

Key odourants of pressure-cooked hen meat

P. Farkaš,^{a*} J. Sádecká,^a M. Kováč,^a B. Siegmund,^b E. Leitner^b
& W. Pfannhauser^b

^aFood Research Institute, Priemysel'ná 4, P.O.Box 25, SK-820 06 Bratislava, Slovakia

^bInstitute for Biochemistry and Food Chemistry, University of Technology Graz, Petersgasse 12/II, A-8010 Graz, Austria

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The volatile aroma compounds of pressure-cooked hen meat isolated by simultaneous distillation-extraction and extraction-high vacuum distillation were investigated by GC/MS and GC-olfactometry. We found out that 2-furfuryl thiol, 3-(methylthio)propanal, 4-hydroxy-2,5-dimethyl-3(2H)-furanone, (E,E)-2,4-decadienal, 2-methyl-3-furanthiol and 2-ethyl-3,5-dimethyl pyrazine are the most potent odourants of the pressure-cooked hen meat. We assume that aromatic compounds such as 2,4,6-trimethyltetrahydro-1,3,5-thiadiazine, 3,5-dimethyl-1,2,4-trithiolane, 5,6-dihydro-2,4,6-trimethyl-4H-1,3,5-dithiazine and some unknown compounds, which could only be found in extracts obtained by simultaneous distillation-extraction, are artefacts formed during this isolation process.

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INTRODUCTION

The meat of spent hen layers is used in food industry as a raw material for the production of dehydrated meat as an important constituent of convenient soups formulations. Hen meat is usually worked up by cooking in the pressure cookers. Higher temperature in pressure cooker shortens the cooking time and promotes the formation of desired flavour compounds generated through lipids oxidation process, Maillard reactions as well as interactions between these reaction products (Whitfield, 1992). The flavour of cooked hen meat or broth is very similar to that of cooked chicken. However, hen broth is described as more intense and flavourful.

A lot of effort has been devoted to the research of cooked chicken meat and broth flavour which resulted in the identification of hundreds of volatile compounds (Pippen & Nonaka, 1960; Minor *et al.*, 1965; Nonaka *et al.*, 1967; Hobson-Frohock, 1970; Harkes & Begemann, 1974; Horvath, 1976; Rothe *et al.*, 1981; Schroll *et al.*, 1988; Schliemann *et al.*, 1988; Gasser & Grosch, 1989; Ramaratham *et al.*, 1991, 1993). Gasser and Grosch (1989) indicated primary odourants of chicken broth using aroma extract dilution analysis (AEDA) (Ullrich & Grosch, 1987).

In all of the quoted works, the chicken meat was cooked in hot water or boiled at atmospheric pressure. To our knowledge, no work, published in the available literature, was devoted to the study of volatile compounds of pressure-cooked chicken or hen meat.

Therefore, the aim of this work was the identification of characteristic aroma compounds of pressure-cooked hen meat by means of GC/MS and GC-olfactometry (Ullrich & Grosch, 1987).

MATERIALS AND METHODS

Chemicals

Diethyl ether (Microchem, Bratislava, Slovakia) was released from peroxides and rectified using a 120 cm × 2 cm Vigreux column. The pure compounds (Table 1): 7, 8, 10, 15, 26 were obtained from Bedoukian Research Incorp., USA, 17, 19 purchased from Aldrich and 12 was synthesised from furfural and thiourea (Vogel, 1948). Compounds 9, 16 were prepared as qualitative mixed reference material by model reactions of D-Ribose, D-Glucose and L-Cystein (120°C, 60 min, pH = 5.0 and 5.8), 20, 22, 23 and 24 were prepared by reaction of acetaldehyde, ammonia and hydrogen sulphide (Boelens *et al.*, 1974).

*To whom correspondence should be addressed.
Tel: (+00421 7) 526 46 22; Fax: (+00421 7) 526 14 17.

