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# Modification of Method for the Determination of Organochlorine Pesticides in Meat Samples

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## Abstract

In the paper the modification of standard method SIST EN 1528 1-4: 1998: Method D for measurements of organochlorine pesticides (OCPs) in food products is presented. The modifications were made in the extraction step in which cold extraction technique was replaced by Soxhlet extraction. For the clean-up step smaller columns were introduced and for the concentration step a rotary evaporation was replaced by Kuderna-Danish concentration technique. Introduced modifications improved the efficiency of the procedure for samples with high fat content. Recovery values for all analyzed pesticides were over 60% and the reproducibility expressed as relative standard deviation was in the range of 10%. The method is suitable for the determination of OCPs in meat products with high content of fat from low ppb concentration range onward. The limits of detection for examined OCPs were in the range from 0.1 ppb to 2 ppb for lindane and α-endosulfan, respectively.

**Keywords:** Organochlorine pesticides; soxhlet extraction; florisil; ECD; meat

## 1. Introduction

Pesticides present a great danger for human health and significantly influence the environment owing to their extensive and uncontrolled use. The negative impact on human organism depends on concentration, susceptibility to degradation, stability in the environment, possibility of bioaccumulation and bioconcentration, ability of insertion into the human food chain etc. Basic classes of pesticides are insecticides, herbicides, rodenticides, fungicides and fumigants. Regarding their chemical structure, pesticides can be divided into organophosphorus pesticides (OPP), organochlorine pesticides (OCP), carbamates, and pyrethroids. The chlorine-containing pesticides include dichloro-diphenyl-trichloroethane (DDT) and metabolites, hexachlorocyclohexane (HCH) isomers, hexachlorobenzene (HCB), aldrin, dieldrin, endrine, heptachlor and heptachlor epoxide. Their classification as organochlorine pesticides is based on their common chemical properties and similar impact on the environment.[1]

Organochlorine pesticides are known to be very resistant in the environment. Their lipophilic nature is the reason for their concentration and bioaccumulation in the food chain, therefore they can be found in foodstuffs of animal origin, mostly in meat and tissues that contain fat, in milk and dairy products, eggs and fish. It was ascertained that because of their long term usage they have accumulated in the environment and have reached critical concentrations even in regions where they have not been produced or used for longer periods. Because of these negative effects their application was abandoned in many countries.[2,3] The Stockholm convention is a global treaty to protect human health and the environment from persistent organic pollutants (POPs). In implementing the convention, governments take measurements to eliminate or reduce the release of POPs into the environment.[4]

There are several procedures developed for the determination of pesticide residues in matrices such as vegetables, honey, beer, baby food, and meat.[5–8] However, their analysis in fatty matrices such as meat, represents much more difficult analytical tasks due to problems of fat

removal. Matsumoto et al.[9] investigated residues of polychlorinated biphenyls (PCBs) and organochlorine pesticides in different meats (beef, pork and poultry) and in processed meat products in Osaka, Japan. They indicated reducing trend in the concentrations of poly chlorinated biphenyls, HCH isomers, DDT analogues and dieldrin in all meats during the past 35 years. However, the residual values of organochlorine (chlorinated) pesticides in processed meat products remained at the same level for the past 15 years. It was also determined that concentration of each individual pesticide is lower in the processed meat products in comparison to raw meat.[9] Lazaro et al.[10] analyzed different meals of the average diet consumed in Aragon (Spain) for organochlorine pesticide residues. Of the 21 OCPs only HCB, lindane, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT and β-endosulfan were detected in samples and their levels were below the limits set by current European regulations.[10] Frenich et al.[3] optimized and validated the method for simultaneous determination of residues of OCPs and OPPs in chicken, pork and lamb meat samples. Extraction was carried out by a Polytron mixer, gel permeation chromatography was applied for the clean-up step and the final determination was performed by gas chromatography coupled to a triple quadrupole mass spectrometry detection system.[3] Juhler optimized the method for the determination of OPPs in meat and fatty matrices.[11]

