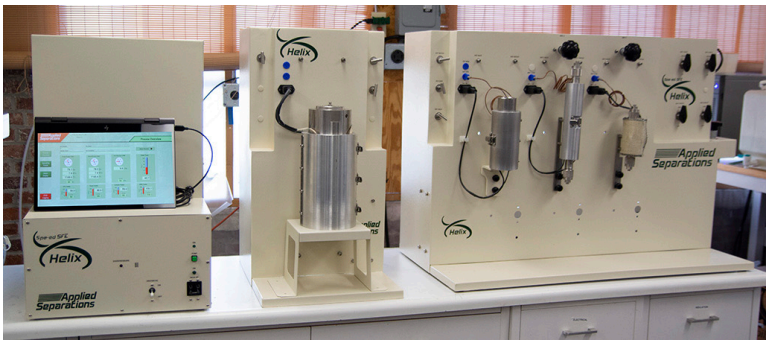
Spe-ed SCF Helix Line of Supercritical CO_2 and Subcritical Water Systems

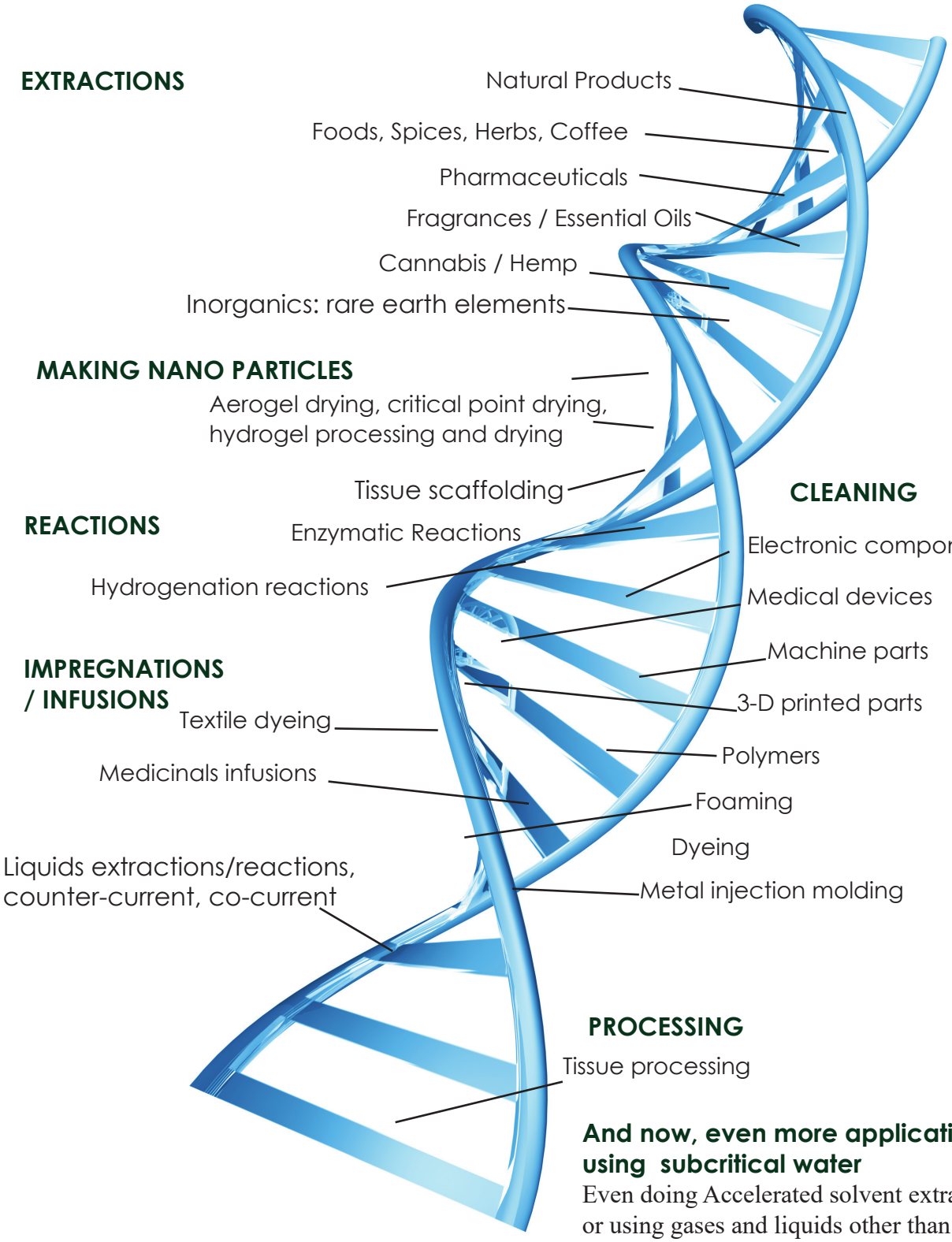


Working with Supercritical Fluids the Endless Applications

Applied Separations

Investigate the Myriad of Applications with the Helix using Existing CO_2 for Supercritical (SC)CO_2 Work

Just as the DNA "double helix" can produce a large variety of different protein configurations, so too does the Applied Separations' Helix give you the opportunity to make system configurations to investigate the widest variety of supercritical fluid applications.