Table 1. Aroma active compounds of pressure-cooked hen meat

No.	Compound	RI _p		FD Factor		Aroma character
		Ultra 1	DB-Wax	SDE	EVD	
1	unknown ^d	< 500	—	256	—	sulfury
2	unknown ^d	569	—	—	8	rotten, pungent
3	unknown ^d	590	—	8	—	garlic
4	unknown ^d	598	—	—	4	onion
5	unknown ^d	711	—	64	—	sulfury
6	unknown ^d	753	—	128	—	garlic
7	hexanal ^a	774	1090	8	—	green, grass
8	butyric acid ^a	780	1645	—	8	butyric acid
9	2-methyl-3-furanthiol ^b	844	1312	128	32	meaty, sweet
10	3-(methylthio) propanal ^a	864	1465	64	128	cooked potato
11	unknown ^d	867	—	—	8	gasolene
12	2-furfuryl thiol ^a	879	1447	256	256	roasty, coffee
13	unknown ^d	891	—	8	16	musty, mousy
14	unknown ^d	952	—	—	4	chickeny, caramel
15	1-octen-3-one ^a	955	1312	8	8	mushroom
16	4-hydroxy-5-methyl-3(2H)-furanone ^a	1007	2143	—	16	caramel, fried chicken
17	4-hydroxy-2,5-dimethyl-3(2H)-furanone ^a	1030	2057	—	128	caramel, sweet
18	unknown ^d	1056	—	—	16	sweet – pleasant
19	2-ethyl-3,5-dimethyl pyrazine ^b	1058	1643	—	32	nutty, roasty
20	2,4,6-trimethyltetrahydro-1,3,5-thiadiazine ^c	1060	2152	8	—	popcorn, burnt
21	unknown ^d	1067	—	—	4	curry
22	3,5-dimethyl-1,2,4-trithiolane ^a	1097	1617	64	—	putrid
23	5,6-dihydro-2,4,6-trimethyl-4H-1,3,5-dithiazine, A ^a	1169	1776	8	—	roasty, musty
24	5,6-dihydro-2,4,6-trimethyl-4H-1,3,5-dithiazine, B ^a	1200	1812	128	—	putrid
25	unknown ^d	1211	—	64	—	sweet, meaty
26	(E,E)-2,4-decadienal ^a	1286	1827	64	64	fatty, chickeny
27	unknown ^d	1290	—	64	—	bread crust, coffee

Compounds identified on the basis of the following criteria:

^aMS(EI), RI, reference compound, odour quality,

^bRI, reference compound, odour quality (MS too weak),

^cMS(EI)(published), odour quality,

^dcompound detected only by GC olfactometry.

Cooked hen meat

Frozen hen carcasses of an average weight of 2.5 kg were obtained from a local poultry slaughtering company and stored at -18°C in polyethylene bags until use. Before cooking, the carcasses were carefully (40 min) thawed in a microwave oven, set to the defrost regime, and cut into four pieces. The meat, together with skin and bones, was cooked at 119°C for 60 min in a pressure cooker. A commercial 7-litre pressure cooker (Marcato, Italy) was adapted for our purposes. The lid of the pot was equipped with a case for the glass thermometer, pressure gauge and pressure controller allowing the cooking temperature to be set in the range of $105\text{--}119^{\circ}\text{C}$. The meat was placed in the pot onto an aluminium frame and cooked in the steam phase of the boiling distilled water (1500 ml). After cooling to room temperature, the meat was deboned, homogenised in a commercial food processor (Masterchef 750, Moulinex,

France) and used immediately for the isolation of the volatiles.

Simultaneous distillation–extraction (SDE)

600 g of homogenised cooked meat and 800 ml of redistilled water was placed into a 2-litre round-bottom, two-neck distillation flask. The flask, equipped with mechanical stirrer, was connected to a *Likens-Nickerson* SDE apparatus (Likens & Nickerson, 1964) and the volatiles were extracted continuously with 15 ml of diethyl ether for 2 h. The extract was dried with anhydrous sodium sulphate and concentrated to 0.2 ml using a 20 cm \times 1 cm Vigreux column.

Extraction–high vacuum distillation (EVD)

300 g of homogenised cooked meat was placed into a Soxhlet apparatus and extracted with 300 ml of purified

diethyl ether for 4 h. The ethereal extract was dried with anhydrous sodium sulphate and concentrated to 50 ml using a 40 cm × 1 cm Vigreux column at 45°C. Volatile and non-volatile constituents of the extract were separated by high vacuum distillation (Guth & Grosch, 1989) at 3 Pa and 23°C for 3 h and at 50°C for another 3 h. After drying with anhydrous sodium sulphate, the distillate was concentrated on a 20 cm × 1 cm Vigreux column to 0.2 ml.

