl ether (4×75 mL). The combined organic layers were washed with water, acidified to pH 4, and dried over anhydrous sodium sulfate. The purity and yield of the addition product ethyl 3-mercapto-3-methylbutyrate was determined by GC–MS (yield 3.2 g, 79%).

The following reduction of ethyl 3-mercapto-3-methylbu-

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Table 1. Concentrations of the Odorants in the Coffee Sample and Recipe for the Aroma Model

compound	concn ^a	stock solution ^b	volume ^c
acetaldehyde (1)	139		70 ^d
propanal (2)	17.4	70	100
methylpropanal (3)	32.3	130	100
2-methylbutanal (4)	20.7	85	100
3-methylbutanal (5)	18.6	75	100
2,3-butanedione (6)	48.4	200	100
2,3-pentanedione (7)	34.0	140	100
2-ethyl-3,5-dimethylpyrazine (8)	0.249	1.05	100
2-ethenyl-3,5-dimethylpyrazine (9)	0.052	0.22	100
2,3-diethyl-5-methylpyrazine (10)	0.073	0.30	100
2-ethenyl-3-ethyl-5-methylpyrazine (11)	0.018		^e
3-isobutyl-2-methoxypyrazine (12)	0.059	0.25	100
4-hydroxy-2,5-dimethyl-3(2 <i>H</i>)-furanone (13)	112	225	200
2-ethyl-4-hydroxy-5-methyl-3(2 <i>H</i>)-furanone (14)	16.8	67	100
3-hydroxy-4,5-dimethyl-2(5 <i>H</i>)-furanone (15)	1.36	2.73	200
5-ethyl-3-hydroxy-4-methyl-2(5 <i>H</i>)-furanone (16)	0.104	0.43	100
(<i>E</i>)- β -damascenone (17)	0.222	0.70	130
guaiaicol (18)	3.04	6.20	200
4-ethylguaiaicol (19)	1.42	2.85	200
4-vinylguaiaicol (20)	55.2	220	100
vanillin (21)	3.41	7.0	200
2-methyl-3-furanthiol (22)	0.068	0.07	400
dimethyl trisulfide (23)	0.028	0.13	100
2-furfurylthiol (24)	1.68	3.40	200
methional (25)	0.228	0.91	100
3-mercapto-3-methylbutylformate (26)	0.077	0.75	40
3-methyl-2-buten-1-thiol (27)	0.0086	0.07	50
methanethiol (28)	4.7		0.88 ^d

^a Values in milligrams per kilogram. Data are means of at least two assays that did not vary more than 15%. ^b The odorants (mg/mL) were dissolved in ethanol with exception of **8**, **9** (diethyl ether), **22** (pentane), **26**, and **27** (dichloromethane). ^c Volume (μ L) of the stock solution used for the preparation of the models (cf. Materials and Methods). ^d Volume (**1** in μ L liquid, **28** in mL gas) added to water, oil, or cellulose. ^e The concentration of pyrazine **8** was increased by the amount found for pyrazine **11**.

tyrate with lithium aluminum hydride to 3-mercapto-3-methyl-1-butanol was performed according to Masanetz et al. (1995). The yield of the alcohol was 2.0 g (85%, impurity: 3-methyl-2-buten-1-ol).

Formylation of 3-mercapto-3-methyl-1-butanol to 3-mercapto-3-methylbutylformate was not carried out with formic anhydride (Masanetz et al., 1995) because of the poor yield of this reaction. Instead, according to Engel (personal communication, 1997), 3-mercapto-3-methyl-1-butanol (1.2 g, 10 mmol) was dissolved in isopentane (50 mL), formic acid (2.8 g, 60 mmol), and some crystals of *p*-toluenesulfonic acid were added. The mixture was then refluxed for 10 h at 32 °C in a water separator until 1.4 mL of water were separated from the organic layer. Subsequently, the reaction mixture was washed with sodium hydrogen carbonate (0.5 mol/L, 10 mL) to remove the excess of formic acid and *p*-toluenesulfonic acid. The organic layer was separated, dried over anhydrous sodium sulfate, and diluted with pentane. MS and NMR data of **26** agreed with the corresponding data published by Masanetz et al. (1995): yield 1.46 g, 100%.

Quantification. The odorants were quantified in the coffee sample by stable isotope dilution assays: **1–5** and **23** (Masanetz and Grosch, 1998); **6**, **7**, **12**, **27**, and **28** (Sammelroch and Grosch, 1996); **8**, **10**, **13–21**, and **24–26** (Sammelroch et al., 1995); and **22** (Kerscher and Grosch, 1998). Some modifications of the above-cited assays and of the method used for pyrazines **9** and **11** were reported elsewhere (Mayer et al., submitted).

