Successful Methods to Eliminate Toxic Contaminants from Cannabis Extracts: Part II

Rose Habib, A-1 Extract
David Schurer, Robert Kerr, and Rob Cotta, Sorbent Technologies
WHO IS SORBENT TECHNOLOGIES?

Established in 2000, Sorbent Technologies specializes in products for chromatography and purification. We assist universities, government labs, and industries—Pharma, Biofuel, Cannabis, Food and Beverage, CMOs and CROs—with method development and optimization.

We provide one of the industry’s broadest product lines and the scientific expertise to assist our clients with projects ranging from analytical methods to full-scale process with both standard and complex chemistries.

Sorbtech delivers solutions for small and large production, while working with biomolecules, synthetic, and natural products. Our company is committed to helping chemists identify and offer the most efficient, cost-effective solutions for their applications.

We offer various high quality chromatography products:
- TLC plates
- HPLC columns
- GC columns
- SPE cartridges
- Flash cartridges
- Bulk adsorbents
Sorbtech and Cannabis Purification

For the past six years, we’ve worked with companies in the cannabis industry to assist in extracting high quality CBD, separating cannabinoids, providing analytical products for testing and delivering solutions for the challenging process of decontaminating cannabis extracts in production.

As the cannabis industry continues to grow rapidly and becomes legal in more states, we’ve received daily calls from customers asking to help them decontaminate their cannabis extracts.

While Sorbtech is committed to offering the best solutions to purification challenges, this decontamination issue has become a challenge for us. Why? There are no current standardized methods to utilize stationary phases—such as florisil or silica gel—for this application by the manufacturers of these products.
The Purpose of This E-book
In Part II of this e-book series, we review the current state of the cannabis industry, introduce results we’ve accomplished over the last year, and share new techniques and applications. We’ll also provide direction to those who have contaminated products based on different pesticides that have failed their concentration levels.

Introducing the Kerr Decontamination Method
Dr. Robert Kerr, PhD, the chief scientist at Sorbtech, has developed a new methodology and is conducting ongoing research regarding the decontamination of cannabis and hemp extracts.

Dr. Kerr’s methodology is called the: Kerr Decontamination Method (KDM)
The Challenge
Contamination of cannabis/hemp is a major problem for those in the cannabis industry. We know this because virtually all of our cannabis customers have contacted us to find a solution to remove contaminants from their cannabis and hemp extracts.

We’re committed to providing our customers solutions to produce safe products for consumers. Since we have significant experience in the use of adsorbents in numerous areas, including natural products, we have been able to advise them on what is best for their needs.

You May Be Wondering
Why did we do this? How did we achieve these results? More importantly, how can you achieve these results?

General Objective
To eliminate toxic contaminants (pesticides, herbicides, fungicides, plant growth regulators, and synergists) from contaminated cannabis/hemp extracts with simple cost-effective procedures.
Background
Though it’s an agricultural product, the level of contamination of cannabis and cannabis extracts are not effectively covered by current federal regulations. The reason is simple; under federal law, it’s illegal to possess cannabis. However, FIFRA (Federal Insecticide, Fungicide, and Rodenticide Act of 1910) as Federal law in the United States, still protects applicators, consumers, and the environment in ALL products of ALL types.

Toxic Contaminants—factors to consider:
- Pesticides, herbicides, fungicides, and synergists are used by growers to improve crop quality and increase crop yield profitability
- Even with “organically grown” cannabis, some contaminants are frequently found due to their use on other crops in adjacent fields or persistence from previous applications—both in the aquifer and the soil
- They’re commonly found at the 1 to 50 ppm level per contaminant in plant material
- Extraction processes to remove and concentrate cannabinoids also removes and concentrates the toxic contaminants
- A concentration increase—by a factor of ten of the toxic contaminants—in the extracts, by comparison with the starting plant material, is the usual case

Total Contaminant Load (TCL)
TLC includes all the contaminants (pesticides, herbicides, fungicides, plant growth regulators, and synergists) added together. This is extremely important because many contaminants have a common mode of hazardous action. That is, their toxicity is due to interactions with the same receptor in the human body. If there are six contaminants, each with a passable level, together they pose a threat equal to six times any one of them because they work in the same way in the human body.

Risks
These levels are clearly health risks to users. State health authorities have already begun to mandate “action levels” for specific pesticides, and eventually federal health authorities will follow suit. Currently, only cannabis and its extracts are regulated, although hemp and hemp extracts will be regulated as well.

Questions you should always be asked:

Why?
There’s only a small amount of pesticides in my cannabis/hemp. How can this hurt me?

