8. Analytical Methods

Sensory analyses of meat from old culling ewes fed on different levels of fish meal.

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In an attempt to reduce carcass fatness 16 old culling ewes from purebred Leicester and Oxforddown were fed on low energy diets (barley straws) supplemented with different levels of fish meal.

The objective of the present study was to clarify whether the fish meal supplementation gave rise to fishy off-flavours as a result of oxidation of the unsaturated lipids during processing and/or storage.

For this purpose sensory analyses were performed by a taste panel and the amounts of thiobarbituric acid reactive substances (TBARS) were measured from meat/fat blends in raw samples, heated samples (70°C), and 2-days stored, reheated samples. In addition lipid samples extracted from backfat of *M. longissimus dorsi* were analyzed for fatty acid composition.

A trend towards increased off-flavour and decreased overall acceptability was found as a result of using fish meal in the diet. However, due to large variation between animals, mainly within the fish meal supplemented groups, no clear systematic effects were found. No significant effect of fish meal supplementation on any of the fatty acids were observed. This supports the general hypothesis that ruminants hydrogenate unsaturated lipids.

AMINO ACID ANALYSIS IN FRESH PORK AND DRY-CURED HAM BY HPLC OF PHENYLISOTHIOCYANATE DERIVATIVES

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One of the most important biochemical changes in ham during the dry-curing process consists in a substantial increase in its free amino acids concentration. This increase may result in an enhance of the natural characteristic taste of dry-cured hams.

Precolum phenylisothiocyanate (PITC) derivatization combined with reverse phase liquid chromatography has been tested for free amino acids analysis in muscle and adipose tissue from both fresh pork and dry-cured ham. Preliminar chromatograms obtained by using a waters pico-tag amino acid analysis with a Supelcosil^{TM} LC-18-DB column showed the presence of an unidentificated peak coeluting with the arginine. Furthermore, a reagent peak also coeluted with ornithine and tryptophan. These problems could be overcome by introducing some modifications which consisted in changes in column temperature (50 ºC instead of 46 ºC), pH of mobile phase (6.75 instead of 6.4) and gradient conditions (including flow rate gradient in the initial six minutes step).

The 21 amino acids identified in meat were succesfully separated and their quantitation completed. A noticeable increase in the free amino acid concentration along the dry-curing process has been detected. In fact, the concentration of free amino acids in dry cured hams is extremely high as compared to raw meat with special increases in aspartic acid (216.8 vs 1.03 mg/100 g), glutamic acid (410.5 vs 11.2 mg/100 g), leucine (240.6 vs 8.3 mg/100 g), lysine (542.7 vs 8.5 mg/100 g), valine (215.8 vs 6.1 mg/100 g).

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Rapid Determination of Collagen in Meat Products

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For the determination of collagen in meat products hydroxyproline is analysed photometrically after acidic digestion of the sample. The time for digestion takes approximately 16 hours. So, this method cannot be used in meat processing enterprises for control of raw material or intermediate products before or during the production process. This problem can be solved by using a commercially available microwave digestor. In this case the time for digestion takes only 30 minutes. Within the scope of this investigestion the digestion by microwave was compared with the method usually used for routine analysis by the Institute for Meat Hygiene.

Materials of investigation were raw beef, ham, sausages, spread, gelatine and mixtures of gelatine with meat products. The microwave digestions was carried out using hydrochloric acid (6 mol/l) and a pressure limit of 85 psi according to the instructions of the producer. For comparative analysis sulphuric acid (3 mol/l) was used. The ratio between the amount of the sample and the digestion liquid was identical in both cases. The photometric determination of hydroxyproline was carried out by ISO-Method 3496.2.

The comparison of the results shows that the mean values of the hydroxyproline contents obtained by the described techniques were only slightly different, the relative standard deviations between 2 and 10% in both cases. There exist no significant differences using the WILCOXON U-test. The analysis speed could be further reduced using a small volume (e.g. 100 l) of the first dilution of the hydrolysate for colour reaction instead of carrying out a second dilution. This comparative investigation demonstrates that the digestion of proteins by a microwave equipment is suitable for rapid determination of collagen in meat products.

