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**POSTERS  
ABSTRACTS**

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**F-01**

**Dissipation of Pesticide Residues on Spent Grains**

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The mash filter grains are nutritionally similar to brewer's grains. A moist by-product from the brewing industry, made up of spent grains, is widely used as a buffer or as forage or concentrate replacer for cattle and sheep. Therefore, although the nutritional potential of the spent grains for animals has been demonstrated, it is important to ascertain the pollution load of the same and how any pesticide residue evolve during storage. For this reason, this study examines the evolution of pesticide residues (pendimethalin, trifluralin, fenitrothion, malathion, myclobutanil, propiconazole and nuarimol) during the storage of spent grains. For this purpose milled malt, once spiked (2 mg/kg) was subjected to a mashing process. At the end of this stage, soluble substances and residual solid particles were separated by filtration into sweet wort and spent grains, respectively. To know the dissipation rate of residues in the spent grains, the experimental data were fitted according to the usual first-order kinetics equation ( $r > 0.91$ ). Five sampling points (0, 15, 30, 60 and 90 days) were used to calculate the statistical parameters. According to the calculated values for the constant rate (K) and half-lives, the following dissipation rate was observed: myclobutanil > fenitrothion > nuarimol > trifluralin > propiconazole > pendimethalin = malathion. The necessary times to reach their respective LMRs ranged from 408-958 days for nuarimol and propiconazole, respectively, which indicates a high persistence level and minimum degradation for the pesticides, especially in the case of propiconazole, fungicide capable of inhibiting ergosterol biosynthesis.

**Keywords :** *Dissipation, pesticide residues, spent grains, brewing*

**F-02**

**Influence of Propiconazole Residues on the Primary  
Fermentation of Young Lager Beer**

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Propiconazole is a systemic fungicide that interferes with ergosterol biosynthesis by inhibiting the demethylation of steroids. Ergosterol is a crystalline sterol, C<sub>28</sub>H<sub>43</sub>OH, synthesized by yeast from sugars or derived from ergot. Azole compounds target the ergosterol biosynthetic enzyme, lanosterol 14 $\alpha$ -demethylase, and are a widely applied class of antifungal agents. In this study, after mashing and boiling processes, wort samples were spiked (1 mg/l) with propiconazole. Later, lager yeasts (bottom-fermenting yeast) were added to each fermentation vessel (n=3) containing the oxygenated pitching wort, which was maintained at 10 $\pm$ 1 $^{\circ}$ C for thirteen days. From the third day onwards, there is a significant decrease in the fermentation rate in the samples with fungicide residues compared with the blank (sluggish fermentation) and after eight days, the fermentation prematurely ceases (stuck fermentation). At the end of the fermentation process the mean concentration of sugars in the blank samples (alcohol content as %, v/v = 6) were 0.03, 0.09, 0.12, 1.64 and 0.27 mg/l for fructose, glucose, sucrose, maltose and maltotriose, while in the case of the treated samples (alcohol content as %, v/v = 1.4), the residual values were 0.13, 0.60, 0.02, 31.99 and 7.85 mg/l, respectively. Significant differences were also observed for total polyphenol and flavonoid contents (59.04 and 35.17 mg/l for blank samples and 17.22 and 21.44 for treated samples, respectively). As conclusion we can affirm that propiconazole residues, strongly affect the growth and fermentability of brewer's yeast, influencing the fermentative kinetic and, depending on the dose, able to cause stuck fermentation.

**Keywords :** *Propiconazole residues, fermentation, lager beer*

**F-03**  
**Organochlorine Analysis Residues in Tunisian Soils**

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Under the increasing contamination of the environment with organic chemicals, soils are more and more polluted with pesticide residues. The aim of this work is to determine the residue level of some organochlorine (about 10 species) in Tunisian soil. In this purpose, 4 agricultural fields are selected and are different in culture type and soil properties. Soil samples are collected from the top 40 cm of the soil profile. In this research, Chromatography Phase Gas (CPG) analysis was carried out using an F.I.D. The investigation of the results confirmed that organochlorine residues are present in the different fields even these pesticides have been removed from the market in the early 1980.s. and included in the WHO class (I a) products. The results showed that: - The rates of pesticide residues are in the order of the ppm. However at this low level, the organochlorine are considered toxic in any level they are found according to their toxicity and their impact on the environment such as soil fertility or on plants or on Humans under the food chain. - Two groups of pesticide are characterised: "major pesticide" which are at high level and "minor pesticide" in low quantities. - Pesticide residues distribution in analysed soils which are characterised with a different structure and mineralogy compositions, show some soils are best filtered than others and will have the consequence of ground water contamination. Thus, the sandy soils contains the lower level of residues despite the massive treatments that such soils receive (about 10 treatments per year), when the clay and silt soils contain a high level of pesticide residues. - In the first top 40cm, we don't find a correlation between mineralogy soils composition and the level residue pesticide. - The presence of these types of residue in Tunisia soils can be explained either by the remanence of the organochlorine or by the currently use of these products by the farmers. Farmers have insufficient information and are using these products because they are relatively low-cost, easy to use and versatile.

**F-04**

**The Residues of Some Pesticides Common Used in  
Canopy Growing at the Region of Aegean and  
Investigation of Their Extraction Methods**

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In this study, two different multi-residue pesticide methods were used for extraction of tomatoes, which are growing in greenhouse and they were compared according to their recovery factors. The residue situation of imazalil, iprodione and procymidone against white mold, gray mold and early blight in greenhouses were investigated at the provinces of İzmir, Aydın and Muğla in Aegean Region. For these analyses, the recovery ratios of two different multi-residue analysis methods were compared and the samples were extracted according to multi-analyze method, which has a high ratio in recovery. First Method: Samples were homogenized with adding acetone. The mixture was filtered. The pesticides were partitioned with addition of methylene chloride:petroleum ether mixture (1:1). Second Method: Pesticides were extracted by a water/acetone mixture. The sample was re-extracted with dichloromethane. The pesticides were partitioned with addition of hexane. NaCl was added at the second partitioning step. Extracts were allowed to pass through an Envi-Carb SPE cartridge, which is connected with an Aminopropyl Sep-pak. The pesticides are eluted from the clean up column with acetonitrile: toluene 3:1. The recoveries of the first method were 19,59%, 58,40% and 64,49% for imazalil, iprodione and procymidone, respectively. The recoveries of the second method were 91,75%, 96,15% and 89,55%, for imazalil, iprodione and procymidone, respectively. At the result of analysis, the second multi-residue method was better than the first method according to their recovery. The second method, which had a high ratio in recoveries, was used for extraction of the tomato samples. The extracts of samples were analyzed GC(ECD) and confirmed by GC/MS. At the end of these analyzes acquired chromatograms were investigated and samples content suspicious with pesticides were evident by weigh. As a result of these analyses, no residue encountered for imazalil, iprodione and procymidone in investigated 20 tomato samples, which were collected at the beginning of harvest period from greenhouses.

**Keywords : Tomato, pesticide, residue, GC(ECD), GC/MS**

F-05

**Household Processing Factors and Diet Portion  
Variability Of Acrinathrin and Kresoxim-Methyl Residues  
in Green Beans**

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Acrinathrin is a pyrethroid acaricide/insecticide commercialised in Spain under the tradename of "Rufast" to be mainly used for the control of "Trips" and "Red Mite" in many fruit and vegetable crops, including green beans (3 days preharvest interval; 0.30 mg/kg MRL). On the other hand, Kresoxim-Methyl is a broad-spectrum fungicide with preventive and curative activity, which is commercialised in Spain under the tradename of "Stroby" to be used on different vegetable crops, but it is not yet registered to be used on green beans (0.05\* mg/kg EU-MRL). The objectives of this work were to assess the influence of household washing and boiling processes on the residue levels of Kresoxim-Methyl and Acrinathrin in green beans, and to determine the residues variability in individual "diet portion" samples of green beans. The study was conducted on (2.2-3.1 kg) green beans samples ("Festival" variety) from an experimental greenhouse, which were collected at 1, 2, 3, 4, 5, 6, 7, 8, 10 and 14 days after a treatment with Acrinathrin and Kresoxim-Methyl at exaggerated application rates (400 and 500 g a.i./ha, respectively). In all cases, the greenhouse samples were properly subdivided to carry out the variability and processing studies. Analyses were carried out by means of the QuEChERS extraction method adapted for conventional GC-ECD determination and GC-MS confirmation. The variability factors, which were determined analysing 4-10 different "diet portion" samples (150 g) of unprocessed green beans from each greenhouse sample, ranged between 1.1 and 1.7. The processing factors (n = 20) obtained for Acrinathrin resulted to be  $0.6 \pm 0.2$  for washing and  $0.5 \pm 0.1$  for boiling, whereas the processing factors obtained for Kresoxim-Methyl were  $0.4 \pm 0.1$  (washing) and  $0.6 \pm 0.2$  (boiling). Pesticide levels in the unprocessed green beans samples were within the ranges 0.7-0.1 mg/kg (Acrinathrin) and 1.1-0.2 mg/kg (Kresoxim-Methyl).

**Keywords :** *Acrinathrin, kresoxim-methyl, grean beans, processing factors, residue variability*

**F-06**

**Assessment of Pesticide Residues in Cotton Honey from  
Greece**

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Cotton plants are very popular among the beekeepers due to their unique nectar secretion during the summer, a period of year with no other blooming of beekeeping importance. Cotton honey has been reported as a high quality product, exhibiting high antibacterial and antioxidant properties compared with the other types of floral honey. The main disadvantage of cotton plants is the great number of different pesticide treatments, resulting in loss of a part of bee population. Moreover cotton honey was accused to be contaminated with residues. The presence in cotton honey of pesticide residues originating from cotton plants or beehive treatments, was investigated in this study. A multiresidue method was developed in order to determine residues of organochlorine, organophosphate and pyrethroid pesticides. A C18 Solid Phase Extraction procedure was used for cleanup and isolation of pesticides, eluted with ethylacetate and petroleum benzene. The final extract was injected through a cool on column inlet to an Agilent HP6890 Gas Chromatograph, equipped with a HP7683 autosampler, an intermediate polarity DB-608 mega bore column and a  $\mu$ ECD detector. A HP5890 GC with a HP7673 autosampler, a split/splitless inlet, a non-polar HP-5 capillary column and an ECD was used for the confirmation of the results. Beehives were transferred and placed in cotton plant fields early in the season, before the application of any pesticide and thirteen samples were collected from them. In addition, sixty two samples of cotton honey were collected from beekeepers. The botanical origin of each sample was confirmed by organoleptic, macroscopic and melissopalinalogical methods. Acaricides used by beekeepers against *Varroa destructor* Anderson were determined (coumaphos, tau fluvalinate and malathion), while no pesticide applied to cotton plants was detected in samples.

**Keywords :** *Cotton honey, residues, GC-ECD, solid phase extraction*

**F-07**

**Analysis of Pesticide Residues in Olive Oil by Gas  
Chromatography / Electron Capture Detector (GC/ECD)**

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In this study, pesticide residues of 29 olive oil were investigated, which were taken from different part of Aegean Region. Pesticides are parathion-methyl, malathion, fenithrothion, chlorpyrifosmethyl, guthion, endosulfan, bromopropylate and cypermethrin. Pesticide residues were extracted by Solid Phase Extraction (SPE) method after 1,5 ml of olive oil were dissolved in 1,5 ml of asetonitrile, and residue analysis was made by using GC/ECD. At the result of residue analysis by GC/ECD, some pesticide residues were detected in 5 olive oil samples, but the amounts found were pretty low concentrations. In this study, a modified analysis method was used.

**Keywords : *Pesticide residues, olive oil, GC/ECD***



**F-08**

**Monitoring Pesticide Residues in Vegetables in Serbia**

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In 2004, Ministry of Agriculture, Forestry and Water Management of Republic of Serbia launched a project of monitoring pesticide residues in foodstuffs of vegetable origin, soil and water, with the objective of gaining information on the level of contamination by these chemical substances. In the first year of the study, vegetable samples (onions, peppers, tomatoes, cabbage, carrots and cucumbers) were taken from retail shops and routine controls by the customs service (border inspection). All samples were analyzed for residues of organophosphorus insecticides. Also cabbage samples were analyzed for pyrethroids and herbicide residues. GC- ECD, NPD and GC-MS were used for residues analyses. A total of 193 samples were analyzed. Pesticide residues were detected in 83 samples or 43%. Five different pesticides were found in four vegetable samples, four pesticides in four samples, three pesticides in 10 samples and two pesticides in 26 samples. The remaining samples contained active substance residues of a single pesticide. The largest number of positive finds was registered in the cabbage samples, with chlorpirifos and bifentrin being the most frequently detected substances. Pesticide residue values above the national MAC were found in 21 vegetable samples or 12.5%. In further study it is necessary to increase the number of analytes searched for in vegetable samples and to improve analytical uniformity, in order to obtain a more accurate insight into the pesticide status of these important foodstuffs.

**Keywords :** *Pesticides, residues, vegetables*

**F-09**

**Control of Pesticide Residues in Sweet Pepper in Souss  
Massa Valley in Morocco**

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In Morocco, sweet pepper crops are considered high value cash crop for farmers and an import outsource of hard currency. Souss Massa valley is the major region for the sweet pepper production in Morocco exporting about 90% to the European Union (EU). To control the pest and deasece growers are frequently obliged to spray pesticides during the plant cycle which thus may increase the risk of pesticide residues in the fruits. Pesticide residues can potentially be harmful to consumer's health, the environment and hold up the exchange trade between Morocco and European Union Pesticides residues analysis from 100 samples of sweet pepper fruits picked up from 20 packinghouses in the area of Souss Valley, in the southern part of Morocco, revealed that about 4% of samples exceeded the European tolerance MRLs.

