## Cleanup of PR


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## Alternative Methods

LLE
Column Chromatography
Sweep co-distillation
Sulfonating Purification
Precipitation with coagulants
SPE

## Necessary to cleanup

Interferences in matrix:
lipids, wax, protein, pigment, amine, phenols, organic acid, saccharides, etc.

It depends on the property of pesticide or sample, the detect method selected, requirement on speed and accuracy.

## Liquid-Liquid Extraction

**LLE, solvent extraction and partitioning,**
is a method to separate compounds based on their relative solubility in **two different immiscible liquids,** usually water and an organic solvent. It is an extraction of a substance from one liquid phase into another liquid phase.

performed using a separatory funnel**.**

**Solvent pairs**
acetone-hexane, acetonitrile-hexane (petroleum ether), DMF-hexane, DMSO-hexane…

## Disadvantages of LLE

**1 Solvent consuming**

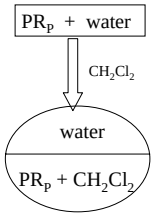
**2 Difficult to separate when emulsion occurs.**
pH change, adding methanol or filtration to solve it.

**3 Labor-consuming, time-consuming**

## Extraction solvent and solvent pairs selection

High water content sample:
PR from polar extraction solvent（water, acetone, methanol）to lower polar solvent.

Non-polar solvents ($CH_2Cl_2$ or ethyl acetate) could extract low polar pesticides (OP and Carbamates).

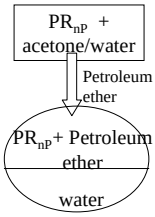


## Extraction solvent and solvent pairs selection

High water content sample:
PR from polar extraction solvent（water, acetone, methanol）to lower polar solvent.

Non-polar solvents (benzene or petroleum ether) could extract non-polar pesticides, leaving the polar interference in the water phase.

Water-$CH_2Cl_2$, acetone-water-$CH_2Cl_2$, methanol-water-$CH_2Cl_2$, water-petroleum ether, acetone-water-petroleum ether, methanol-water-petroleum ether




## Extraction solvent and solvent pairs selection

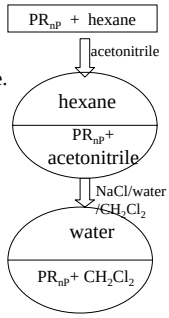
Low water, high fatty content sample

Non-polar pesticide：the purpose of clean up procedure is to remove oil and fat in the sample.

Extraction with n-hexane or petroleum from matrix.

Polar solvents such as acetonirtrile or DMF were used in LLE.

Low polar solvents such as $CH_2Cl_2$, benzene, petroleum were used in LLE.




## Extraction solvent and solvent pairs selection

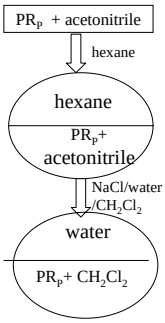
Low water high fatty content sample

Polar pesticides

Extract with acetonitrile, DMSO or DMF from matrix.

LLE, with n-hexane or petroleum ether

Add sodium chloride or sodium sulfate, then extract pesticides with petroleum ether, dichloromethane or n-hexane.




## Principles：*p* value

*K*ow: octanol-water partition coefficient

$K=p/q$

*K*——partition coefficient
*p*——pesticide in non-polar solvent
*q*——pesticide in polar solvent

$p+q=1$

*p* value represents the degree to which pesticide will preferentially dissolve in a non-polar solvent, which has the equal volume with the polar solvent.

**P56**

## *p* value

***p* is a constant for a pesticide in stable environment, fixed solvent pairs.**

**T is mainly factors to affect *P*.**
*p* increased with the increasing of T.

**Higher *p*，**
extract the pesticide in polar solvent with non-polar solvent.
**Lower *p*，**
extract the pesticide in non-polar solvent with polar solvent.

## Application of *p* value

**Single extraction with equal volume：**

***K=p/q*, *p=Kq***

**E**xample:

10 mL of water sample (the polar phase) contained PR was extracted with 10 mL (the equal volume) of n-hexane (the non-polar phase), the recovery of PR in hexane was equal to *p* value.