DSC and NMR Studies of lipids in "FOIE GRAS"

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The quality of "foie gras" was studied in France during the last years. The determination of lipids in "foie gras" was a basic experiment to characterize the melting type of "foie gras". This is presently determined by tactile and visual test and means loss of fats by cooking. The feeding of animals and the processing of product (slaughter, plucking) play an important role in the quality of "foie gras". We propose to apply NMR methods (spectroscopy and micro-imaging) to estimate lipids in liver cells by assessment of fatty globules size in different parts of the "foie gras" and characterization of unsatured and satured lipids by ^{13}C NMR. The micro-imaging technique, using pulsed gradients, allows by selection of a slice to obtain images with contrast of proton density and times relaxation. With a resolution of ten micrometers and slices of less than millimeter, it is possible to measure the size of adipocytes in "foie gras". Furthermore, the differential scanning calorimetry might enforced this attribution by pointing out the proportion of polyinsaturated fatty acids. These techniques might support the previous observations and develop new parameters for estimation of "foie gras" quality.

Volatile Components of Dry Cured Ham: Identification and Sensory Characterization by Sniffing

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The volatile components of dry cured ham were extracted by Dynamic Headspace and analyzed by Gas-Chromatography-Mass-Spectrometry (GC-MS). The structure of the more volatile molecules was identified by mass spectrometry and measure of Kovats indices. More than 60 compounds were identified including a number of aldehydes, ketones, alcohols, esters, aromatic and heterocyclic compounds. These compounds come from the catabolism of the main constituent parts of the meat (carbohydrates, lipids and proteins) during the curing of the hams, from the pig feed or technological processes. Flavor tests showed the existence of several aromatic molecules which may contribute to the characteristic odor of dry cured products.

Determination by enzyme immunoassay of clenbuterol levels in urine and tissues of treated cattle.

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We have developed an enzyme immunoassay (EIA) using an antiserum raised in rabbits by immunization against a clenbuterol diazo-derivative coupled to human serum albumin. Horse radish peroxidase coupled to the clenbuterol diazo-derivative was selected as a tracer. The dosis of clenbuterol that caused 50% binding inhibition was 18 pg and the limits of detection of EIA was 0.2 ppb and 0.3 ppb in urine and liver respectively (taking into account the variability of blank values in samples from untreated animals) This assay was applied to urine and tissue samples collected from veal calves and cows that have received in their feed different doses of clenbuterol during various periods.

It appeared that clenbuterol levels in urine from treated animals (20 µg/kg live weight and per day) differ largely even when the animal received the same treatment. Large differences were also observed with time in the same animal. A quantitative relationship did not appear between clenbuterol concentration in urine and the level of the administered dose. The clenbuterol concentrations in tissues, with the exception of liver, were to low to identify treated animal 28 days after stopping the treatment. On the other hand, the veal calves treated with clenbuterol during the whole period (without stopping the treatment before slaughtering) can be easily identified owing to the presence of detectable clenbuterol in the various tissues examined, higher concentrations being observed in liver, kidney, and spleen.

Veal calves have also been treated with a much lower dose of clenbuterol (2 µg/kg.d) that is probably closer to the doses suspectely used in illegal treatment for meat production. In this case, the concentration of clenbuterol in tissues are lower than the detection limit of our assay when the withdrawal period was equal or longer than 4 days. If the daily administration of 2µg/kg of liveweight of clenbuterol is stopped 3 days before slauthering, levels of clenbuterol residues are close to the limit of detection of our immunoassay in liver, whereas they were undetectable in bile, kidney, spleen and muscles.