**Keywords :** *Pesticide residues, sweet pepper*

**F-10**

**Pesticide Residues in Food Samples Originating from  
Mediterranean Countries from the Point of View of a  
Trading Laboratory**

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Summaries of residue data of several thousands of raw agricultural commodities, processed material and other foodstuff originating from the Mediterranean belt are presented, and an interpretation of their phytosanitary status is given. Most export-relevant crops are being highlighted. Modern analytical methodology applied in private trading as well as in monitoring laboratories of the public sector is addressed in the light of the need for quick turnaround times and reliable results. General conclusions are drawn on the legal status of the produce and its impact on consumer safety.

**Keywords :Residue monitoring, mediterranean production, consumer safety, MRL, analytical methods**

**Posters Abstracts: Food**

F-11

**Effect of Fruit Epicuticular Waxes on the  
Photodegradation of Chlorpyrifos-Methyl**

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The effect of epicuticular waxes of three fruits (grapefruit, strawberry, and apple) on the photodegradation of chlorpyrifos methyl was studied. Chlorpyrifos methyl is a broad-spectrum pesticide with relatively low toxicity and persistence. The photodegradation experiments were conducted exposing the chlorpyrifos methyl to light of xenon lamp in the presence of a film of wax extracted from the fruit surface. The chlorpyrifos methyl decay rate was calculated as a pseudo first order kinetic. Apple, grapefruit and strawberry waxes decrease the photodegradation rate compared to control. The half-life of pesticide irradiated without waxes was 9.6min, while in presence apple, grapefruit and strawberry waxes it was of 83, 34, and 26 min, respectively. Some compounds present in the waxes probably absorb radiations and may act as a filterreducing the decay rate. During the photodegradation experiments the main metabolite TCP was measured but was more photolytically unstable than chlorpyrifos-methyl.

**Keywords :** *Epicuticular waxes, fruits, photodegradation, chlorpyrifos methyl*

**F-12**  
**Effect of Fruit Cuticle and Epicuticular Waxes on  
Pesticide Residue Sorption**

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The effect of cuticle and epicuticular waxes of three fruits (grapefruit, strawberry, and apple) on the penetration of chlorpyrifos methyl was studied. Chlorpyrifos methyl is a broad-spectrum pesticide of relatively low toxicity and persistence. Fruits with and without wax layers, were treated with an aqueous suspension of pesticide. After 24 hours-contact the residue penetration from the cuticle to the pulp was measured. Fruit samples without waxes showed a higher total amount of chlorpyrifos methyl. Penetration rate was then depending from the species. This pesticide penetration into the grapefruit and apple was lower, probably due to the structure of their cuticle and the chlorpyrifos methyl residues weren't detected into their pulp, while they were determined in all the fractions of strawberry. The highest penetration and accumulation of pesticide into the pulp of no-waxes strawberries was measured. The waxes and cuticle seem affect the pesticide penetration and are a rate-limiting barrier of pesticides.

**Keywords :** *Epicuticular wax, fruit, chlorpyrifos ethyl, sorption*

F-13

**Uncertainty Estimation of Sample Processing in Pesticide  
Residue Analysis**

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In accordance with the new requirement of ISO 17025 Standard (Anon, 1999) and the recently approved Codex Standard (Codex Secretariat, 2003) testing the efficiency of sample processing shall be included in the general requirements of method validation and internal quality control of a laboratory. Our objective was to set up a robust method for determination of the uncertainty of sample processing and to present some results that highlighted its importance. Cucumbers, jackfruit, and papaya and star fruit were selected as representative matrix. The method used was based on the surface treatment of crops with radio-labelled chlorophosphorus. The samples were processed with Stephan Chopper at ambient temperature. Replicate analytical portions of different size were withdrawn from homogenised material and extracted with EtAc. Determination was carried out by Liquid Scintillation Counter. Study describes calculation of recoveries, the statistical elaboration of data, estimation of the sampling constant introduced by Wallacae and Kratachovil (1987) and its use in estimation of sampling uncertainty. Our results showed that, the use of radio labelled compounds gives a great advantage, since precise (typical relative standard deviations 1.04% of 130 sets of measurements) and quick direct determinations of the analyte in the extract could be taken with, and thus eliminating the effects of the rest of the analytical procedure. When the processed sample was statistically well mixed, the efficiency of sample processing was characterised by sampling constant ( $K_s = W \cdot CV_{sp}^2$ ). Typical sampling constant was calculated as 1.89 kg for Stephan Chopper at ambient temperature. This concept was used to predict the uncertainty of sample processing (CVSP) for different test portion sizes. A method of analysis that uses a 5 g analytical portion may have great advantages in terms of solvent consumption and analysis time, but it is not the best choice in terms of uncertainty of sample processing. It was observed that if representative section from each unit can be taken, relative uncertainty around 8% may be expected with Stephan Chopper for 30 g analytical portion. On the other hand analysis a 100 g or 150 g analytical portion does not improve the situation in terms of uncertainty, it increases costs.

**Keywords :** *Pesticide, uncertainty, sample processing*

**F-14**

**Fate of Endosulfan and Deltamethrin Residues During  
Tomato Paste Production**

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Fate of Endosulfan and Deltamethrin Residues during Tomato Paste Production In this study, the effects of tomato paste processing steps on pesticides with active ingredient endosulfan and deltamethrin were investigated in Biga/Canakkale. Residue data were obtained by analyzing samples taken during harvesting, taken after washing and chopping, taken after pulping (pulp and pomace) and taken from the tomato paste with GCC. Data were analyzed using SAS V8. In the process of making tomato paste, washing decreased endosulfan and deltamethrin, 30.62% and 47.58%, respectively. Pre-heating, pulping, evaporation and half-pasteurization increased deltamethrin 2.33% while decreasing endosulfan 66.5% after washing. The whole process decreased endosulfan and deltamethrin, 76.8% and 46.3%, respectively. The residues were mostly collected in pomace.

**Keywords :** *Endosulfan, deltamethrin, pesticide residue, GC/ECD, tomato paste*

**F-15**

**The Fate of Chlorpyrifos-ethyl During Processing  
Procedures in Apples**

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Pesticides are commonly used for protecting apple from pests and diseases. Apples are also consumed as processed product. Pesticide behavior changes during processing procedures. In this work, the fate of chlorpyrifos-ethyl was investigated during processing procedures such as washing, peeling, cooking and juice extraction. Field treatments were carried out on Starking variety of apple in a farmer garden in Denizli province, well known apple producing. National Instruction Manual was taken into account on pesticide treatments. The pests were controlled according to Forecasting System. Consequently, six sprayings against Codling moth was required. The pesticide was applied to three plots (three repetitions). Samples were randomly collected as 30 kg from all parts of trees every plot 3 days and 14 days after last application and mixed thoroughly. They were processed in same day. Having been processed, the samples were homogenized in kitchen robot and kept in a jar in deep freeze at  $-20\text{ }^{\circ}\text{C}$ . The residue of chlorpyrifos-ethyl was investigated during processing steps as; harvested apples, apples washed with water, peeled apple, apple juice and apple puree. A GC (FPD) method was used for residue analyses. As a result of analyses, chlorpyrifos-ethyl residues 3 days after application on harvested apples, washed apples, peeled apple and apple puree were 0,472, 0,475, 0,016 and 0,156 mg/kg, respectively. The residue level of chlorpyrifos-ethyl in apple juice was below LOD (0.001mg/kg). Chlorpyrifos-ethyl residues 14 days after application on harvested apples, washed apples and apple puree were 0,396, 0,366 and 0,096 mg/kg, respectively. The residues of chlorpyrifos-ethyl in peeled apple and apple juice were below LOD (0.001mg/kg).

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**Keywords :** *Apple, pesticide, chlorpyrifos-ethyl, residue, GC(FPD)*



**F-16**

**Behaviour of Some Organophosphorus Pesticide  
Residues in Peppermint Tea During Infusion Process**

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Abstract In order to investigate dissipation behaviour of malathion, fenitrothion, dimethoate, chlorpyrifos and pirimiphos-ethyl during infusion process, pesticide-free dried peppermint leaves were spiked with the pesticides. Infusions were prepared according to the usual process of infusion preparation. The effect of infusion process on the transfer of the pesticides from the spiked peppermint leaves into brew was examined at intervals of 5, 10, 15 and 20 minutes. Residues were determined using gas chromatography equipped with a FID. The decrease in pesticide levels during infusion was found to be statistically significant ( $p < 0.05$ ). Transfer of residues decreased significantly with infusion time. The carryover of the residues of dimethoate, which has the highest water solubility, into the infusion was the highest. Satisfactory relationships were found between water solubility ( $W_s$ ), partition coefficient ( $K_{ow}$ ) and Henry's Law constant ( $H$ ) of the pesticides with the transfer of pesticides to brewed tea. It was observed that not only water solubility or  $K_{ow}$  but also  $H$  controls the dissipation of the pesticides from water or their air-water partitioning.

**Keywords :** *Malathion, fenitrothion, dimethoate, chlorpyrifos, pirimiphos-ethyl, peppermint tea*

**F-17**

**Determination of Carbendazim in Cherry Samples from  
Domestic Market**

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The occurrence of carbendazim in 65 cherry samples from local market during 2004 was investigated. A reverse phase high performance liquid chromatography method was used for determining carbendazim residues. The residues were extracted with ethyl-acetate followed by liquid-liquid extraction clean-up. The fungicide was then separated on RP column using mobile phase acetonitrile-water (40:60) and finally determined by UV detection. The method of standard addition was used for quantitative analysis. Its performance was evaluated and validated: the detection limit (UV-Vis detection  $\lambda = 285$  nm) was 0,03mg/kg, the relative standard deviations were 0,35 and 1,80% and the recoveries were between 73,6 to 88,4% at the 0,20, 0,80 and 2,00 mg/kg fortification levels. Concentration range was from 0,080 to 2,650 mg/kg. Only four samples exceed MRL for carbendazim ( 2 mg/kg). Positive samples were confirmed by normal phase chromatography using Diol column and determined by fluorescence detection. ( $\lambda_{ex}=280$  nm  $\lambda_{em}=310$  nm).

**Keywords :** *Carbendazim, HPLC, cherry*

**F-18**

**Determination of Iprodione, Procymidone and Vinclozolin  
in Cherry Samples from Domestic Market**

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A reverse phase high performance liquid chromatography method was used for determining iprodione, procymidone and vinclozolin in 65 cherry samples from local market during 2004. The residues were extracted with ethyl-acetate following by solvent exchange with acetonitrile. The fungicides were then separated on Zorbax Eclipse XDB column using an acetonitrile-water in gradient mode and finally determined by DAD. The method of standard addition was used for quantitative analysis. Its performance was evaluated and validated: the detection limits ( $\lambda = 222 \text{ nm}$ ) were 0,01mg/kg, 0,01mg/kg and 0,05mg/kg for iprodione, procymidone and vinclozolin respectively. The relative standard deviations were 0,73 and 3,61% and the recoveries were between 83,7 to 93,2% at the 0,10, 0,50 and 1,00 mg/kg fortification levels. Concentration range was from <0,01 to 0,35 mg/kg. No samples exceed MRLs for iprodione( 10 mg/kg), procymidone(10 mg/kg) and vinclozolin (5 mg/kg).

**Keywords :** *Iprodione, procymidone, vinclozolin, HPLC, cherry*

**F-19**

**Pesticide Residues in Serbian Food Produce, Soil and  
Water**

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Although there is increasing world-wide importance given to issues related to food safety and pesticides, Serbia lacks adequate data on pesticide residues in food commodities, necessary for updating national MRLs and doing risk/hazard evaluations. A similar situation exists regarding pesticide residues in soil and water. A project, »Pesticide residues in food commodities of plant origin, soil and water« funded by the Directorate for Plant Protection was initiated to remedy this problem. The main objectives are:

- a) Creation of a general strategy for residue analysis and establishing the required pesticide residue data base.
- b) Control of of pesticides residues in selected fruit and vegetables at the farm level, domestic market and for export purposes.
- c) Systemization of information and existing data required for up-dating existing regulations for pesticide usage in Serbia and Montenegro.
- d) To enhance the knowledge of agricultural workers and food producers in pesticide usage and food safety.

The project will comprise of three phases but preliminary results of phase I will be reported. This involves a study with 368 samples of 10 food commodities (apples, carrots, cabbage, citrus, sour cherries, raspberries, tomato, peppers, cucumber, raspberries) analyzed for 38 pesticides. General conclusions will be discussed. A more detailed account of the analysis of vegetable samples are described in a separate presentation.

**Keywords : Pesticides, MRLs, regulation, fruit, vegetables**

**F-20**

**Residues of Azoxystrobin from Grapes to Raisins**

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Azoxystrobin is a fungicide of the strobilurin group, introduced in the European Union in 1998 and an MRL of 2 mg/kg was set for grapes. The aim of this work was to assess the magnitude of residues on fresh grapes, on washed berries and on raisins after the following two types of processing. Treatment 1 (commonly applied treatment): submersion for 3 minutes in an aqueous solution of 3% K<sub>2</sub>CO<sub>3</sub> and 1% ethyl oleate, and sun drying for 15 days. Treatment 2: sun drying only for 21 days. The commercial formulation QUADRISO 25% SC was applied according to GAP for two consecutive years on a typical cv. Thomson seedless and a seed-producing clone. Samples were collected at 0, 15 and 21 days postapplication. Residues were analysed using gas chromatography with ECD; recoveries were 86 ± 12% for grapes and 99 ± 15% for raisins. All residues were below the EU MRL, ranging between 0.49 and 1.84 mg/kg. Washing of berries removed 75% of the residue. For seedless grapes processed with treatment 1, residues in the raisins ranged from 0.51 to 1.49 mg/kg. With treatment 2 (21 days) residues were higher, 1.42-2.08 mg/kg, despite the longer drying time. This is consistent with the high proportion of residues removed by washing. Although processing treatment 1 reduced residues considerably, in some cases the residues in raisins were higher than those in fresh grapes due to high (3.2 to 4.4) concentration factors. The residue transfer factors calculated were 0.72 to 2.4. In order to avoid trade problems, a higher MRL for raisins is necessary.