So what?
More government regulations? More hoops to jump through for business?

Why do this?
Here’s why: Edibles and Smoke/Vape

The “safe” concentration of pesticides in food products (edibles in the cannabis industry) is established, well-known, and relatively high due to poor adsorption through the digestive tract or the skin.

The safe concentration levels of pesticides in cannabis cigarette smoke or vapor from vape products (including hemp) is NOT known. It IS known that substances in smoke are well-absorbed through the mucus membranes into the bloodstream.

Moreover, pyrolysis occurs during partial combustion and numerous secondary products can be generated. Many of these secondary products are toxic.

For example, heat applied to Myclobutanil (Eagle 20) is a common fungicide used for cannabis/hemp and grapes, and when heated (both vape and plant material) it generates Hydrogen Cyanide—which is definitely toxic.

We do know that up to 70% of the toxic contaminants found in the plant material or extracts are also found in the smoke (or vape).

How do we know this?

Scientific experts in our field have measured it. Nicholas Sullivan, Sytze Elzinga, and Jeffrey C. Raber at The Werc Shop, Inc. in Pasadena, California in the USA published the following article in April 2013 in the Journal of Toxicology. Volume 2013. Article ID 378168. 6 pages.
Abstract
The present study was conducted to quantify to what extent cannabis consumers may be exposed to pesticide and other chemical residues through inhaled mainstream cannabis smoke.

Three different smoking devices were evaluated to provide a generalized data set representative of pesticide exposures possible for medical cannabis users. [Four substances] Three different pesticides, bifenthrin, diazinon, and permethrin—along with the plant growth regulator paclobutrazol—which are readily available to cultivators in commercial products, were investigated in the experiment.

Smoke generated from the smoking devices was condensed in tandem chilled gas traps and analyzed with gas chromatography-mass spectrometry (GC-MS). Recoveries of residues were as high as 69.5% depending on the device used and component investigated. This suggests that the potential of pesticide and chemical residue exposures to cannabis users is substantial and may pose a significant toxicological threat in the absence of adequate regulatory frameworks.
Decriminalization – 1973 to 2001

- 1973: Oregon
- 1975: California, Colorado, Maine and Ohio
- 1976: Minnesota
- 1977: North Carolina and Rhode Island

The rest of the states have followed since 2001.

Current Status (July 2019)

- **34** Medical Marijuauna Laws
- **39** MML + CBD Oil
- **11** Recreational Marijuauna
- **11** Marijuana and Hemp Fully Illegal

“First Wave” States
Oregon Was First
Oregon decriminalized cannabis in 1973 and effectively was “first in” with serious regulations of cannabis extracts—and has the most experience in regulating cannabis and cannabis extracts.

Oregon is the model for other states and probably will be at the federal level later on. Since 2019, Oregon requires testing of cannabis products BEFORE they are sold.

Currently, California and Oregon are the ONLY states to require testing for contaminants of non-medical marijuana products.

Background: Current Legal Status in Georgia (July 2019)

Question:
So, now that Hemp is legal in Georgia, what can we expect in terms of regulations concerning contaminants?

Answer:
Let’s look and see what other states have done, starting with Oregon.
Current Regulations
What contaminants are being regulated/tested?

According to the Oregon Health Authorities Technical Report 8964, some 59 contaminants (pesticides, fungicides, plant growth regulators, and synergists) must be tested in cannabis and cannabis-containing products before the products can be sold.

Action levels of individual contaminants range from 0.2 to 2 ppm depending on the type of contaminant.

Being below an “action level” is OK.

Being above an “action level” is NOT OK.

The permitted Total Contaminant Load is 25 ppm.

Note that thousands of pesticides are registered at the US-EPA under FIFRA – but only 59 are required to be tested for in Oregon. The other states will probably follow Oregon’s lead.

Oregon Regulations
Garden Variety Pesticides Versus Cannabis

Comparing the average pesticide consumers purchase and use in their homes and gardens with the concentration of pesticides in cannabis extracts—the numbers are virtually the same.

There can be as much pesticides in Cannabis/Hemp extracts as in the stuff you buy to rid your home of spiders, ants, and roaches.

