

Designed polymers for purification of flavor oils

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- » The company
- » Separation techniques
- » Chromatography, adsorbents and selective separations
- » Case study: selective pesticide removal from lemon oil
- » Summary

About Biotage and MIP Technologies



Where we are

 **Charlotte** ●



 **Uppsala** ● ●
Corporate headquarter



 **Lund** ●



 **Cardiff** ● ●



 **Shanghai** ●



● **Tokyo** ●



Legend
● Sales office
● Production and R&D

Nearly 300 employees – ca 500 M SEK turnover

- » Founded in 2000, Lund Sweden
- » Acquired by Biotage AB in 2010
- » Total of 12 chemists (9 PhD)
- » R&D team to develop novel adsorbents
- » Full analytical lab to test and QC
- » Pilot plant to scale-up and manufacture



» **Activities:**

- » **Polymeric adsorbents**
- » **Development of new adsorbents and applications**
- » **Problem solving service for customers:**
 - » High success rate (~ 75 %) in conducted projects
 - » Worked with many of top 100 companies
 - » ~ 15 years in the business



- » The company
- » **Separation techniques**
- » Chromatography, Adsorbents and Selective separations
- » Case study: Selective pesticide removal from lemon oil
- » Summary

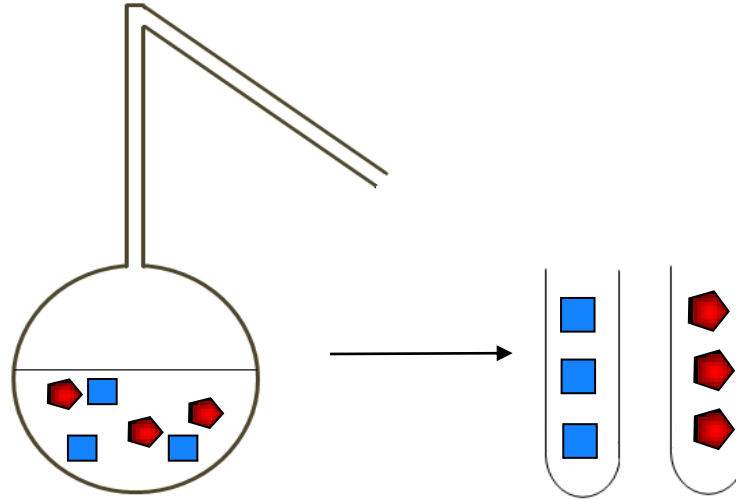
- » Separations/Purifications basically have 2 goals:
- » 1) to **remove** *unwanted* substances
 - » Example: remove toxic or allergenic from complex mixtures
- » 2) to **recover** a *desired* substance
 - » Example: extract a valuable compound from a mixture

- » Selectivity is the ability to distinguish one molecule from other molecules
 - » Target compound 
 - » Other compounds  etc
- » Adsorbent = *Polymer* = *Resin*