8

Quantitative and Qualitative Analysis of Free Fatty Acids in Meat and Meat Products using Ion Exchange Resin

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A rapid and reliable method for quantitative and qualitative analysis of free fatty acids (FFA) is described. This method is a modification of Needs et al. (1983) one which allows separation of FFA from milk using anion exchange resin Amberlyst 26. After investigations with standard lipid mixtures, the following procedure were adopted:

50 to 100 mg of lipids extracted according to Folch et al. (1957), which contain 50 µg to 1 mg of FFA, were shacked with 100 mg of resin and 15 ml of acetone/methanol (2:1, v/v) during 30 minutes. Non resin bound lipids were washed with 5 x 5 ml of acetone/methanol. FFA were methylated directly with resin according to Morrison and Smith's method (1964). Individual fatty acids were quantified using internal standard (17:0) by gas liquid chromatography with a 30 m fused silica capillary column coated with polyethylene glycol. FFA recovery is almost complete (95 -100%) whatever the chain length and the insaturation; interferences by triglycerides, monoglycerides or phospholipids account for less than 5 % of the FFA fraction.

Results of FFA determination in meat and meat products were presented.

Relationship between the Content of Skatole and Indole in Backfat and Lean Pork Obtained by a New HPLC Method

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The aim of this study was to find out the relationship between the concentration of skatole (3- methylindole) and indole in backfat and lean meat. For the investigation slices from M. longissimus dorsi of boars with adhering backfat were used. These were exactly separated in backfat and lean meat, and then analyzed on their content of skatole and indole. Therefore a rapid and specific testing method was developed, which is able to detect even very small concentrations of both substances. Skatole and indole were extracted with methanol from the sample material. The clean-up of the methanol extract was realized by freezing to eliminate the fat, followed by a solid-phase extraction. The concentrations of skatole and indole in the backfat and lean meat were determined by high-performance liquid chromatography (HPLC) with fluorimetric detection. As expected, the skatole and indole content in lean meat was distinctly lower than in backfat because of the more lipophile characteristics of both substances. Furthermore the tests have demonstrated that the content of skatole in lean meat correlates clearly with the content in backfat. The coefficient of correlation ($r = 0,96$) is significanly ($P < 0.001$) and demonstrates a strong linear relation between the content of skatole in backfat and lean meat. A lower significant ($P < 0.001$) correlation is obtained between the content of indole in lean meat and backfat ($r = 0.76$).

Occurence of Nitroso- and Nitrophenols in Cured and Smoked Meat Products

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In cured and smoked meat products, nitrite from the curing salt and phenols from the smoking process are supposed to react to nitrosophenols or nitrophenols. Some nitrophenols could be detected in experimental sausages, but nitrosophenols were not found in meat products. Because of their toxicological properties and of catalytic effect on the synthesis of nitrosamines, the question had to be answered, whether nitrosophenols can occur as stable residues in cured and smoked meat products. The application of three "conventional" extraction - and clean up procedures together with massspectrometric analysis gave no hint about the occurence of nitrosophenols in meat products. Nitrophenols, however, the oxidation products of nitrosophenols were detected.

Therefore a method was elaborated in order to prove the existence of nitrosophenols in phenol standard solutions, in liquid smoke, in "model sausages" and in cured and smoked meat products. The idea of the method was to stabilize a possible intermediate by a specific reagent. In the case of nitrosophenols, a Grignard reagent, methyl-magnesiumbromide was applied to the extracts to give N-methyl-N-hydroxyphenylhydroxylamine, which then would be detectable by massspectrometric procedures. With this method different substituted hydroxylamines could be detected in model systems. This confirms, that the nitrophenols, detected in meat products are synthesised via a pathway which includes the nitrosophenol step. However, these substances could not be detected in meat products. Thus the occurence of nitrosophenols in cured and smoked products as stable residues is very unlikely. The main reason for this result seems to be the numerous and fast side-reactions between the intermediate nitrosophenols and the constituents of meat products and smoke and because of the numerous different phenolic substances. Therefore the detection limits of the single nitrosophenols were not reached in real meat products.

