## Volatile monocarbonyl compounds in hams manufactured by different technologies, at the final stage of ageing

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Using combined thin-layer and gas-liquid chromatography, an analysis was made of volatile carbonyl compounds, such as 2,4-dinitrophenylhydrazones, in hams prepared by an express technology or in the classical manner.

The contents of most of the monocarbonyl substances in conventional ham are higher than those in express ham.

The subfraction of alkanones changes more sensitively compared to that of alkanals and some more polar unsaturated monocarbonyls.

## Flüchtige Monokarbonylstoffe in nach verschiedenen Technologien hergestelltem Schinken in der Reifungsendstufe

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Es wurden die flüchtigen Karbonylverbindungen wie 2,4-Dinitrophenylhydrazon mittels kombinierter Dünnschicht- und gasflüssiger Chromatographie in nach einer Expresstechnologie und nach der klassischen Methode hergestelltem Schinken analysiert.

Der Gehalt an den meisten Monokarbonylstoffen in dem nach der klassischen Methode hergestellten Schinken ist höher als dieser in dem nach der Expresstechnologie hergestellten.

Bedeutend grösser ist die Veränderung der Unterfraktion der Alkanone im Vergleich mit dieser der Alkanale und einiger polareren ungesättigten Monokarbonyle.

8.4

## Les monocarbonyles volatils dans le jambon, fabriqué d'après des technologies différentes, dans la période finale de la maturation

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On analyse les carbonyles volatils comme 2,4-dinitrophénylhydrazones au moyen d'une chromatographie combinée - en couches minces et en phase gazo-liquide - dans du jambon, fabriqué d'après une technologie intensifiée et du jambon, fabriqué d'après une méthode classique.
La majorité des monocarbonyles présentent une teneur plus élevée dans le jambon, fabriqué d'après la technologie classique, par rapport à celui, fabriqué d'après la technologie intensifiée.
La sous-fraction des alcanones subit une modification plus sensible par rapport à celle des alcanaux et de certains monocarbonyles plus polaires non saturés.

## Летучие монокарбонильные вещества в ветчине, произведенной по различным технологиям, на конечном этапе созревания

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Путем комбинированной тонкослойной и газо-жидкостной хроматографии анализированы летучие карбонильные соединения, как 2,4-динитрофенилгидразоны, в ветчине, приготовленной по экспрессной или по классической технологии.
Содержание большинства монокарбонильных веществ в классической ветчине выше, чем в экспрессной.
Чувствительнее изменяется подфракция алканонов по сравнению с алканалами и некоторыми более полярными ненасыщенными монокарбонилами.

8.4

# Volatile monocarbonyl compounds in hams manufactured by different technologies, at the final stage of ageing

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The degree of lipid oxidation and hydrolyses is one of the dominating factors accounting for the flavour and taste of fresh and canned foods,fats and oils.Known is the role of the polyene fat acids,whose speed of hydrolyses and oxidation is the fastest,for the formation of a wide specter of volatile compounds, defining a given flavour of foods. Among the different degradation products obtained as a result of autooxidation, hydrolyses or mutual oxidation of the food lipids, carbonyl compounds are believed quite an important group,responsible for the appearance of or the loss of an wanted , very becoming, or respectively not wanted, unfavourable flavour in canned or shelved for long time meat products,fats and oils. The methods for the isolation of carbonyls from the lipids of meat are based either on destilation with water steam in the presence of an inert gas, or on extraction in acid media and low temperature. The last cited methods assure total degradation of the proteinlipid complexes,without any change in the lipids, while at the same time is ensured the necessary for the formation of 2,4 dinitrophenyl-hydrazine derivates in an acid media.

Two types of cooked hams were analysed for volatile carbonyl substances after the method of acid extraction at low temperature, namely:

- 1, ham express in foil and a period of curing of 24 h (H.Exp)
- 2, ham pressed in foil and a period of curing of 14 days (H.14)

For muscle injection of the ham produced by the express method with a production cycle of 24 - 48 hours was used a complex salt solution with a density of 21°Bé. "H.14" was injected with a part of the above salt solution and for dipping was used a solution of a smaller Bé density, 12 - 14.

