

Pressurized Fluid Extraction

Application : Pesticides Residues in Cereal

ASE : Accelerated Solvent Extraction

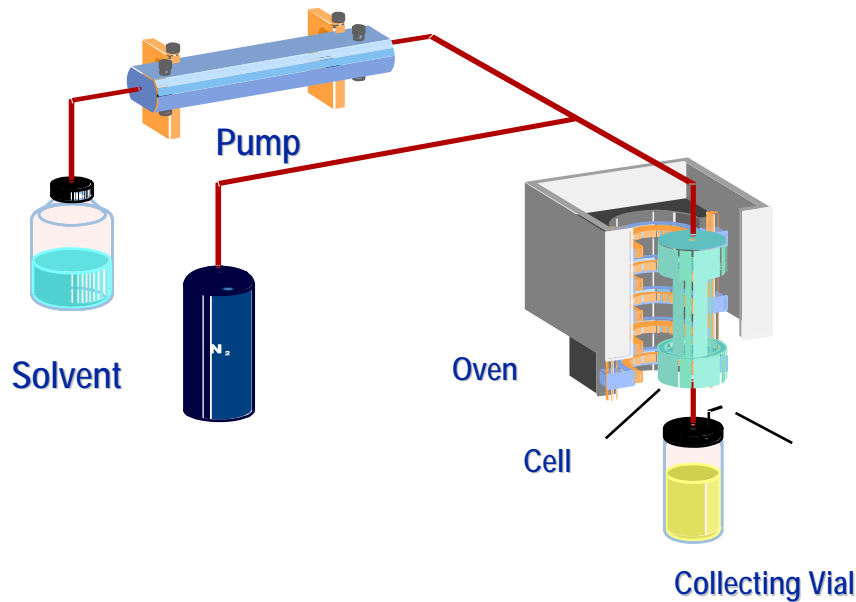
Cell



Collecting Vial

Jeudi 18 Octobre 2008

PROCESS OF EXTRACTION



Extraction Conditions

Pressure : 100 bars

Flush : 90 %

Temperature : 70 °C

Purge : 120 min

Cycles : 3

Solvent : 100 % Acetonitrile

Filling Cell

0,5 à 1 min

Heating

5 min

Static

5 min

Collecting extract

0,5 min

Purging

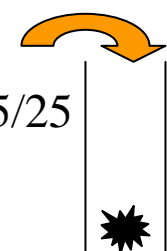
1 à 2 min

Total Time

12 à 14 min

Upper layer

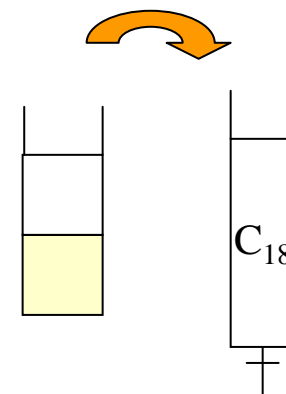
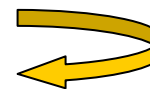
10 ml : Acetonitrile /
Dichloromethane : 75/25



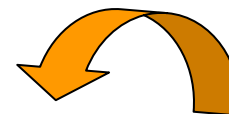
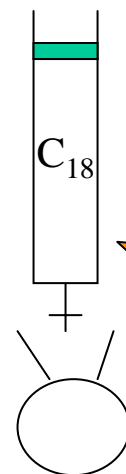
Vortex



Centrifugation
20 min / -10 °C



1- 10 ml Acetonitrile / Water : 25/5
2 - 5 ml Acetonitrile



30 ml Acetonitrile -
« azéotrope »
acetonitrile /eau

Influence of solvent

Sample : Wheat

Solvent : Methanol,
Hexane
Cyclohexane,
Acétate d'éthyle



Recovery : 60-70 %

Cleaning step :
aim : to replace Dichloromethane

Acétone, Hexane, Cyclohexane,



Recovery : < 50 %

Influence of Temperature on the extraction

Sample : Wheat

	50 °C	70 °C
Dichlorvos	68	71
Pirimiphos Méthyle	79	94
Chlorpyriphos Méthyle	84	99
Malathion	88	97
Parathion Ethyle	91	96

Pesticides Residues in Rye

	1	2	3	4	5	6	7	8	Average	σ	CV(r)
Bifenthrine :	0,22	0,22	0,28	0,25	0,24	0,26	0,22	0,25	0,24	0,02	9 %
Malathion	1,6	1,6	2,4	2,1	2,1	2,0	1,6	1,9	1,9	0,29	15 %

mg/kg

Proficiency Test - BIPEA - Pesticides Residues in Wheat

	P.F.E.	NF EN 12393	Assigned Value
Pyrimiphos Méthyle	0,19	0,19	0,19
Chlorpyriphos Méthyle	0,19	0,19	0,17
Malathion	0,32	0,28	0,30
Lindane	0,13	0,12	0,11
Deltaméthrine	0,23	0,18	0,21
Lambda-Cyhalotrine	0,17	0,16	0,15

Results of EUPT on Pesticides in Cereal

EUPT - C1 / C2

Azoxystrobine	C1	C2
Assigned Value	0,24	0,24
Lab	0,29	0,19

mg/kg