Concentrations of the Odorants in the Synthetic Mixtures. Pure samples of odorants **1** and **28** were directly used for preparing the model (cf. Table 1). The amounts of the odorants **2–7**, **10**, **12–21**, and **23–25** were determined by weight. The compounds were then dissolved in ethanol to prepare the stock solutions listed in Table 1. The concentrations of **8**, **9**, **22**, **26**, and **27** were determined by high-resolution gas chromatography (HRGC) using the fused silica capillaries and the conditions reported by Semmelroch et al. (1995), Semmelroch and Grosch (1996), and Kerscher and Grosch (1998). The compounds given in parentheses were used as internal standards without correction factors: **8**, **9** (2,3-diethyl-

5-methylpyrazine), **22** (2-furfurylthiol), **26** (*n*-butyl thioglycolate), and **27** (3-methyl-2-buten-1-ol).

Sensory Analyses. Sensory evaluations were performed in an isolated sensory panel room as described by Guth and Grosch (1994). The test panel consisted of 10 experienced assessors, six males and four females, aged 26–35 years. The samples (coffee 1 g; cellulose model 1 g; oil, water, and oil–water mixture 20 g) were presented in covered glass beakers (diameter, 40 mm; capacity, 45 mL) at 21 \pm 1 °C. The glass cover was removed, and the sample was sniffed by the panelist. The oil–water mixture was vigorously shaken for 15 s before removal of the lid. All samples were prepared shortly before the sensory analysis.

Models. With the exception of 2-ethenyl-3-ethyl-5-methylpyrazine (**11**), stock solutions were prepared for 27 of the 28 odorants (Table 1). To compensate for pyrazine **11**, the concentration of pyrazine **8** was correspondingly increased in the models. This is admissible as both the odor quality and the odor threshold of the pyrazines **8** and **11** are almost identical in air (Czerny et al., 1996). The volumes of the stock solutions detailed in Table 1 were added to the following bases: 400 g of water (model with water as base); 400 g of sunflower oil (oil); 20 g of sunflower oil, an aliquot (1 mL) of which was then pipetted together with water (19 g) into a glass beaker (oil–water mixture); and 20 g of cellulose of which, after shaking, an aliquot (1 g) was mixed with 19 g of fresh cellulose (cellulose).

Flavor Profile Method. The procedure followed the comments of Jellinek (1985). In the first sessions, the panel was acquainted with the flavor of the coffee sample. Then the assessors generated the following attributes to describe the flavor profile of roasted coffee: “sweetish/caramel”, “earthy”, “roasty/sulfury”, and “smoky”. In further sessions, the intensities of these flavor attributes were scored for the coffee sample and for the corresponding models using a category scale of 0, 0.5, 1.0, ..., 3.0 (Table 2). After an outlier test according to Nalimov (Kaiser and Gottschalk, 1972), the results were expressed as means \pm standard deviations.

Omission Experiments. In the model for the coffee sample, one or more components were omitted (Table 3). In

Table 2. Flavor Profiles of the Coffee Sample and Four Aroma Models Differing in the Base^a

attribute	coffee sample	aroma model using the base			
		cellulose	oil	water	oil/water ^b
sweetish/caramel	1.0 (0.0)	1.6 (0.5) ^c	2.1 (0.5) ^c	1.0 (0.8)	1.4 (0.9)
earthy	1.6 (0.5)	1.3 (0.5)	1.1 (0.8)	2.1 (0.6)	1.3 (0.5)
roasty/sulfurous	2.3 (0.5)	1.7 (0.5) ^c	1.8 (0.7)	1.6 (0.5) ^c	2.1 (0.2)
smoky	1.7 (0.5)	1.2 (0.3) ^c	1.3 (0.5)	2.0 (0.9)	1.4 (0.9)
similarity ^d		1.8 (0.3)	1.6 (0.6)	1.0 (0.8)	2.3 (0.3)

^a The intensity of the attributes was scored on a scale of 0 (absent) to 3 (strong). The results obtained by 10 panelists were averaged; the standard deviations are given in parentheses. ^b Mixture of sunflower oil–water (1:20, v/v). ^c Significant difference ($p < 0.05$) to the corresponding attribute in the flavor profile of the coffee sample. ^d Similarity rating scale: 0 (no similarity) to 3 (identical with the coffee sample).