<table>
<thead>
<tr>
<th>Percent</th>
<th>Parts</th>
<th>PPM</th>
</tr>
</thead>
<tbody>
<tr>
<td>10%</td>
<td>1/10</td>
<td>100,000</td>
</tr>
<tr>
<td>1%</td>
<td>1/100</td>
<td>10,000</td>
</tr>
<tr>
<td>0.5%</td>
<td>1/500</td>
<td>5,000</td>
</tr>
<tr>
<td>0.1%</td>
<td>1/1000</td>
<td>1,000</td>
</tr>
<tr>
<td>0.05%</td>
<td>1/5000</td>
<td>500</td>
</tr>
<tr>
<td>0.01%</td>
<td>1/10,000</td>
<td>100</td>
</tr>
<tr>
<td>0.005%</td>
<td>1/50,000</td>
<td>50</td>
</tr>
<tr>
<td>0.001%</td>
<td>1/100,000</td>
<td>10</td>
</tr>
<tr>
<td>0.0005%</td>
<td>1/500,000</td>
<td>5</td>
</tr>
<tr>
<td>0.0001%</td>
<td>1/1,000,000</td>
<td>1</td>
</tr>
</tbody>
</table>

Concentration of Pesticides at Cannabis Extracts

<table>
<thead>
<tr>
<th>Percent</th>
<th>Parts</th>
<th>PPM</th>
</tr>
</thead>
<tbody>
<tr>
<td>10%</td>
<td>1/10</td>
<td>100,000</td>
</tr>
<tr>
<td>1%</td>
<td>1/100</td>
<td>10,000</td>
</tr>
<tr>
<td>0.5%</td>
<td>1/500</td>
<td>5,000</td>
</tr>
<tr>
<td>0.1%</td>
<td>1/1000</td>
<td>1,000</td>
</tr>
<tr>
<td>0.05%</td>
<td>1/5000</td>
<td>500</td>
</tr>
<tr>
<td>0.01%</td>
<td>1/10,000</td>
<td>100</td>
</tr>
<tr>
<td>0.005%</td>
<td>1/50,000</td>
<td>50</td>
</tr>
<tr>
<td>0.001%</td>
<td>1/100,000</td>
<td>10</td>
</tr>
<tr>
<td>0.0005%</td>
<td>1/500,000</td>
<td>5</td>
</tr>
<tr>
<td>0.0001%</td>
<td>1/1,000,000</td>
<td>1</td>
</tr>
</tbody>
</table>

Question: Is this a REAL problem?

Answer: Yes!

Two Other “Little” Problems

1. There are a LOT of farmers (growers), extractors, formulators, and distributors. In all states, there are an insufficient number of analytical laboratories certified (and qualified) to do the testing. The ratio is 10 to 30 cannabis businesses to one analytical laboratory depending on the state.

2. Cannabis/hemp plant material and extracts are hard to work with. Samples submitted to 10 different laboratories could come back with 10 different answers. Sometimes these answers vary by a factor of 10 (1000%)—sometimes more.
What can be done about this?
Let’s evaluate potential methods to eliminate contaminants from cannabis.

Pesticide-Free Crop Growth
- Right, sounds easy enough—just don’t use pesticides
- But it’s not as easy as it sounds due to persistence of contaminants in the soil and contaminants applied to adjacent fields
- Also, contaminant free water must be used, which requires a significant capital expenditure on equipment and high cost of production in the required volumes

Selective Extraction
- All solvents extract a relatively broad range of substances, and substances with similar characteristics are always extracted together
- If your initial cannabis plant material has toxic contaminants—no matter what solvent you use—then your extract will have the same toxic contaminants at a higher level (10X) than in the original plant material
- The reason—contaminants and cannabinoids have the same properties

RESULT: Selective extraction, for this matrix, is a **MYTH**.

Selective Adsorption
- It works well with the right products and equipment, that is, there are selective adsorbents that achieve the objective.

Chromatographic Purification
- This will work—it DOES work—with the right equipment and trained, experienced people.

Deconstruction of Contaminants by Chemical Reaction
- We transform one or more functional groups required for biological activity into different functional group(s) such that the biological activity (pesticide action, toxicity, whatever) is gone. It’s not as difficult, painful, or expensive as it sounds.
- The reaction must transform the substance into a non-toxic entity. It’s fairly easy to accomplish, but the chemical reaction must also leave the substance(s) of interest intact (cannabinoids in our case). It can be done. See page 20 for more information on this process.
Selective Adsorption
There are numerous available adsorbents—from A to Z.