The European Committee for Standardization recommends several methods for the determination of pesticide residues and PCBs in fatty foodstuffs. They proposed eight methods (from A to H) and each of them is suited for determination and quantification of different sets of OCPs and OPPs in fatty foods. All methods consist of four steps: extraction, clean-up, identification and quantification.[12] Several approaches can be applied in the extraction step. The most frequently used techniques are cold extraction (recommended by the standard Method D), Soxhlet extraction,[13] solid-phase extraction,[14,15] sonification[16] and more recently supercritical fluid extraction,[17] microwave assisted extraction,[13] fluidized-bed extraction[18] and accelerated solvent extraction.[19] An important step in the analytical procedure is clean-up of the extracts. In this step interferences should be eliminated and the analyte is prepared for chromatographic analysis. Several techniques such as gel permeation chromatography, adsorption chromatography on different sorbents (Florisil – recommended by the standard Method D, silica etc.) have been applied for sample clean-up prior to the chromatographic detection.[11,20–24] For the detection of organochlorine pesticides usually gas chromatography with either electron capture detector or mass selective detector is proposed.[25] Among newly established techniques the application of capillary electrophoresis is described.[26]

The aim of our work was to modify the standard SIST EN 1528 1-4: 1998: Method D in order to improve recovery values and reliability of the results. From eight proposed methods,[12] method D based on gas chromatography was chosen because it is suitable for the determination of 20 OCPs, 6 OPPs and PCBs in fatty foodstuffs. Each part of this method was thoroughly examined and improvements were made in extraction, clean-up and concentration steps.

# 2. Experimental

## 2. 1. Chemicals and Reagents

Pesticide standards of lindane, α-endosulfan, 4,4'-DDE, aldrin, dieldrin and HCB were purchased from PolyScience (Niels, IL, USA) and Serve (Hiedelberg, Germany) and their purities were > 99%. The pesticide mixture EPA CLP/625 (aldrin, a-BHC, b-BHC, d-BHC, g-BHC, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, dieldrin, endosulfan I, endosulfan II, endosulfan sulfate, endrin, endrin aldehyde, heptachlor, heptachlor epoxide) was purchased from Supelco and was of analytical grade. Organic solvents (acetone, n-hexane, petroleum benzene, diethyl ether, dichloromethane) were purchased from Merck (Germany) and were of HPLC grade. Florisil and anhydrous $Na_2SO_4$ (purities in both cases > 99%) were purchased from Fluka (Germany) and Merck (Germany), respectively. Partially deactivated Florisil was prepared with heating to 550 °C and left overnight. After cooling it was stored in a sealed container. Before usage it was heated for 5 hours (130 °C) and then MilliQ water (Molsheim, France) was added to obtain 3% final mixture.[8] Stock standard solutions were prepared by weighing appropriate amounts of standard substances, which were dissolved in 50 mL of n-hexane. Concentrations of stock solutions were 500 μg $mL^{-1}$. Liquid pesticide mixture was dissolved in 25 mL of n-hexane. All standard solutions were kept in the refrigerator at 4 °C. Nitrogen (99.996%, Messer, Austria) was used for GC analysis and solvent evaporation.

The glassware used for analysis was thoroughly washed: first with detergent and rinsed with Milli-Q water and ethanol. After drying it was rinsed with acetone, dried up and again rinsed with n-hexane.

## 2. 2. Extraction

Meat samples and their products were stored at –31 °C until analysis. Approximately 100 g of meat sample was taken and chopped with a food chopper. Ten grams of chopped homogenized sample were weighed into a glass beaker and 10 g of anhydrous $Na_2SO_4$ were added. When spiking samples, 1.0 mL of standard solution (concentration range from 1 to 10 μg $mL^{-1}$) was added in this phase. The sample was transfered on a filter paper (type 388, Sartorius, Germany) which was folded and put into a cellulose thimble. On top of the thimble a piece of cotton wool soaked with n-hexane, which was used for cleaning (collecting remaining parts of sample) the glass beaker, used for sample weighing, was placed. The thimble was

placed into a Soxhlet equipment assembled with a condenser on top and a round bottom flask that contained 100 mL of n-hexane and perforated glass boiling beads on the bottom. The sample was allowed to reflux for at least 18 hours on a water bath (95 °C), then it was cooled down and a part of the solvent was evaporated on a water bath.