Gas chromatography–Mass spectrometry (GC/MS)

GC/MS analyses were performed on a Hewlett-Packard HP 5971A mass-selective detector directly coupled to an HP 5890II gas chromatograph. A fused silica capillary column Ultra 1, (H-P), 50 m × 0.2 mm × 0.33 μm was employed using helium as carrier gas. The samples were injected by splitless technique at 250°C. The column temperature was programmed from 30°C (1.0 min) to 250°C with a gradient of 1.7°C min⁻¹. The ionisation energy (EI) was 70 eV.

Gas chromatography (GC) and aroma extract dilution analysis (AEDA)

For the determination of retention indices and GC-olfactometry analyses, a Hewlett-Packard HP5890II gas chromatograph with an FID and a sniffing port was used. The samples were injected splitless on an Ultra 1(H-P), 50 m × 0.32 mm × 0.5 μm column operated at a temperature programme from 30°C (0.5 min) up to 250°C with a gradient of 2°C min⁻¹ and on a DB-WAX (J&W), 30 m × 0.32 mm × 0.5 μm column with a temperature programme from 35°C (0.5 min) up to 250°C with a gradient of 4°C min⁻¹. The linear velocity of the carrier gas hydrogen was 36 cm s⁻¹ for Ultra 1 and 30 cm s⁻¹ for DB-WAX column (measured at 143°C). The linear retention indices (RI_p) were calculated after Van den Dool and Kratz (1963) using n-alkanes C₆–C₂₂ as reference compounds. For AEDA experiments, the effluent of the column Ultra 1 was split with the split ratio 1:1 to the FID and the sniffing port, where humidified air (200 ml min⁻¹) was added. For determination

