

Volatile Components in Roast Beef

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The volatile components of roast beef and roast beef drippings have been analyzed by gas-liquid chromatography (glc) and mass spectrometry (ms). Samples were prepared by low temperature, vacuum distillation of meat and drippings, followed by extraction of the distillates with diethyl ether. Major volatile components are alkanals, alk-2-enals, and alka-2,4-dienals. In addition to these compounds the meat samples show high concentrations of

3-hydroxy-2-butanone and γ -butyrolactone. Further classes of constituents are 2-*n*-alkylfurans, 2-alkanones, 3-alkanones, 2,3-alkadiones, pyrazines, primary and secondary alcohols, acids, γ - and δ -lactones, alkanes, aromatic compounds, sulfur compounds, and acetylpyrrole. By comparing the lean meat portion and the fat portion of roast beef it was found that the fat is the prime site of the aldehydes.

Many investigations have been devoted to the identification of volatile constituents of meat from several different species of animals. Studies of components in beef, pork, lamb, and chicken have been reviewed by Hornstein and Crowe (1964), Hornstein (1967), Phippen (1967), and Solms (1968). The volatiles of boiled beef were investigated by Kramlich and Pearson (1960), Yueh and Strong (1960), Hornstein *et al.* (1960), Bender and Ballance (1961), Sanderson *et al.* (1966), and Chang *et al.* (1968). Tonsbeek *et al.* (1968) and Copier *et al.* (1970) identified compounds in beef broth.

This study was made to determine the volatile components of roast beef and roast beef drippings. Several concentrates were analyzed by glc on open tubular columns and identified by mass spectrometry. It was also intended to compare the constituents in the lean meat portion and the fat portion of roast beef.

EXPERIMENTAL

The roast beef was prepared by Westreco, Inc., Marysville, Ohio. A rib roast weighing approximately 6 lb was cooked without the addition of any condiment for 22 min per lb at a temperature of 163° C. After cooking, the roast was cooled to room temperature, sliced, packed in aluminum foil, wrapped in a plastic bag, frozen in a Dry Ice chest, and sent to Houston by air. The drippings of the rib roast were collected, frozen, and sent to Houston in the same parcel. Roast beef and drippings were kept in a freezer for 1-4 days before processing.

Preparation of Concentrates of Roast Beef Volatiles. Concentrates of volatile constituents were prepared by combined distillation and extraction. In a representative sample preparation, 1000 g of roast beef was cut into small pieces and distilled for 20 hr at a vacuum of 2 μ . The 3-l. round-bottomed flask containing the meat pieces was heated in a water bath at 55° C. The distillation apparatus used here

has been described by Liebich *et al.* (1970). Apiezon M grease was used to lubricate the ground glass joints of the assembly. After the 20-hr distillation period, the first trap had collected most of the aqueous sample, while the second trap contained no more than 2 ml. After removing the Dewars of liquid nitrogen and disconnecting the assembly, 30 ml of redistilled, anhydrous Baker Analyzed diethyl ether was added to the traps. Subsequently the traps were closed with ground glass stoppers and allowed to warm up. The combined contents of both traps were extracted for 24 hr with 80 ml of the above described diethyl ether in a continuous liquid-liquid extractor. By distillation of the ether extract at atmospheric pressure over a Vigreux column, most of the solvent was removed and a sample of 1 ml was obtained. A further concentration of the extract was accomplished by leaving the sample vial uncapped at room temperature until approximately 35 μ l remained. Blank distillations, extractions, and concentrations were made to ensure that no components in the concentrate originated from contaminations. Samples from 500 g of the lean meat portion and 180 g of the fat portion of roast beef were prepared according to the same method.

Preparation of a Concentrate of Volatiles of Roast Beef Drippings. The drippings from 6 lb of roast beef amounted to approximately 120 g and contained about 70% fat. The entire amount of drippings was distilled under the same conditions as described for the meat and yielded 23 ml of aqueous distillate. After extraction with 60 ml of diethyl ether and concentration of the extract, 50 μ l of a concentrated sample were obtained.