Table 3. Flavor of the Model (Base: Oil/Water) for the Medium Roasted Arabica Coffee Sample As Affected by the Absence of Compounds

exp no.	compound(s) omitted ^a	number ^{b,c}	
		duo test	triangle test
1	acetaldehyde (1), propanal (2), methylpropanal (3), 2- and 3-methylbutanal (4, 5)	8	17 ^d
2	acetaldehyde (1), propanal (2)	np ^e	12 ^d
3	2,3-butanedione (6), 2,3-pentanedione (7)	7	6
4	2-ethyl-3,5-dimethylpyrazine (8), 2-ethenyl-3,5-dimethyl-pyrazine (9), 2,3-diethyl-5-methylpyrazine (10), 3-isobutyl-2-methoxypyrazine (12)	9 ^d	13 ^d
5	pyrazines 8–10 as in exp 3	np	12 ^d
6	4-hydroxy-2,5-dimethyl-3(2H)-furanone (13), 2-ethyl-4-hydroxy-5-methyl-3(2H)-furanone (14)	6	8
7	4-hydroxy-4,5-dimethyl-2(5H)-furanone (15), 5-ethyl-3-hydroxy-4-methyl-2(5H)-furanone (16)	6	9
8	furanones 13–16	np	11 ^d
9	β -damascenone (17)	4	6
10	diones 6 and 7, furanones 13–16, β -damascenone (17)	np	17 ^d
11	guaiaicol (18), 4-ethylguaiaicol (19), 4-vinylguaiaicol (20), vanillin (21)	10 ^d	13 ^d
12	guaiaicol (18)	10 ^d	10
13	4-vinylguaiaicol (20)	10 ^d	11 ^d
14	vanillin (21)	6	np
15	2-methyl-3-furanthiol (22), dimethyl trisulfide (23), methional (25), 3-mercapto-3-methylbutylformate (26), 3-methyl-2-buten-1-thiol (27), methanethiol (28)	8	10
16	2-furfurylthiol (24)	9 ^d	15 ^d
17	2-methyl-3-furanthiol (22)	8	10
18	methional (25)	8	np
19	3-mercapto-3-methylbutylformate (26)	7	10
20	3-methyl-2-buten-1-thiol (27)	6	10

^a Numbering of the compounds refers to Table 1. ^b Number of correct answers. ^c Maximum number of correct answers in the duo test 10 and in the triangle test 20. ^d Significant flavor difference ($p < 0.05$) between the complete and the reduced model (Jellinek, 1985). ^e np, test not performed.

the first set of experiments, 10 assessors compared each reduced model with the complete model for flavor differences. In this duo test, the sample containing the complete model was marked. After the duo tests, triangle tests were performed with freshly prepared samples. In a random order, two complete models and one reduced model were presented in each test. The assessors had to find out by sniffing the sample what was different in the overall flavor. On the following day, this test series was repeated with fresh samples. The number of correct answers in each triangle test (maximum 20) was summed up, and the significance of the result ($p < 0.05$) was evaluated according to Jellinek (1985). The confidence limit of 95% was reached when at least 9 answers out of 10 (duo test) or 11 out of 20 answers (triangle test) were correct.

RESULTS AND DISCUSSION

The concentrations of the 28 odorants that were found in the coffee sample are provided in Table 1. A number of these odorants have been quantified earlier in a sample of medium-roasted Arabica coffee that also originated from Colombia (Semmelroch et al. 1995; Semmelroch and Grosch, 1996). The amounts of odorants 6–8, 10, 12–15, 17–20, 24, 25, and 27 agreed very well in the two coffee samples as they differed not more than 25%. However, the sample analyzed in the present study was essentially lower in furanone 16 (35%), phenolic compound 21 (30%), and thiol 26 (40%).

The sensory experiments were started with the search for a base for the aroma model. To this purpose, 27 odorants were adsorbed on cellulose, dissolved in sunflower oil, in water, and in a sunflower oil/water mixture. The flavor profiles of these models were compared with that of the coffee sample. According to the results in Table 2, the flavor profile of the model containing the odorants dissolved in the oil/water mixture was the closest to the original. In particular, the very characteristic roasty/sulfurous note of coffee was as intense as in the real coffee sample. This was in contrast to the models with cellulose or water as base in which the intensity of this note was significantly lower. On the other hand, the sweetish/caramel impression was much too intense when cellulose or sunflower oil was the base. Addition of an emulsifier (e.g., 3-*sn*-phosphatidylcholine) to the oil/water mixture impaired the flavor profile of the model due to its odor (data not shown). These results prompted us to use the oil/water mixture without an emulsifier as base for the aroma models that were compared in the following experiments.