**Keywords : Azoxystrobin, residues, grapes, raisins, processing**

## F-21

### Determination of Endosulfan Residues in Raspberries

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Raspberries are regarded one of the Serbia and Montenegro's most profitable export commodities. Consequently, maximum attention should be given to the technology of raspberry production in order to secure high and stable yields, as well as high quality of produce. Besides the application of optimum quantities of fertilizers, it is necessary to use readily degradable pesticides. Endosulfan is a cyclodiene organochlorine insecticide widely used on fruit and vegetables to control sucking, chewing and boring insects. Endosulfan is a mixture of two stereoisomers: alpha-endosulfan and beta-endosulfan.

Endosulfan residues were extracted from raspberries with acetonitrile and the mixture was blended with an Omnimixer. The resulting slurry was filtered through Celit 545 into a cylinder containing sodium chloride. The cylinder was shaken vigorously and phases were allowed to separate. Coextractives were removed by C18 solid-phase extraction cleanup cartridge (Supelco part no. 57012), followed by second cleanup on ENVI Carb cartridge (Supelco part no. 57094) coupled to an amino propyl cartridge (Macherey-Nagel part no. 730180). The tube was eluted with a mixture of acetonitrile/toluene and the solvent was changed to acetone using rotovap. The final extract was analyzed by capillary gas chromatography with Electron Capture Detection. The separation was achieved using HP 5890 gas-chromatograph and DB-5MS column (Agilent part. No 123-5563).

The method was validated on its specificity, accuracy, repeatability, linearity of the detector response and limit of detection (LOD). The response of endosulfan is linear in the range from 0.002 µg/ml to 1 µg /ml. A limit of detection is 0.005 mg/kg. The average recovery for endosulfan on raspberries is 96.79% (n=5, RSD 7%) at fortification level of 0.05 mg/kg, and 128.17% at fortification level of 0.01 mg/kg (n=5, RSD 35%).

In this study endosulfan residues have been monitored in the period from 2000-2004. The number of analyzed samples was 79. Endosulfan was found in 53.16% analyzed samples. Their concentrations ranged from 0.004 mg/kg to 2.122 mg/kg. The concentration of the endosulfan exceeded the EU MRL value, which is 0.05 mg/kg for raspberries, in 16.45% of all analyzed raspberry samples.

References

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**Keywords :** *Pesticides, residues, raspberries*

**F-22**

**Disappearance of Three IGRs Residues in Pepper and  
Zucchini by Vegetable Freezing**

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The influence of food industry freezing process on the degradation of Insect Growth Regulator Pesticides was studied to discover the levels of residues which could reach consumers and suppose human risks.

Flufenoxuron and Lufenuron were applied to pepper and Pyriproxifen was used in pepper and zucchini, because it is one of the most representative situations of agriculture in Southeast Spain. The treatments were carried out according to the commercial product licenses. Good Practice Laboratory was followed and two types of treatments, one for Good Practice Agriculture (GPA) and the other for the critical one (cGPA), were done. The freezing process had place like in a usual factory, with two minute steps: washing up with water; heating up at 95-98°C; cooling down with sodium hypochlorite at 100 ppm in water and freezing in food bags at -18°C during one month. Analyses were practiced in each of them and in the treatment days for GPA and cGPA; and in the first, third or seventh day from the GPA treatment.

As results, neither Pyriproxifen in zucchini nor Lufenuron in pepper were found at levels over the LOQ (0.05 ppm). Pyriproxifen was found at 0.05 and 0.16 ppm in pepper for the initial and first day and Flufenoxuron in pepper at 0.07, 0.12, 0.05, 0.06 ppm for the initial, first and third day and initial cGPA, respectively.

In the end, low levels of residues are left in these vegetables after treatment and freezing process, although they could contact with consumer with our continued diet.

**Keywords : *Pepper, zucchini, disappearance, freezing***



**F-23**

**Influence of Modified Atmosphere Processing (MAP) in  
the Elimination of IGRs Residues in Cucumber and  
Lettuce**

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The influence of food industry Modified Atmosphere Processing (MAP) on the degradation of Insect Growth Regulator Pesticides was studied to discover the levels of residues which could reach consumers and suppose human risks.

Flufenoxuron was applied to lettuce and cucumber, because it is one of the most representative situations of agriculture in Southeast Spain. The treatments were carried out according to the commercial product license. Good Practice Laboratory was followed and two types of treatments, one for Good Practice Agriculture (GPA) and the other for the critical one (cGPA), were done. The MAP had place like in a usual factory, with two minute steps: washing up with water; washing up with sodium hypochlorite at 100 ppm in water; washing up with water again; centrifugation at 800 rpm for five minutes only for lettuce and modified atmosphere packing with Biogon. Analyses were practiced in each of the steps, in the fifth and tenth day from the packing, and in the treatment days, for GPA and cGPA cases; and in the first, third and seventh day from the GPA treatment.

As results, Flufenoxuron was not found in cucumber at levels over the LOQ (0.05 ppm) and in lettuce the residues were between 0.50-0.09 and 1-0.30 ppm approximately, for GPA and cGPA food processes, respectively.

In the end, low levels of residues are left in these vegetables after MAP, although they could contact with consumer with our continued diet.

**Keywords :** *MAP, elimination, IGR, lettuce, cucumber*

**F-24**

**Pesticides in Wheat, Their Health Hazards and Alternative  
Pest Control Measures**

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Pesticides used in order to protect grains against insects, mites, rodents, pathogens and diseases, on the other hand cause adverse effects on human beings, environment, birds and animals. Although pesticides used in wheat control wheat diseases effectively, other pest control measures for crop management have to be discussed for safer crop production. Alternative pest control measures are organic agriculture, Integrated Pest Management (IPM), biological control and biotechnology. Among these systems, IPM that keeps pest population within tolerable limits and increases crop yield has received the most attention. In this study, the pesticides used in wheat and other grains, their health effects on human beings and alternative pest control measures are discussed.

**Keywords :** *Pesticides, pest control, organic agriculture, IPM, wheat*

**F-25**  
**Italian Progress to Face Minor Crops Problem**

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In the European Community a crop or a product is classified as a major if: daily dietary intake contribution is; 7,5 g for a 60 kg person and/or cultivation area is; 10000 ha and the production is; 200000 tonnes per years. Other crop are classified as minor or very minor crops. Since minor uses are of minor importance to industry in all Member States of the E.U. there are serious problems regarding the legal availability for use of plant protection products on minor crop and for other minor uses. In 2004 Italian regions decided to spend the 20% of funding of Project "Residue control of pesticide with aim of rationalising the crop protection" trying to solve the problem of minor crop uses. So a Commission was formed with members of Ministry of Agriculture, Ministry of Health, Plant protection Services, Farmer's organisations, Agrochemical companies under co-ordination of Plant Pathology Research Institute. The Region wish lists on minor crop with possible solution were collected and the Commission evaluated them and established work's criteria to select priority needs. A working group of experts (authors of this abstract) devised a priority table with problematic crop, pest- diseases- weeds and active substances for solving the matter. The most problematic crop are: chestnut, pistachio, plume tree, blackberry, raspberry, blueberry, fig, persimmon, beetroot, radish, cauliflower, chicory, valerianella, spinach, beet leaf, basil, mint, parsley, tarragon, rucola, asparagus, artichoke, fennel, leek, celery, caper, saffron and sorghum. On the basis of the priority table approved by the Commission, works are in progress to produce studies for registration. Also a rapid procedure for authorisation will be supported

**Keywords :** *Minor crops, registration, priority list*

**F-26**

**Studies on the Effect of Processing on Pesticide Residues in Plant Products Intended for Baby Food**

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The health protection of infants and little children is priority problem in EU. Very strict limit of 0.01 mg/kg has been established for any individual pesticide in cereal-based food and baby food.

The study was aimed at evaluation of processing parameters efficiency for minimization of residues in potentially contaminated raw commodities intended for baby food. Two commodities of importance for baby food production

–wheat grain and peaches were selected and various combinations commodity/pesticides were included in the study. The study was focused on the effect of storage intervals and milling on dissipation and distribution in processed products of chlorpyrifos-methyl, pirimiphos-methyl and deltamethrin applied post harvest on wheat grain. Samples of whole grain and eight products of milling

–bran, semolina, groats (3 fractions), flour (3 fractions) were analyzed during 9 months storage period.

The effect of processing of peaches to baby puree on residues of chlorpyrifos-methyl, fenitrothion, procimidone and vinclozolin applied preharvest in the field was studied. Samples taken at the crucial steps of the technological process were analyzed and the results were discussed for evaluation of particular procedures efficiency on pesticide residues dissipation. The data obtained in this study give a reason to propose prohibition of the use of post-harvest treated wheat grain for baby foods.

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**Keywords : *Pesticide residues, baby food, wheat grain, peaches, processing***

**F-27**

**Determination of the Pre-Harvest Intervals of Iprodion,  
Vinclozolin and Imazalil in Raisin**

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Botrytis cinerea Pers. is one of the most important pathogens of vineyards in Turkey. To control B. cinerea, repeated applications of different fungicides are applied during the growing season. Dicarboximide fungicides such as procymidone, iprodion and imidazole fungicides such as imazalil are most often used for protection of different fruits and vegetables and especially of grapes from fungus as: B. cinerea. The main aim of this work was to study the degradation rates of these fungicide. Formulated products of procymidone (Sumiscler 50 WP), iprodione (Rovral 50 WP), imazalil (Magnate 50 EC) were applied on vineyard at recommended rates. Vineyards were sprayed tree times within a summer of two months. Analyses were carried out on the samples, which were taken 0, 3, 7, 14, 21 and 28 day after treatment and were dried. Residues for iprodion and procymidone were extracted from samples with acetone, followed by partition with dichloromethane and ethyl acetate. Detection limit of both fungicides was 0.004 mg/kg and the recovery was 86-91%. Residues for imazalil were extracted with hexane: ethyl acetate (90:10). Detection limit of imazalil was 0.003 mg/kg and the recovery was 80%. All of the samples were analysed by GC using capillary column equipment with a NP detector. Degradation equations were found to be  $\log y = 0,741 - 0,028 x$  ( $y = \text{mg/kg residue}$ ;  $x = \text{day}$ ;  $r = -0,995$ , for procymidone),  $\log y = 0,588 - 0,023 x$  ( $r = -0,968$ , for iprodione),  $\log y = -0,48 - 0,045 x$  ( $r = -0,932$ , for imazalil).

**Keywords :** *Degradation, iprodion, vinclozolin, imazalil in raisin*

**F-28**

**Short-Term Dissipation Curves for Fungicides Used in  
Tomato Crops from Gran Canaria, Canary Islands, Spain**

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Several dissipation curves have been carried out with four different anti-Botrytis fungicides, Cyprodinil, Fludioxonil, Procymidone and Iprodione in four different tomato farms at Gran Canaria, Canary Islands. A short period of study, less than one week, has been considered to get estimations for the 50% and the 90% dissipation of the total active substance applied. The four fungicides assayed has been selected due to their traditional use, Procymidone and Iprodione, and for their recently introduction, Cyprodinil and Fludioxinil. These four active substances are currently in correct use for Tomato crops. Iprodione is actually included in the Annex I of the Council Directive 91/414 and, Procymidone, Cyprodinil and Fludioxonil are included in the list of active substances under revision. A total of 22 short-term dissipation curves have been estimated from the results of the pesticide residue analysis from the samples collected during the monitoring in the four farms. The results showed maximum residues for the studied pesticides of 0.25, 0.55, 0.75 and 1.70 for Fludioxonil, Cyprodinil, Procymidone and Iprodione respectively, and DT50 and DT90 parameters below 2 and 7 days for DT50 and DT90 respectively for all fungicides except for Procymidone with 3 and 9 days for DT50 and DT90. Taking a brief look to the security periods and MRLs for these fungicides, we can accept 7 days as a broad safe period in Gran Canaria for these four fungicides application in Tomato crops. Shorter security periods could be even evaluated based upon these preliminary results.

**Keywords : *Tomato, Cyprodinil, Fludioxonil, Iprodione, Procymidone, dissipation curve***

F-29

**Short-Term Dissipation Curves for Buprofezin and  
Tebufenpyrad in Tomato Crops from Gran Canaria,  
Canary Islands, Spain**

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The most used acaricide for the tomato cultivations in the Canary Islands has been Bromopropylate. Recently, the Council Directive 91/414/EEC has banned the use of this active substance in Europe. However, the Commission Regulation (EC) 2076/2002 has evaluated the use period referred in the Council Directive 91/414 for several active substances, considering essential uses and extending the time period of use. In Spain, the use of Bromopropylate has been extended until June 30th 2006 with essential use in tomato crops. The Commission Directive 2004/59/EC established a Maximum Residue Limit of 1.00 mg/kg for Bromopropylate in tomatoes. Therefore, it is necessary to look for an alternative to Bromopropylate to be used in the tomato crop. Buprofezin and Tebufenpyrad have been introduced recently in the Canary Islands as new active substances. A preliminary study has been carried out to estimate short-term, less than one week, dissipation curves of Buprofezin and Tebufenpyrad in tomato cultivations. A total of 2 dissipation curves for Buprofezin and 3 for Tebufenpyrad have been conducted. The maximum residues detected after the pesticide application, following the usual doses, were 0.20 mg/kg for Buprofezin and 0.60 mg/kg for Tebufenpyrad, while the estimated DT50 and DT90 for both active substances have shown values lower than 2 and 5 days respectively. The security period for both, Buprofezin and Tebufenpyrad, fixed in Spain is 7 days.