## 2. 3. Florisil Clean-up

To remove matrix compounds, the clean-up procedure was performed on a glass column that was filled with 10 mL of n-hexane. Then 3 g of Florisil (an activated magnesium silicate) were slowly added. After approximately 15 minutes, when Florisil was settled, the sample solution was introduced into the column. The level of n-hexane was adjusted (redundant n-hexane was washed through the column) so that it was 1 cm above Florisil. The sample was slowly and quantitatively transferred into a column. Elution was carried out at flow rate of 5 mL $min^{-1}$ with 30 mL of n-hexane and dichloromethane mixture (w/w 4:1). The resulting samples were collected in glass beakers.

## 2. 4. Kuderna-Danish Sample Concentration

The eluent of the sample from Florisil clean-up step (around 35 mL) was transferred with a boiling chip into a flask and receiving vessel of a Kuderna-Danish concentrator. A Snyder column was put on top and the concentrator was submerged (so that almost the whole receiving vessel was under water) into a water bath with temperature close to the boiling point of water. Samples were concentrated to 1 mL and n-hexane was added to adjust the final volume to 2 mL. The concentrated sample was then transferred into a glass vial and kept in a refrigerator until GC analysis.

## 2. 5. Chromatographic Analysis

Chromatographic measurements were performed using a HP 6890 gas chromatograph (Hewlett-Packard, CA, USA) equipped with $^{63}$Ni electron capture detector (ECD). The RTX-5MS column with dimensions 60 m × 0.25 mm × 0.5 μm was purchased from Restek (USA). For quantification an HP Chemstation software (Rev. A. 05. 04) was used. Before analysis 1 μL of n-hexane was injected three times to stabilize the response of the detector. Then 1 μL of sample was manually injected three times and the result was the mean value of three replicates.

# 3. Results and Discussion

Our preliminary results based on the application of the recommended standard EN 1528 1-4: 1998: Method D for the determination of OCPs in fatty foodstuffs showed several weaknesses. Very low recovery values (below 10%) for all tested spiked samples were observed, which influenced the precision and accuracy of the method. Since the standard method is based on a cold extraction technique which can be the reason for the low recoveries, firstly this part of the procedure was taken under investigation. Cold extraction was replaced by Soxhlet extraction. To specify other sources of possible errors, also all analytical steps were examined. Our aim was to modify an existing standard method with incorporating other well-established concentration techniques in order to lower solvent consumption, gain higher extraction yields, obtain better reproducibility and lower limit of detection (LOD).

## 3. 1. Chromatography

The standard procedure allows a wide range of different separation columns regarding the selection of stationary phases as well as column dimensions (column length, internal diameter and stationary phase thickness) to be used. That is why no universal temperature program

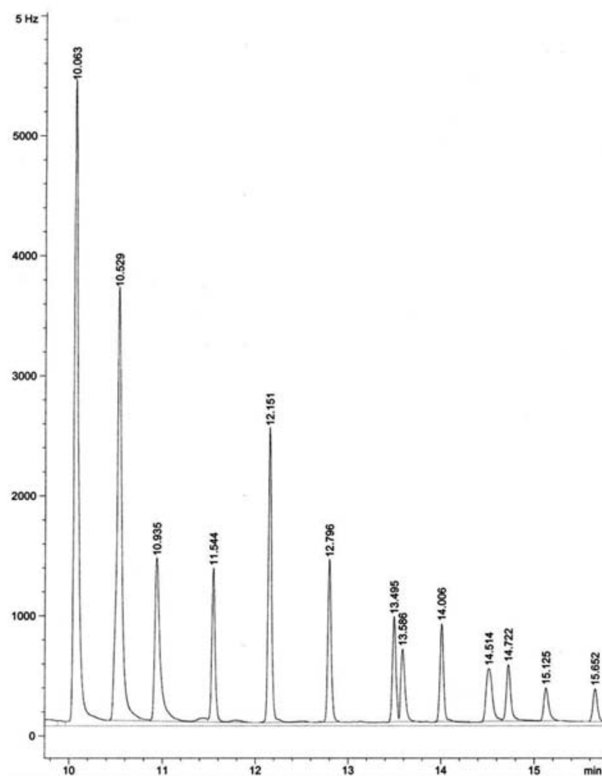


**Figure 1.** Segment of a chromatogram for separation of OCPs. Peaks from left to right correspond to: α-lindane (10.063 min); β-lindane (10.529 min); γ-lindane (10.935 min); heptachlor (11.544 min); aldrin (12.151 min); heptachlor epoxide isomer B (12.796 min); 4,4'-DDE (13.495 min); dieldrin (13.586 min); 4,4-DDD (14.006 min); endrin (14.514 min); α-endosulfan (14.722 min); endrin aldehyde (15.125 min) and 4,4'-DDT (15.652 min).