The results of the omission experiments are summarized in Table 3. The first experiment (exp) indicates that the model in which acetaldehyde (1), propanal (2), and the Strecker aldehydes 3–5 were lacking was sensorially discriminated by the panel from the complete model. It was concurrently established that the reduced model smelled less malty. Experiment 2 without acetaldehyde (1) and propanal (2) in the reduced aroma model confirmed this valuation. Due to the presence of the Strecker aldehydes 3–5, the malty odor quality was perceptible. However, as the overall odor of the reduced model differed significantly from that of the complete model, acetaldehyde (1) and propanal (2) also contributed to the coffee flavor. In experiment 3, the model without diones 6 and 7 was not clearly differentiated from the complete model. This result indicated that the two diones did not play a significant role in the coffee flavor.

The omission of the pyrazines in experiments 4 and 5 affected the flavor, but the panel was not able to

recognize which note had been changed in the flavor profile; only that the intensity of the overall flavor had somewhat decreased. Absence of furanones **13** and **14** in experiment 6 as well as furanones **15** and **16** in experiment 7 was not recognized by a significant number of panel members. However, when the four furanones **13–16** were lacking (experiment 8), the odor of the model was significantly different. The sweetish/caramel-like note was weaker, and a cooked potato-like odor note that was most likely generated by methional (**25**) was perceived by the panel members. As shown in experiment 9, β -damascenone (**17**) did not belong to the key odorants of the coffee flavor. Lack of the diones **6** and **7**, the furanones **13–16**, and β -damascenone (**17**) resulted in a significant flavor difference between the complete and the reduced aroma model (experiment 10). As expected on the basis of experiment 8, the sweetish/caramel-like note of the reduced model was weaker than that of the complete imitate and a cooked potato-like note was to perceive. A comparison of the results obtained in experiments 6–10 suggested that at least two of the four furanones are necessary to maintain the sweetish, caramel-like note in the odor profile of the model and to mask the cooked potato-like note.

The absence of the four phenolic odorants **18–21** were clearly recognized (experiment 11), and some panelists noticed a decrease in the intensity of the smoky flavor note. Triangle tests in which either guaiacol (**18**) or 4-vinylguaiacol (**20**) were omitted (experiments 12 and 13) showed that the former odorant missed the state of being a significant component ($p \leq 0.05$) of the complete model by one answer whereas the latter reached this state. The panel found that the sulfurous odor note was too strong when **20** was lacking. Vanillin (**21**) did not play a role in the coffee flavor, since its absence in the model was not clearly perceived in the duo test (experiment 14).

A comparison of experiments 15 and 16 indicated that 2-furfurylthiol (**24**) was the outstanding odorant in the group of sulfur compounds. When **24** was lacking (experiment 16), the intensity of the sulfurous/roasty note was lowered. In contrast, the remaining six sulfur compounds were only missed by a number of panelists that was too small to reach the 95% confidence limit (experiment 15). The important role established here for 2-furfurylthiol (**24**) confirmed the assumption of Reichstein and Staudinger (1955) that **24** is a key component of the coffee flavor. The authors had based their assumption on the observation that the odor of a highly diluted solution of **24** is reminiscent of coffee.

In experiments 17–20, the sulfur compounds **22** and **25–27** were individually omitted in the flavor model. As expected from the result of experiment 15, the flavor difference caused by the lack of each of these odorants could be recognized by several assessors, but the number of 10 was somewhat smaller than the confidence limit of 95%. However, one or the other sulfur compound might exceed this confidence limit in those coffee samples in which it occurs at higher concentrations than in the coffee sample that was here used as a reference.

CONCLUSION

The results indicate that the characteristic notes in the flavor profiles of a sample of medium-roasted Arabica coffee could be imitated by mixing the potent odorants that were identified by instrumental analyses. The flavor profile of coffee is mainly caused by 2-fur-

furylthiol, 4-vinylguaiacol, several alkyl pyrazines, furanones, acetaldehyde, propanal, and the "malty" smelling Strecker aldehydes. In contrast to 2-furfurylthiol, the other sulfur compounds (e.g., 3-methyl-2-buten-1-thiol, 3-mercapto-3-methylbutylformate) only have a limited influence on the coffee flavor.

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