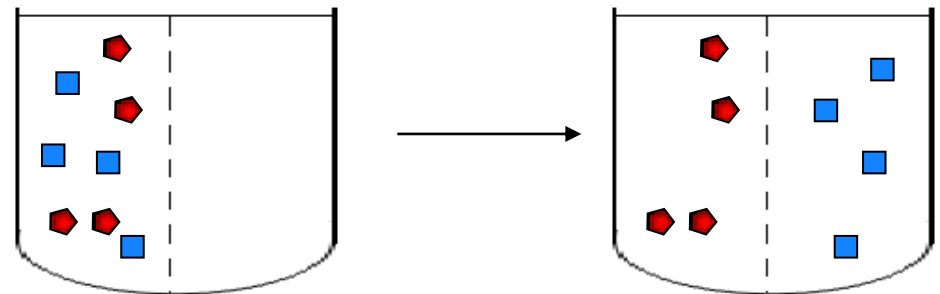
Separation techniques

A few generic examples

- » Distillation
 - » By volatility

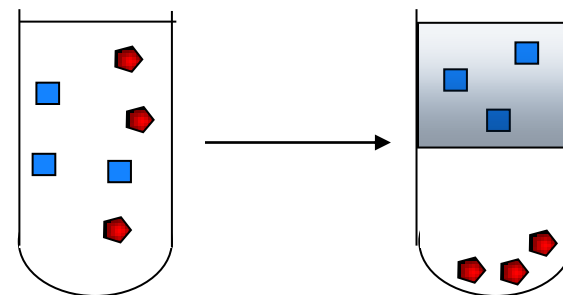


- » Membranes /Filtration
 - » By permeability



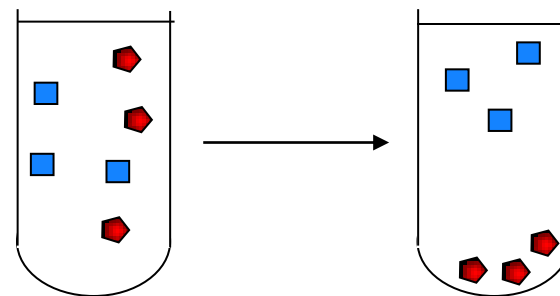
» Liquid/Liquid Extraction

» By solubility



» Precipitation / Re-crystallisation

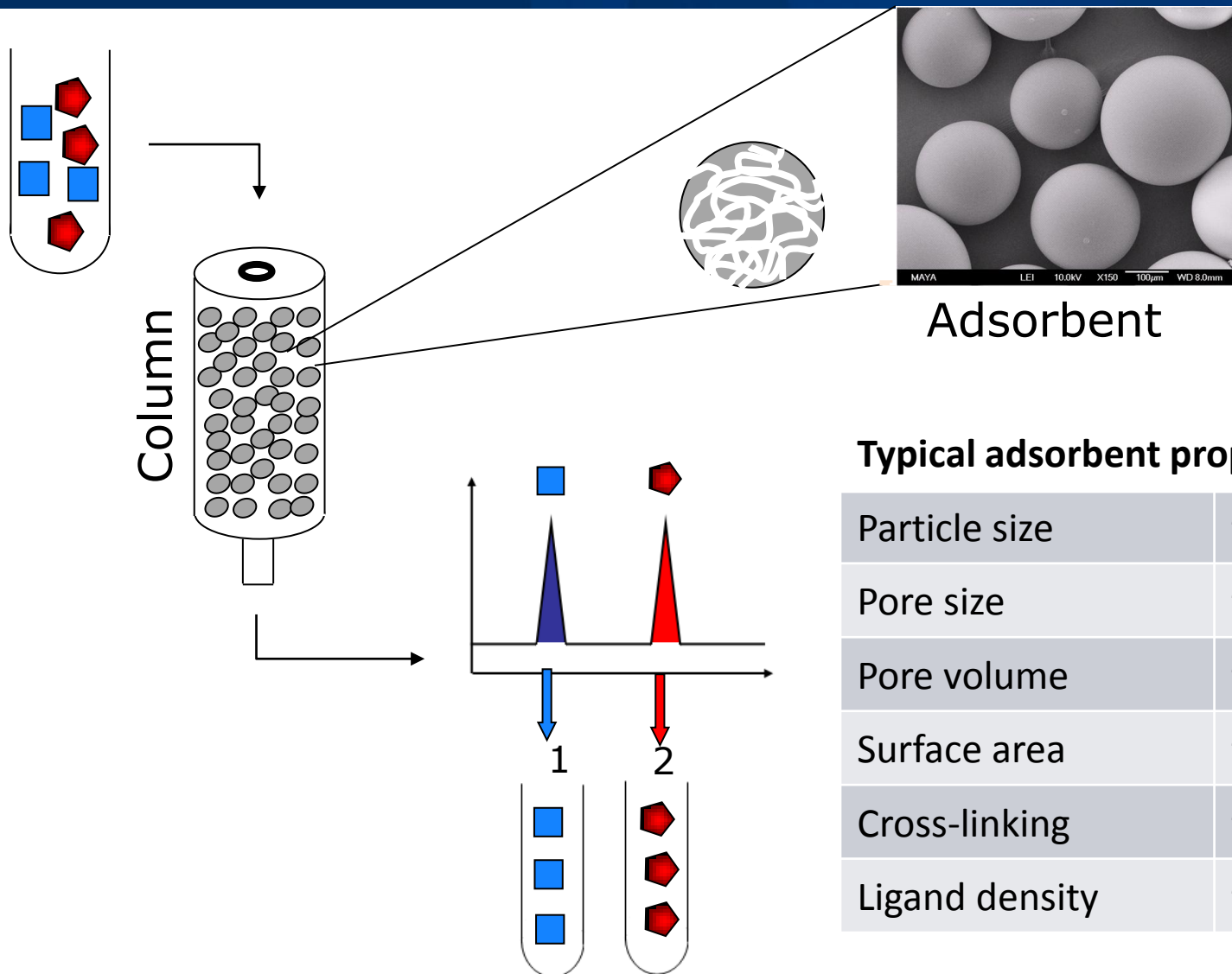
» By solubility



- » The company
- » Separation techniques
- » **Chromatography, Adsorbents and Selective separations**
- » Case study: Selective pesticide removal from lemon oil
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Chromatography