And now, even more applications using subcritical water

Even doing Accelerated solvent extraction or using gases and liquids other than CO_2

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Applied Separations

Helix

For Supercritical Fluids AND/OR Subcritical Water

The Helix is made up of several “base” components. The basic components are put together in a variety of standard or custom configurations to make the system perform the applications you need.

With the base system you will be able to use some of the same components to do separations and extractions as well as many other applications. One day do extractions and on another day do something completely different, like infusions or making nano particles.

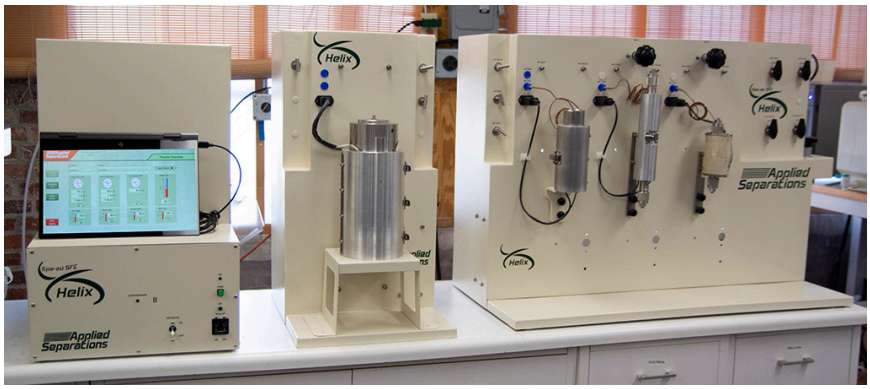
Basic Requirements for All Extractions

All extractions require just these 3 Applied Separations, Inc. components:

- Base unit (Helix Basic)
- CO_2 Pump
- Pressure vessel assembly

and these utilities:

- Recirculating bath (chiller)
- A source of air delivered at 7 BAR Electrical power: 240v or 120v
- Source of liquid CO_2 or water.



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The compact Helix Basic is the simplest configuration.

It consists of the:

- CO_2 pump module,
- computer/laptop interface and
- platform holding the pressure vessel

The system can accommodate pressure vessels ranging in size from 24 ml to 1 liter*.

Input, output and vent lines are controlled by shutoff valves located on the front of the unit.

Pressure vessels are heated by specially designed heater assemblies that accurately control the temperature.

An additional CO_2 preheater is employed to ensure that the CO_2 is at the designed temperature before entering the pressure vessel.

A back pressure regulator controls the flowrate of gaseous CO_2

The Helix Basic is controlled through ASI vision software.

The Helix Basic and ALL Helix systems have these features:

- temperatures up to 240°C – no technical limitations because of not having sufficient heat
- pressure up to 10,000 psi (690 BAR) – get maximum density and solvating power, don't leave extracts in the pressure vessel
- holds 24mL to 1L vessels standard; up to 5 liter available – start with a small sample and scale up—no problem
- pump flow rates up to 400mL/min (more with 5 liter vessel add-in) extract quickly and efficiently**
- fully-adjustable, non-clogging, micrometering valves control CO_2 flow rate
- collection into standard glassware, chilled collection to capture volatile compounds, on SPE for further chromatographic separations
- in-vessel chromatographic capabilities
- modifier addition capability – extract polar compounds, too
- multiple flow path – efficiency, efficiency, efficiency
- extract liquid samples as liquids
- stirrer capability –
- view cell…capability to look inside of vessel – keep a video record
- configurations for multiple/varied applications, check applications sections

* larger vessels can also be added in customized systems

**flow rate based on incompressible liquid

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Pressure Vessels and Separators

Pressure vessels are heated by specially designed heater assemblies (“clamshell”) that accurately control the temperature.

An additional CO_2 preheater is employed to ensure that the CO_2 is at the designed temperature before entering the pressure vessel.

A back pressure regulator controls the flow rate of gaseous CO_2 if exiting to ambient collection or regulating the pressure in a downstream pressure vessel (e.g. cyclone separator).

Fractionation and Separators

One of the major advantages of supercritical CO_2 is to be able to fractionate samples by manipulating the pressure in the separator. You can go from extracting at a high pressure to discharging an extract at ambient pressure and temperature. Or, the Helix gives you the option of reducing the pressure in a series of steps.

You pick the number of pressure drops - separators - you need for your evaluations. The Helix offers you up to 3 intermediate separators before ending at an ambient condition. With the new Helix configuration, the extractor and the separators are loaded on to a single, operator friendly frame.