is suggested within these standard documents. The temperature programs found in the literature[13,17] using similar separation columns did not give satisfactory separation of 4,4'-DDE and dieldrin. Both literature procedures used shorter separation columns. In general longer separation column would enable better separation, however one should not forget that we have used just equivalent stationary phase. It was already shown that equivalent separation columns can give different resolution for the particular pair of chromatographic peaks, even more they can even cause the change in the elution order of different chromatographic peaks.[27] Therefore the temperature program was optimized for our experimental configuration. Satisfactory separations of OCPs under investigation were obtained with the following parameters: temperature of injector 250 °C, temperature of detector 320 °C, temperature program for column: initial temperature 70 °C, heating with rate of 30 °C $min^{-1}$ to 250 °C, with rate of 5 °C $min^{-1}$ to 270 °C and with rate of 10 °C to 300 °C (hold time 5 min). From Figure 1 it is evident that under such conditions, compounds are well separated, which enables their reliable quantitative determination. At selected conditions the precision of measurements for all compounds expressed as relative standard deviation was below 10% for manual injections of 1 μL of standard solution with concentration of 1 μg $mL^{-1}$. For most of the of investigated compounds the linearity was obtained in concentration range from 0.08 to 7 μg $mL^{-1}$ (lindane: y = 15538 x – 163.4, $R^2$ = 0.993, n = 7, α-endosulfan: y = 13733 x + 20.5, $R^2$ = 0.995, n = 7, dieldrin: y = 14869 x – 388.5, $R^2$ = 0.990, n = 7). For 4,4'-DDE linear range was even broader, that is from 0.08 to 10 μg $mL^{-1}$ (y = 8370 x – 1721.9, $R^2$ = 0.998, n = 8).

## 3. 2. Concentration Step

For concentration of extracts, besides rotary evaporation, other procedures were tested: purging with nitrogen and the application of Kuderna-Danish concentrators. For this purpose standard solutions of lindane, α-endosulfane, dieldrin and 4,4'-DDE in concentration range from 0.2 to 1.0 μg $mL^{-1}$ (for lindane 10 times lower) were chosen. In all cases 35 mL of initial solution of standards were concentrated to 5 mL.

For rotary evaporation concentrating procedure several disadvantages were observed. Due to large apparatus surfaces it is very difficult to efficiently concentrate analytes to low volumes and in addition, analyte losses or problems related to the contamination are more pronounced in comparison to other two above mentioned techniques. When low-volatility solvent xylene was added to serve as a keeper, the recovery values were within the experimental uncertainty of previous measurements. However the presence of the keeper hindered the subsequent concentration of the extract, therefore it was excluded in further experiments. From Table 1 we can see that satisfactory recovery values were obtained only for concentrations greater than 0.2 μg $mL^{-1}$. At lower concentrations the contamination problems are evident. For all examined concentrations the relative standard deviation for three separate runs was in the range of 20%.

The concentration efficiency when using nitrogen purging (not shown) gave overall lower recovery values (around 50%), however the reproducibility throughout the concentration range in comparison to rotary evaporation was improved. The relatively low recovery values can be attributed to losses caused by formation of an aerosol.

The results obtained with Kuderna-Danish concentrators showed the best results regarding reproducibility as well as recovery values. Table 2 represents average recovery values for lindane, α-endosulfan, 4,4'-DDE and dieldrin throughout the concentration range from 0.2 to 1.0 μg $mL^{-1}$ (in case of lindane from 0.02 to 0.1 μg $mL^{-1}$) with corresponding RSD values. In comparison with rotary evaporation and nitrogen purging, Kuderna-Danish concentration technique yields higher recovery values and better reproducibility (RSD below 10%). This technique also provides better pre-concentration rates, since it is possible to reduce final sample volume to 1 mL or less.