## Application of *p* value

**Multi extraction with equal volume：**

**Case 1**
**Extraction pesticide in non-polar solvent with polar solvent.**

$E_{非}=p^n$

$E_{极}=1-E_{非}$

**Case 2**
**Extraction pesticide in polar solvent with non-polar solvent.**

$E_{极}=(1-p)^n$

$E_{非}=1-E_{极}$

## Application of *p* value

**Example 1: *p* 0.12 lindane in hexane-acetonitrile**

Case 1:
Lindane residue sample (in hexane) was extracted with $CH_3CN$ 3 times.

In hexane: $E_{非}=p^3=0.12^3=0.0017$
In $CH_3CN$: $E_{极}=1-E_{非}=1-0.0017=0.9982$,
**Recovery: 99.82%**

Case 2:
Lindane residue sample (in $CH_3CN$) was extracted with hexane 3 times.

In $CH_3CN$: $E_{极}=(1-p)^n=(1-0.12)^3=0.6814$
In hexane: $E_{非}=1-E_{极}=1-0.6814=0.3185$
**Recovery: 31.85%**

## Application of *p* value

**Example 2: *p* 0.73 aldrin in hexane-$CH_3CN$**

Case 1:
Aldrin residue sample (in hexane) was extracted with $CH_3CN$ 3 times.

In hexane: $E_{非}=p^3=0.73^3=0.3890$
In $CH_3CN$: $E_{极}=1-E_{非}=1-0.3890=0.611$
**Recovery: 61.1%**

Case 2:
Aldrin residue sample (in $CH_3CN$) was extracted with hexane for 3 times

In $CH_3CN$: $E_{极}=(1-p)^n=(1-0.73)^3=0.0197$
In hexane: $E_{非}=1-E_{极}=1-0.0197=0.9803$
**Recovery: 98.03%**

## Application of *p* value

**Single extraction with unequal volume：** $\alpha=\frac{非极性溶剂的体积}{极性溶剂的体积}$
***a*，volume ratio of solvents**

$$E_{非}=\frac{\alpha P}{\alpha P-P+1} \qquad E_{极}=\frac{1-P}{\alpha P-P+1}$$

when $a=1$，$E_{非}=p$、$E_{极}=1-p$

when $a\neq 1$
If $a<1$，$E_{非}<p$
If $a>1$，$E_{非}>p$

$p=0$，$E_{非}=0$，all pesticide resolved in polar solvent
$p=1$，$E_{非}=1$，all pesticide resolved in non-polar solvent

## Comparison of pesticide in solvent pairs with different a

| pesticide | *p* value | $E_{非}$（$a=0.2$） | $E_{非}$（$a=0.1$） |
|---|---|---|---|
| aldrin | 0.7 | 0.32 | 0.25 |
| a - chlordan | 0.57 | 0.22 | 0.13 |
| dieldrin | 0.58 | 0.22 | 0.13 |
| P.P'－DDT | 0.61 | 0.25 | 0.14 |
| heptachlor | 0.73 | 0.34 | 0.20 |

*a* is very low in LPME, usually <0.1, it is impossible to absolute recovery.

## *p* of some pesticides in different solvent pairs

| Pesticide | hexane ＋ACN | 异辛烷＋二甲基甲酰胺 | 异辛烷＋85%二甲基甲酰胺 | 庚烷＋90%乙醇 | 异辛烷＋80%丙酮 |
|---|---|---|---|---|---|
| Aldrin | 0.73 | 0.38 | 0.86 | 0.76 | 0.98 |
| P.P'－DDT | 0.38 | 0.083 | 0.36 | 0.64 | 0.93 |
| allethrin | 0.21 | 0.14 | 0.59 | 0.41 | 0.84 |
| dicofol | 0.15 | 0.043 | 0.18 | 0.32 | 0.84 |
| Lindane | 0.12 | 0.052 | 0.14 | 0.41 | 0.78 |
| Trifluralin | 0.23 | 0.21 | 0.81 | 0.72 | 0.93 |
| parathion | 0.044 | 0.029 | 0.082 | 0.30 | 0.76 |
| malathion | 0.042 | 0.015 | 0.037 | 0.14 | 0.46 |
| Parathion-methyl | 0.022 | 0.012 | 0.015 | 0.11 | 0.40 |
| carbaryl | 0.02 | 0.02 | 0.01 | 0.06 | 0.20 |