Manupulate the Supercritical Pressure

To Change the Density

Which will Change the Solvating Power

Allowing You to Selectively Dissove Compounds

and Fractionate

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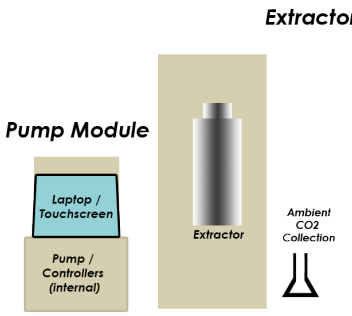
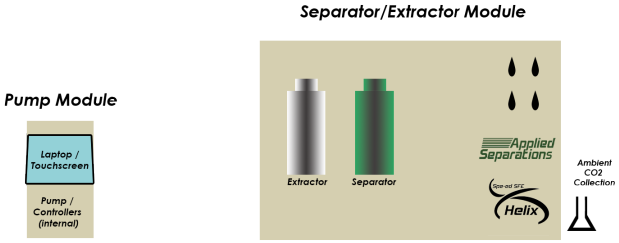
The Many Configurations of the Helix

0 CO2 Separators



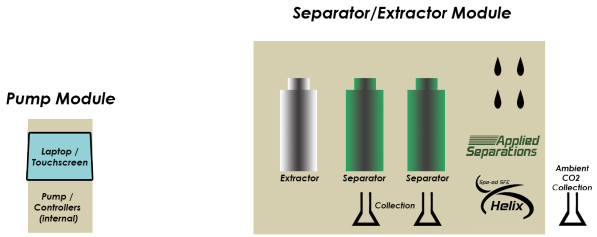
Basic Helix
Helix CO_2 Extraction System with *No CO_2 Separators*

1 CO2 Separator



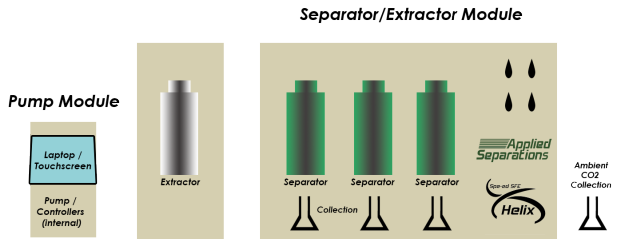
Helix CO_2 Extraction System with *1 CO_2 Separator*

2 CO2 Separators



Helix CO_2 Extraction System with *2 CO_2 Separators*

3 CO2 Separators



Helix Extraction System with *3 CO_2 Separators*

Exact configurations of extractors and separators may vary with a system designed for your specific application.

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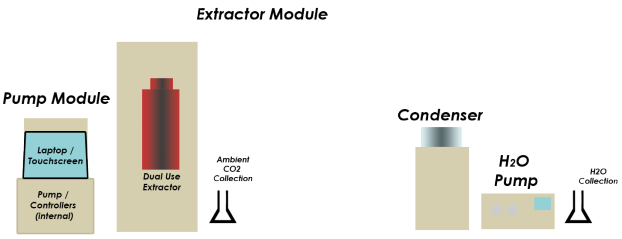
Applied Separations

Now let's give you even more options with the Helix... *Subcritical Water*

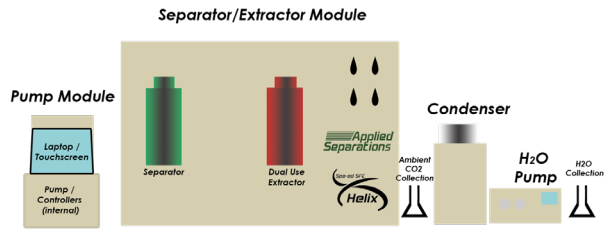
You can combine both technologies on a single Helix platform. Do both supercritical CO_2 and Subcritical water

0
CO2 Separators



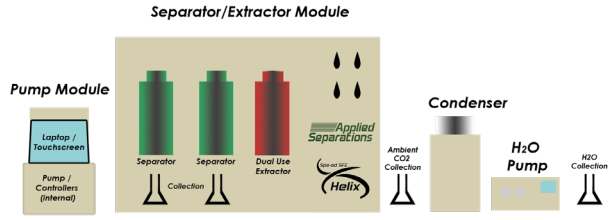
Helix CO_2 / Subcritical H_2O Extraction System with
- No CO_2 Separators
- Dual Use Extractor Vessel
- Subcritical Water

1
CO2 Separators



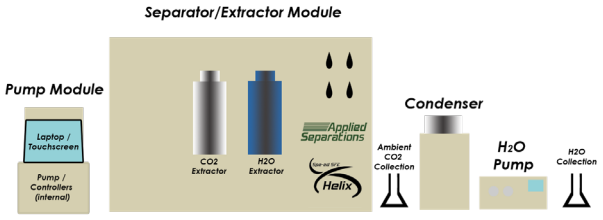
Helix CO_2 / Subcritical H_2O Extraction System with
- 1 CO_2 Separator
- Dual Use Extractor Vessel
- Subcritical Water

