GAS–CGROMATOGRAPHIC INVESTIGATION OF RESIDUAL SOLVENTS IN PACKED MEAT PRODUCT

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INTRODUCTION

In the production of conposite packaging material organic solvents are used for lamination, lacquering and printing. Theoretically, it is possible to remove completely the solvents, however, in normal production it is not the case. The residual solvents in the packaging (solvent traces) may enter the content and influence the changes of sensory characteristics of the packed meat product.
The solvent traces can be determined by sensory and instrumental methods. There are few papers on this problematics (1, 2, 3, 7, 8, 9, 10). In the available literature no data were found on the investigation of solvent migration from the packaging into the packed content. The solvents are determined in the package only by head–space analysis (2, 8, 10). Appropriate systems are constructed for sampling and sample concentration (4, 5, 6, 11).
For the mentioned reasons, the task of this work was the investigation and definement of conditions of gas-chromatographyc determination of solvents traces in the packed meat product. The column with appropriate packing and conditions of chromatography had to be chosen.

MATERIAL AND TACHNICS

The investigation was performed by head-space gas-chromatography, using the HEWLWTT PACKARD 7620 A gas chromatograph. The following columns were employed for the investigation:
- metal column, 3,o m long, i.d. 3,2 mm, packed with Carbowax 1540 on Chromosorb W-AW-DMCS (column 1) and
- glass capillary column Chrompack WCOT FUSED SILICA 7725, 51,0 m long, i.d. 0,24 mm, thicknes of packing (SP Sil 43 CB) on the column walls 0,19 μm (column 2).

Ethyl–acetate (ETA), methyl–ethyl–keton (MEK) and toluol (TOL) were used for the elution. As the model–content cured porc ham was used.

RESULTS AND DISCUSSION

Selektion of column and chtomatography conditions

The columns were investigated at different chromatography conditions. The injector temperature ranged from room temperature to 150°C. Detektor temperature was 230°C, and the column temperature ranged from 20°C to 80°C and was either constant or programmed with temperature increase 4 e.g. 6°C/min.
During investigation of column 1, the following conditions were determined: injector temperature – 150°C, column temperature – 70°C, helium flow rate 24 ml/min, air flow rate – 30 ml/min, hydrogen flow rate – 24 ml/min and sensituvity – 10^2 x 1. Under the conditiones determined, it was possible to separate and quantitatively determined the solvents, however, 0,5 ml of head–space gas has to be injected and this is a relatively big sample (chrom. 1).

Column 2 has pronounced advantage (chrom. 2) as the amount of injected head–space gas is 5 μl. At such a small sample, a need arose for the concentration of sample either using a pre–column or at the inlet end of the column. The sample concentration at the inlet end was chosen (fig. 1). The capillary column next to the injection blok, in the lenght of 5 cm was immersed in liquid nitrogen (–196°C). One minute after column immersion, the sample was injected into the gas–chromatograph and 4 minutes after injection the column was removed from the liquid nitrogen and chromatography started. In this way the impurities from carrier gas are also concentrated (the peaks as high as the ones of sample), so the tube of carrier gas was also immersed in liquid nitrogen, but throughout the investigation. The following conditions were determined for the use of column 2: injection temperature – 50°C; column temperature 20°C/1 min, 6°C/min, 80°C/5 min; heliun flow rate through the column – 0,6 ml/min; heliun flow rate (make–up) – 23 ml/min; air flow rate – 30 ml/min; hydrogen flow rate – 25 ml/min; sensitivity – 10x1.

Sensitivity threshold of the method

A model system was used for the determination of sensitivity threshold of GC analisys of solvents in real content using column 2. Cured pork ham (sample 1) was packed in glas jars and solvent were on the inner side of the cover. Sample was pasteurized. The smollest added amounts of solvents that were determined are presented in Table 1.
Wery low amounts of added solvents could be determined, and the results depend on the investigation temperature (lower amounts of MEK were determined at higher temperature), kind of content and solvent characteristics.

CONCLUSION

On the basis of the investigations, obtained and discussed results it can be concluded:

- The Capillary column Chrompack, type WCOT FUSED SILICA is the most convenient for the investigation of solvent which remain in the packing and later migrate into the meat;
- The impurited in the carrier gas can bi immersing the carrier gas tube into liquid nitrogen, while the concentration of the sample is performed by immersing the inlet end of the column into liquid nitrogen;
- Lower amounts of added solvents (MEK) were determined in samples thermostated at 40°C, by instrumental methods;
- The determined amount of solvent depends on the characteristics of the investigated solvents.

Literature

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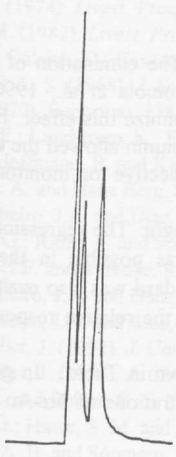
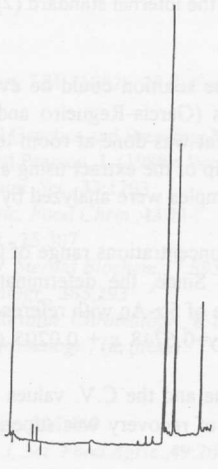
Table 1. Smallest solvent amount in 100 ml content – determined instrumentally

Sample	Investigation temp. (°C)	Determined amount of added solvent (µl)		
		ETA	MEK	TOL
1	20	1.54	3.85	0.10
	40	1.54	3.08	0.10



Chrom. 1.



Chrom. 2.

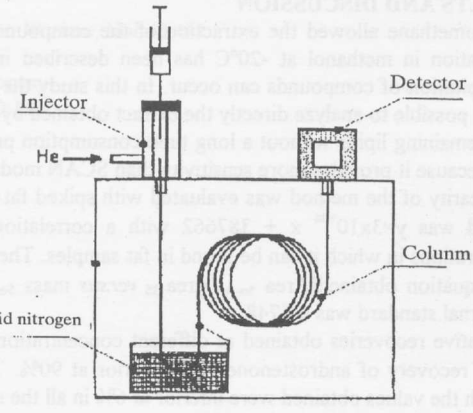


Fig. 1.