**Keywords :** *Tomato, Buprofezin, Tebufenpyrad, dissipation curve*

**F-30**

**Dissipation Curve of Fenitrothion for Banana in Gran  
Canaria, Canary Islands, Spain**

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Banana crop is the most important agricultural activity in the Canary Islands. However, this crop is a minor one in the European list of agricultural activities. The application of the Council Directive 91/414/EEC in addition to the low interest of the agrochemicals companies to defend pesticides to be used in minor crops has reduced the broad spectrum of possible insecticides to be applied in banana to a short list of active substances, a total of seven have authorisation today. One of these pesticides is the insecticide Fenitrothion, with a security period of 15 days, it is one of the two insecticides authorised to be used against the pest commonly named "taladro" (*Opogona sacchari*). Besides, Fenitrothion is authorised to be applied for other pests in banana. In the present effort, a dissipation curve for Fenitrothion has been carried out in banana cultivation as a preliminary study for future rigorous characterisation of dissipation curves of pesticides used in the banana crop in the Canary Island. A 21 days field experiment was conducted and the dissipation curve estimated shows a period of 5 days for the dissipation of the 50% of the initial residue detected and a period of 16 days for the dissipation of the 90% of the initial residue. After 21 days, the residue of Fenitrothion in banana was 0.01 mg/kg.

**Keywords :** *Banana, Fenitrothion, dissipation curve*



**F-31**

**Dissipation Curves of Fungicides for Grapes in Tenerife,  
Canary Islands, Spain**

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A total of 19 dissipation curves for seven different fungicides have been estimated in 8 different locations in the North of the Island of Tenerife during the ripening period of the grapes in the summer of the year 2004. The estimated dissipation curves corresponds to the last two pesticide applications before the grape harvest. All the fungicides studied were applied against Botrytis. The most used fungicide for this case-scenario has been Procymidone. However, other fungicides have been studied as alternatives or as a better option, based upon the pest threat conditions, to be. Nine of the dissipation curves were for Procymidone. The other ten were for Cyprodinil, Fludioxonil, Kresoxim-methyl and Pyrimethanil, two estimations each one, and one curve for both, Iprodione and Vinclozolin. The averaged periods of time needed for the dissipation of the 50% and the 90% of each applied fungicide, DT50 and DT90, were between 15 and 21 days and between 30 and 75 days respectively. Influences of the initial residue levels, environmental temperature, grape variety and the monitoring time for each dissipation curve are discussed in the present effort. The Maximum residue levels, MRLs, for each fungicide, lead to accept security periods between 15 and 30 days as acceptable before the harvest.

**Keywords :** *Grapes, Fungicides, Procymidone, dissipation curve*

**F-32**

**Dissipation Curve of Insecticides for Grapes in Tenerife,  
Canary Islands, Spain**

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Field dissipation curves for the two most used Insecticides, Malathion and Fenitrothion, during the ripening period of grapes in the North of the Island of Tenerife, have been studied. The dissipation curves came from 8 eight different farms for Malathion and from 6 different farms for Fenitrothion. In all cases, there was no experimental controlled application conditions, therefore the initial residue levels depends upon each farmer application performance. The initial residue levels were between 2.50 and 8.50 mg/kg for Fenitrothion and between 1.00 and 7.00 mg/kg for Malathion. Using the correct application doses, the initial residue level should be between 2.00 to 3.00 mg/kg for both. The period of time needed for the dissipation of the 50% and the 90% of the applied insecticides, DT50 and DT90, were between 3 to 8 days and 10 to 27 days for Fenitrothion and, between 4 to 9 and 14 to 30 days for Malathion. The average DT50 and DT90 were 6 and 17 days for both active substances, showing a coefficient of variation of 35% for the all cases studied. The results lead to the conclusion that a security period of one week, 7 days, is enough to the dissipation of the 50% of the total insecticide applied, and a security period of three weeks, 21 days, is enough to a minimum residue of both. However, to assure that the residue levels will be below the MRL, 0.5 mg/kg for both insecticides, after three weeks, it is necessary a correct application dose.

**Keywords :** *Grapes, Malathion, Fenitrothion, dissipation curve*

**F-33**

**Monitoring of Copper Residues in Grapes from Tenerife,  
Canary Islands, Spain**

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Monitoring of inorganic copper residues in grapes from the north of the Island of Tenerife was carried out during the ripening period in the summer of 2004. A total of 16 farms were monitored for copper residues, including farms where applications of fungicides based upon different chemical forms of copper and farms where copper was not applied. The maximum residue level, MRL, for inorganic copper is 20 mg/kg. After pesticide applications including fungicides based upon copper, usual blue colouring and sometimes blue spots could be observed in the grapes, besides the higher levels of copper residues have been found when more than two treatments were applied during the ripening period. As intense the blue colouring and spots are, as high the applied dose of copper is. By the end of the ripening period, usually three or four weeks after the last pesticide application, if blue spots still could be observed in the grapes, high levels of copper will be found. During the monitoring performed in this effort, a good correlation between blue spots observed by visual inspections and residue levels above 10 mg/kg have been observed. In the North of Tenerife, no more than two applications of copper fungicides are recommended for grapes at the beginning of the ripening period.

**Keywords :** *Grapes, Copper*

**E-01**

**Pesticide Using in Agriculture in Şanlıurfa**

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SAP (South and East Anatolian Project) region is a large area which has an intensive agricultural production and an increasing food consumption connected with a continuously increasing population. Nowadays as a matter of fact, the aim in agriculture is to get the yield of good quality and high in amount from the unit area. Therefore, pesticides and chemical fertilizers have been used widely in agriculture. Distribution of different crops in the agricultural area in Sanliurfa and the annual amount of pesticides that has been used on the crops for this purpose during the SAP period has been researched on this study. The fate of pesticide species in environment that were used at maximum amount on the large fields and the effect of pesticide residues in environment have been also investigated. The solutions to prevent the pesticide contamination in environment have been submitted in the research. To educate the farmers and the conscious pesticide application, to research the natural products and to support the studying on this research were the priority solutions on the conclusion.

**Keywords:** *Pesticide, SAP, region, crop, contamination*

**E-02**

**Monitoring of Fungicide Carbendazim in the Surface  
Waters of the Lake of Albufera, Valencia, Spain**

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Albufera Natural Park sited in Valencia (Spain) is a flora and fauna very rich area surrounded by rice fields in which pesticide spraying is a regular practice. The lake has functioned since the last century as a freshwater reservoir for the demands of rice cultivation. The lake water level is regulated by sluices situated at its three outlet channels which flow into the Mediterranean Sea. So that, a knowledge of realistic environmental concentrations of sprayed pesticides has to be taken into account to assess the adverse impacts these chemicals may have on non target organisms in aquatic ecosystems. Carbendazim is very common fungicide in this area for to prevent or control the main rice disease in the ANP, *Pyricularia oryzae* during July and early August. The residue occurrence of this compound in water of the Albufera lake was monitored nine times after fungicide application, during 2003. Water samples were collected from six stations: four located in the lake and two others at their related irrigation channels. They were chosen because they represent important inlets and define two spatial zones of the lake with different nutrient loadings. Water samples were also collected from an experimental rice plot. Solid phase extraction procedure and chromatographic techniques were applied to quantify carbendazim. The results showed that water pollution due to carbendazim residues was in the same level in the case of lake and channels. In general, the highest values in samples of Albufera lake were obtained in the second week of August, 2,19 and 4,97 µg/L, in the sampling points of Overa channel and in the middle of the lake, respectively.

**Keywords :** *Carbendazim, Natural Park, monitoring, pesticides*

**E-03**

**Determination of Pesticides in Soils by GC/NPD**

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A multiresidue method for the simultaneous determination of 25 pesticides in soil was developed. Soil samples are extracted by sonication with a water-acetonitrile mixture and the pesticides are partitioned into dichloromethane. Final determination is by gas chromatography (GC) with nitrogen-phosphorus detection (NPD). The identity of all compounds was confirmed by GC with mass selective detection (MSD) in the selected-ion monitoring mode. Pesticides were confirmed by their retention times and their qualifier and target ions. The average recovery through the method obtained for these compounds varied from 72.3 to 119.9% with a relative standard deviation between 1.8 and 6.2%. The method is linear over the range assayed 50 – 2000 µg/L and the detection limit for the different pesticides studied varied from 0.1 to 10.4 µg/Kg. The proposed method is rapid, simple and sensitive, requiring small volumes of solvents. Another advantage of the method is the application to the analysis of pesticides in soil samples collected in greenhouses of peppers from the Region of Murcia.

**Keywords :** *Environmental analysis, soil, pesticides*

**E-04**

**Effects of Exogenous Dissolved Organic Matter on  
Fenhexamid Sorption by Soils**

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Fenhexamid (N-(2,3-dichloro-4-hydroxyphenyl)-1-methyl-cyclohexanecarboxamide, Teldor®) is a fungicide representative of the new chemical class of hydroxanilides. It is a locosystemic compound with excellent activity against *B. cinerea*. Dissolved organic matter (DOM) plays an important role on the mobility of pollutants in environment. The aim of this study was to investigate fenhexamid adsorption on soils after addition of DOM coming from different composts. Two soils were chosen, a calcareous soil with low organic matter (OM) content and an acidic soil rich in OM. The empirical Freundlich equation describes well the adsorption behaviour of fenhexamid on soils. The adsorption isotherms on calcareous soil resemble L-type isotherms characteristic of a high affinity of the fungicide for the adsorption sites. The adsorption on acidic soil shows a C-type isotherm suggesting a constant partitioning of adsorbate between solution and adsorbent sites. In general, the DOM addition to soils increases fenhexamid adsorption. In fact, the Freundlich constant (KF) increases from 4.86 to 6.70-8.16 on calcareous soil and from 35.2 to 44.1-49.2 on acidic soil. FT-IR spectra indicate that the fungicide does not undergo significant changes as a consequence of the adsorption. These findings are indicative of a weak interaction like a hydrophobic bond. The fungicide adsorption on soils is hysteretic and the amount of fenhexamid desorbed by water decreases as the soil OM content increases. Fenhexamid is scarcely soluble in water because of its scarcely polar character, therefore water is not suitable as extracting solvent. A strong increase of desorbed fenhexamid is obtained using dichloromethane as desorbing solvent.

**Keywords :** *Fenhexamid, fungicide, adsorption, desorption, dissolved organic matter, soil*

**E-05**

**Study of Adsorption of the Bio Insecticide "Spinosad"  
on Three Moroccan Soils**

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Spinosad is a mixture of the two most active naturally occurring metabolites (spinosyn A + spinosyn D) produced by a new species of; *Saccharopolyspora spinosa* isolated in the soil[1]. The sorption study of Spinosad was carried out on three soils representative of the intensive agriculture in Morocco. The soils chosen for this study have distinct physicochemical characteristics in terms of organic matter and other soil constituents. Two grams of air-dried soil sieved to 2 mm is placed in centrifuge tube containing an aqueous solution of Spinosad with pesticide concentration varying from 1 to 20 µg/ml. Tubes are agitated for 24 hours. This period is necessary to reach sorption equilibrium as determined in a preliminary test. After centrifugation, the adsorbed quantity was determined by HPLC/UV analysis of the supernatant. The obtained results show that the kinetics of adsorption are fast and corresponding isotherms are S type. The isotherms of adsorption follow the Freundlich model. The study of relationship between the obtained parameters of adsorption and the physicochemical characteristics of the various soils indicate that the role of the clay-humic complexes is determinant in the phenomenon of Spinosad adsorption on the soil. The results obtained in this study, enable to conclude that Spinosad is probably not very mobile in the studied soils, and can provide consequently a solution of replacement of the chemical pesticides such as the organophosphorus. [1]: Boek L.D., C. Hang, T.E. Eaton, O.W. Godfrey, K.H. Michel, W.M. Nakatsukasa, and Yao R.C. 1994 Process for producing A 83543 compounds. US Patent N° 5,362,634. Assigned to Dow Elanco. (Dow Agro Science).

**Keywords : Adsorption, bio insecticide, Spinosad, isotherms of adsorption, mobility**



**E-06**

**Pesticide Intoxications Detected by Two Years Study in  
Turkey**

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Pesticides are quite widely used in agriculture in Turkey as used in the world. Depending on this, intoxications are faced in animals. Although some of these cases are seen accidental consumption of or subjection to the pesticides, it is thought that most cases occur due to purposely usage of pesticides to kill animals. In Central Veterinary Control and Research Institute Toxicology Laboratory, pesticides analyses are routinely carried out. In the present study, the analyses results of the animal tissue and feed samples sent with the suspicion of intoxication in 2003 and 2004 were evaluated. Total 216 samples were analyzed in 2003 and several pesticides were detected in 39 samples (18,05%) by GC and GC-MS. It was shown that Endosulfan was the most often encountered pesticide and it was detected in 27 (12,5%) cases. Methomyl was found in 6 (2,7%) cases; and Tetramethrin, Phenothrin, Trifluralin, Carbaryl, Chlorpyrifos and Parathion-methyl in 1 (0,40%) case each. Total sample number analyzed in 2004 was 246 and 42 (17,07%) cases were found to be positive regarding to several pesticides. Endosulfan was the most commonly used pesticide as in 2003 and it was detected in 28 (11,38%) cases; Cypermethrin in 8 (3,25%); Methomyl in 2 (0,81%); and Malathion, Parathion-methyl, Chlorpyrifos and Tetramethrin in 1 (0,40%) case each. In two years period, total 462 samples sent with the intoxication suspicion were analysed. In 2003 and 2004, according to the pesticides groups, the percentages of intoxications of Organochlorines, Pyrethroids, Carbamates and Organophosphorus were 11,90 (55 cases); 2,38 (11 cases); 2,16 (10 cases) and 1,08 (5 cases), respectively. And in one case (0,22%), Herbicide intoxication was detected. According to the present study, it was detected that 17,53% of death in some species of animals were caused by several pesticides in the cases sent with intoxication suspicion.