2
CO2 Separators



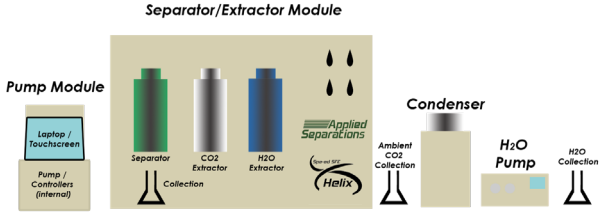
Helix CO_2 / Subcritical H_2O Extraction System with
- 2 CO_2 Separators
- Dual Use Extractor Vessel
- Subcritical Water

0
CO2 Separators



Helix CO_2 / Subcritical H_2O Extraction System with Separate CO_2 /H_2O Extractor Vessels and
- CO_2 Extractor Vessel
- No CO_2 Separator
- Subcritical Water

1
CO2 Separator



Helix CO_2 / Subcritical H_2O Extraction System with Separate CO_2 /H_2O Extractor Vessels and
- 1 CO_2 Separator
- Subcritical Water

Exact configurations of extractors and separators may vary with a system designed for your specific application.

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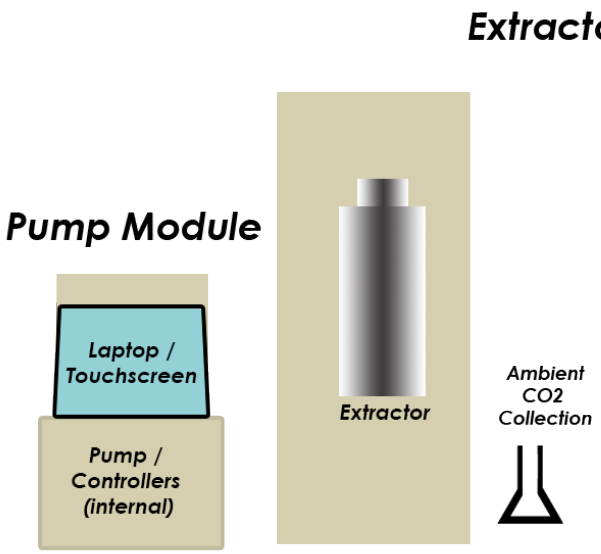
Helix Basic
CO_2 Extraction System with
No CO_2 Separator

The Helix Basic give you the opportunity to extract samples up to one liter in volume at up to 10,000 psi and up to 240°C and discharge the extract to an ambient condition.

- temperatures to 240°C – no technical limitations because of not having sufficient heat
- pressure up to 10,000 psi (690 BAR) – get maximum density and solvating power, don't leave extracts in the pressure vessel
- holds 24mL to 1L vessels standard – start with a small sample and scale up—no problem
- pump flow rates up to 400mL/min - extract quickly and efficiently*
- fully-adjustable, non-clogging, micrometering valves control flow rate
- collection into standard glassware, chilled collection to capture volatile compounds, on SPE for further chromatographic separations
- in-vessel chromatographic capabilities
- modifier addition capability – extract polar compounds, too
- multiple flow path – efficiency, efficiency, efficiency
- extract liquid samples as liquids
- stirrer capability
- view cell…capability to look inside of vessel – keep a video record
- configurations for multiple/varied applications, check applications sections



One of the many thousands of applications might be the collection of soluble oils from natural products. Soluble oils may be dissolved in high pressure supercritical CO_2 and collected when depressurized to atmospheric conditions. For example, soluble lipid components from rose-hip seeds, flax seeds etc can be easily dissolved in CO_2 and collected in an atmospheric collection container.

*flow rate based on incompressible liquid

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Applied Separations

Helix CO_2 Extraction System with *1 CO_2 Separator*

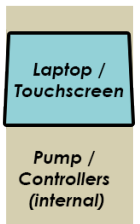
The Helix CO_2 Extraction System with 1 CO_2 separator give you the opportunity to extract samples up to one liter in volume at up to 10,000 psi and up to 240°C and discharge the extract to a separator or an ambient condition.

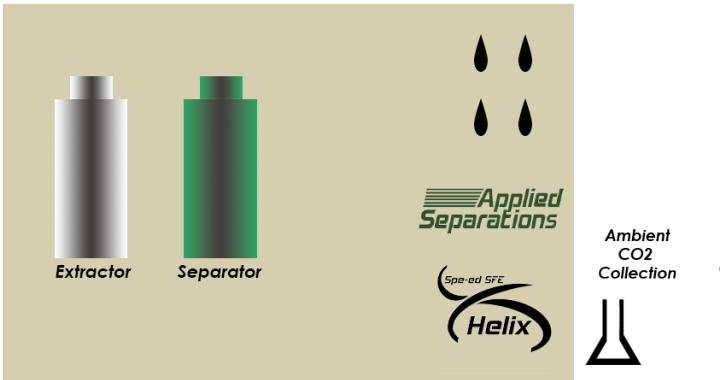
An example might be cloves contain 2 major components that may be dissolved in supercritical CO_2 and separated.
A dark oily resin can be collected in separator 1 and an essential oil fraction in the final atmospheric collector.