## Application of P value

**Multi extraction with unequal volume：**

**Case 1**
**Extraction pesticide in non-polar solvent with polar solvent.**

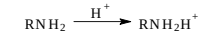
$$E_{非}=\left(\frac{\alpha p}{\alpha p-p+1}\right)^{n}$$

**Case 2**
**Extraction pesticide in polar solvent with non-polar solvent.**

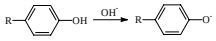
$$E_{极}=[(1-p)/(ap-p+1)]^{n}$$

## Application of *p* value

**-$NH_2$ contained compound**



**Ar-OH contained compound**



## Column Chromatography

Separation method of pesticide and interferences, based on the chromatography.

Conventional column chromatography

Glass column: diameter 0.2-2cm，length 15-30cm

**P58**

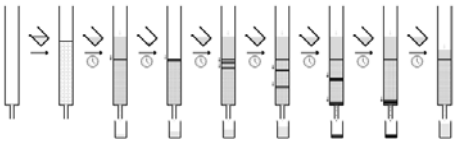
## Column Chromatography

Column preparation:

**dry method**
the column is first filled with stationary phase powder, then eluted with mobile phase, which is flushed through the column until it is completely wet. It is never allowed to run dry.

**wet method**
a slurry is prepared of the eluent with the stationary phase powder and then carefully poured into the column. Care must be taken to avoid air bubbles.

## Adsorption Column chromatography



Sorbents:
silica gel, alumina, florisil, active carbon……

## Adsorption Column chromatography

**Requirements to absorbent：**

（1）Large surface area, porous particle
（2）Strong adsorption and reversible adsorption
（3） Non-reactive and inert with ordinary usage, not dissolved in the eluent
（4）Columns were quickly and easily packed
（5） Repeatable

## Silica gel

Used to clean up the polar pesticide

∵ Various of" ≡Si-OH" on the surface，high adsorption of moisture，can be removed by heating （≡Si-OH…O≒$H_2$）

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## Silica gel

∵The adsorption capacity of silica gel is relevant with water content. Heating removes water adsorbed on the surface which enhance the adsorption capacity. This procedure is called activation；Adding some water will decrease the adsorption capacity which is called deactivation.

∵ weak acidity on surface.

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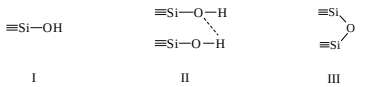
## Silica gel

Silica gel will lost its adsorption capacity at 200℃，for Si-OH was changed to Si-O.

Activation of silica gel should not exceed 150 ℃.

Usually at 110 ℃.




I II III

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## Alumina

Absorb lipid and wax

Basic alumina：1,3,5-triazine herbicide
Neutral alumina (Acidic alumina) :
OCl or OP are not stable under basic conditions

Commercially available alumina for adsorption chromatography (Neutral or acidic).

Activated at 130℃ for about 4h，then 5～10％(W/W) distilled water was added for deactivation.

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## Alumina

| Activity class | Water content in silica gel | Water content in alumina |
|---|---|---|
| Ⅰ(strong absorbability) | 0 % | 0 % |
| Ⅱ | 5 | 3 |
| Ⅲ | 15 | 6 |
| Ⅳ | 25 | 10 |
| Ⅴ(weak absorbability) | 38 | 15 |

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## Alumina

| | *n*-hexane+dichloromethane （100mL） | | | | | | |
|---|---|---|---|---|---|---|---|
| | 8:2 | 7:3 | 6:4 | 1:1 | 4:6 | 3:7 | 2:8 |
| Ⅴ | | Few | ~90% | Few | | | |
| Ⅳ | | | | 10% | 85% | Few | |
| Ⅲ | | | | | Few | 85% | Few |

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## Florisil

Florisil was made by magnesium sulfate and sodium silicate，which is filtrated and dried to be magnesium silicate. It is cellular solids with large surface area (specific surface area was 297).

Florisil can be used to separate classes of neutral lipids. The eluent was chosen according to the polar of analyte .

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## Florisil

**Activation**：at 650℃ for 1～3h. It will maintain active for four days in the dryer**.** Once losing activation, keep it in oven at 130 ℃ over night before used.

Usually, it is estimated that, 1g of Florisil is of appropriate activation to absorb 100mg of compounds with molecular weight of 200.