Gas Chromatographic and Mass Spectrometric Analysis. The glc analyses were performed on a Perkin-Elmer Model 900 gas chromatograph. It was operated with prepurified nitrogen as the carrier gas at an inlet pressure of 14 psi. All samples were separated on a 500 ft \times 0.02 in. i.d. stainless steel column coated with Dowfax 9N15 (Column A). The column was programmed from room temperature at 150° C at 2° C/min. Roast beef drippings were analyzed on an LKB 9000 gas chromatograph-mass spectrometer at 70 eV, with an ion source temperature of 270° C and a separator temperature of 230° C. The glc column used with this instrument was a 700 ft \times 0.03 in. i.d. stainless steel column coated with Dowfax 9N15 (Column B).

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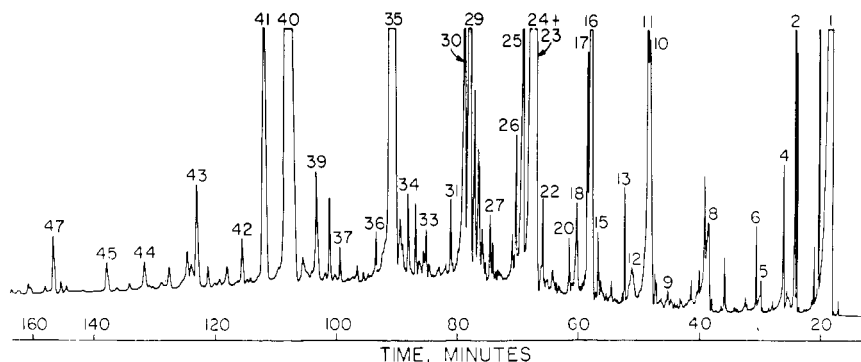


Figure 1. Gas chromatogram of a concentrate of volatile constituents from roast beef drippings. Attenuation 256, sample size 0.3 μ l, Column A

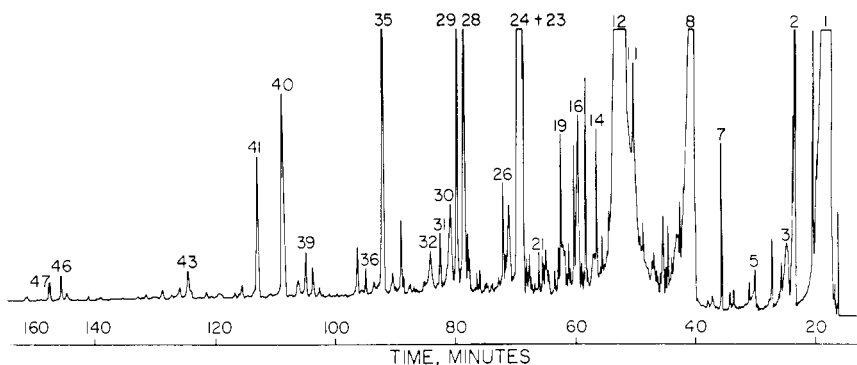


Figure 2. Gas chromatogram of a concentrate of volatile constituents from roast beef. Attenuation 256, sample size 1.8 μ l, Column A

RESULTS AND DISCUSSION

The concentrated samples exhibited pleasant meaty odors with a somewhat fatty nuance for roast beef concentrates and slightly burned for the drippings. The odors of the drippings samples were considerably stronger than the meat odors and were distinctive of drippings.

Figures 1 and 2 show chromatograms of concentrates from drippings and roast beef, respectively. Odor differences are reflected in differences in the chromatograms. The concentrations of *n*-hexanal, 3-hydroxy-2-butanone, and γ -butyrolactone (peaks 8, 12, 28) are much higher in meat than in drippings, while the drippings chromatogram shows higher concentrations for *n*-heptanal, *n*-octanal, 1-heptanol, 2-nonenal, 1-octanol, 2-decanal, 2-undecenal, and 2,4-decadienal (peaks 10, 16, 25, 29, 30, 35, 40, and 41, respectively).