- temperatures to 240°C – no technical limitations because of not having sufficient heat
- pressure up to 10,000 psi (690 BAR) – get maximum density and solvating power, don't leave extracts in the pressure vessel
- holds 24mL to 1L vessels standard;– start with a small sample and scale up—no problem
- pump flow rates up to 400mL/min - extract quickly and efficiently*
- fully-adjustable, non-clogging, micrometering valves control flow rate
- collection into standard glassware, chilled collection to capture volatile compounds, on SPE for further chromatographic separations
- in-vessel chromatographic capabilities
- modifier addition capability – extract polar compounds, too
- multiple flow path – efficiency, efficiency, efficiency
- extract liquid samples as liquids
- stirrer capability
- view cell - capability to look inside of vessel – keep a video record
- configurations for multiple/varied applications, check applications sections

Separator/Extractor Module

Pump Module





Exact configurations of extractors and separators may vary with a system designed for your specific application.

*flow rate based on incompressible liquid

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Applied Separations

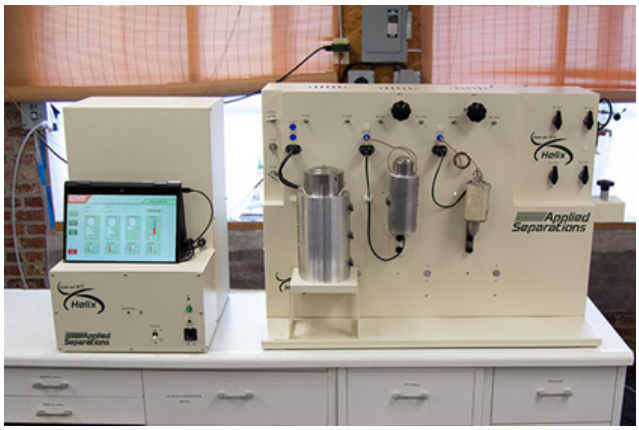
Helix CO_2 Extraction System with *2 CO_2 Separators*

The Helix CO_2 Extraction System with 2 CO_2 separators gives you the opportunity to extract samples normally up to one liter in volume at up to 10,000 psi and up to 240°C and discharge the extract to 2 different separators; cascading in decreasing pressure from separator #1 to separator #2 and then to ambient collection.

- temperatures to 240°C – no technical limitations because of not having sufficient heat
- pressure up to 10,000 psi (690 BAR) – get maximum density and solvating power, don't leave extracts in the pressure vessel
- holds 24mL to 1L vessels standard;– start with a small sample and scale up—no problem
- pump flow rates up to 400mL/min - extract quickly and efficiently*
- fully-adjustable, non-clogging, micrometering valves control flow rate
- collection into standard glassware, chilled collection to capture volatile compounds, on SPE for further chromatographic separations
- in-vessel chromatographic capabilities
- modifier addition capability – extract polar compounds, too
- multiple flow path – efficiency, efficiency, efficiency
- extract liquid samples as liquids
- stirrer capability
- view cell - capability to look inside of vessel – keep a video record
- configurations for multiple/varied applications, check applications sections

Where this configuration might be used is the extraction black pepper. Black Pepper contains several distinct components that may be dissolved in supercritical CO_2 at 500 bar and 90°C and separated as the pressure is sequentially lowered.

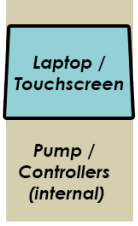
- A resinous material is collected in separator #1,
- a piperine concentrate in separator #2 and
- an essential oil in the final collection container at atmospheric pressure.

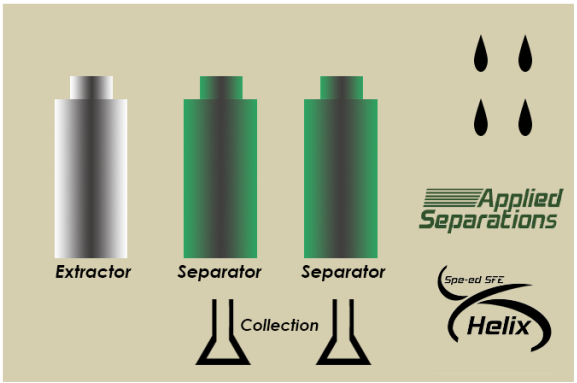


Exact configurations of extractors and separators may vary with a system designed for your specific application.