By reversible surface interactions

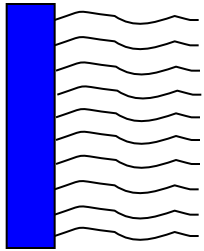
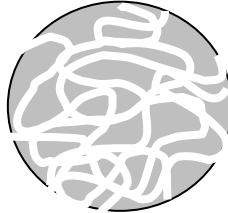


Adsorbent

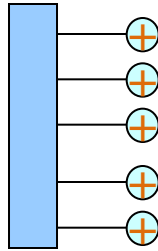
Typical adsorbent properties

Particle size	~ 100 μm
Pore size	~ 100 \AA
Pore volume	~ 0.5 ml/g
Surface area	~ 500 m^2/g
Cross-linking	~ 50 %
Ligand density	~ 0.5 mmol/g

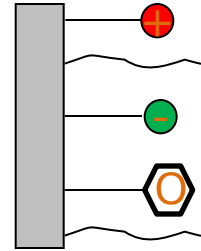
Adsorbent surface landscape



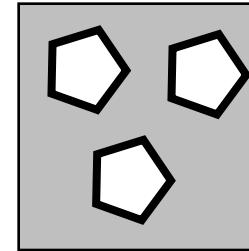
Reversed Phase



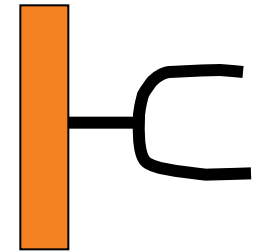
Ionic/Polar



Designed



Imprinted



Affinity

Selectivity

Non-selective materials (hydrophobic, ionic/polar): wide-spread general use.
For difficult separations, selective materials are needed:
Affinity, imprinted adsorbents, designed adsorbents...

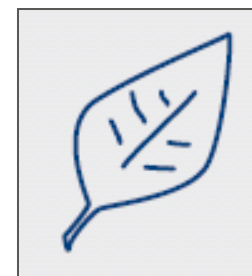
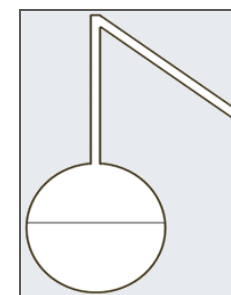
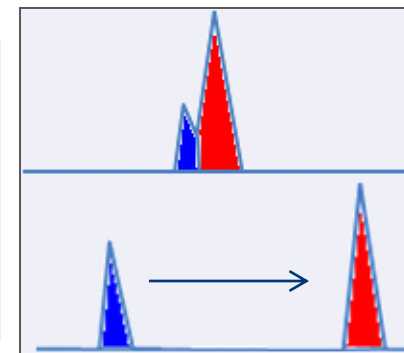
In general: non-selective materials are cheap, selective materials cost more
Most affinity materials are fragile (biological molecules as ligands)

Potential of selective adsorbents

- » Resolution of difficult-to-separate compounds
- » Selective removal of unwanted contaminants
- » Targeted recovery or enrichment of valuable compounds
- » Cumbersome purifications with many or large columns

- » Replacing distillation or liquid-liquid extraction
 - » Energy consumption, purity, contaminants...

- » Avoiding undesired chemicals e.g. salts, flammable organic solvents or toxic chemicals
 - » Green chemistry...



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Extracted from the peel

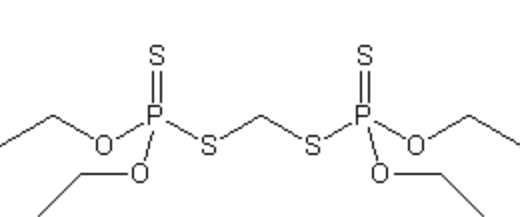


Flavor oils may be contaminated
with agricultural residues

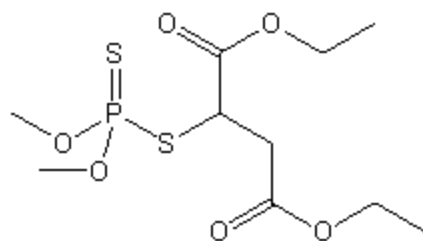
Important ingredient in

- » Food industry
- » Drinks industry
- » Fragrances
- » Etc...