Species Identification of Heated Meat and Meat Products by Isoelectric Focusing Followed by Silver Staining

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Isoelectric focusing (IEF) of proteins of **heated** meat and meat products extracted with 0.075 M tris buffer, pH 8.9, enables the identification of beef, pig, horse, sheep, deer and several other meat species after visualization of the protein patterns in the IEF gel by a very sensitive silver staining method.

The samples investigated were canned products heated to different levels of stability (F_C-values: 0.2, 1.0 and 6.0). A corresponding cooperative trial in which 22 institutes took part demonstrated the reliability of the method. Polyacrylamide gels of different thickness (0.15 - 0.5 mm) and different pH gradients (6 - 9; 3.5 - 9.5; 3 - 10) were used by the participants successfully.

Investigations about meat mixtures of different species are in progress.

8

Liquid Chromatographic Determination of Ochratoxin A in Animal Matrixes

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The Danish meat industry has recognised the increasing demands from governments and consumers for residue free food.

To meet these demands and to use them as a competition parameter the industry has decided to develop a system, which is able to check as many carcasses as possible for unwanted residues. Such a system must be fast enough to follow the slaughter speed and in addition be sturdy and give the best possible identification of the residues. Mass spectrometry (MS) is the only technique that meets these demands.

The system will be based on blood samples as it is easier to analyze blood than tissue samples. During the development of the MS-procedures it is necessary to use "reference" methods to make sure that no systematic error occurs. In addition it is necessary to study the ratio between concentrations in blood and tissue as most regulatory limits are set for tissue.

The present work concerns a "reference" method for ochratoxin A. Ochratoxin A is a potential carcinogenic mycotoxin, produced under certain conditions by mould species that may be present on grain. When such grain is used for pig feed, ochratoxin residues may be present in pork for human consumption.

The analysis proceeds as follows. Plasma samples are acidified with hydrochloric acid and are extracted with 3:2 hexane: tert. butyl acetate. Kidney and muscle samples are acidified with phosphoric acid and sonificated to disperse the tissue. For tissue samples celite is added to bind proteins and major cell components. The ochratoxin is extracted with 1:1 hexane: ethyl acetate. The ochratoxin is then extracted from the organic phase with aqueous sodium bicarbonate and after acidification ochratoxin is back-extracted to an organic phase. (Plasma samples: Chloroform; tissue samples: tert. butyl acetate). The organic phase is evaporated and the residue is dissolved in the eluent (48:52 acetonitrile: acetic acid). Samples are injected to an HPLC-system (C-18 column) and detected by fluorescence (Em. 460 nm, Ex. 330 nm).

The method gives a linear response for blood samples between 1 and 50 ppb and for tissue samples between 5 and 40 ppb. Correlation coefficients are always better than 0.995. Recovery is between 60 and 90%, being rather constant within a day.

Errors in pH-Measurement of Meat and Meat Products by Dilution Effects

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The pH of meat and meat products is generally be measured by using a needle-shaped glass electrode, which is pierced directly into the sample. If either the use of this kind of electrode is not adviseable or the sample is to dry or the sample has to be homogenized, usually some distilled water is added. The official method (Amtliche Sammlung von Untersuchungsverfahren nach § 35 LMBG) limits the water to sample ratio to 1: 1. But according to literature, many investigators increased the amount of water from 1 : 2 up to 1 : 10 (w/w). However, the pH-values measured by the direct method differed significantly from these values, found out with the addition of distilled water. The aim of this investigation was to determine the deviation between both methods and to develop an improved procedure. Homogenized meat was divided into samples, where the pH was chemically adjusted to different initial values with glucono- delta-lactone. The change in pH of a 10 g sample with increasing amounts of water added (2..500 ml), was measured and compared to the initial pH without added water. The error was constantly increasing with increasing amount of water, especially at the first few milliliters. At high initial values (pH 6) the pH constantly increased with increasing amount of water, whereas at low initial values (pH 5) the pH constantly decreased. Thereby the maximum error could be as high as + 0,2 pH and - 0,3 pH (at ratio 1 : 10), respectively. These high deviations have to be called as unacceptable. The reason for the deviation probably depends on the change of ionic strength and with it the charge of proteins by dilution effects, which in the end leads to a shift of pH. If the distilled water was replaced by a solution of NaCl, the deviation was reduced remarkably to ± 0,03..0,1 pH. While the concentration around 1% (w/w) gave the lowest deviation with meat, the optimum concentration for sausages apparently depends on the salt content of the product. If the pH determination requires the addition of a solvent, the use of an appropriate salt solution instead distilled water is therefore suggested.