Separator/Extractor Module

Pump Module





*flow rate based on incompressible liquid

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Applied Separations

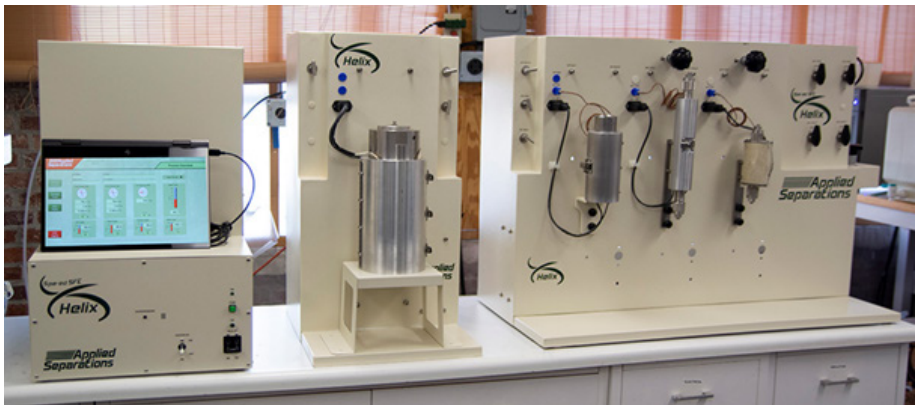
Helix Extraction System with *3 CO_2 Separators*

The Helix CO_2 Extraction System with 3 CO_2 separators gives you the opportunity to extract samples up to one liter in volume at up to 10,000 psi and up to 240°C and discharge the extract to 3 different separators; cascading in decreasing pressure from separator #1 to separator #2, to separator #3 and then to ambient collection.

- temperatures to 240°C – no technical limitations because of not having sufficient heat
- pressure up to 10,000 psi (690 BAR) – get maximum density and solvating power, don't leave extracts in the pressure vessel
- holds 24mL to 1L vessels standard;– start with a small sample and scale up—no problem
- pump flow rates up to 400mL/min - extract quickly and efficiently*
- fully-adjustable, non-clogging, micrometring valves control flow rate
- collection into standard glassware, chilled collection to capture volatile compounds, on SPE for further chromatographic separations
- in-vessel chromatographic capabilities
- modifier addition capability – extract polar compounds, too
- multiple flow path – efficiency, efficiency, efficiency
- extract liquid samples as liquids
- stirrer capability
- view cell - capability to look inside of vessel – keep a video record
- configurations for multiple/varied applications, check applications sections

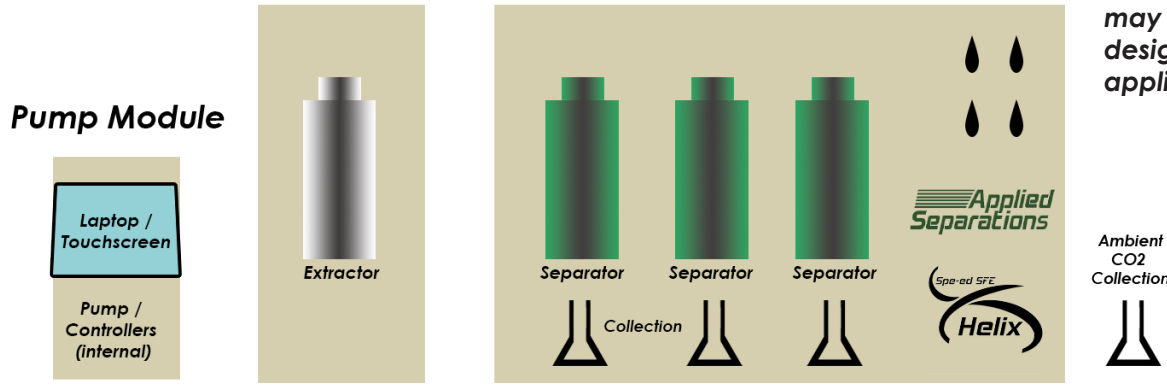
Many CO_2 soluble polymers may be fractionated by molecular weight including silicones, polycarbosilanes, and perfluoropolyethers. For example, polydimethylsiloxane with an average molecular weight of approximately 90,000 can be dissolved in supercritical CO_2 at 80°C and 600 bar and fractionated as the pressure is reduced sequentially from 450 bar to atmosphere.

- Separator# 1 contains an average molecular weight of 150,000,
- Separator #2 contains an average molecular weight of 75,000
- Separator#3 contains an average molecular weight of 46,000
- The ambient collector contains an average molecular weight of 1500.



Separator/Extractor Module

Exact configurations of extractors and separators may vary with a system designed for your specific application.