Activity was calculated by weight adsorbtion of lauric acid (Molecular weight about 200) by 1g of florisil.

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## Active carbon

Strong adsorption on pigment, but not on fat and wax .

Active carbon mixed with neutral alumina or florisil can be used to clean up pigments, fat and wax.

**Example**：
Prepare column with active carbon mixed with 5-10 times weight neutral alumina or florisil, Celite 545 and clay.
Elute with acetonitrile: Benzene = 1：1
**Used to clean up OP**

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## Others

Graphitized Carbon Black, GCB
Porous Graphitic Carbons, PGC**多孔石墨碳**
Multiwalled Carbon Nanotubes, MWNT **多壁碳纳米管**
Graphene **石墨烯**
。。。

Homework title?

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## Gel permission chromatography

GPC separates based on the size or hydrodynamic volume of the analytes. This differs from other separation techniques which depend upon chemical or physical interactions to separate analytes.

The smaller analytes (pesticides) can enter the pores more easily and therefore spend more time in these pores, increasing their retention time. Conversely, larger analytes (Fat, protein, chlorophyll) spend little time in the pores and are eluted quickly. Each column has a range of molecular weights that can be separated.

**Common used gel and** elution system：
Bio Beads $SX_2$、$SX_4$ or $SX_8$, cyclohexane system；
Bio Beads $SX_3$, Toluene - ethyl acetate 1:3 system.

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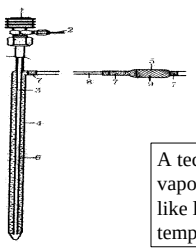
## Sweep co-distillation，SCD



1.进样口; 2.载气进口; 3.分馏管内管; 4.分馏管外管; 5.收集管; 6.硅烷化玻璃珠(1.5 mm); 7.硅烷化玻璃棉; 8.无水硫酸钠; 9.弗罗里硅土

吹扫蒸馏法装置图
（据 S. M. Watters）

A technology to separate the easily vaporized pesticides from impurities like lipids by introducing $N_2$ at high temperature.

Suitable for high oil content sample

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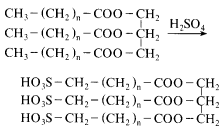
## Sulfonation Purification

Remove fat and wax by adding concentrated sulfuric acid.

OCl, some pyrethroids can be clean up by sulfonation .

Some OP, carbamate and pyrethroid pesticides which were not stable in strong acid should not use this method.



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## Sulfonation Purification

By ways of adding acid:

Column sulfonation（**硫酸硅藻土柱法**）：在等量的浓硫酸和20％发烟硫酸（9ml）中，加入30g Celite 545，与硅藻土混合后装柱，使用已烷或石油醚等非极性溶剂淋洗，当样本杂质含量多时常用此法。

Direct Sulfonation： directly add $H_2SO_4$ into extract in a separatory funnel. The amount of $H_2SO_4$ was 1/10 of extract volume. If oil content of sample was very high，2～3 times clean up procedures were needed.

## Precipitation with coagulants

Precipitate protein with coagulants

Coagulant was prepared by mixing $NH_4Cl$ and $H_3PO_4$ in a suitable proportion.

Use to clean up polar and water soluble pesticides such as OP, carbamate or other N-containing pesticides .

Sample extract was concentrated then dissolved in a certain concentration of acetone aqueous solution. Precipitate disruptors with coagulants then removed by filter.

Other coagulants : $Pb(AC)_2$

## Solid Phase Extraction

SPE is used most often to prepare liquid samples and extract semivolatile or nonvolatile analytes, but also can be used with solids that are pre-extracted into solvents.

SPE products are excellent for sample extraction, concentration, and cleanup. **Compared with LLE，SPE takes 1/12 of time, and 1/5 of cost .**

**In generally，the advantages of SPE：**
① Batch Analysis；②quick；③ low consuming of solvent；④ high selectivity；⑤enrichment of trace level pesticide；⑥ eliminate emulsification；⑦ automatic analysis

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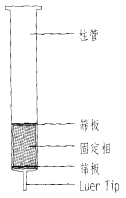
## Solid Phase Extraction



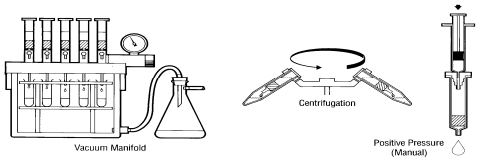

（1）**Syringe barrel-like body:** usually polypropylene, sometimes glass.