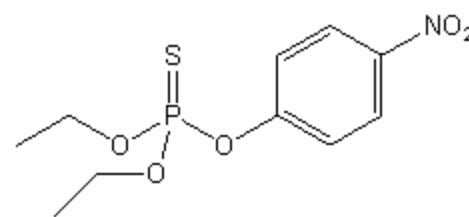
Pesticides in lemon oil



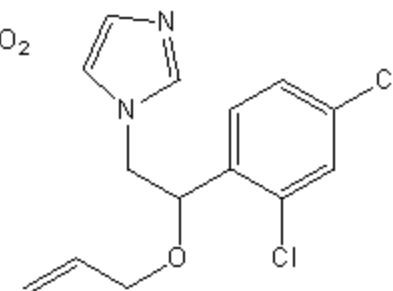
Ethion



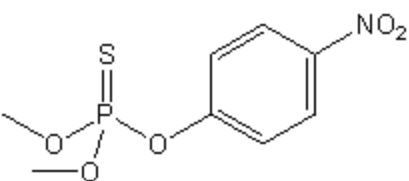
Malathion



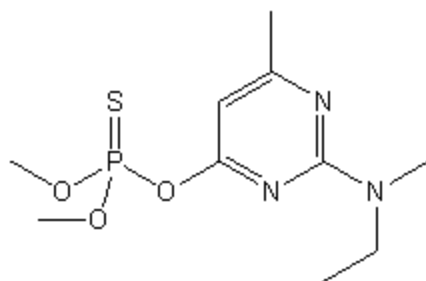
Parathion



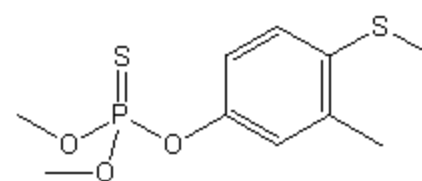
Imazalil



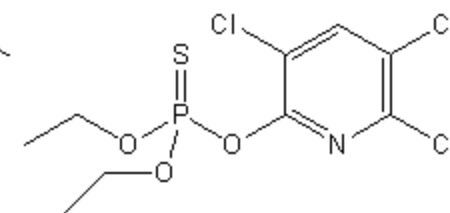
Methylparathion



Pirimiphos methyl



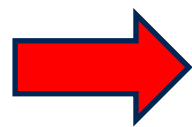
Fenthion



Chlorpyrifos

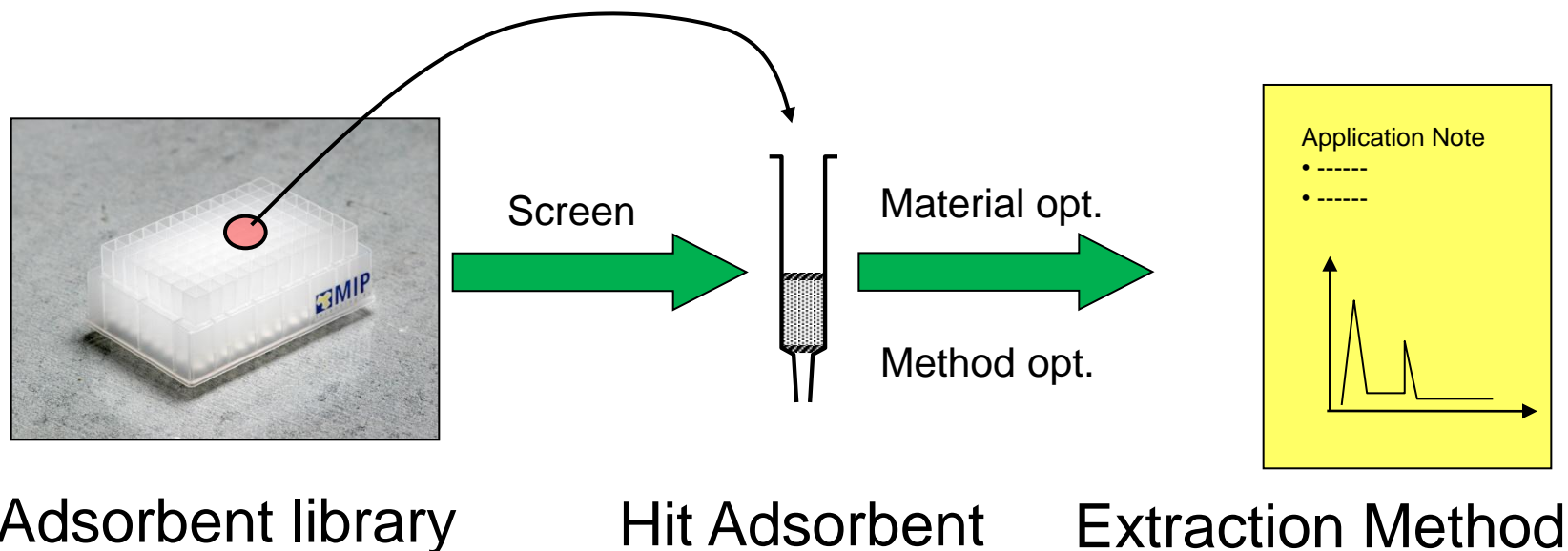
- Toxic: similar to nerve agents (e.g. sarin)
- Many pesticides have a thiophosphoester moiety
- Present at ppm levels