*flow rate based on incompressible liquid

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Helix CO_2 /Subcritical H_2O Dual Use Extraction System with *No CO_2 Separators*

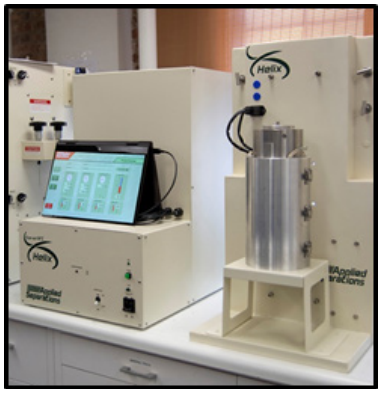
In this Helix configuration with $SCCO_2$ and Subcritical H_2O, the same extractor vessel is used for both types of extractions. They cannot be used simultaneously. However, after clean-up from one operation, the other technique can be employed.

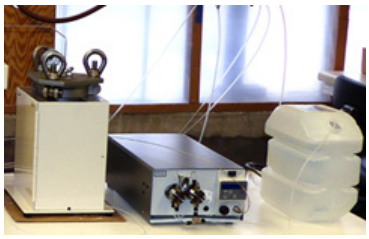
The supercritical CO_2 part of the configuration is the same as the Basic Helix, shown on page 8.

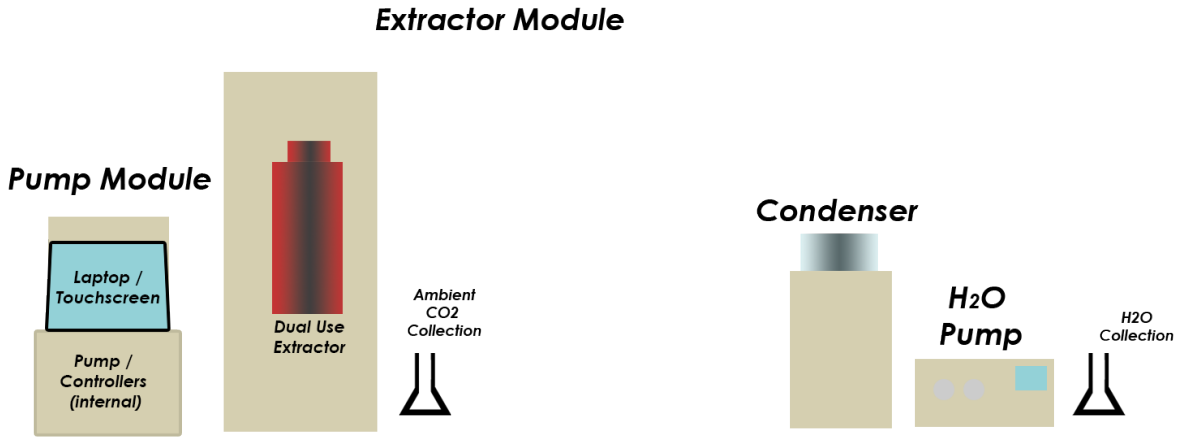
In order to do subcritical water extractions, the Helix employs:

- a separate liquid pump to pump the water into the extractor vessel
- a separate collection/condensation system to safely condense the "superheated steam" and collect the desired extracts
- Applied Separations, Inc. standard vessels configured to subcritical water achieve 240°C. Optionally, vessels can be configured to achieve 300°C.

A potential operation would be to perform a supercritical CO_2 extraction like doing a cleanup and then do a subcritical water extraction like extracting flavonoids and polyphenolics from orange peels including the flavonones, hesperidin and narirutin.







Exact configurations of extractors and separators may vary with a system designed for your specific application.

Helix CO_2 /Subcritical H_2O Dual Use Extraction System with *1 CO_2 Separator*

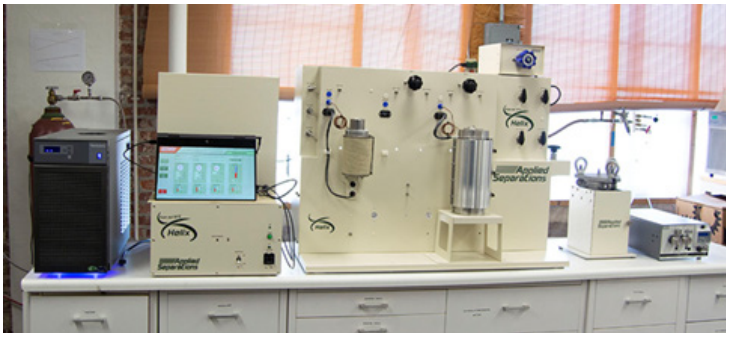
In this Helix configuration with $SCCO_2$ and Subcritical H_2O, the same extractor vessel is used for both types of extractions. They cannot be used simultaneously. However, after clean-up from one operation, the other technique can be employed.