Influence of Storage, Curing and Heating on the Electrophoretic Behaviour of Sarcoplasmic Proteins.

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The influence of storage, salting, curing and heating on the electrophoretic behaviour of sarcoplasmic proteins was investigated in order to test the suitability of electrophoretic methods for reliable species identification in meat and meat products.

Materials under investigation were raw beef and pork stored at different temperatures, raw and heated beef and pork salted and cured with nitrite using different concentrations of salt and nitrite. For protein separation isoelectric focusing and zone electrophoresis were used. Whole sarcoplasmic proteins, myoglobin and esterases were visualized by different staining methods.

The protein patterns of stored meat showed no differences if the sensoric quality of the meat was not impaired. In the case of spoilage different changes in the patterns could be obtained such as decreasing of the intensity of the myoglobin bands or of the acidic esterases. The addition of salt with or without nitrite results in a decreasing or a fading of sarcoplasmic proteins in the neutral region with increasing salt concentration. This effect could also be recognized in samples heated to 65° and 70°C for 15 minutes. Heating of meat to 75°, 80° and 100°C reduces or neutralizes the described influence of salt. In contrast to this the myoglobin and esterase bands of raw samples are not influenced by curing. Heating of cured meat effects a reduction of the intensity of the myoglobin bands visualized using their peroxidase activity in comparison to samples without addition of nitrite.

Raw meat can be identified by electrophoretic methods independent of the age of meat with the exception of spoiled samples. Differences of protein patterns caused by heating and curing can be equalized by second heating of the samples to 80°C for reliable species identification.

Meat Species Identification: Rapid Electrophoretic Methods and Staining Techniques

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Electrophoretic methods and staining techniques usually used for meat species identification are time consuming and require a great deal of instrumentation. In this study a simple equipment for isoelectric focusing which doesn't require any cooling was tested for its suitability to meat species identification. Furthermore, a method should be developed in order to combine different staining techniques. Investigated materials were raw pork, beef, mutton, horse-, rabbit-, chicken-, and turkey meat. Isoelectric focusing was carried out in 0,5 mm polyacrylamide gels with a distance between the electrodes of 5 cm. The separated proteins were visualized using Serva Violet 17 for whole sarcoplasmic proteins and specific stainings of myoglobin and esterases.

Using this isoelectric focusing cell meat species mentioned above could be identified within 2 to 3 hours depending on the staining procedure. The sharpness of the protein bands could be improved in comparison to conventional IEF cells. Furthermore no wavy bands occured in the acidic region of the gel. The staining method using Serva Violet 17 enables a rapid visualization of the whole sarcoplasmic proteins. The number of samples of a single run could be increased using a self made "triple electrode electrofocusing cell". For increasing reliability the electrophoretically separated proteins were stained by two different methods in order to profit from both techniques. Therefore, a sheet of nitrocellulose was rolled on the gel. The proteins diffuse partially to the nitrocellulose and are bound there without denaturation. After this "incomplete diffusion-blot" the myoglobins bound to the nitrocellulose were detected specifically. The proteins remaining in the gel were stained with Serva Violet 17. The described procedure enables to combine the advantages of the visualization of the sarcoplasmic proteins with the specific detection of myoglobin for reliable and rapid meat species identification.