Many of the volatile constituents in roast beef and drippings could be identified by mass spectrometry. The chromatograms obtained with the 0.03-in. column on the LKB 9000 gas chromatograph-mass spectrometer were very similar to the chromatograms obtained with the 0.02-in. column on the Perkin-Elmer instrument. Checks on the identity of the compounds indicated by the spectra were made by comparing the gas chromatographic retention time of the component with that of the authentic compound, by using the reference spectrum from the literature, and in some cases by measuring the spectrum of the authentic compound. When standard compounds were not available for comparison, identifications were assigned if reference literature spectra were available and if the mass spectral fragmentation pattern and the retention time suggested a compound homologous to other components identified in roast beef or drippings.

All the constituents identified in roast beef and drippings are listed in Table I. Peak numbers refer to numbers in the

figures. Identified compounds which are not labeled appear in the chromatograms between two labeled peaks in the order given in the table. No numbers were assigned to these constituents because the experimental conditions for the glcms analyses were different from the conditions for glc analyses, resulting in slight changes in peak sizes and retention times. The chromatograms shown in the figures were recorded on the regular chromatograph. The main classes of compounds identified are alkanals, alk-2-enals, alka-2,4-dienals, 2-*n*-alkylfurans, ketones, 2,3-alkadiones, primary and secondary alcohols, acids, γ - and δ -lactones, alkanes, aromatic compounds, dimethyl disulfide, dimethyl sulfone, and acetylpyrrole. Several of the compounds reported in this investigation have been found in other types of meat (Hornstein, 1967; Pippen, 1967; Solms, 1968; Nonaka *et al.*, 1967; Lillard and Ayres, 1969; Hobson-Frohock, 1970). Some low-boiling constituents not found in the concentrates from roast beef and drippings have been identified in boiled beef (Kramlich and Pearson, 1960; Yueh and Strong, 1960; Hornstein *et al.*, 1960; Bender and Ballance, 1961; Sanderson *et al.*, 1966).

The carbonyl compounds seem to be the basis for the roast beef and especially the drippings volatiles. According to Hornstein and Crowe (1960, 1963) many of the aldehydes are formed by heating the fat portion of meat in air. This is in accordance with results obtained by comparing the volatiles of the lean meat portion with the fat portion of the same piece of roast beef. Unlike the lean meat, the fat shows high concentrations of several saturated and unsaturated aldehydes. The relatively low concentration of *n*-heptanal and *n*-octanal in the meat fat is remarkable as opposed to their higher concentrations in drippings.