## 3. 3. Extraction Step

The cold extraction technique (as proposed in the standard method) was replaced by a Soxhlet extraction. For the study of this step standard mixture of lindane, α-endosulfane, dieldrin and 4,4'-DDE with concentration of 1 μg $mL^{-1}$ was used (1 mL of mixture was added to a filter paper, which was then put in a cellulose thimble). Different extraction times were investigated. Initially we let the sample to reflux for 3 hours. The yielded recoveries were very low (around 5%) so the time was prolonged to 5 hours and further to 10 hours, 18 hours and 24 hours. Recovery values increased with the prolonged time and the maximum was achieved after approximately 18 hours. Se-

**Table 1.** Average recovery values for lindane, α-endosulfan, 4,4'-DDE and dieldrin after evaporating the solvent with rotary evaporator and corresponding RSD values (n = 3) in brackets.

| Concentration (μg $mL^{-1}$) | η (%) α-endosulfan | η (%) 4,4'-DDE | η (%) dieldrin | Concentration n (μg $mL^{-1}$) | η (%) lindane |
|---|---|---|---|---|---|
| 0.2 | 113 (21%) | 119 (18%) | 110 (17%) | 0.02 | 118 (23%) |
| 0.6 | 54 (16%) | 47 (17%) | 67 (14%) | 0.06 | 72 (17%) |
| 1.0 | 59 (18%) | 50 (14%) | 62 (15%) | 0.1 | 64 (18%) |

**Table 2.** Average recovery values for lindane, α-endosulfan, 4,4'-DDE and dieldrin after evaporating the solvent with Kuderna-Danish concentrator and corresponding RSD values (n = 3) in brackets.

| Concentration ($\mu$g mL$^{-1}$) | η (%) α-endosulfan | η (%) 4,4'-DDE | η (%) dieldrin | Concentration ($\mu$g mL$^{-1}$) | η (%) lindane |
|---|---|---|---|---|---|
| 0.2 | 82 (4%) | 74 (8%) | 80 (6%) | 0.02 | 74 (9%) |
| 0.6 | 69 (5%) | 66 (6%) | 68 (7%) | 0.06 | 60 (6%) |
| 1.0 | 87 (5%) | 79 (4%) | 84 (9%) | 0.10 | 78 (8%) |

veral different solvents with different polarities were also tested: n-hexane, mixture of n-hexane and acetone (1:1) and diethyl ether. Among all tested solvents, n-hexane (as the standard method suggests) showed best results. While the use of n-hexane and acetone mixture (1:1) resulted in higher background of the chromatogram and slightly lower peak areas, the diethyl ether gave significantly lower recovery values. Further different temperatures and solvent volumes were also tested. For the extraction 200 mL round bottom flasks were used. At least 100 mL of n-hexane should be used to prevent drying of the bottom part of flask during cycles. When performing extractions at 70 and 80 °C respectively the reflux was not satisfactorily, therefore the temperature was increased almost to the temperature of boiling point of water (water bath was set to 95 °C). At those conditions the best reflux was achieved.

## 3. 4. Clean-up

For a clean-up agent, Florisil was applied, which is also recommended by the standard method. Using standard mixture of lindane, α-endosulfane, dieldrin and 4,4'-DDE (1 mL of mixture with concentration of 1 μg mL$^{-1}$ was added to 5 mL of n-hexane and elution was performed with 30 mL of eluting mixture) we have shown that Florisil does not affect the overall losses of examined substances. However it has to be mentioned that it is very important to prepare Florisil by partial deactivation before usage (this process is described in the experimental section). Chromatographic peaks of analytes obtained with clean-up on partially deactivated, compared to unconditioned Florisil, are higher. This indicates possible losses due to the adsorption of analytes on the unconditioned Florisil.

For the elution several compositions of n-hexane : dichloromethane mixture: (w/w) 1:1, 1:2, 1:3 and 1:4 were tested. No improvement over the 1:4 (w/w) elution mixture (as suggested in the standard method) was observed.