- » Distillation and other standard unit operations cannot always remove the pesticides well
 - » Pesticides and lemon oil constituents are chemically similar (e.g. vapor pressure)
- » Heat may influence aroma / taste profile
- » Standard adsorbents do not exhibit sufficient selectivity



Development of a selective adsorbent to remove pesticides without affecting key aroma molecules

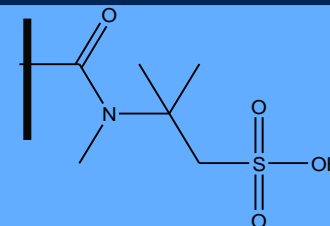
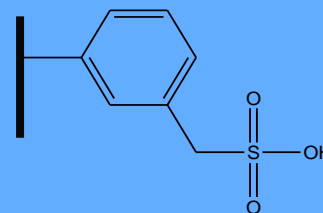
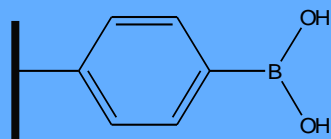
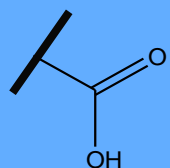
How to find the best adsorbent?



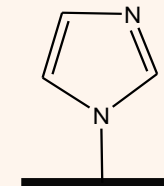
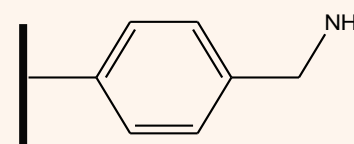
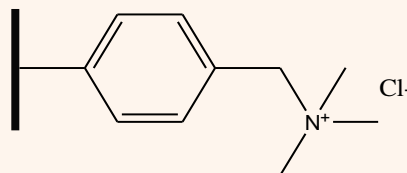
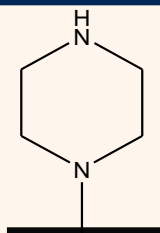
2500 candidates

Adsorbent surface chemistries

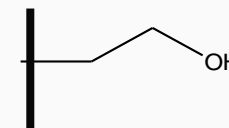
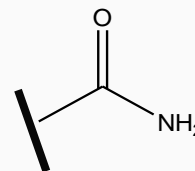
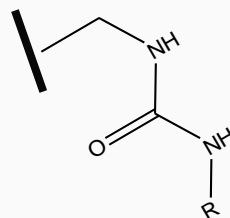
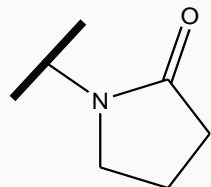
Acidic/
Anionic



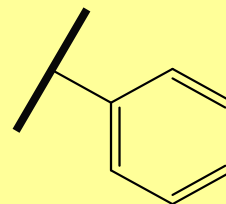
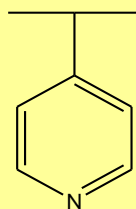
Basic/
Cationic