**Keywords : Pesticides, intoxication, animals, feed, GC, GC-MS.**

**E-07**

**Biodegradation of the (2,4-dichlorophenoxyacetic acid) by  
Pure and Mixed Bacterial Cultures**

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In this research, biodegradation of 2,4-D which is a herbicide frequently used in agricultural area of Eskişehir by mixed bacteria cultures isolated from soil have been examined. Soil samples were collected from four different agricultural areas and two different depths from fields grown wheat. Mixed cultures able to degrade 2,4-D were obtained and these mixed cultures have been used for assays of biodegradation. All of the mixed cultures were able to utilize 2,4-D as the sole source of carbon in the biodegradation medium. Bacteria take part in mixed cultures was identified at species level. According to research results, 8 different mixed cultures and 4 pure bacteria have displayed degradation activity towards 2,4-D in several rates. Mixed bacterial culture designated as MC3, when 2,4-D (500 mg/l) was used in biodegradation medium as single carbon source, degraded 86% of 2,4-D within 5 days. Key Words: 2,4-D, Biodegradation, Mixed Bacterial Culture, *Acinetobacter lwoffii*, *Xanthomonas maltophilia*, *Pseudomonas putida*, *Alcaligenes eutrophus*.

**Keywords : 2,4-D, Biodegradation, Mixed Bacterial Culture,  
*Acinetobacter lwoffii*, *Xanthomonas maltophilia*, *Pseudomonas  
putida*, *Alcaligenes eutrophus***

**E-08**

**Adsorption of 2,4-Dichlorophenoxy Acetic Acid (2,4-D)  
from Water Environment**

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One of the current environmental concern is the contamination of aquatic systems due to pesticide discharges from manufacturing plants, surface runoff, leaching accidental spills and other sources. Indiscriminate use of pesticides in the modern agricultural practices leads to the contamination of soils, surface and ground waters. Among the numerous agrochemicals in use today, the herbicide 2,4-D has been widely applied because of its low cost and good selectivity. However, it is considered as moderately toxic and the maximum allowable concentration is 100 ppb in drinking water. Water utilities have been using powdered activated carbon (PAC) and granular activated carbon (GAC) since the 1930s for taste and odor control. Since the 1970s GAC has played an increasing role in removal of organic molecules from water. In this work the adsorption of 2,4-D from an aqueous solution using PAC was studied. The effect of dissolved organic carbon (DOC) on the adsorption equilibrium was also investigated by using natural organic matter. Adsorption equilibria of the single substance and multisolite system were determined. Consequently ultimate capacity of the PAC for both cases was defined.

**E-09**

**A Model to Predict Pesticide Residue Behaviour in Grape**

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This work attempts to describe a mathematical procedure to predict the concentration of pesticides in the edible part of crop at the time of harvest. The model is applicable for cases where the most important exposure route is by direct spray on the canopy of the crop and where uptake by roots into the plant can be disregarded. The dose arriving on the edible part of the crop is calculated by application rate, taking into account the crop-cover of the whole plant and the crop-cover of the edible part. The loss processes include photodegradation, volatilization, uptake by leaves and wash-off. Photodegradation is considered to occur according to first order kinetics. The cumulative volatilization for the first 7 days is approximated by the empirical equation of Smit and colleagues (1998). Wash-off is calculated as a fraction of the quantity arriving on the edible part of the crop, by taking the amount rainfall (m) times a wash-off factor (m-1). Uptake by leaves is calculated take intoaccount the LogKow. The model was tested against analysis data pesticide residues in grape. The samples originate from six farms in Lombardia province in northern Italy. All data used as model input were provided by farmers involved in this study (Garcia et al. 2004, personal communication). Comparison between model output and analysis data suggest that the results are in the same order of magnitude. This study is performed within the HAIR project (SSPE-CT-2003-501997)

**Keywords :** *Model, residue, pesticide*

**E-10**

**Photocatalytic Degradation of Imazethapyr Herbicide by  
Powder and Supported TiO<sub>2</sub>**

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The imidazolinone herbicides were developed for use in a wide range of crops including oil palms, sugar cane, forestry, cereals, sunflower and lentils. Some imidazolinone herbicides have been tested for weed control in cereal crops and sugar beat in Morocco. The aim of this work was to investigate the photocatalytic degradation of imazethapyr using UV-irradiated PC500-Millennium TiO<sub>2</sub> photocatalyst in suspension or deposited on a paper support. A comparison between the results obtained with the catalyst either as a powder or supported on papers was performed. The effect of the initial concentration of the pesticide on the kinetics of its disappearance was studied, especially with respect to the true reaction order.

**Keywords :** *Imazethapyr photocatalytic degradation, TiO<sub>2</sub>-P25*

## E-11

### Pesticides Dermal Exposure of Workers in Greenhouses

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In this work the risk of pesticide dermal exposure for workers in greenhouse in the summer time is evaluated. Dermal exposure has been evaluated during the growing of some crops in two years (2003 – 2004). In summer 2003, the risk of pesticide dermal exposure, re-entering in greenhouses after treating, has been evaluated for different crops (tomato, strawberry and cucumber) through indirect measures of exposure by Dislodgeable Foliar Residue (DFR) measurements. In summer 2004, dermal exposure has been evaluated directly for the only tomato crop either during treatments and the following greenhouse re-entries. The pesticides monitored were acrinathrin, azoxystrobin, chlorpyrifos ethyl, fenarimol, metalaxyl and tetradiphon in the first year and azoxystrobin, chlorpyrifos ethyl and metalaxyl in the second year. As analytical techniques we used the Gas Chromatography (GC) with electron capture detector (ECD) and Nitrogen-Phosphorous detector (NPD). Indirect evaluation of dermal exposure has been obtained measuring DFR and applying a transfer coefficient (Ct) from the foliar to the dermal surface. Direct evaluation has been instead obtained applying polypropilene patches on worker's different body surface areas. The residues on both patches and cotton gloves worn by worker were quantified and by these values the dermal exposure was directly evaluated. In two cases, direct and indirect evaluation, dermal exposures were transformed in the absorbed dose values (D) through an absorption coefficient (Ca) and the two methodologies were compared each other. Then we built decay curves (dose Vs time function) that, intercepted with the Acceptable Operator Exposure Levels (AOEL) or the Acceptable Daily Intake (ADI), allowed us to evaluate the (acceptable) re-entry times.

**Keywords :** *Pesticides, greenhouses, dermal exposure*

## E-12

### Biodegradation of Cyclodiene Insecticide Endosulfan by *Nocardia* spp

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Endosulfan, a chlorinated cyclodiene insecticide, is of environmental concern because of its apparent persistence and toxicity to many non-target organisms. It is used extensively throughout the world to control the insect pests wide range of crops including cereals, tea, coffee, cotton, fruits, oil seeds and vegetables. The technical endosulfan is a mixture of two stereoisomers, alpha and beta- endosulfan in the ratio of 70:30. Endosulfan is a great concern because of its persistence and extreme toxicity to fish and aquatic invertebrates. Since the deregistration in many countries of most cyclodiene insecticides, the ongoing availability of endosulfan has become important as a alternative option in resistance management strategies of pest species. Because of such abundant usage, and potential for environmental transport, endosulfan contamination is found throughout the environment. Endosulfan has been detected in atmosphere, soils, sediments, surface waters, rain waters and food stuffs. As with most pesticides, the persistence and degradation of endosulfan are affected by the environmental conditions in which is found. Endosulfan does not undergo direct photolysis but is transformed by chemical hydrolysis under alkaline conditions. In soil, endosulfan has been shown to be degraded by a wide variety of microorganisms. However, degradation rates are usually low and metabolism often results in the formation of endosulfan sulfate, an oxidative metabolite shown to be equally as toxic and persistent as the parent compound, endosulfan. Because of its persistence and toxicity, endosulfan contamination poses a significant environmental concern In this study, *Nocardia* spp which was isolated from soil was used for the degradation of Endosulfan in soil. Degradation of soil-polluted endosulfan in uninoculated and inoculated soil by *Nocardia* spp was studied for variable and time point of the study and the results were compared. To study the degradation in soil twelve bakers were maintained. 150 grams of endosulfan spiked soil was added to each baker. Inoculated and uninoculated mineral mediums were mixed with soil. Soil samples were taken weekly for two months. The endosulfan concentrations were determined by GC ECD. The degradation of endosulfan in soil by *Nocardia* spp was evaluated.

**Keywords :** *Biodegradation, Endosulfan, Nocardia Spp, soil*

**E-13**

**Studies on Adsorption of Cyhalofop Buthyl**

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Cyhalofop-butyl (CB) is an herbicide recently introduced for rice crop in Gharb region, dominated with clay soils. Information on its behaviour in the soil is still rare. This work aimed study adsorption of CB on montmorillonite clay free and saturated with different cations (Na<sup>+</sup>, K<sup>+</sup> and Mg<sup>2+</sup>). Clays were prepared by agitation of 15g of commercial montmorillonite in 0,01M aqueous solution of CaCl<sub>2</sub> (100ml) saturated with the given cation for 24h. The complex is then washed with distilled water and dried at 110°C for 48h and stored at ambient temperature. 100 mg of clay complex were agitated in 10 ml of aqueous solution of CB at 15, 10, 2.5, 1 and 0.5 ppm for 24h. Tubes are centrifuged at 2500rpm. The supernatant is extracted with 5 ml of dichloromethane. Residue is transferred to 5 ml methanol for analysis. Isotherms of adsorption were found to fit the linear model of Freundlich ( $\text{Log } x/m = \text{Log } K_f + 1/n_f \text{ Log } C_e$ ), where  $x/m$  represent the ratio of the amount adsorbed on the amount of soil used,  $K_f$  and  $n_f$  are Freundlich coefficient and  $C_e$  stands for equilibrium concentration; with high coefficients of correlation (0.882 – 0.984), Data collected showed that values of  $K_f$  varied from 3.16 to 15.58 for Montmorillonite saturated with Mg<sup>2+</sup> and free cation montmorillonite (FCM) respectively. According to the classification of Giles et al., except for the isotherm of FCM, all the other complexes were L type isotherm. In terms of affinity of CB towards these clays the following order could be proposed: FCM > M – K<sup>+</sup> > M – Na<sup>+</sup> > M-Mg<sup>2+</sup>

**Keywords :** *Cyhalophobuthyl, adsorption, clays, rice*



**E-14**

**Possible Effects of Pesticides on Human Biorhythms**

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Even though international health organizations are striving to bring public awareness about the hazardous effects of pesticides, third world countries are not fully aware of the danger at their social and cultural cognitions. This is unfortunate since many of those countries are heavy-pesticide users. In this presentation we will shed some light on some possible effects of pesticides on human biorhythms. Ultradian Rhythm has been recently targeted as a hot area of study. We will then talk about Ultradian Stress Response (USR) and Ultradian Healing Response (UHR) in relation to pesticidal effects on the human cybernetic system. In particular, we will talk about how some of those pesticides may disrupt psychogenetic, neuroendocrinological, and mind-body synchronization leading to human stress. We will also discuss the role of some cultural and psychosocial conditioning on the values of life and death in these countries.

**Posters Abstracts: Environment**

**E-15**

## Examining the Biological and Biochemical Effects of Erosion on Agricultural Soils of Marand Region (North West of Iran)

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Marand Region is one of the main agricultural regions of Iran where Horticulture and Agriculture have been developed. This research tries to investigate the effects of erosion on the biological and biochemical properties of the soil. Although the biological and biochemical factors existing in the soil have great role in human, animal and plant life, the amount of these material existing in the soil is very low. So they are important in the changing process of the soil. Thus, it can be used as a criterion for the measurement of the quality of the soil in the region; More ever, these factors are very useful for health, and have direct effects on the functions of the most important organs of human beings. It should be mentioned that microelements have basic effects in the process of life and they can be found in the combination of enzymes. Furthermore, microorganisms and microelements have great effect on fertilization of soils and increasing plants , farming and improving their index of qualities, Mu , Cu , I , B , accelerates the physiological and biochemical process in the organs of living being and it functions as catalyze for this process. In this respect an experimental research was done and three sites were selected, each site with qualitatively and quantitatively different soils. And the samples of soils were taken. After wars the amount of activities of the micro organisms, bacteria s, actinomycetes, fungus, and microelements (the biological and biochemical factors) in each sample were tested. Finally it was found that gray brown soils had the best index for fertilization and growth of different plants in the site of selected samples. Therefore the erosion of soil on slops was over 30% with out natural plant cover (less than 60% of region) which resulted indistruction of biological activities, existing in the soil. It is obvious from the figures and statistics that each 35 to 40 tons of soil per hectare are destroying.