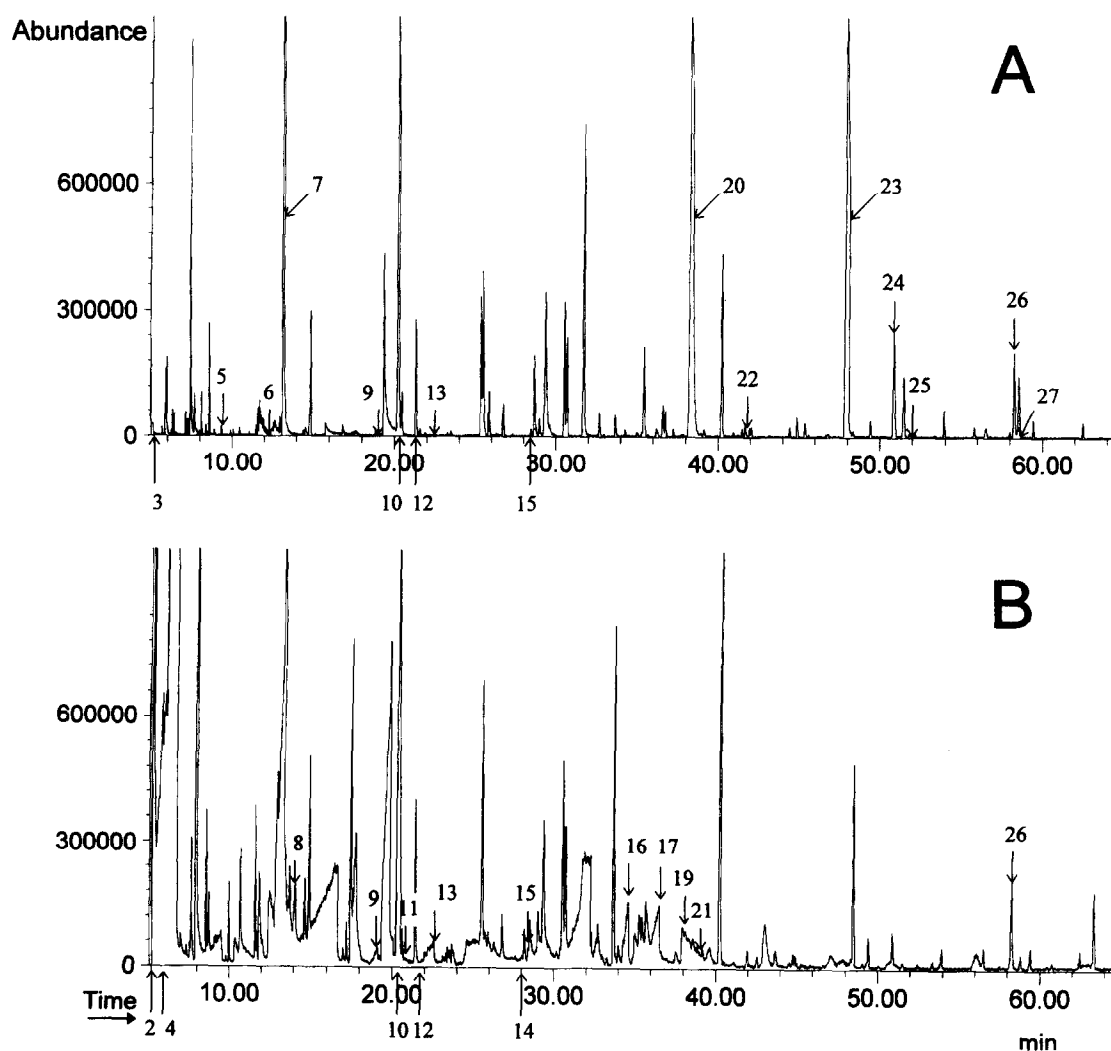


Fig. 1. Gas chromatograms of the volatiles isolated from pressure-cooked hen meat by SDE (A) and EVD (B). The numbers indicate the positions on the chromatogram where aroma-active compounds (FD ≥ 4) were registered by GC olfactometry, column Ultra 1, 50 m × 0.20 mm × 0.33 μm. The numbering corresponds to Table 1.

of aroma potency of the flavour compounds by means of flavour dilution (FD) factors, the analysed samples were stepwise diluted with diethyl ether from 1:4 up to 1:256 and each dilution was examined by GC-sniffing. The AEDA was performed by two trained assessors with two repeats for each dilution.

RESULTS AND DISCUSSION

The smell of the extract of volatiles prepared by SDE was slightly meaty but with a prevailing roasty, fatty odour. The smell of the flavour extract obtained by EVD was strong meaty, sweet with a roasty, fatty undertone. The EVD extract gave a very similar smell impression to the aroma of fresh pressure-cooked hen meat. The GC/MS analyses and AEDA (Fig. 1) indicated compounds with high FD factor common for both types of isolates, but also some flavour-active compounds present only in one type of the isolates. As can be seen from Table 1, the common compounds with high FD factors are 2-methyl-3-furanthiol, 3-(methylthio)propanal, 2-furfurylthiol and (E,E)-2,4-decadienal. All these compounds have also been identified as primary odourants of chicken broth (Gasser & Grosch, 1989). The compounds with high FD factors (≥ 64) present only in the isolate prepared by SDE are 3,5-dimethyl-1,2,4-trithiolane (DMT) with putrid odour, one isomer of 5,6-dihydro-2,4,6-trimethyl-4H-1,3,5-dithiazine (isomer marked as B) (TMDT) with roasty, dusty odour, unknown compounds 1, 5, 6 with sulphurous, garlic, compound 25 with meaty and compound 27 with bread crust, coffee-like odour. The aroma-active compounds present only in the isolate prepared by EVD were 4-hydroxy-2,5-dimethyl-3(2H)-furanone (HDMF), 4-hydroxy-5-methyl-3(2H)-furanone (HMF) both with caramel like aroma, 2-ethyl-3,5-dimethyl pyrazine with nutty, roasty aroma and unknown compound 18 with sweet, pleasant aroma. We found out in a model experiment that HDMF cannot be recovered from an aqueous solution by SDE using a *Likens-Nickerson* apparatus due to its high polarity. The 2,4,6-trimethyl-tetrahydro-1,3,5-thiadiazine (TMTD), TMDT, HDMF, HMF and 2-ethyl-3,5-dimethyl pyrazine were identified in cooked chicken meat for the first time. As shown in Table 1, the difference in the composition of the volatiles extracts obtained by the two different isolation procedures is considerable. The TMTD has been found to be an unstable reaction product of acetaldehyde, ammonia and hydrogen sulphide (Boelens *et al.*, 1974). The TMDT has been identified as a component of volatiles of different meats (Brinkman *et al.*, 1972, Nixon *et al.*, 1979) and as a product of some model reactions (Zhang *et al.*, 1988, Zhang & Ho, 1989). Both these compounds are readily formed but at the same time they are thermally labile. It is claimed (Mottram, 1991) that they can also be formed during the extraction of volatiles. As DMT, TMTD, TMDT, and the unknown

compounds 1, 3, 5, 6, 25, 27 were not found in the sample prepared by EVD, we assume that they are artefacts formed during the SDE process. This finding can possibly refer to the need of reevaluation of some earlier studies of meat-aroma compounds and some model reactions.

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