One of the aims was also to reduce the amounts of organic solvents used in the analytical procedure. The object was to reduce the amount of n-hexane and dichloromethane in the clean-up step without deteriorating the efficiency of the procedure. Regarding the standard method approximately 10 times smaller columns were tested (amounts of sample, Florisil and solvents were appropriately adjusted). It was ascertained that in case of smaller columns the efficiency of the clean-up step with Kuderna-Danish concentration technique was even slightly improved. With the reduction of the column size we also managed to lower the amounts of sample, Florisil and organic solvents while at the same time the efficiency of Florisil clean-up step was improved.

## 3. 5. Application of Method for Meat Samples

The developed analytical procedure as described in the experimental part and shortly summarized in the paragraph below was critically evaluated regarding reproducibility and recovery values. The results for optimization of different steps of procedure (concentration, extraction and clean-up step) for the determination of OCPs in fatty foodstuffs were initially focused on lindane, α-endosulfan, 4,4'-DDE and dieldrin because they represent a wide range of polarities from non polar lindane to the more polar α-endosulfan with 4,4'-DDE and dieldrin in the middle range. After the optimization of individual parts using the standard mixtures without matrix, the same procedures were tested with spiked samples of meat and different meat products (bacon, sausages, lean stag meat). Other organochlorine compounds present in the EPA CLP/625 standard mixture were also evaluated.

Approximately 10 g of homogenized meat was transferred into a glass beaker and approximately 10 g of anhydrous $Na_2SO_4$ were added. Samples were spiked with 1 mL of EPA CLP/625 standard mixture with concentration of 1.0 μg mL$^{-1}$. Soxhlet extraction with 100 mL of n-hexane was performed for 18 hours. Sample clean-up procedure on Florisil was applied and the samples were concentrated using Kuderna-Danish concentrators to 2 mL. 1 μL of sample was injected into the gas chromatograph. Recovery values for all examined OCPs were over 60% and the results presented in Table 3 are an example for analysis of lean stag meat. Reproducibilty of our modified method was tested by analyzing meat samples on different days using different operators. In Table 3 results of measurements where individual runs were performed more than 1 month apart are presented. The reproducibility among 9 different runs is satisfactory – the average error of 9 different parallels among all tested analytes is around 10% (RSD). Similar results were also obtained for analysis of bacon and sausages. Compared to the standard SIST EN 1528 1-4: 1998: Method D, where recovery values of all examined OCPs were below 10%, substantial improve-

**Table 3.** Recovery values (%) of α-lindane; β-lindane; γ-lindane; heptachlor; aldrin; heptachlor epoxide isomer B; 4,4'-DDE; dieldrin; 4,4-DDD; endrin and α-endosulfan for sausage and corresponding RSD values.

| Analyte | η1[1] | η2[1] | η3[2] | η4[2] | η5[2] | η6[2] | η7[3] | η8[3] | η9[3] | RSD (%) |
|---|---|---|---|---|---|---|---|---|---|---|
| α-lindane | 67 | 69 | 71 | 79 | 84 | 84 | 71 | 66 | 72 | 9 |
| β-lindane | 70 | 67 | 68 | 77 | 81 | 81 | 70 | 68 | 72 | 8 |
| γ-lindane | 58 | 58 | 61 | 66 | 75 | 73 | 67 | 62 | 67 | 9 |
| heptachlor | 65 | 66 | 69 | 74 | 82 | 79 | 67 | 63 | 69 | 9 |
| aldrin | 64 | 67 | 69 | 74 | 79 | 76 | 76 | 70 | 75 | 7 |
| heptachlor epoxide isomer B | 59 | 64 | 63 | 70 | 75 | 74 | 73 | 69 | 73 | 8 |
| 4,4'-DDE | 66 | 67 | 65 | 67 | 71 | 69 | 80 | 79 | 82 | 9 |
| dieldrin | 60 | 66 | 64 | 70 | 75 | 76 | 74 | 68 | 73 | 8 |
| 4,4-DDD | 69 | 80 | 88 | 93 | 95 | 99 | 79 | 77 | 83 | 11 |
| endrin | 55 | 59 | 55 | 63 | 71 | 73 | 68 | 64 | 69 | 10 |
| α-endosulfan | 58 | 62 | 59 | 67 | 74 | 78 | 64 | 60 | 64 | 11 |

[1] Date of analysis: 1st February 2008, [2] Date of analysis: 14th February 2008, [3] Date of analysis: 5th March 2008

ment of the method is evident. The main weakness of the standard method is low efficiency of the cold extraction. When Soxhlet extraction is applied, where the sample is allowed to reflux at an elevated temperature (95 °C), the distribution between solvent and analytes is more effective, resulting in higher recovery values.