Neutral/
hydrophilic



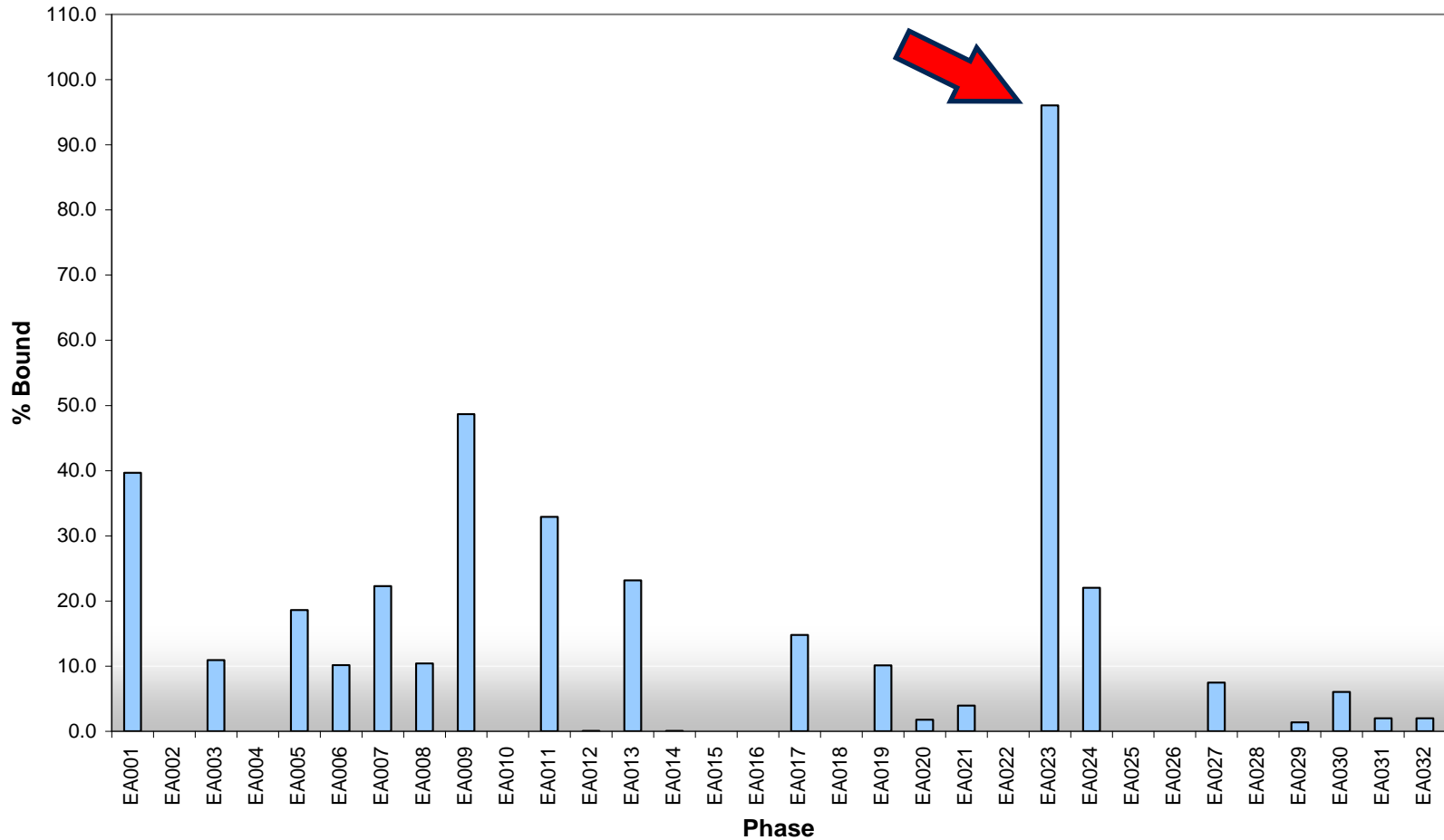
Neutral/
hydrophobic



Adsorbents have combinations of chemistries...