（2）**Frits**: 20μm pores (usually polyethylene, sometimes Teflon or stainless steel) **.**

（3）**Stationary phase**: Reversed Phase, Normal Phase, Ion Exchange

## SPE

Vacuum manifold, positive pressure or centrifugation




**You can decide either analytes or impurities retained by the sorbent.**

## SPE

1. Solid phase adsorption
2. Bonding technology

The carrier (stationary phase) was coated with stationary liquid which is insoluble with mobile phase. Pesticide and extract distribute in two phases.

## SPE

| | **Normal SPE** | **Reversed SPE** |
|---|---|---|
| Polarity of stationary phase | Polar of mid-polar | Non-polar or mid-polar（C-18） |
| Solvent polarity | Non-polar or mid-polar | Polar or mid-polar |
| Elution order | Non-polar compound will be eluted first | Polar compound will be eluted first |
| Increase solvent polarity | Reduce the elution time | Increase the elution time |

## SPE

**How to select an SPE sorbent：** choose the one that will bind selected components of the sample — either the compounds of interest or the sample impurities.

Similar polar sorbent was preferred.

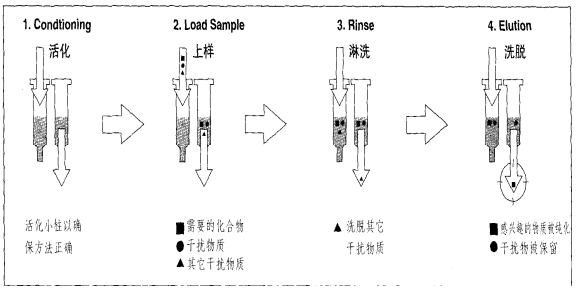
Consider the polarity of sample solvent.

## SPE

| 极性溶剂溶剂强度大 | 正相固定相 | 反相固定相 | 非极性溶剂溶剂强度大 |
|---|---|---|---|
| | 水 | 已烷 | |
| | 甲醇 | 异辛烷 | |
| | 异丙醇-2 | 甲苯 | |
| | 乙腈 | 氯仿 | |
| | 丙酮 | 二氯甲烷 | |
| | 乙酸乙酯 | 四氢呋喃 | |
| | 乙醚 | 乙醚 | |
| | 四氢呋喃 | 乙酸乙酯 | |
| | 二氯甲烷 | 丙酮 | |
| | 氯仿 | 乙腈 | |
| | 甲苯 | 异丙醇 | |
| | 异辛烷 | 甲醇 | |
| | 已烷 | 水 | |

| Type | Stationary phase |
|---|---|
| Non-polar | 十八烷基C18 |
| | 辛烷基C8 |
| | 乙基$C_2$ |
| | 环己烷基CH |
| | 苯基PH |
| | 氰基CN |
| Polar | 氰基CN |
| | 二醇基Diol |
| | 硅胶Si |
| | 氨基$NH_2$ |
| Cation exchange | 苯丙磺酸SCX |
| | 丙磺酸PRS |
| | 甲羧酸CBA |
| Anion-exchange | 三甲基丙基胺SAX |
| | 二乙基丙基胺 |
| | 一元或二元胺基 |

## SPE procedures




C18-SPE

## Condition

**Object：** Create an environment which is compatible with the sample and solvent, remove impurities in the column

**Two solvent: A pre-conditioning solvent** is used to remove any impurities on the SPE tube that could interfere with the analysis. **Final Solvents** was used to establish a proper environment to make sure analyte keeping in sorbent.

Final Solvents should not be stronger than the sample solvent

## Load sample

The process when the sample was added to SPE column and the sample was driven to pass the column. The analytes and the interference remained on the stationary phase in the process.

Weak solvent should be used. Strong solvent will not keep analyte in column, which cause low recovery（breakthrough）.

Strong solvent should not be loaded on the column directly. Dilute with a weak solvent in a proper concentration.

**E.g.** Soil sample was extracted by 50％ methanol，2ml extract should be diluted with 8ml water. Then it can be loaded on reversed phase column and breakthrough will not happen.