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**Keywords :** *Biological and biochemical effects, erosion, agricultural soils*

**E-16**

**Disturbances in Energy Reserves of Daphnia Magna**

## after Exposure to Pesticides

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Tetradifon and Propanil are pesticides extensively used in agriculture for crop protection. These pesticides seem to produce many physiological and biochemical changes, others disturbances of these pesticides were reduction of feeding behaviour in freshwater organisms. Feeding behaviour is an important physiological function because food intake is related to energy reserves and determined de nutritional state of the organisms. Biochemical indicators of stress are potentially useful because of their sensitive to sublethal concentration of chemicals and may provide the earliest possible warning of future adverse effects. Sublethal exposure to different Tetradifon (0.10, 0.18, 0.22 and 0.44 mg/L) and Propanil concentrations (0.07, 0.10, 0.21 and 0.55 mg/L) reduced energy reserves as glycogen, lipids and proteins in *Daphnia magna*. Analysis of glycogen, proteins and total lipids were made after 24, 48, 72, 96 and 120 hours of continuous exposure to both pesticides, these results were used to calculate the energy content in daphnids. The results showed that energy content decreased as concentration of the selected pesticides increased. A reduction of 41% was found after 96 h of Tetradifon exposure (0.44 mg/L) and a 65% after 120 h of Propanil intoxication (0.55 mg/L). Results are discussed in relation with data of a previous study about the effects of these pesticides on feeding behaviour. The results suggest that there are an important relationship between the food intake and energy content in *Daphnia magna*.

**Keywords :** *Daphnia magna*, energy content, biochemical indicators, Tetradifon, Propanil

**E-17**

**Hepatic-Somatic Index (HSI) and Water Content (WC)  
Used for Assessing Fish Exposure to Herbicides**

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Propanil is widely used in the rice crop area of the Albufera Natural Park (Valencia, Spain). This wetland is traditionally inhabited by the European eel. To investigate the sub-lethal effects of propanil, eels from the Albufera Lake were exposed to two herbicide concentrations (1/50 and 1/10LC50) for 3 days. After exposure, animals were transferred to clean water for 6 more days in a recovery period. HSI and WC from liver and muscle were evaluated at 0, 2, 12, 24, 56 and 72 hours of exposure and at 8, 24, 48, 96 and 144 hours of the recovery period. Finally, another experiment was performed as control, to test if the handling of animals and the laboratory conditions had any effect on these parameters. Eels from control experiment did not show any significant difference ( $p > 0.05$ ) in neither HSI nor WC. Results showed that propanil induced significant ( $p < 0.05$ ) increases of HSI during the exposures. Moreover, animals exposed to the highest concentration showed increased HSI during the entire experiment. WC from liver and muscle were also increased ( $p < 0.05$ ). At the end of exposure to 1/10LC50, increases were 166% for muscle and 45% for liver. Once in clean water, WC from liver was recovered rapidly in animals pre-exposed to 1/50LC50 and after 144 h in those exposed to 1/10LC50. At the end of the experiment the WC from muscle remained increased. A raised HSI, joint to an increase in WC, may be suggesting a probable hepatic dysfunction. The obtained results reflect disorders in the homeostasis of the selected tissues as consequence of herbicide presence.

**Keywords : Eel, HSI, WC, propanil, liver**

**E-18**

**Muscular Metabolism of the European Eel Under Propanil Exposure**

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The effects of propanil on certain parameters of muscular metabolism were evaluated in the fish *Anguilla anguilla*. Two groups of eels were exposed to 0.63 and 3.16 ppm for 72 hours in a flow-through system, and later transferred to propanil-free water for 144 hours. An additional group was kept in clean water as controls. Muscle samples were isolated from exposed, recovery and control fish at several times, and used for estimation of glucose, lactate, glycogen and lactate dehydrogenase (LDH) activity. Control animals did not showed differences statistically significant ( $p>0.05$ ) during the entire period. However, propanil induced alterations in those parameters, showing a concentration-dependent response. An increase of both glucose and lactate, during propanil exposure periods, was observed compared with levels at 0 hours. The parameters recovered in eels previously exposed to 0.63 ppm after 24 hours in clean water, but did not do in animals pre-exposed to the highest concentration. Glycogen strongly decreased from the first hours in both experiments and remained decreased after fish transferred to clean water. LDH rapidly increased from the first hours. It exhibited a recovery response after 48 hours in clean water in animals exposed to the lowest propanil concentration, whereas remained perturbed during the entire experiment with 3.16 ppm of propanil. These results reveal that propanil has an important impact on the carbohydrate metabolic pathway at muscular level in the European eel.

**Keywords : *Eel, LDH, Lactate, glucose, Glycogen, propanil, recovery period***

**E-19**

**Behavior of Pirimicarb in Greenhouses of Peppers from  
the Region of Murcia**

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The growing of peppers in greenhouses is one of the main cultivation activities in the Region of Murcia (Spain). Pirimicarb is an aphicide commonly used in the growing of peppers in greenhouse. The contamination of soil and underground water systems by pesticides as a result of agricultural practice is one of today's most worrying environmental issues. Taking this into account, one of the main objectives in this work is to study the behaviour in soil and in peppers of pirimicarb in fields. The study in field, also aims to provide information on the leaching of pirimicarb, primarily with a view to evaluating any possible risk of contamination of underground water systems which might be used for human consumption.

Pirimicarb residues in leaves, waters, peppers and soils were analysed by gas chromatography (GC) with nitrogen-phosphorus (NPD) detectors and with a mass spectrophotometer (MSD). The results showed the possible contamination by pirimicarb of underground water and aquifers.

**Keywords :** *Pirimicarb, degradation, leaching, mobility, persistence, pepper*

**E-20**

**Detection and Degradation of Trifluralin in Soils and Their Uptake by Carrots**

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Commercial grade product of trifluralin (Tefralin EC) was sprayed in dilute solution from at recommended rate to the surface of field plot. The treatment was trifluralin in 1 application. Trifluralin was applied to soils, as spray treatment at rate 2 kg/ha, pre-em. Carrot was grown in weed free plots of soil and treated with pre-em. application of 2 kg/ha trifluralin. Detection of trifluralin were studied in trifluralin-treated soil and sampled over a 23 week period, beginning after spray treatment. Pesticide residue concerns in soil samples taken in the top of 20 cm of the soil profile, at different days after application. Residues were extracted from soil and carrot samples with methanol, followed by partition with n-hexane. Detection limit was 0,002 mg/kg. The recoveries were 100% for soil and 87% for carrot.

The trifluralin concentration was observed to decline from 0,617 to 0,058 mg/kg. Degradation equation was found to be  $\log y = -0,275 - 0,006 x$  ( $y =$  mg/kg residue;  $x =$  day;  $r = -0,920$ ). The persistence of the trifluralin and its residues in carrots (root) at harvest time were studied under field conditions. Residues were lower than the tolerance level in Turkey (0,2 mg/kg).

**Keywords :** *Degradation, trifluralin, soils, carrot*

**M-01**

**Validation of Ethyl Acetate / LC-MS/MS Multiresidue  
Method for the Analysis of Pesticides Residues in Fruits  
and Vegetables.**

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A multiresidue method was validate for the analysis of Acetamiprid, Azoxystrobin, Carbendazim, Demeton-S-methylsulfone, Imidacloprid, Metiocarb, Methomyl, Oxamyl, Oxydemeton-methyl, Spinosad (A+D), Thiabendazole, Thiocloprid and Thiamethoxam in fruits and vegetables. The multiresidue method procedure consists in an organic solvent extraction with Ethyl Acetate (60+30 mL). After the filtering, solvent is evaporated to dryness and reconstituted with Methanol (10 mL). The final volume is adjusted to 15 mL with water. The analysis is perform by Liquid Chromatography – Electrospray Ionisation Tandem Mass Spectrometry in positive ion mode. The injection of samples were perform with autosampler loop following this sequence: 45  $\mu$ L mobile phase + 10  $\mu$ L sample + 45  $\mu$ L mobile phase. The identification and quantification of these pesticides are conducted with a single run of 50 minutes. Validation studies were performed using matrix blank of pepper, tomato and water melon. The linearity in the response was studied with matrix matched calibration solutions. Seven point calibration curves were constructed in the concentration ranges of 5 to 500  $\mu$ g/L. The Limit of Detection (LoD) was determined as the analyte concentration that give a Signal to Noise ratio (S/N) of 5. Recovery studies were perform on spiked samples of pepper, tomato and water melon at two different levels. The recovery data were calculated by external standard calibration using matrix matched standard. All pesticides had good selectivity, linearity in response ( $R^2 \geq 0.99$ ), good sensitivity and recovery values higher than 75% in most of cases with good precision ( $RSD \leq 20\%$ ).



**M-02**

**Multiresidue Method for the Rapid Determination of  
Several Pesticides in Malted Barley, Spent Grains, Wort  
and Beer**

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Malted barley is the main ingredient for beer production. Unmalted cereals, especially maize, and rice are often used as adjuncts. It is important to note that several pests and diseases can attack these crops, and good weed control is also essential. For this reason, farmers need to protect their crops with pesticides. The problem is that traces of these pesticides may remain in the beer and by-products used as animal feed like spent grains obtained from the treated ingredients. With this aim, seven pesticides (pendimethalin, trifluralin, fenitrothion, malathion, myclobutanil, propiconazole and nuarimol) were determined in malted barley, spent grain, wort and beer by GC-ECD and GC-ITMS. The correlation coefficients derived from the linear regressions (2-2000 ng/ml) were higher than 0.999 in all cases. The repeatability was in the range of 4-8% (RSD). Pesticides were extracted with n-hexane/dichloromethane (50/50, v/v). Clean-up was necessary for malt and spent grain samples in the case of ECD system. In the clean-up step the compounds were eluted from the cartridge (Florisil) with n-hexane+ethyl acetate (20/80, v/v). Because of the high selectivity of the ITMS detector no interferences were found and clean-up was not necessary. Recoveries from spiked samples (n=5) ranged from 81 to 113% with a maximum coefficient of variation of 11%. Limits of quantitation varied from 0.05-5 ppt. Therefore, the analytical methodology used, rapid and reliable allows the correct determination of the studied compounds at levels well below the maximum residue limits (MRLs) established for barley by Spanish legislation (0.02-8 ppm).

**Keywords :** *Analysis, pesticide residues, malted barley, spent grains, wort, beer*

**M-03**

**Determination of Pesticides in Soils by GC/ECD**

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In the application of pesticides in agricultural crops, a fraction of the amount used reaches the soil, even when the pesticide is applied to plant foliage. A novel analytical multiresidue method using gas chromatography (GC) with electron-capture detection (ECD) has been developed for the rapid and sensitive determination of various classes of pesticides in soil, requiring small volumes of solvents, reducing the risk for human health and the environmental. Soil samples are extracted by sonication with a water-acetonitrile mixture and the pesticides are partitioned into dichloromethane. The identity of all compounds was confirmed by GC with mass selective detection (MSD) in the selected-ion monitoring mode. Good recovery and low detection though method was obtained for all the pesticides studied. The proposed method was used to determine pesticides levels in real soil samples.

**Keywords :** *Multiresidue, soil, pesticides, gas chromatography, environmental analysis*

**M-04**

**Ruggedness of Fast GC-MS Utilizing Narrow-Bore  
Capillary Columns in Pesticide Residues Analysis**

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The most straight-forward approach for speeding-up GC is the utilization of narrow-bore capillary column. Its main advantage over low pressure GC (with short wide-bore capillary columns) is substantially higher separation efficiency. However, narrow-bore capillary columns may demonstrate several disadvantages, such as: low sample capacity, low tolerance against matrix co-extractives and low carrier gas flow – complicating splitless injection. In this work, two narrow bore capillary columns were compared with regards to ruggedness of columns performance, CP-Sil 8 CB 10 m long, 0.1 mm I.D., 0.1 µm film thickness and CP-Sil 8 CB/Low Bleed 15 m long, 0.15 mm I.D., 0.15 µm film thickness, both from Varian Inc. For injection PTV inlet in cold splitless mode was utilized. As a model sample, apples were used. Samples were prepared according to the procedure described elsewhere [1] (acetonitrile extraction followed by SPE –NH<sub>2</sub> clean-up and solvent exchange to toluene). To search for deterioration of column performance, at the beginning of the sequence and then always after 20 injections, solution of pesticide standards in neat toluene and matrix matched standards were injected (both n=3). Between the standard solutions, real samples prepared according to the same procedure were analyzed. The studied parameters of separation were: shifts in elution times, peak areas and its repeatability, peak widths, peak tailing and signal-to-noise ratios. From the comparison, column with 0.15 mm I.D. proved significantly better ruggedness with regards to all parameters investigated.

Acknowledgement: The authors acknowledge support of this research by NATO project No. SfP 977 983 and Slovak Grant Agency VEGA No. 1/2463/05. [1] A. Hercegová, M. Dömötöröová, E. Matisová, M. Kirchner, R. Otrekal, V. Štefuca.: J. Chromatogr. A 2005 (in print).

**Keywords : *Fast GC, fast GC-MS, ruggedness, pesticide residues, ultra-trace analysis***

**M-05**

**Analysing of Some Herbicides in Water Samples Using  
SPE and LC with DAD**

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Herbicides of various chemical structures that belong among the most common agrochemicals applied for weed control. They are partially soluble in water and may leach into surface and ground waters at greater than ppb levels. Thus, the simple and sensitive analytical methods for monitoring in drinking and surface waters are required. The aim of this work was to develop a simple and sensitive method for simultaneous analysis of some herbicides (chlorsulfuron, diuron, bentazone, linuron, chlorpropham, fenoxoprop-ethyl, MCPA, diclofop-methyl, fluazifop-butyl, trifluraline) in water samples using SPE-HPLC-DAD. Sample preparation procedures included the use of octadecyl (C18) and styrene divinylbenzene (SDB) solid phase extraction (SPE) sorbents were proposed for the determination of these herbicides. Reversed-phase high-performance liquid chromatography (RP-HPLC) using a Luna C18 5mm column and ACN:Milli-Q water acidified with phosphoric acid pH:3.5 as mobile phase, with 220 nm UV-diode array detection (DAD), was used for separation and quantification of the herbicides. Method optimisation and validation parameters were presented in this work. Recoveries varied from 76.0% to 99.0% for C-18 disks, from 75.1% to 100.0% for C-18 cartridges and from 54.0% to 98.0% for SDB cartridges over concentrations at 0.025-0.4 µg L<sup>-1</sup>. The limits of detection were 0.012-0.035 µg L<sup>-1</sup>. Also the method has been applied to the monitoring of these herbicides in surface water samples from the Trakya area (Istanbul).