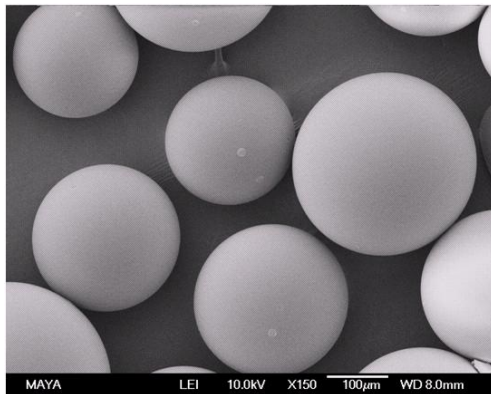
Outcome of a screening example

Using the MIP Tech adsorbent library



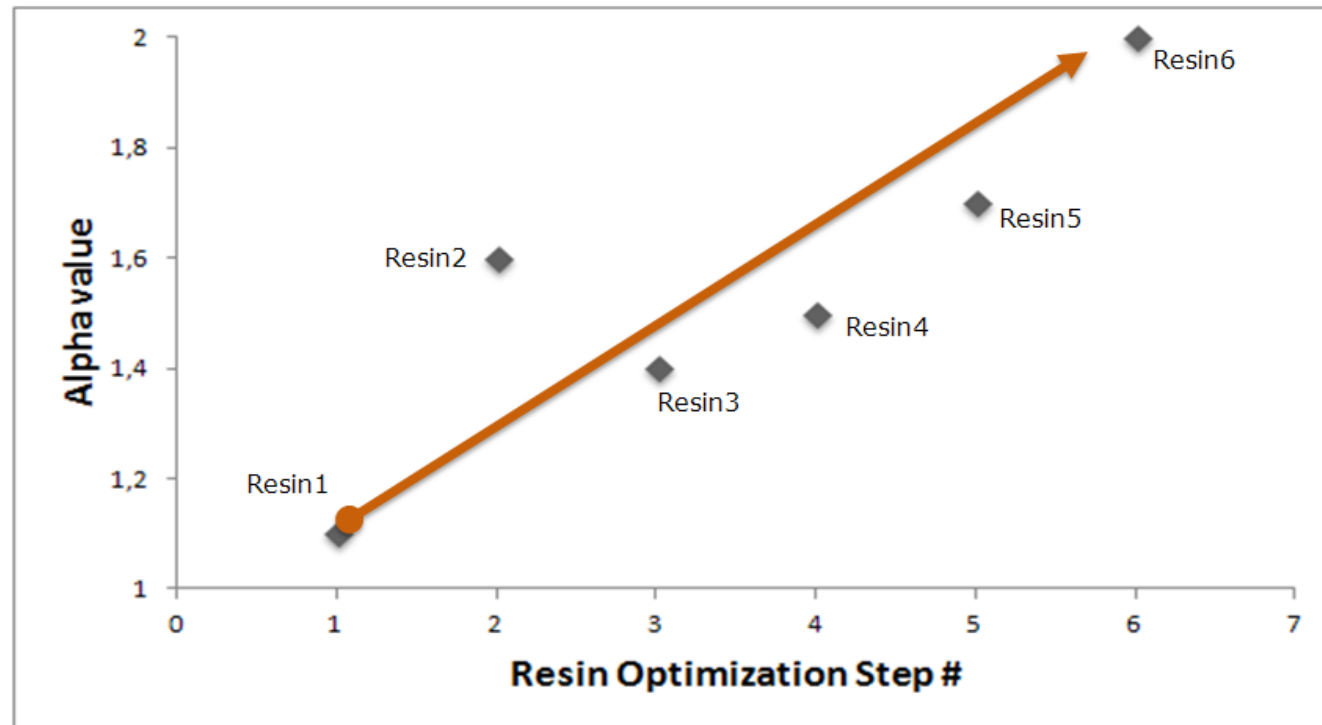
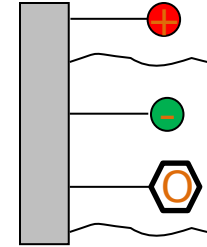
» Material morphology

- » Pore size
- » Surface area
- » Pore volume
- » Bead size
- » Rigidity



Surface chemistry

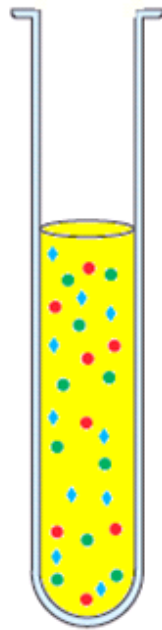
- » Interaction design
- » Ligand density
- » Binding site design