The isolation of the carbonyl derivates with 2,4 DNPhydrazine from the hams is a multi stage operation : (1) freeing of the low, water soluble substances of the carbonyl class by treatment with perchloric acid with consequent destruction of the protein-lipid complexes; (2) forming of the derivates either with 2,4 DNPhydrazine and the consequent extraction with organic solvents,executed in conditions ensuring the passage of the hydrazones of monocarbonyles,semialdehydes and cetoglycerides as whole in the organic solvent; (3) adsorption of the obtained derivates on MgO-Celite coloumn (w/w),followed by elution with a mixture of nitromethane - chloroform; (4) fractionation of the monocarbonyl derivates from these of the ceteglycerides on $Al_2O_3$; (5) determination of their total quantity by absrbtion of the solutions at 340 nm; (6)deviding of the monocarbonyl hydrazones in classes on thin layer Mg-Celite 545; (7)identification of the obtained hydrazones by combination of GLC and thin.layer chromatography (after $R_f$ and $R_t$ values).

By measuring the absorbtion of the ethylacetate solutions of 2,4 DNPhydrazones of the monocarbonyls isolated from the hams at $\lambda$ = 340 nm it was found the total monocarbonyls quantity, contained in the two types of product. For the Celite was calculated a standart straight of the function C(D) at 340 nm for a standard substance (2,4 DNPhydrazone of propionic aldehyde) in concentrations of 1 - 9 $\gamma$/ml. The data from the experiment are treated after the method of least squares and K of the standard straight is calculated,

by the formula:

$$K = \frac{n\sum c_i D_i - \sum c_i \sum D_i}{n\sum c_i^2 - (\sum c_i)^2}$$

Each unknown concentration could be found after the formula $C_x = \frac{D_x}{K}$ $\gamma$/ml, where $D_x$ is experimentaly determined. The experimentaly found total quantity of 2,4 DNPhydrazones of monocarbonyls as well as the relative ration between the subfraction of cetons and and aldehydes derivates, calculated after Schwartz and Paris, is shown on fig.1.

From the figure it is seen that the quantity of 2,4 DNPhydrazones of the monocarbonyls isolated from ham produced by a long period of curing is approximately twice as high as the one of ham with a period of curing of 24 hours (32 to 59.$10^{-5}$ mM/100g). It must be noted as well that the fraction of ceton derivates changes significantly, while that of the alcanales exibits very little changes. These consclusions are confirmed also in comparing the results obtained in our laboratory in the identification of the spots which the separate subfractions (alcanolic and alcanalic) produce on thin layer Kieselgel F (fig.2), as well as by their gaschromatographic profiles (fig.3) obtained by using a column of 1,8 m length, id 2 mm and filling 4%SF-30 on Varaport DMCS-AW (80 - 100 mesh).

The richer in components ham "normal" with a period of cubing of 14 days, contains methyl-propylceton[X], which lacks in the ham produced after the express method, while at the same time in the last type of ham exist also thraces of octanol[XX] (spots N° 1 on both plates). In respect to the subfracton of alcanales, which is poor of components, should be noted the presence of acetaldehyde in the two samples as well as traces of propionaldehyde[XXX] in the normal ham with a period of curing of 14 days.

In conclusion we can say, that the total chromatographic picture reveals, that alcanones subfraction of the ham cured for 14 days contains more significant quantities of alcanones in comparison to the ham produced by the express technology. In general these changes are less significant in the subfraction of alcanales, while both products contain more significant quantities acetaldehyde, and the ham produced by the 14 days technology contains also propionaldehyde.

FIG 1. TOTAL MONOCARBONYLS

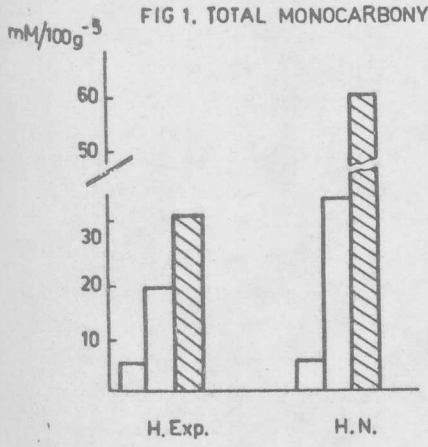


FIG.2 TLC ON KIESELGEL F

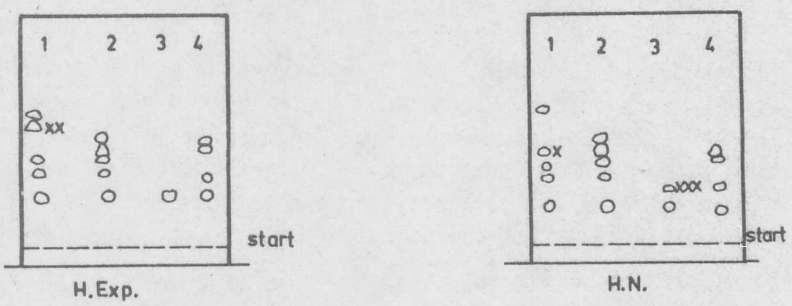


FIG. 3