**Keywords :** *HPLC-DAD, Solid-phase extraction, Water analysis, Herbicides*

**M-06**  
**Determination of Various Pesticides in Water by  
HPLC-DAD**

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Since it is known that pesticides can induce a wide array of human health effects ranging from myelotoxicity to cytogenetic damage and carcinogenicity, pesticide residue analysis in environmental samples has received increasing attention in the last few decades. The misuse of pesticides by concerned individuals and lack of or weak national controlling plans are behind the outbreak of adverse effects in developing countries. The purpose of this study was to determine 5 pesticides (benomyl, chlorothalonil, folpet, tetradifon, quinomethionat) commonly used in Turkey in water samples. Analytical separation was performed on a C18 column by reversed-phase high performance liquid chromatography (RP-HPLC) with UV-diode array (DAD) detection. Silica based octadecyl (C18) and styrene divinylbenzene (SDB) solid-phase extraction (SPE) sorbents were used for sample preparation. Reanalysis of the sample extracts on a cyano (CN) column were used to confirm the identity of compounds. Limits of detection and quantification show that the method developed can be used to detect the pesticides in concentrations below the maximum residue levels (MRL) established by the European Union legislations. Methods optimisation and validation parameters were presented in this study.

**Keywords :** *HPLC-DAD, pesticides, solid-phase extraction*

**M-07**

**Practical Application of the QuEChERS Extraction and  
Concurrent LC & GC Triple Quadrupole MSMS for the  
Routine Monitoring of Pesticide Residues in Foods**

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With the demand from consumers for 'residue free' foods; the requirement to meet lower statutory maximum residue levels (MRLs) and the pressures placed on the food supply chain by the 'Global Marketplace', there has never been a greater need for accurate, sensitive & rapid methods to monitor pesticide residues in foods. Taking advantage of the selectivity provided by triple quadrupole mass spectroscopy (GC & LC) and the recent development of a 'quick-easy-cheap-effective-rugged and safe extraction technique

(1) our laboratory has developed and implemented a multi-class multi-residue procedure for the routine monitoring of pesticides at or below UK/EU maximum residue levels. The procedure uses a single extract which is run concurrently on GC & LC triple quadrupole systems. It has been validated in accordance with the EU Quality Control Procedures for Pesticide Residue Analysis

(2) and is accredited to the ISO 17025 standard as a generic technique. The method has been used routinely in our laboratory for over a year to monitor a wide range of pesticides and metabolites in fruits & vegetables and with minor modification for milk samples.

(1) Anastassiades, M., Lehotay, S.J., Stajnbaher, D., Schenck, F., Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and "Dispersive Solid-Phase Extraction" for the Determination of Pesticide Residues in Produce (QuEChERS method) J. AOAC Int., 86 (2003) 412-431.

(2) EU Document No. SANCO/10476/2003, 5th February 2004.

**Keywords : Pesticide, residues, QuEChERS, food, LC-MSMS, GC-MSMS.**

**M-08**

**A Monoclonal Antibody-Based Piezoelectric  
Immunosensor for the Analysis of Carbaryl**

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Immunosensors combine immunoassay selectivity with the high sensitivity often provided by electronic signal transducers. In this communication, the development of a monoclonal antibody-based Piezoelectric Immunosensor for the analysis of carbaryl is described and its main analytical characteristics are presented. Specific carbaryl haptens coupled to BSA were covalently attached to gold coated AT-cut 9 MHz quartz crystals, previously functionalized with a self-assembled alkanethiol monolayer. The crystal was inserted in a homemade Arnite cell that allowed only one face of the crystal to be in contact with the reagents. The cell was integrated in a flow-through system and pesticide detection was accomplished by competitive immunoassay in the conjugate-coated format. The assay consisted of the incubation of the analyte with a specific monoclonal antibody, followed by the immunoreaction of the mixture with the carbaryl derivative immobilized on the sensor surface. The frequency variations of the piezoelectric crystal were measured with a commercial Research Quartz Crystal Microbalance (RQCM) and correlated with the carbaryl concentration in the standards. The total time required for a complete assay cycle, including regeneration, was 20 min. As it corresponds to binding inhibition immunoassays, the frequency signal provided by the sensor was inversely proportional to the analyte concentration. A sigmoidal calibration curve was obtained by fitting experimental points to a four-parameter equation. The immunoassay sensitivity, expressed as the carbaryl concentration that reduced the assay signal by 50% (I<sub>50</sub>), was around 25 µg/l. The inhibition curve would allow the detection of carbaryl in fruits and vegetables at European regulatory levels.

**Keywords :** *Biosensor, Immunosensor, Piezoelectric, Antibodies, Carbaryl*

**M-09**

**A GC-MS Method to Determine Formic Acid Levels in  
Honey**

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Formic acid, an organic acid found as a natural component in honey, is used as varroacide against the mite *Varroa destructor*. In this work, levels of formic acid in honey were determined using a fast a liquid-liquid extraction (LLE) with diethyl ether followed by a gas chromatographic with mass spectrometry detector (GC-MS) analysis. This method has several advantages of being fast and inexpensive. Due to the absence of interfering peaks in the analytical region and to the high selectivity of the MS detector no cleanup step was necessary. Limit of detection of the proposed method was 0.2 mg/kg. Good linearity was achieved in the range from 0.2 to 600 mg/kg, with a coefficient of correlation of 0.9994. Recoveries of formic acid were from 81 to 92% with a maximum coefficient of variation of 10%.

**Keywords :** *Formic acid, honey, GC-MS*



**M-10**

**Application of Method Validation Procedures to Pesticide  
Residue Analyses**

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The growth of international trade in food commodities brings about the great need for reliable and comparable analytical data in food analyses, which can be achieved by implementation of an effective internal quality control/quality assurance system (QA/QC) and proving of the proficiency of the testing laboratories. The performance of the individual methods should be regularly verified during its use as part of the internal quality control program of the laboratory. Method validation is the process of defining an analytical requirement, and confirming that the method under consideration has performance capabilities consistent with what the application requires. The objective of this presentation is to elaborate the subject of method validation and its specific application to pesticide residue analyses and the statistical tools used for evaluation. Some of the parameters to be assessed for method validation in pesticide residue analysis include analyte stability during sample storage and sample processing; extraction efficiency; selectivity of separation; specificity and selectivity of analyte detection; calibration range for determination of analyte and matrix effect; analytical range; recovery through extraction, clean-up, derivatisation and measurement; limit of detection; limit of quantitation; reporting limit; accuracy and also precision. The extent of validation or revalidation required depends on the nature of the changes made in laboratories, instrumentation, operators, and the circumstances in which the method is going to be used. Based on the validation data generated, a QC scheme should be designed for the procedure, including appropriate limits, frequency of checks and system suitability tests for equipment.

**Keywords :** *Pesticide, method validation*

**M-11**

**Analytical Methodology for the Control of Fenoxycarb,  
Flufenoxuron, Lufenuron and Pyriproxifen Residues by  
HPLC-DAD.**

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Recently pesticides called Insect Growth Regulators (IGRs), are being used more and more in vegetables in Spain. A new extractive and analytical method was developed in order to study the residues of Fenoxycarb, Flufenoxuron, Lufenuron and Pyriproxifen in such important crops like lettuce, pepper, cucumber and zucchini.

The liquid-liquid microextraction is carried out with: 10 g of vegetable (blank or spiked) + 30 ml of acetonitrile + 1g of sodium sulphate anhydrous. Then, this mixture must be homogenized with Polytron for three minutes, filtered through phase separator paper of 150 mm diameter, evaporated until dryness with rotaevaporator at 40°C and the extract must be redissolved in 10 ml of acetonitrile:water 50:50 (V:V). In the end, 20 microliters of this are injected in the HPLC chromatographer from a capped vial. The analytical method is shown above:

- Reverse Stationary Phase: C8 Zorbax XDB Eclipse.
- Mobile Phase: AcN:W 50:50-70:30,7min; AcN:W 70:30, 8 min.
- Flow: 1 ml/min.
- DAD: 230 nm.
- Temperature: 25°C.

Validation of the method was made under ISO 17025 conditions and the results were: LOQ = 0.05 ppm;  $r_2 > 0.99$  in linearity; R.S.D < 20% in precision; and recuperations between 70-110% in exactness.

Hence, a new extractive and analytical method has been obtained, which is economical, simple and with no time-consuming. In the future, this method could be used in the control of pesticide residues in vegetable food.

**Keywords : IGRs, residues, microextraction, HPLC-DAD**

**M-12**

**High-Performance Liquid Chromatographic Quantification  
of Guazatine Residues Using Solid-Phase Extraction and  
Spectrophotometric Detection.**

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Guazatine,"a mixture of the reaction products from polyamines, comprising mainly octamethylenediamine, iminodi(octamethylene)diamine, octamethylenebis(imino-octamethylene)diamine, and carbamonitrile" is a non-systemic contact pesticide which disturbs the membrane function of fungi, decreasing the cellular permeability. The decrease in oxidative capacity is probably due to the inhibition of the uptake of certain substrates rather than a direct effect on enzymes. The main uses of guazatine are for the seed treatment of cereals and post-harvest application to citrus fruits. On citrus fruits, guazatine is used as a bulk dip after harvest, in the packing line as a spray and in washing installations to disinfect the process water. It controls sour rot (*Geotrichum candidum*), green mould (*Penicillium digitatum*) and blue mould (*Penicillium italicum*).

The aim in this study was to develop a liquid chromatographic method suitable for evaluating trace amounts of guazatine. For this purpose, an HPLC method with spectrophotometric-diode array detection is proposed. Factors affecting the chromatographic separation such as mobile phase composition, pH, type of column or flow rate and those related to spectrophotometric detection were optimised and excellent results were obtained. Also, an automated solid-phase extraction system (SPE) was used for purification and concentration of the analyte in real matrices. The method is shown to be an excellent alternative for the determination of this pesticide in real samples.

**Keywords :** *Citrus fruits, guazatine, HPLC, solid-phase extraction*

**M-13**

**Determination of Ethylenbis(Dithiocarbamates) in Food -  
Comparison of Two Analytical Approaches**

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Ethylene-bis (dithiocarbamates) (EBDC) belong to a widely applied group of non-systematic fungicides, used mainly to control the fungal disease of fruits, vegetables and cereals. The main compounds in this pesticide group are methiram, zineb, mancozeb and maneb, the last two of which are used and legislatively regulated in the Czech Republic. The maximum residue limits (expressed as CS<sub>2</sub>) range from 0.05 to 5 mg/kg. Special regulations apply to baby foods, for which the maximum residue limits of all pesticides are lowered to 0.01 mg/kg according to EU legislation.

The purpose of our study was to develop a method sufficiently sensitive to determine low EBDC levels in baby foods. With respect to this requirement solid-phase microextraction (SPME), known as a very sensitive technique, was tested. EBDC was hydrolysed with sulphuric acid in the presence of stannous chloride and the carbon disulphide released was then analysed by headspace SPME using polydimethylsiloxane/divinylbenzene/carboxene fibre. Finally the amount of CS<sub>2</sub> was determined by gas chromatography/mass spectrometry (ITD). The various factors affecting the efficiency of the SPME procedure were investigated during method optimisation. Limit of detection (LOD) and limit of quantification (LOQ), 0.0005 mg/kg and 0.001 mg CS<sub>2</sub>/kg of the sample respectively, were achieved using the optimised SPME method.

A comparison is made with the liquid-liquid extraction method, which uses the trapping of CS<sub>2</sub> released after acid hydrolysis into isooctane. This "classical" extraction method is clearly not sensitive enough to meet the low residue limit requirements for baby foods (LOQ 0.015 mg CS<sub>2</sub>/kg of the sample). Linearity, sensitivity and limits of quantification are used to compare the performance of both methods.

**Keywords : Pesticides, Ethylene-bis(dithiocarbamates), baby food,  
solid-phase microextraction**

**M-14**

**Comparison of Application Potential of UPLC-MS/MS and  
HPLC-MS/MS for Polar Pesticides Residue Analysis**

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In view of the perpetual growth of demands on laboratory throughput the chromatographic systems used for routine pesticides residue analysis are progressively improving. One of the new approaches to get a fast separation is the application of a special separation column packed with small particle size sorbents (less than 2 µm), which can provides higher resolution allowing faster separation. To the technical solutions how to operate that columns belongs the system for Ultra Performance Liquid Chromatography (UPLC), which is able to pump mobile phase at pressure up to 15 000 psi (approx. 1034 bar). In comparison with that High Performance Liquid Chromatography (HPLC) employs systems capable to pump mobile phase at pressure up to 6000 psi (approx. 414 bar).

