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Proficiency Tests on Pesticide Residues in olive oil

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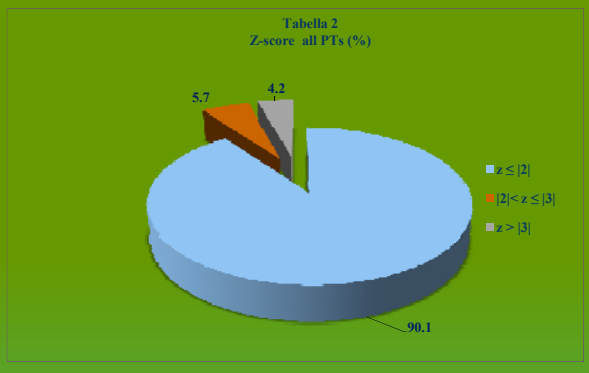
INTRODUCTION

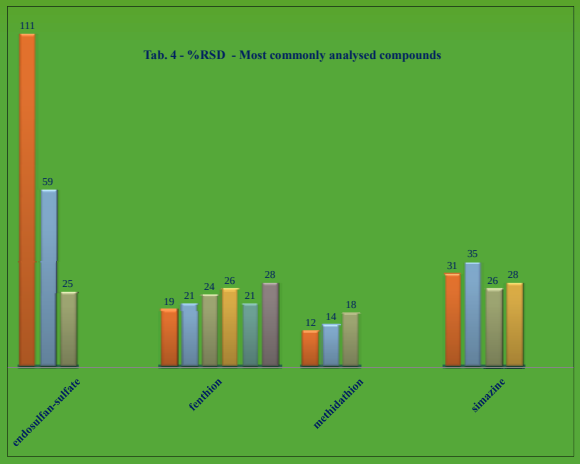
The aim of this paper is to present the experience of our Laboratory as organizer of Proficiency Tests (PTs) on pesticide residues in olive oil on behalf of International Olive Council (IOC). The (IOC) is the only intergovernmental organization in the world in the field of olive oil and table olives. IOC members are producers and exporters of olive oil and table olives located primarily in the Mediterranean region.
Istituto Superiore di Sanità (ISS) has been appointed since 1997 to coordinate the Working Group on Pesticides of the IOC. In order to compare analytical performances of laboratories in Mediterranean area and to promote mutual acceptance of pesticide residues data, the Working Group decided to organize Proficiency Tests on pesticide residues in olive oil. Nine PTs have been organized in the past years and one is in progress at the moment.

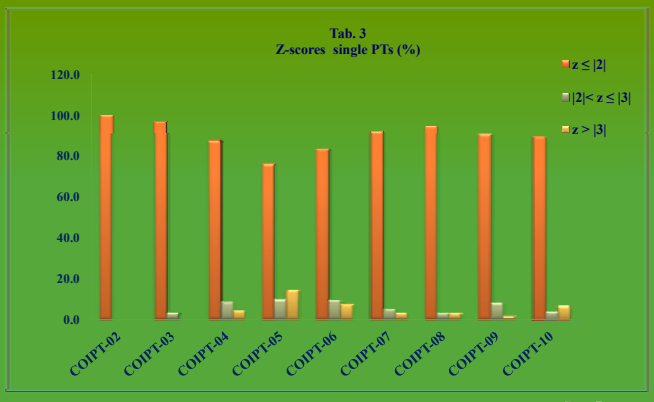
Tab. 1 - Proficiency Tests organized

PT name	COIPT-02	COIPT-03	COIPT-04	COIPT-05	COIPT-06	COIPT-07	COIPT-08	COIPT-09	COIPT-10	COIPT-11
Participating Laboratories	6	6	14	10	11	17	15	18	19	45 in progress
Tested compounds										
buprofezin									X	X
chlorpyrifos	X		X							
chlorpyrifos-methyl										X
deltamethrin				X						
diazinon	X		X							
diflufenican							X			
dimethoate	X		X			X			X	X
alfa-endosulfan				X	X					
beta-endosulfan				X	X					
endosulfan-sulfate				X	X	X				X
fenthion	X	X	X			X	X	X		
fenthion-sulfoxide		X	X			X	X	X		
fenthion-sulfone						X	X	X		
fenitrothion		X						X		
formothion		X								
lindane				X						
malathion		X								
methidathion	X		X						X	
methoxychlor				X						
omethoate									X	X
oxyfluorfen						X	X			
parathion	X		X							
permethrin				X	X					
pirimiphos-methyl								X		
procymidone						X				X
simazine						X	X	X	X	
terbuthylazine						X				
trifloxystrobin									X	
trifluralin								X		

In Tab. 1 all PTs organized at ISS are listed, together with the numbers of participating laboratories and the pesticides tested. The test material for all PTs consisted of commercial olive oil, spiked with pesticides in the range 0,050– 0,300 mg/kg. The spiked pesticides have been chosen out a list of the most used compounds on olive tree or contaminants that can be found in olive oil.
As the main scope of these PTs was to compare different methodologies on pesticide residues analysis in olive oil, the laboratories were asked to quantify the residues of known pesticide using their own standard procedures. In the first three PTs a calibration solution was also supplied to participants by the organizer.
The Z-score approach was used in all PTs to evaluate the performance of laboratories. Table 2 shows the overall performances of the laboratories express (all PTs). The 90,1 % of laboratories obtained a Z-score value $z \leq |2|$; 5,7% of laboratories a Z-score value $|2| < z \leq |3|$ and 4,2% of laboratories a Z-score value $z > |3|$.
In Tab.3 is reported the Z-score% for each PT. It is possible to note that as the number of participating laboratories increases the Z-score values become more significant. In the first two PTs only six labs participated and all of them presented acceptable Z-score values ($z \leq |3|$). In the first three PTs the mean of the laboratories means as "consensus value" was used. Increasing the number of laboratories to 15-20 it was decided to choose the median as "consensus value". In a similar way also the "Target Standard Deviation" value was changed with the time. Firstly was used the Standard Deviation value of all the results, then the $\sigma_{Horwitz}$ calculated according to the Horwitz equation; in the last two PTs we then adopted the σ_{EUPT} approach: a fit-for-purpose relative target standard deviation (FFP RSD%) = 25% of the median value. This last statistical approach is used by the European Reference Laboratories for Pesticide Residues in Fruits and Vegetables and in Food of Animal Origin and Commodities with High Fat Content, that organize PTs on pesticides residues in food.
Some pesticides were analysed in various PTs as showed in Table 4. Two pesticides fenthion and methidation present good RSD% values, ≤ 25%. The two high RSD% values for endosulfan sulfate refer to samples containing very low concentration of incurred pesticide.
Simazine is an herbicide used on olive tree and that can be give problems in the final step of analytical determination. RSD% values for simazine demonstrate an improvement of the laboratory performances over the time.







CONCLUSIONS

The aim of PTs on residue of pesticides in olive oil is to improve the quality, accuracy and comparability of the pesticide residue data in olive oil.
Results of these PTs could be more significant with the increase of the number of participating laboratories to PTs.