8

Rapid Method for Determining Fat Content in Meat by Nuclear Magnetic Resonance

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Rapid methods are needed for the in-line control of quality parameters (e.g. fat or moisture content) in the meat industry. Nuclear Magnetic Resonance (NMR) has been used for a long time in the oil and confectionery industry, however, there are several references in the literature using continouos wave (CW)NMR or pulsed NMR for the quality control of meat and meat products. The investigations presented here were carried out with a Newport Analyser MKIII.A, using a 40 cm^3 sample tube. At about 20 g of sample was dried in a microwave oven, the dried residue was transferred quantitavely into the NMR-tube and the NMR-signal was obtained in 2-3 s. The fat content was calculated from different calibration curves obtained by measuring the signal for different amounts and types of fat and meat (lard, tallow, pork and beef with known fat contents). The fats were dehydrated with sicc. Na_2SO_4, the meats were dried in a microwave oven. Equations were formulated separately with these materials and their combinations (lard-tallow; pork-beef; lard-pork; tallow-beef; lard-beef; lard-tallow-beef; lard-tallow-pork, etc.). Results obtained by applying the various calibration curves and results of the Soxhlet extraction were compared. Calculations were carried out with the help of Deming's regression. It was established that the type of the fat influenced the results, however, reliable calibration curves could be obtained with the mixtures of the different meats and fats. So, the NMR method seems to be applicable for the determination of fat content in the meat.

Analytical Methods

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According to the scheme of analysis sophisticated methods in meat analysis are reviewed. Emphasis is centered on separation techniques using solid phase extraction technique, gel-permeation, immunoaffinity clean up and solid-phase dispersion to isolate the analyte. Trends and perspectives in analytical methodology e.g. quick tests and multi-residue methods for regulatory and monitoring purposes as well as screening tests are discussed.
TLC technique is a cheap and quick method. The automated multiple development mode (AMD) of TLC offers a new approach to screening analysis.
Gaschromatographic separation of organic residues by programmed temperature vaporizing, on-column injection and GC/MS in the ppt level using GC/NCI-MS technique are reviewed.
Examples of separation by liquid chromatography including pre- and post column derivatisation and combination of HPLC/MS using thermospray device are given.
Flavour substances of meat and meat products play a increasingly important role in quality assurance and quality assessment. The combination of sensorial and instrumental analytical methods is demonstrated.
Trace elements, preferably analyzed by multielemental methods and electrophoretic methods, able to solve the problem of identifying certain meat products are stressed.

Fingerprinting Meat Quality through Spectroscopy - On-Line

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Most chemical components in meat can be rapidly determined by spectroscopic methods. Light is transmitted via optical fibres and an insertion probe into the carcass. Here the constituents of the meat will absorb light at various wavelengths. Some of the attenuated light is reflected and transmitted through other optical fibres to a spectrometer. The spectra are treated with multivariate data analysis to correlate these to meat quality parameters such as pigment and protein contents.

We have developed an equipment to measure the intrinsic colour (pigment) with a spectrometer working in the visual wavelength range. A method for determining protein using near infrared light is under development.

The correlation regarding determination of pigment content (myoglobin/haemoglobin) between the laboratory method and the above-mentioned method is 0.98. At the moment the correlation for protein content is 0.85.

Investigation of Bovine Blood Plasma Utilizing Advanced Identification and Separation Techniques

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Spray-dried, frozen, freeze-dried and freshly drawn bovine blood plasma was subjected to various separation and chemical identification tests. The separation tests included: Electrophoresis (Urea- and SDS PAGE systems), HPLC and UV spectrophotometry. The various fresh plasma samples were prepared by centrifuging the bovine blood at 3, 6, 8 and 14 thousand r.p.m. (1090, 4360, 7740 and 23700 g-values, respectively).