- Activated Alumina
- Bonded Phase Silica
- Calcium Carbonate
- Carbon Nanotubes
- Cellulose
- Charcoal (Activated Carbon)
- Florisil (Magnesium Silicate)
- Graphitized Silica
- Ion Exchange Resins
- Iron Oxide
- Lignin clays
- Magnesium Oxide
- Modified Cellulose
- Polymeric Adsorbents (non-ionic Reversed Phase Resins)
- Silica (Silicon Dioxide)
- Sorption clays
- Titania (Titanium Dioxide)
- Zeolites
- Zinc Oxide
- Zirconia (Zirconium Dioxide)

Adsorbent Selection Criteria
There are numerous available adsorbents—from A to Z.

1. **Cost**—must be substantially inexpensive or it’s too cost prohibitive
2. **Availability**—must be able to obtain the material
3. **Disposable**—must be able to safely dispose of spent material
4. **Efficient**—must be acceptably efficient that it’s economically viable
5. **Reusable or regenerable**—preferably, should be regenerative or reusable
6. **Selective**—must either selectively retain either the contaminants or the substances of interest
7. **Capacity**—must have sufficient capacity to be useful
Potentially Viable Adsorbents
The following adsorbents pass all the criteria:
- Silica gel
- Bonded phase silica
- Activated alumina
- Florisil (magnesium silicate)
- Polymeric adsorbents

All other potential adsorbents fail one or more criteria.

Procedure for Selective Adsorption Using Silica Gel
1. Dissolve the extract (crude or distillate) in a water-free alkane solvent (such as butane, pentane, hexane, heptane, isooctane, etc).
2. Pass the mixture through a column filled with a drying agent, such as magnesium sulfate or sodium sulfate, to prevent the silica gel from being deactivated by water.
3. Load the mixture (up to 50% by weight) through a column filled with a good quality chromatographic silica gel.
4. After loading, pass more alkane solvent through the column (~2-4CV). This contains the lipophilic pesticides. The cannabinoids are completely retained by the silica gel and cannot be recovered here.
5. Pass about 2-4CV of alkane with 10-25% polar modifier (dichloromethane, ethyl acetate, chloroform) to recover the cannabinoids.
6. Wash the column for regeneration with 100% polar modifier, then 100% methanol.
7. Go back to the initial conditions for the next run with 100% polar modifier, then 100% alkane.
Results from Selective Adsorption Using Silica Gel

Example:
We have customers who are using our procedure daily for large lots of cannabis that are failing primarily due to chlordane.

- 250g extract for 1,000g of silica gel (25% Loading/run, 10 to 20 runs/day).
- Recovery of cannabinoids is virtually 100%.

This procedure is very effective for contaminants that have no retention on silica gel such as: chlordane, etenoprox, chlorfenapyr, and other contaminants.

We have other customers using nearly identical procedures from us to achieve the objective.

Advantages:
- Stable
- Easy to do
- Relatively inexpensive
- Relatively high capacity
- Can be used repeatedly

Limitations:
- It isn’t free
- Equipment is required (pumps, columns, connecting lines, evaporators, etc.)

Procedure for Selective Adsorption Using Florisil PR (Magnesium Silicate)

1. Dissolve the extract (distillate) in a water-free alkane solvent (such as butane, pentane, hexane, heptane, isooctane, and so on)
2. Pass the mixture through a column filled with a drying agent such as magnesium sulfate or sodium sulfate--prevents deactivating the Florisil PR by water
3. Pass the mixture through a column filled with Florisil PR
4. Pass more solvent through (about 5CV) to recover all the extract
5. Evaporate the solvent

Results from Selective Adsorption Using Florisil PR
We have numerous customers in California, Oregon, Colorado, and other states using our procedure to decontaminate cannabis/hemp for lots large and small.

- 150g extract (maximum) for 1,000g of Florisil PR (15% loading).
- Don’t use this procedure if your Total Contaminant Load is more than 10-20ppm.
Another Example:

![Total Contaminant Load](image)

**Note:**
The dilution ratio extract: Heptane used to generate these results was 1:1.

<table>
<thead>
<tr>
<th>Ration of Cannabis to Florisil</th>
<th>ppm</th>
<th>Percent Contaminants Eliminated</th>
<th>Weight of Cannabis Extract (g) per kg of Florisil</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>96.49</td>
<td>0.0%</td>
<td>UNTREATED</td>
</tr>
<tr>
<td>0.125</td>
<td>1.71</td>
<td>98.2%</td>
<td>125</td>
</tr>
<tr>
<td>0.25</td>
<td>2.36</td>
<td>97.6%</td>
<td>250</td>
</tr>
<tr>
<td>0.375</td>
<td>7.78</td>
<td>91.9%</td>
<td>375</td>
</tr>
<tr>
<td>0.5</td>
<td>16.51</td>
<td>82.9%</td>
<td>500</td>
</tr>
<tr>
<td>0.5</td>
<td>11.73</td>
<td>87.8%</td>
<td>500</td>
</tr>
<tr>
<td>1</td>
<td>48.36</td>
<td>49.9%</td>
<td>1000</td>
</tr>
<tr>
<td>2.5</td>
<td>65.73</td>
<td>31.9%</td>
<td>2500</td>
</tr>
<tr>
<td>5</td>
<td>83.66</td>
<td>13.3%</td>
<td>5000</td>
</tr>
<tr>
<td>10</td>
<td>89.9</td>
<td>6.8%</td>
<td>10,000</td>
</tr>
<tr>
<td>15</td>
<td>91.56</td>
<td>5.1%</td>
<td>15,000</td>
</tr>
</tbody>
</table>