## Load sample

**The break through volume**:

• the sample volume which can be loaded on the sorbent bed without the loss of the analytes;

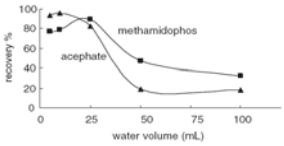
or

• maximum sample volume which can be applied with a theoretical 100% recovery

**Multi-Residue Analysis of Some Polar Pesticides in Water Samples with SPE and LC–MS–MS**

CHROMATOGRAPHIA 2006, 63, 233–237

Fengmao Liu[1,2], G. Bischoff[2], W. Pestemer[2], Wenna Xu[3], A. Kofoet[3]




## Rinse

**Interference elution：** The impurities are rinsed through with wash solutions that are strong enough to remove them, but weak enough to leave the compounds of interest behind.

**The volume of solvent : 0.5-0.8ml/100mg sorbent**

The adsorbed compounds of interest are eluted in a solvent that leaves the strongly retained impurities behind**. The elution solvents should not be too strong or too weak.**

## Elution

Elution volume was calculated **by 0.5-0.8ml/100mg sorbent** 。

Choose proper solvent：too strong； too weak。

Elute with strong solvent, **5-10 times of bed volumes**

e.g. column with 500 mg sorbent， bed volumes : **0.6mL，** solvent volume for elution will be **3-6 mL。**

**溶剂互溶性。后流过柱床的溶剂必须与前一溶剂互溶。**

Vol 2 农药学学报 No 2
2000 年 6 月 CHINESE JOURNAL OF PESTICIDE SCIENCE 89~93

水中 12 种农药的固相萃取及 GC-MS 测定方法研究

刘丰茂 钱传范 江树人

ISOLUTE™ ENV+ 200mg/3mL SPE

**Pretreatment of water sample:**
Adjust pH, 2%$CH_3CN$, 2% NaCl, filtration
**Condition:**
2*2.5mL n-hexane, 2*2.5mL acetone, water
**Loading:**
vacuum, 5mL/min
**Eluting (without rinsing):**
2*2.5mL acetone, 2*2.5mL n-hexane
**Concentration**
50 uL n-butanol, $N_2$ concentration

**Multi-Residue Analysis of Some Polar Pesticides in Water Samples with SPE and LC-MS-MS**

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Fengmao Liu[1,2], G. Bischoff[2], W. Pestemer[2], Wenna Xu[3], A. Kobet[3]

Methamidophos
Acephate
SPE

Oasis HLB, Chromabond HR-P, LiChrolut EN, C18 … SPE were reported in previous literature, however, some were not reproducible.

Oasis HLB
The water sample was passed through the cartridge. The cartridge (if necessary, it was stored at about -15℃ until analysis) was dried, the analytes were eluted. The eluate (if necessary, it was stored at about -15 ℃ until analysis) was evaporated to dryness with weak nitrogen stream without disturbing the surface of the solution.

· 202 · 农药与环境安全国际会议论文集

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土壤中农药多残留分析方法及淋溶性研究

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**SPE was used in Soil samples**

Soil sample was
Extracted with mixture of acetone and water, ultrosonic method

Filtrated
Evaporated to remove acetone
Addition of 2g NaCl
Loading to SPE

ISOLUTE™ ENV+ 200mg/3mL SPE

## Ion exchange extraction

**Definition：**由一带电荷的农药或其代谢物与相反电荷的柱填料互相吸附，从而实现与其它杂质分离的技术。

**Two classes：**

Cation exchange phase：Keep the compound with positive charged
**Note：organic amine and carboxylic acid！**Change the pH to ionize it

Anion-exchange phase： Keep the compound with negatively charged

**P67**

## Ion exchange extraction

**LC-SCX（苯丙磺酸），Strong cation exchange column，extract the pesticide with cation ⊕**

$R\text{-}NH_2$ + HCl（稀）→ $R\text{-}NH_3^+Cl^-$
organic base pesticide 与酸结合形成带质子的化合物

$NH3^+$-R （酸性，带阳离子胺类农药与相反电荷的柱填料互相吸附）

LC-SCX-$SO_3^-$ $Na^+$

$NH_4^+$ (用强阳离子交换柱填料的对离子将被吸附农药淋洗下来)