Preliminary separation data showed few differences between the plasma samples prepared at different g-values. Significant differences (presence and/or absence of bands and/or band shifts) were, however, recorded for spray-dried and freshly prepared bovine blood plasma. These differences were indicative of the disappearance of some proteins whilst the molecular weight of others was altered significantly. The results were confirmed by the data on the various chemical analyses. From the results it is speculated that the "wild" taste of meat products (as well as other food products) containing two or more percent dried plasma results from the formation of one or more unidentified, heat induced, high molecular protein-linked molecules.

8

Quantification of Hemoproteins in Seal Meat and Other Muscle Foods

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Myoglobin and hemoglobin of muscle foods are generally separated from each other by precipitation of hemoglobin in a phosphate buffer solution. They are then converted to their corresponding cyano derivatives and are quantified using spectrophotometric methods. Although the myoglobin so obtained was always pure, the hemoglobin fraction showed contamination with different proportions of myoglobin as evidenced by a gel electrophoretic separation technique. The degree of contamination of hemoglobin fraction with myoglobin depended on the initial content of each component as well as the concentration of buffer used. Separation of hemoproteins on Sephadex G-75 revealed that the hemoglobin fraction may contain up to 78% myoglobin. Thus, the quantification method of individual hemoproteins of muscle foods by phosphate buffer precipitation is inadequate and the procedure could only be used for of myoglobin purification.

Thermogravimetric analysis (TGA) of different hog and cattle tissue lipids

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The investigation of volatile compounds formation by TGA, which was recently performed for hog intramuscular lipids, indicated that such an analysis could be very useful for determining the thermal and oxidation stability of lipids. Moreover,other thermal method (DSC) were also successfully used for correlating the heat of fusion or effects of oxidation to the lipids composition. In the present work special attention was paid to determining the rate of volatile compounds formation from lipids extracted from different hog and cattle tissues (muscle and fatty tissue,spinal cord,brain and liver) by TGA.

TGA was performed under the following conditions: 30-200°C; 2.5, 5 and 10°/min -heating rate; air and nitrogen as the carrier gas - 25 cm^3/min. The samples of lipids from different tissues were obtained by a procedure according to Folch and stored at +4°C. The total lipids were also fractionated to neutral lipids,glucolipids and phospholipids according to the procedure described by Johnston. The aim of this work was to quantitatively show that the rate of volatile compounds formation mostly depends on the phospholipids content.

The obtained results show that the mass change of the samples decreased in the following series of total lipids extracted from: the liver, muscle tissue, brain, spinal cord and fatty tissue; as well as that the total mass change during TGA was always about 20% larger in the case of samples extracted from hog tissues. The greatest differences between the results obtained in air and nitrogen atmosphere were detected in the case of lipids from muscle tissue, the liver and spinal cord, which indicated their reactivity to oxygen.

The rate of volatile compounds formation above 130°C, when significant change occurs, could be analysed using a first order kinetic expression. The calculated activation energy for the formation of volatile compounds in air was in the range 14-55 kJ/mol, indicating that a simple relation exists between the content of phospholipids in the samples and the determined activation energy of oxidation.

The Use of Ca-Ion-Selective Electrode in Determination of Ca Content in Mechanically Deboned Meat

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The purpose of this study was the search of possibility for the use of ion-selective electrode to determine Ca content in mechanically deboned meat. The material for examination was mechanically deboned chicken meat containing 0,3 and 3,0% of bone particles. The Ca-ion-selective electrode with plastic membrane (on PCV base) - made in Poland was used, with parametres: pH range 6-11, temperature range 0-40^{o}C.

The determination of Ca content was done in solutions obtained by: 1) soluting ash residues of MDCM, 2) in wet digestion procedure of MDCM. The Ca content of both variants of solutions were determined by: 1) the official AOAC with EDTA titration procedure, 2) atomic spectrophotometric absorbtion method, 3) titrametric method using the Ca-ion-selective electrode as indicator of titration end point. In the last method the equivalence point of titration was determined graphically from ionometric titration curve. The method of the second derivative was applied.