With the proposed procedure better recovery, better reproducibility and lower limits of detection (LOD) in comparison to the standard method were obtained. Calculations of LODs are based on the results of measurements of the complete analytical procedure with a blank sample (only required solvents without matrix were used). LOD values are expressed as a three times standard deviation (3σ) of blank extract. The lowest LOD was found for lindane (0.1 ppb) and the highest for α-endosulfan (2 ppb). Results show that the method is suitable for the determination of wide spectrum of OCPs. Experiments with unspiked meat samples showed that concentrations of OCPs were approximately in the 10–70 ppb (ng per kg of fat) concentration range. From those results it is evident that for all analyzed meat products concentrations of examined OCPs were below the recommended maximum residue levels (MRLs), which are legislated from country to country differently (e.g. for UK and Germany 1 mg per kg of fat (1ppm) and for France 0.2 mg per kg of fat).

## 4. Conclusions

The standard procedure SIST EN 1528 1-4: 1998: Method D was modified in order to make it suitable for the determination of organochlorine compounds in food samples with high fat content. Extraction of OCPs from the food matrix was thoroughly optimized and we proposed the use of a Soxhlet instead of a cold extraction technique. Among different tested solvents n-hexane was chosen because of its better characteristics compared to other tested solvents, such as a mixture of n-hexane/acetone (1:1) or diethyl ether. The time of extraction was prolonged to 18 hours and the temperature of water bath during extraction was held at 95 °C. For the clean-up step the use of smaller columns was proposed to reduce the amounts of organic solvents, Florisil and sample weight. The elution was carried out at an approximate rate of 5 mL min$^{-1}$ with 30 mL of n-hexane and dichloromethane mixture (w/w 4:1). Partially deactivated Florisil has to be freshly prepared before analysis. For sample concentration the use of Kuderna-Danish concentrators was proposed. Also rotary evaporation (as the standard method suggests) and nitrogen purging were tested but in this case results did not meet our expectations. It was shown that with use of Kuderna-Danish concentrators the reproducibility and recovery values are improved. The parameters for GC-ECD determination of OCPs are following: temperature of injector 250 °C, temperature of detector 320 °C and temperature program for column: initial temperature 70 °C, heating with rate of 30 °C min$^{-1}$ to 250 °C, with rate of 5 °C min$^{-1}$ to 270 °C and with rate of 10 °C to 300 °C (hold time 5 min).

The obtained recovery values of OCPs were from 60 to 90% and the reproducibility expressed as relative standard deviation was in the range of 10%. The limits of detection for examined OCPs were in the range from 0.1 ppb to 2 ppb for lindane and α-endosulfan respectively.

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## Povzetek

V članku je opisana prilagojena standardna metoda SIST EN 1528 1-4: 1998: Metoda D, ki je primerna za določevanje organokloriranih pesticidov (OCP) v mesnih prehrambenih izdelkih. Glavne spremembe so bile napravljene v stopnji ekstrakcije, kjer smo hladno ekstrakcijo nadomestili s Soxhlet ekstrakcijo. Za čiščenje vzorcev predlagamo uporabo manjših kolon, za koncentriranje analitov pa smo namesto tehnike izparevanja topila z rotavaporjem uporabili Kuderna-Danish koncentratorje. Vpeljane spremembe so pripomogle k izboljšanju izkoristkov za vzorce z visoko vsebnostjo maščob, ki so bili za vse analizirane pesticide nad 60 %. Natančnost postopka, izražena kot relativni standardni odmik, je bila 10 %. Metoda je primerna za določevanje OCP v mesnih izdelkih z visoko vsebnostjo maščob. Meje zaznav za OCP so bile v območju od 0.1 ppb za lindan ter 2 ppb za $\alpha$-endosulfan.