## Ion exchange extraction

**LC-SAX（三甲基丙基胺）是强阴离子交换柱，可萃取带阴离子的农药，如有机羧酸：**

R – COOH + NaOH → $R\text{-}COO^-Na^+ + H_2O$
(有机酸，不溶于水) （有机酸的钠盐，溶于水）

$R\text{-}COO^-$ (碱性，带阴离子的羧酸农药与相反电荷的柱填料互相吸附）

LC-SAX-$N^+$ $Cl^-$

$PO_4^{3-}$ (用阴离子交换柱填料的对离子将被吸附农药淋洗下来)

## SPE disks

SPE disks can be processed on a vacuum filtration flask-type assembly.

## SPE disks

**Resprep™ C18 and Resprep™ C8 SPE Disks**

- Glass fiber disks embedded with C18 or C8 bonded silica.
- Extract semivolatile organic compounds.
- Deep-pore design reduces clogging and allows faster flow rates.
- Meet requirements for EPA Methods 525.1, 506, 550.1, and 549.1.
- Lower cost than Teflon® disks.

| Description | qty. | cat.# | price |
|---|---|---|---|
| Resprep-C8 47mm SPE Disks | 24-pk. | 24048 | |
| Resprep-C18 47mm SPE Disks | 20-pk. | 24004 | |
| Resprep-C18 90mm SPE Disks | 12-pk. | 25988 | |

**Resprep™ Oil & Grease SPE Disks**

- 47mm glass fiber disks embedded with specialty bonded silica.
- Meet requirements for EPA Method 1664.*
- Reduce emulsion formation and amount of solvent required by previous EPA methods.
- No chlorofluorocarbons needed.

| Description | qty. | cat.# | price |
|---|---|---|---|
| Resprep Oil & Grease SPE Disks | 20-pk. | 26022 | |

*A sodium sulfate drying tube and a 0.45µm PTFE syringe filter (cat.# 26145, page 363) also may be used.

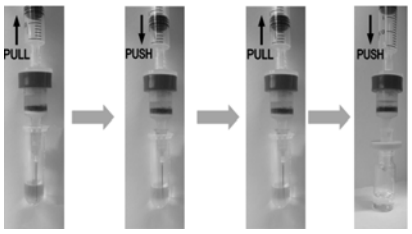
Resprep

Resprep™ disks & flow filters extract analytes of interest at high flow rates, and significantly reduce clogging.

## mPFC method
## (multi-Plug and Filtration Cleanup)

多次推拉过滤

❖**Repeat 2-3 times**




**Do it in seconds!**

**不同萃取方法的比较**

| 萃取方法 | 萃取时间(min) | 样品体积(mL) | 萃取溶剂量(mL) | 适用范围 | 相对标准偏差(%) |
|---|---|---|---|---|---|
| 液－液萃取 | 60～80 | 50～100 | 50～100 | 难挥发性 | 5～50 |
| 固相萃取 | 20～60 | 10～50 | 3～10 | 难挥发性 | 7～15 |
| 固相微萃取 | 5～20 | 1～10 | 0 | 难挥发性与挥发性 | <1～12 |

## Conclusion of extraction and cleanup methods

| | Sample analytes | | | Sample Matrix | | | | Exhaustive Extraction |
|---|---|---|---|---|---|---|---|---|
| Technique | VOC | S-VOC | N-VOC | Solid | S-solid | Liquid | Gas | |
| P&T | √ | | | √ | √ | √ | | √ |
| HS | √ | | | √ | √ | √ | | |
| SPE | | √ | √ | | √ | √ | | √ |
| SPME | √ | √ | √ | | | √ | √ | |
| SFE | | √ | √ | √ | √ | | | √ |
| UWave Extr. | | √ | √ | √ | √ | √ | | √ |
| LLE | | √ | √ | √ | √ | √ | | √ |
| Sonication | | √ | √ | √ | √ | √ | | √ |
| Soxhlet | | √ | √ | √ | √ | | | √ |
| GPC | | √ | √ | | | √ | | √ |

Volatile Organic Compounds

## Questions

1. *p* value
2. Stationary phase in column chromatography
3. Activation and deactivation of the absorbent
4. Cleanup method