Several of the aldehydes found in roast beef, especially

Table I. Volatile Constituents of Roast Beef and Roast Beef Drippings

| Peak no. | Compd | Sample | Peak no. | Compd | Sample |
|----------|----------------------------------|--------|----------|---------------------------------|--------|
| 1 | Diethyl ether (solvent) | M, D | 23 | <i>n</i> -Nonanal | M, D |
| 2 | Ethyl acetate (solvent impurity) | M, D | 24 | 2-Octenal | M, D |
| 3 | Ethanol (solvent impurity) | M, D | 25 | 1-Heptanol | M, D |
| 4 | 2-Methylbutanal | M, D | | Benzaldehyde | M, D |
| 4 | 3-Methylbutanal | M, D | 26 | 2- <i>n</i> -Heptylfuran | M, D |
| | 2,3-Butanedione | M | | C ₄ -benzene | M |
| 5 | <i>n</i> -Pentanal | M, D | | 2,4-Octadienal (tent.) | M, D |
| | <i>n</i> -Nonane | M, D | | 2,3-Butanediol | M, D |
| | 1-Propanol | D | | 2-Decanone | M, D |
| | 2,3-Pentanedione | M | | <i>n</i> -Tetradecane | M, D |
| 7 | Toluene | M, D | 28 | <i>n</i> -Decanal | M, D |
| | Dimethyl disulfide | M | 29 | γ -Butyrolactone | M, D |
| 8 | <i>n</i> -Hexanal | M, D | 30 | 2-Nonenal | M, D |
| | <i>n</i> -Decane | M, D | 31 | 1-Octanol | M, D |
| | <i>n</i> -Butanol | M, D | | 2- <i>n</i> -Octylfuran | D |
| | 1,4-Dimethylbenzene | M, D | | Butyric acid | D |
| | 1,2-Dimethylbenzene | M | | 6(?) -Methyltetradecane | M |
| 9 | 3-Heptanone | D | 32 | Phenylacetaldehyde | D |
| | <i>n</i> -Undecane | M, D | | 2-Octen-1-ol | M |
| | 2-Heptanone | M, D | 34 | <i>n</i> -Pentadecane | M, D |
| 10 | <i>n</i> -Heptanal | M, D | | 2-Undecanone | M, D |
| | 2-Hexenal | D | | γ -Hexalactone | D |
| 12 | 3-Hydroxy-2-butanone | M, D | 35 | <i>n</i> -Undecanal | M, D |
| 12 | 1-Pentanol | M, D | 36 | 2-Decenal | M, D |
| 13 | 2- <i>n</i> -Pentylfuran | M, D | | 2,4-Nonadienal | D |
| | Trimethylbenzene | M | | δ -Valerolactone (tent.) | D |
| | Methylethylbenzene | M | | γ -Heptalactone | D |
| 14 | 3-Octanone | M, D | | Dimethyl sulfone | M, D |
| | Trimethylbenzene | M, D | | 2-Dodecanone | D |
| | 4-Heptanol (tent.) | D | 39 | <i>n</i> -Hexadecane | M, D |
| | 3-Heptanol | D | | 2,4-Decadienal | M, D |
| 15 | 2-Octanone | M, D | 40 | <i>n</i> -Dodecanal | M, D |
| | <i>n</i> -Dodecane | M, D | 41 | 2-Undecenal | M, D |
| 16 | <i>n</i> -Octanal | M, D | | 2,4-Decadienal | M, D |
| 17 | 2-Heptenal | M, D | | δ -Heptalactone | D |
| | 2,3-Octanedione | M | | <i>n</i> -Heptadecane | M, D |
| | Diethylbenzene | M | | Heptadecene | D |
| 18 | 1-Hexanol | M, D | 43 | Acetylpyrrole | D |
| | Acetic acid | D | | γ -Octalactone | D |
| 19 | C ₈ -benzene | M | 44 | 2-Tridecanone | M, D |
| 20 | 2- <i>n</i> -Hexylfuran | D | | <i>n</i> -Tridecanal | D |
| | <i>n</i> -Butylbenzene | M | 45 | 2-Dodecenal | D |
| | C ₄ -benzene | M | | δ -Octalactone | D |
| | Dimethylpyrazine (tent.) | D | 47 | 2,4-Undecadienal | D |
| | 4-Octanol (tent.) | D | | <i>n</i> -Octadecane | D |
| | 3-Octanol | D | | γ -Nonalactone | M, D |
| | 2- <i>n</i> -Butoxyethanol | M | | 2-Tridecenal | D |
| 22 | 2-Nonanone | M, D | | 2,4-Dodecadienal | D |
| | <i>n</i> -Tridecane | M, D | | δ -Nonalactone | D |
| | | | | γ -Decalactone | D |
| | | | | 2-Pentadecanone | D |

^a M: Compound was found in roast beef meat; D: Compound was found in roast beef drippings.

Table II. Volatile Constituents of Boiled Beef

| | |
|-------------------------|-----------------------|
| Dimethyl sulfone | Benzoic acid |
| Dimethyl disulfide | Benzyl alcohol |
| γ -Butyrolactone | Trimethylpyrazine |
| Methylbutyrolactone | Dimethylethylpyrazine |
| Phenol | Vinylguaiaicol |
| Acetylpyrrole | Methylcinnamaldehyde |
| Dimethylpyrazine | Tetradecanal |
| Benzothiazole | Pentadecanal |
| Benzaldehyde | Heptadecanal |

one or two isomers of 2,4-decadienal, have been identified in many other foods such as soybeans (Wilkins and Lin, 1970a,b), potatoes (Buttery *et al.*, 1970), potato chips (Mokherjee *et al.*, 1965), carrots (Buttery *et al.*, 1968), and tea (Bondarovich *et al.*, 1967). It may be of interest to note that some volatile constituents in boiled beef were also determined.

The compounds identified are listed in Table II. No attempt was made to prepare a synthetic mixture of the identified components in order to test its odor, since some of the required reference compounds were not available. Because of the complexity of the mixture, further investigations may be necessary to elucidate the complete nature of the flavor of roast beef.

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