There was no significant difference between values obtained for the same sample of MDCM in different analytic procedures. The experiences gained to determine Ca content use Ca-ion-selective electrode as indicator of titration end point for sample prepared in wet digestion procedure and titration EDTA. This way eliminates the error which caused by subjective estimation of titration end point in the method AOAC.

A rapid method for measuring pigment concentration in porcine and other low pigmented muscles.

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Meat colour is one of the most important characteristics consumers use when selecting and purchasing meat. Colour is determined, to varying extents, by the pigment concentration in the meat. Consequently, methods for measuring pigment concentration are important in the evaluation of meat quality. This paper describes a rapid method for measuring total pigment concentration which is applicable to porcine and other low pigmented muscles. The method involves: 1) Extraction of total pigments by homogenising the muscle in 0.04M pH 6.5 phosphate buffer [sample to buffer ratio 1:10]; 2) Filtration through No 1 Whatman filter paper; 3) Clarification of the filtered extract by addition of 10% Triton X100 [final concentration 2.5%]; 4) Oxidation of the pigment with 65mM sodium nitrite [final concentration 1.2mM); and 5) Measuring absorbance of the oxidised pigments at 409 nm [corrected for turbidity, measured at 730nm]. This method when used with porcine muscle had the same accuracy and precision as the standard Hornsey and cyanometmyoglobin methods but was much more rapid and did not involve the use of toxic and/or flammable reagents. Additionally, the method was more accurate and reproducible than two other rapid methods; the alkaline haematin method and the $Absorbance_{525}$ method. The greater accuracy and precision obtained with this method in muscles with low pigment levels is due to the fact that the absorbance is measured in the Soret band where the absorbance in 5-10 times greater than at the wavelengths used with other methods.

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Monitering on Conditioning of Beef by Biosensors

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For quality control on conditioning of beef we developed the biosensor system which can monitor both the initial stage of bacterial putrefaction and the progress of conditioning of beef. Polyamines, which were known as products of bacterial decarboxylation of amino acids had been studied as a chemical quality index for meat and seafood. Especially cadaverine and putrescine were useful for determining the initial stage of putrefaction of meat. Many index for conditioning had been proposed and hypoxantine and xantine, which were the decomposed products of ATP were considered to be one of useful index.

Our 2-line flow injection analysis (FIA) system were composed of auto injector, microtube pump, oxygen electrode, water bath, system controller and recorder. At the tips of two oxygen electrodes putrescine oxidase and xantine oxidase immobilized membranes were fixed respectivery. Vacuum packed sir loin meat of bullock was stored at 20,10 and 2℃. These samples were used for microbial and chemical analysis. In chemical analysis putrescine, cadaverine, ATP related compounds and fragmentation index were measured. Samples were also measured by 2-line FIA system and compared to the data by HPLC.

In the intermediate temperature conditioning at 10℃ and 20℃ we could monitor the initial stage of putrefaction and the progress of conditioning by 2-line FIA system. However, in the low temperature conditioning at 2℃ putrescine sensor was not useful for detecting the bacterial putrefaction because lactic acid bacteria became the dominant micro organisms in the micro flora and they didn't produce diamines.

True Stomach Enzyme Vibroextraction Process Study

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At present the process of lamb and calf true-stomach enzyme extraction is made in vats by means of salt solution and takes much time.

It is possible to reduce the process of extraction to a large extent using vibration (Limonov G.E. et al.); enzyme activity increases as well. However the design and working conditions of the vibroextractor were not optimal. Hydrodynamics and heatmass moving of a vibrated in a closed vessel liquid have been studied. The data obtained have shown the presence of resonance regime of vibration followed by intensive mixing, gas saturation, cavitation, stagnant wave formation and hydraulic blow which are useful for external heatmass moving as well as for salt diffusion process in true stomach cell and true stomach enzyme in resonance conditions in vibroextractor showed that 20 minutes extraction repeated three times brings about complete extraction of the true stomach enzyme and increases its activity. The data taken made it possible to develop vibroextractor working in resonance regime.