**Example:**
- 1kg of extract to 1kg of Heptane

We tested higher dilutions:
- 1kg of extract to 2kg of Heptane
- 1kg of extract to 3kg of Heptane
- 1kg of extract to 4kg of Heptane

The results were superimposable.
Results of Selective Adsorption with Florisil-PR—continued

Bifenzenate is highly reduced but doesn’t pass.

Bifenthrin is slightly reduced but doesn’t pass.

All other contaminants are completely eliminated.

NOTE: Piperonyl Butoxide is completely eliminated

- Note that this procedure “only” eliminated 98.2% of the contaminants at 100ppm TCL and 15% loading (per run, 10 to 20 runs per day).
- This procedure will eliminate 100% of the contaminants with a TCL of <20ppm and a smaller loading – 5-10%.
Results of Selective Adsorption with Florisil-PR—continued

This procedure is:
- very effective for some contaminants
- ineffective for other contaminants
- is the ONLY adsorbent that can eliminate Piperonyl Butoxide*

*PB is a commonly used non-toxic synergist that increases the insecticidal active found in many pesticides and is in majority of commercially available pesticide mixtures.

Advantages
- Stable (if you pay attention to water content)
- Can be regenerated and used again (with scrupulous attention to water content, can be reused at least 5 times)
- Easy to do

Limitations
- Relatively expensive
- Relatively low capacity
- Very sensitive to water content
- Equipment required (pumps, columns, connecting lines, evaporators and so on)

Destruction of Contaminants by Chemical Reaction
- There are many chemical reactions specific to a given functional group
- Functional groups include: alcohols, ketones, alkanes, alkenes, aromatic rings, amines, oximes, amides, and more
- Cannabinoids have a set of functional groups
- Contaminants have a set of functional groups

Selective Chemical Destruction: General Approach
Selective Chemical Destruction: The KDM
We have found a chemical reaction that does NOT react with cannabinoids (or lower molecular weight terpenes, flavonoids, and other phenols) and DOES react with ~75% of regulated contaminants. We have used this reaction—that includes chromatographic cleanup—to 100% decontaminate cannabis and hemp extracts even at VERY high levels (5,000ppm).

- Example 1: Initial Tests
  We took 250g of cannabis distillate already contaminated with ~10 different pesticides (~100ppm Total) and spiked it with 5,000 ppm of 8 different pesticides – and subjected it to our process.

- Example 2: Pilot Runs
  We have worked with 1 to 5kg of contaminated cannabis distillate and subjected it to our process.

In all cases:
- 100% of the contaminants are eliminated
- 100% of the cannabinoids are recovered

This is the MGM of decontamination: Meat-Grinder Method.
Whatever you feed into a meat grinder—sirloin, ribeye, chuck roast, prime rib - comes out as hamburger. Whatever is fed into this method, comes out COMPLETELY DECONTAMINATED.

This is a Proprietary Procedure.
The KDM Method is available for an affordable fee. If you’d like more information on this procedure or would like Sorbtech to manage the process for you, please contact us. We can help, particularly if you’re stuck with contaminated product.

Decontamination of Cannabis/Hemp Extracts
Today, July 2019, a cannabis/hemp producer can take analytical test results and determine which procedure, or combination of procedures, are required to completely eliminate any and all pesticide residues in cannabis products.

Sorbtech has developed several inexpensive cost-effective procedures using readily available adsorbents to deal with extracts with low contamination levels (<10ppm).

We’ve developed an affordable technique to completely decontaminate any cannabis/hemp extract no matter how contaminated it is.
PHONE: 770-936-0323
EMAIL: cannabis@sorbtech.com
WEBSITE: www.sorbtech.com
ADDRESS: 5955 Peachtree Corners E. Norcross, GA 30071