New UPLC method optimization for residue analysis of 17 polar pesticides using BEH C18 (100 mm x 2.1 mm, 1.7 µm) column built in ACQUITY UPLC™ system (Waters, USA) was carried out. Final method was compared with routinely used HPLC method employing Discovery C18 (150 mm x 3 mm, 5 µm, Supelco, USA) column in Alliance 2695 HPLC system (Waters, USA). Analogous gradient elution with methanol-water at the same flow rate (0.3 ml/min) for both systems was used. For pesticides detection mass spectrometric tandem quadrupole instrument Quattro Premier (Waters, USA) operated in positive electrospray and MS/MS mode was used. UPLC separation was finished for 12 minutes and HPLC separation for 25 minutes including the necessary columns reconditioning. This fact means significant time and solvents consumption savings. On the other hand, parameters measured for characterization of separation process were moderately favorable for UPLC, but did not bring expected significant improvement in comparison with HPLC. Moreover, UPLC is still developing technique accompanied by partial technical imperfection.

**Keywords :** *LC-MS/MS, UPLC, HPLC, pesticides, residues*

**M-15**

**Optimization of Pressurized Liquid Extraction (PLE) in  
Determination of Pesticides in Complex Food Matrices**

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Many advanced extraction procedures based on instrumental techniques such as microwave-assisted extraction (MAE), microwave-assisted Soxhlet extraction (MASE), and pressurized liquid extraction (PLE) have been used for sample processing in pesticide residue analysis. Pressurized liquid extraction represents an alternative extraction technique enabling:

- (i) reduction of the volume of solvents required for extraction,
- (ii) improvement of the precision of analyte recovery,
- (iii) reduction of the extraction times, and
- (iv) reduction of sample preparation costs.

The effects of operational parameters (i.e. temperature of extraction, number of static cycles and extraction solvent) on the PLE efficiency were investigated in this study. Identification and quantitation of target analytes –modern pesticides was performed by high-resolution gas chromatography coupled with high-resolution “time-of-flight” mass spectrometry (GC/TOF MS).

The extraction of strobilurins from cereal matrices using PLE has been optimized. As the extraction solvents methanol, ethyl acetate and acetonitrile were tested employing extraction temperatures 60°C, 90°C and 120°C. Comparison of the extraction efficiency of PLE with those of the other techniques such as conventional ethyl acetate extraction is presented together with the assessment of generated data..

Appraisal of mentioned isolation techniques with respect to the amount of co-extracts, procedure blank levels and time and solvent volume demands was also done. The results proven the substantial reduction of solvent and time consumption without decrease of analyte recoveries.

This study was realized as a part of the European Commission-funded Integrated Project FOOD-CT-2004-06988 “BIOCOP (New Technologies to Screen Multiple Chemical Contaminants in Foods)” co-ordinated by Queen’s University, Belfast, Ireland.

**Keywords :** *Pressurized liquid extraction (PLE), pesticide residues, strobilurins, time-of-flight mass spectrometry*

**PP-01**

**Johnsongrass (*Sorghum halepense*) Control Using  
Brassicaceae Crops**

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Johnsongrass (*Sorghum halepense*) is a troublesome weed species in Turkey. Despite herbicide use to control for two decades, it has been still a problem in crops such as cotton. In addition, trends in agriculture such as organic agriculture and integrated pest management and increasing concerns on environmental issues require development of alternative control methods/strategies. Use of natural products as herbicides and/or of allelopathic effect of plants has been having increasing attention. Brassicaceae species have been known for their allelopathic effects on weeds. In a limited area, garden radish (*Raphanus sativus*) has been used by farmers when population of johnsongrass (*Sorghum halepense*) increased. Despite earlier studies, the use of allelopathic crops has not been expanded or improved. Field and laboratory experiments were conducted to find out how to use radish in cropping systems and to explore the possibilities of using other Brassicaceae crops for the control of johnsongrass. Our results showed that garden radish can be used either as a cover crop or a rotational crop and also that other allelopathic Brassicaceae crops can be used as a substitute of garden radish to control johnsongrass.

**Keywords :** *Allelopathy, Brassicaceae, Johnsongrass, crop residue, Raphanus spp, Brassica spp*

**PP-02**

**Investigations on Chemical Control of Brown Rot  
(*Sclerotinia fructigena* Aderh et Ruhl.) on Sweetcherry  
Fruits in Turkey**

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The study was carried out in two different locations, Uşak-Banaz and Balıkesir-Dursunbey in Aegean Region from 1997 to 2000. Aims of the study were to determine suitable application program against brown rot (*S. fructigena*) on sweet cherry fruits and to detect whether there is a residue problem of the fungicide (Benomyl 50WP) in fruits. Following application programs were tested for their effectiveness. First program: First application was made when fruits reached chickpea size stage and other applications were continued 10- 12 day intervals until 15 days before harvest. Second program: First application was made when fruits reached nut size stage and other applications were continued 10-12 day intervals until 15 days before harvest. Third program: Only one application was made as soon as fruits became red, 15 days before harvest. All of the 3 programs were in the same group statistically. It means they had same effectiveness. In the study carried out to determine residue level of the third program, the residue level was 0.068 ppm and the result was below the tolerance for stone fruits of Turkey and in cherry of EU for benomyl (0.1ppm). There was no residue problem in case pre-harvest interval was 14 days. As a result, the third Program has been advised for control of the disease, because it was in the same group statistically with others, consist of one application and there was no residue problem.

**Keywords :** *Sweetcherry, Sclerotinia fructigena, brown rot, fungicide, residue level*



**PP-03**

**Antifungal Activity of Some Plant Extracts Against Some  
Phytopathogens**

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Chemical fungicides provide the primary means for controlling postharvest fungal decay of fruit and vegetables. The use of synthetic chemicals to control postharvest deterioration has been restricted due to their carcinogenicity, high and acute residual toxicity, long degradation period, environmental pollution, and their effects on food and other side-effects on humans. The ultimate aim of recent research in this area has been the development and evaluation of various alternative control strategies to reduce dependency on synthetic fungicides. Biologically active natural products have the potential to replace synthetic fungicides. The preservative nature of some plant extracts has been known for centuries and there has been renewed interest in the antimicrobial properties of extracts from aromatic plants. Some plant extracted in different solvents have shown inhibitory action against different fungi. In this study some plants extracted with supercritical fluid extraction method. The antifungal activity of these extracts was evaluated against some phytopathogens using paper disk assays.

**Keywords :** *Phytopathogens, biologically active compounds, supercritical fluid extraction, antifungal activity*

**PP-04**

**Potential of Fluorescent Pseudomonads as Component of  
Integrated Management of Ascochyta Blight of Pea**

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Agriculture is increasingly depending on the use of chemical fertilizers, chemical growth regulators, and chemical pesticides to increase yield. This dependence is associated with problems such as environmental pollution, health hazards, interruption of natural ecological nutrient cycling, and destruction of biological communities that otherwise support crop production. Hence, crop improvement and disease management have to be achieved in shorter intervals of time with fewer detrimental inputs. The use of bioresources to replace chemical pesticides, chemical growth regulators, and chemical fertilizers is growing. In this context, fluorescent pseudomonads are often novel and potential tools to provide substantial benefits to agriculture. The object of the research presented here is to determine the most effective isolates of fluorescent pseudomonads for biological control of *Ascochyta pinodes*. For this purpose, two hundred and eight fluorescent pseudomonas isolates from rhizoplane, stems, flowers and capsules from pea plants were screened for their antagonistic activity in vivo against *A. pinodes*. Among them one *Pseudomonas putida* 17 and 3 isolates of *P. fluorescens*, 51, 116 and 122, gave promising results, mostly due to the production of fluorescent siderophores, and selected for field trial. A randomised block field trial was performed with 5 replicates. Antagonists were sprayed to aerial plant parts of peas and also plant debris which was severally infected with the pathogen before. Spraying plants with a suspension of fluorescent pseudomonads at the stage of leaflets with fourth node had expanded reduced ascochyta blight compared with the control by 45 to 60%. The best control of the leaf diseases was provided by 51, which reduced disease by almost 60%. On the other hand, application of the biocontrol agents to the plant debris, 7 days before sowing, resulted in 40-55% disease control and increased yield nearly 15% with the isolate of 51. Moreover, the compatibility of the antagonists with some chemicals, which are commonly sprayed on peas was investigated under in vitro conditions. Fungicides did not adversely affect the colonial development of bacterial strains at the concentration as high as 100 µg/ml in vitro.

**Keywords : *Biological control, ascochyta blight, fluorescent pseudomonads, pea***

**PP-05**

**Testing the Interaction of Pest-Predator-Plant Weather  
Component as a Simple Empirical Model for Predicting  
the Development and Field Generations of Some Cotton  
Insect Pests in Egypt**

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Field studies conducted during 2004 cotton growing season in Fayum Governorate, to detect the adult population of the pink bollworm, *Pectinophora gossypiella*, cotton leaf worm, *Spodoptera littoralis*, cotton aphid, *Aphis gossypiella*, white fly, *Bemisia Tabaci* and red mite, *Tetranychus Urticae*. Field generation numbers, life table parameters for field, thermal requirements and heat unit's accumulation was used. Cotton plant phenology was recorded as well as weather factors. The relationships in cotton complex were detected.

**Keywords :** *Prediction, IPM, cotton*

**PP-06**

**Effect of Some Pesticide Alternatives on the Population  
Size of the Black Melon Bug (*Coridius viduatus*) in the  
New Valley, EGYPT**

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Field experiments were carried out during 2000 and 2001 seasons to study the effect of some pesticide alternatives on the population size of the black melon bug (BMB). During summer plantation, the high initial effects against *C.viduatus* were recorded in case of the treatment with Evisect, Vertimec and dustable powder of sulphur; they induced reductions of 94.25, 75.75 and 73.18%, respectively after one day of application. On the other hand, high residual effects of 81.29 and 75.19% were obtained in the case of the treatment with Vertimec and Evisect after 21 days of application, respectively. During Nili plantation, Evisect, Vertimec and Agerin caused the highest reduction of the BMB population as initial effect than other compounds (74.08, 67.82 and 63.13%, respectively). On the other hand Evisect, Vertimec and *M.anispoliae* showed the highest reduction percentages as residual effect (74.00, 70.39 and 61.05%, respectively). It could be useful recommended that use of Evisect (500gm /100 liter of water), Vertimec (40ml / 100L water), *M. anisopliae* (200gm/ 100L water) and wettable power of sulphur 70% (250gm / 100L water) as alternative for the chemical compounds, to control the nymphs and adults of the BMB.

**Keywords :** *Black melon bug, Pesticide alternatives*

**PP-07**

**Effect of Some Essential Oils Against Citrus Leaf Miner  
Pupal Stage**

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The present investigation was directed to evaluate the effectiveness of seven essential oil constituents that represent four chemical groups of mono- and sesquiterpene, against the pupal stage of citrus leafminer. All essential oils tested were effective on the pupae emergence with different rates. Based on the percentage of the pupae mortality, the essential oils tested can be arranged in three groups. The first one includes the ester oil (methyl anthranilate) and the phenol (carvacrol), which were considered the most effective oils. Their mortality percentages were 94% and 86%. The second group includes the hydrocarbon oil (-3-carene), the phenol oil (Thymol) and the aliphatic alcohol (citronellal) which exhibited moderate effects with a mortality ranging from 63% to 73%. The least effective oils include the two alcohols (Nerol and Alpha-Terpineol). The mortality percentages were 20 and 30%. The dead pupae in the present investigation were in an intermediate stage (between pupal and adult stage). This may indicate a role of the essential oil constituents applied in juvenile hormone synthesis.

**Keywords :** *Citrus leafminer, essential oils*

PP-08

**A New Organic Insecticide Success Bait GF-120: Aerial  
Bait Spraying to Control the Olive Fruit Fly (*Bactrocera  
oleae* Gmel.) in Turkey**

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Olive fruit fly, *Bactrocera oleae* Gmel.(Diptera:Tephritidae) is the major pest in olive growing areas in Turkey like other Mediterranean countries. Aerial-bait spraying applications as area-wide control always gives successful results at the control of the pest. This experiment was carried out in Balıkesir-Havran in Turkey in 2003-2004. The experiment was formed 40.000-45.000 olive trees at the each parcel (character) Population trend of *B. oleae* was observed by means of McPhail traps containing DAP 2% and yellow sticky traps with pheromone capsules. Sprayings were initiated by the time fly catches tended to increase in the traps paralell to observe the first and new fly punctures on olive fruits.

Success-bait GF-120 (Spinosad) were applied with three different doses

a) 1200 ml insecticide+1200 ml water,

b) 1000 ml insecticide+1000 ml water,

c) 800 ml insecticide+800 ml water) and Supertakimethrin (Alphacypermethrin) which is one of the widely used product in the country was used as reference with the dose rate of 200 ml +800 ml Ziray (local food lure).

Success-bait GF-120 was applied as strip spraying whereas Super takimethrin was used as cover spraying. The insecticides were treated two (15.09.2003 and 22.10.2003) and three (21.09.2004, 08.10.2004 and 25.10.2004) times in 2003 and 2004 respectively For assessment, each parcel was divided into 4 unit to check the fruits. The fruits were counted as punctured or not, one day before the 1st. spraying and ten days after the last spraying in the each unit. The effectiveness of the spraying was determined by using Abbott formula and Annova test As a result of this experiment 800, 1000 and 1200 ml doses of Success-bait GF-120 were effective at the rate of 99.21% and 99.46%; 99.70% and 99.86% ; 99.83% and 99.87% according to the years respectively. The effectiveness of the Super takimethrin were 99.83% and 99.80%. The damage rates in the control were 44.72% and 83.90%. According to the results given above, 800 ml dose rate of Success-bait GF-120 has been registered against *Bactrocera oleae* Gmel. In the control of the pest as aerial bait-spraying.

**Keywords : *Bactrocera oleae*, aerial-bait spraying, Success-bait GF-120**