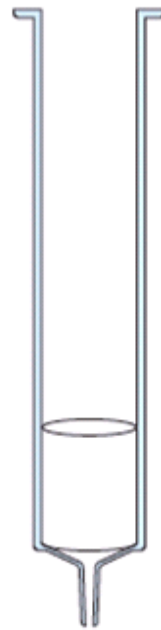
Optimised designed adsorbent

Pesticide clean-up in one step

Citrus oil
containing
pesticides



RENSA™ 101
column



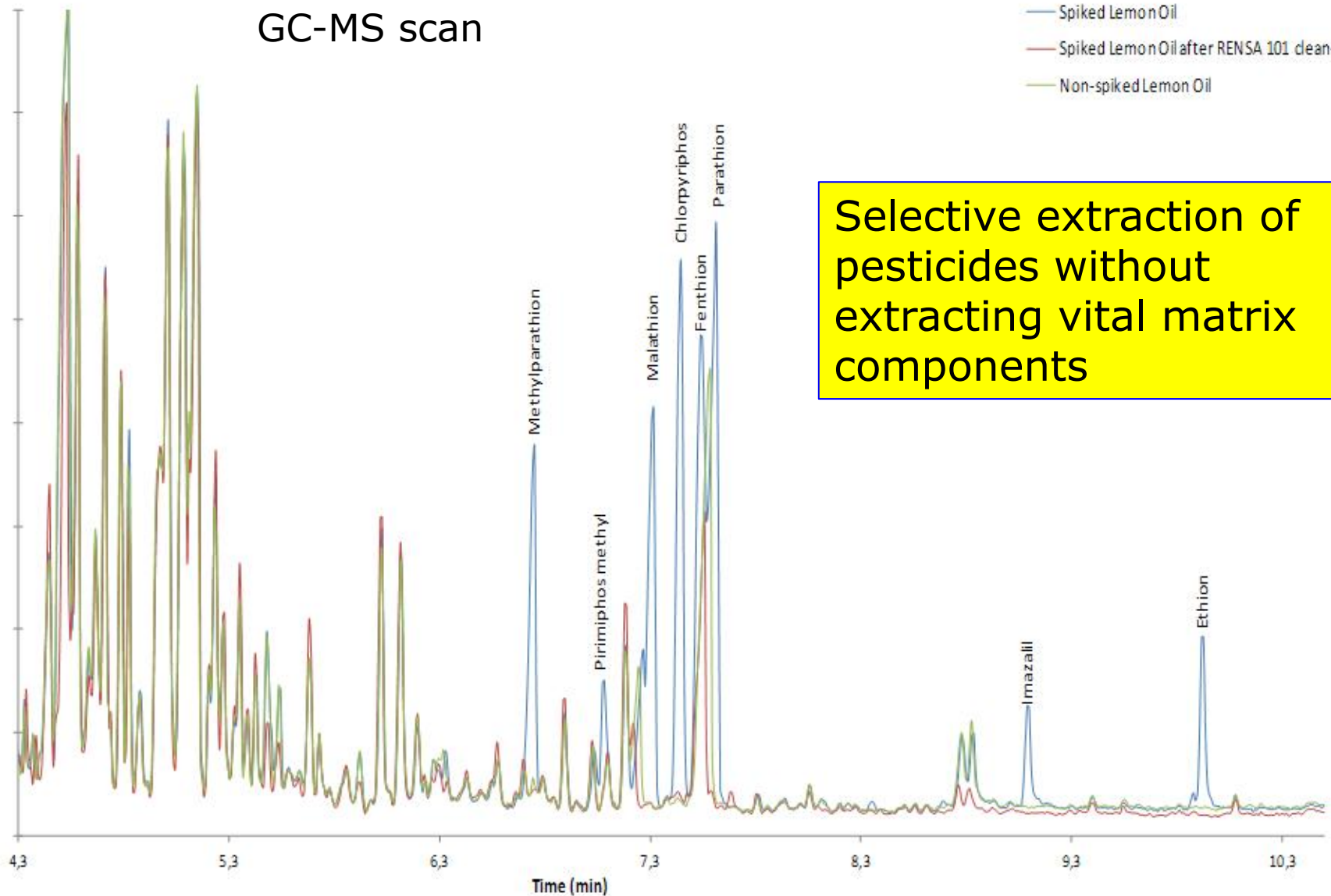
Pesticides ● ◆

Citrus Oil Constituents ●

Selective pesticide removal

GC-MS scan

- Spiked Lemon Oil
- Spiked Lemon Oil after RNSA 101 clean-up
- Non-spiked Lemon Oil



Selective extraction of pesticides without extracting vital matrix components

Unaffected color, flavor and aroma



The cleaned-up lemon oil passed all sensory tests with no impact on the oil quality

- » The company
- » Separation techniques
- » Chromatography, Adsorbents and Selective separations
- » Case study: Selective pesticide removal from lemon oil
- » **Summary**

- » Pressure from regulation will force raw material producers and users to evaluate new technologies
 - » New agricultural residues or other contaminants
 - » New knowledge about problematic natural constituents
- » Separation techniques have pros and cons
 - » Distillation is versatile & established but energy demanding
 - » Old techniques may be expensive and less efficient
 - » Many techniques do not deliver sufficient selectivity
 - » Choosing the right adsorbent + method may be a challenge
- » Novel adsorbents could improve or lead to new quality of essential oils and economics of processing

Thank you!