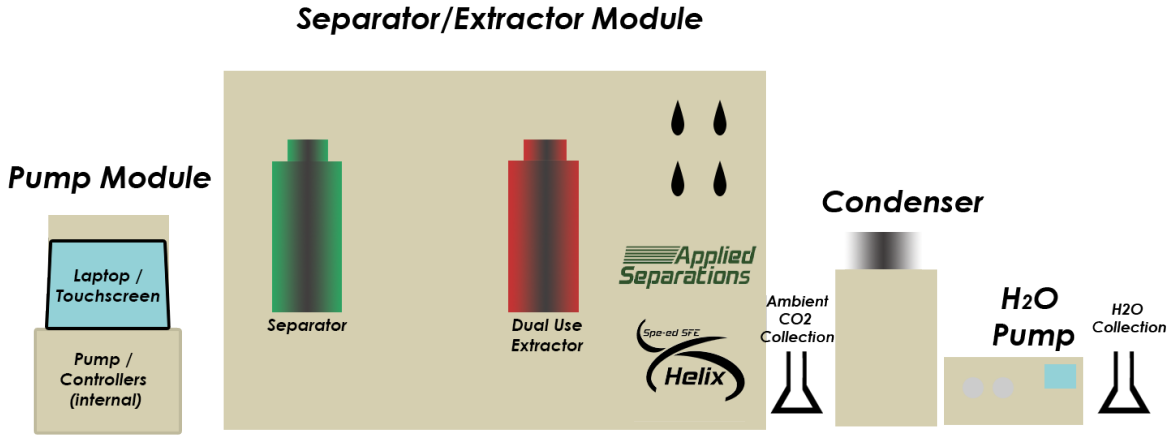
The supercritical CO_2 part of the configuration is the same as the Helix with one separator, shown on page 9.

In order to do subcritical water extractions, the Helix employs:

- a separate liquid pump to pump the water into the extractor vessel
- a separate collection/condensation system to safely condense the "superheated steam" and collect the desired extracts
- Applied Separations, Inc. standard vessels configured to subcritical water achieve 240°C. Optionally, vessels can be configured to achieve 300°C.

A potential operation would be to perform a supercritical CO_2 extraction like doing a cleanup followed by a simultaneous subcritical water extraction of phenolics (eriocitrin) and essential oils (menthol) from peppermint leaves.





Exact configurations of extractors and separators may vary with a system designed for your specific application.

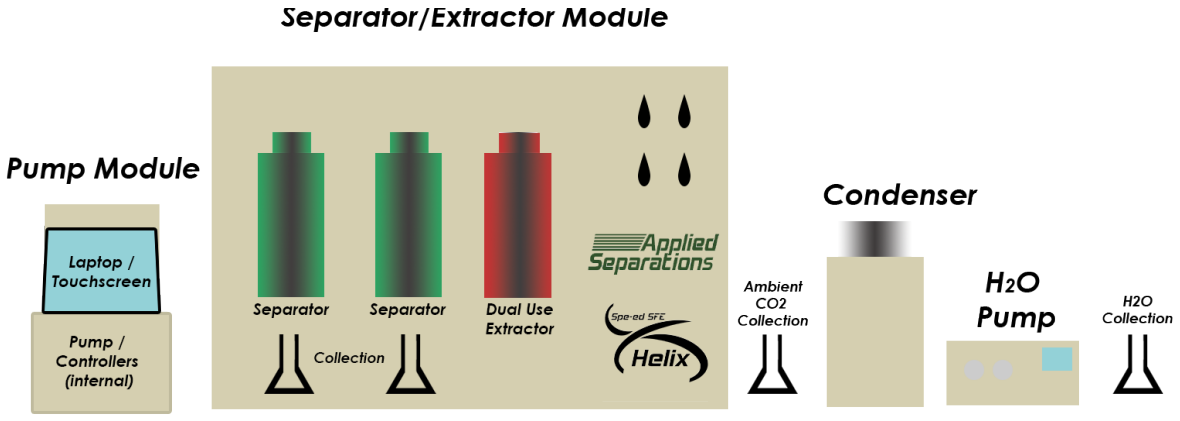
Helix CO_2 /Subcritical H_2O Dual Use Extraction System with *2 CO_2 Separators*

In this Helix configuration with $SCCO_2$ and Subcritical H_2O, the same extractor vessel is used for both types of extractions. They cannot be used simultaneously. However, after clean-up from one operation, the other technique can be employed.

The supercritical CO_2 part of the configuration is the same as the Helix with two separators, shown on page 10.

In order to do subcritical water extractions, the Helix employs:

- a separate liquid pump to pump the water into the extractor vessel
- a separate collection/condensation system to safely condense the "superheated steam" and collect the desired extracts
- Applied Separations, Inc. standard vessels configured to subcritical water achieve 240°C. Optionally, vessels can be configured to achieve 300°C.



Exact configurations of extractors and separators may vary with a system designed for your specific application.

Helix CO_2 /Subcritical H_2O Extraction System with

- ***Separate Dedicated CO_2 Extractor Vessel***
- ***No CO_2 Separator***
- ***Separate Dedicated H_2O Extractor Vessels***

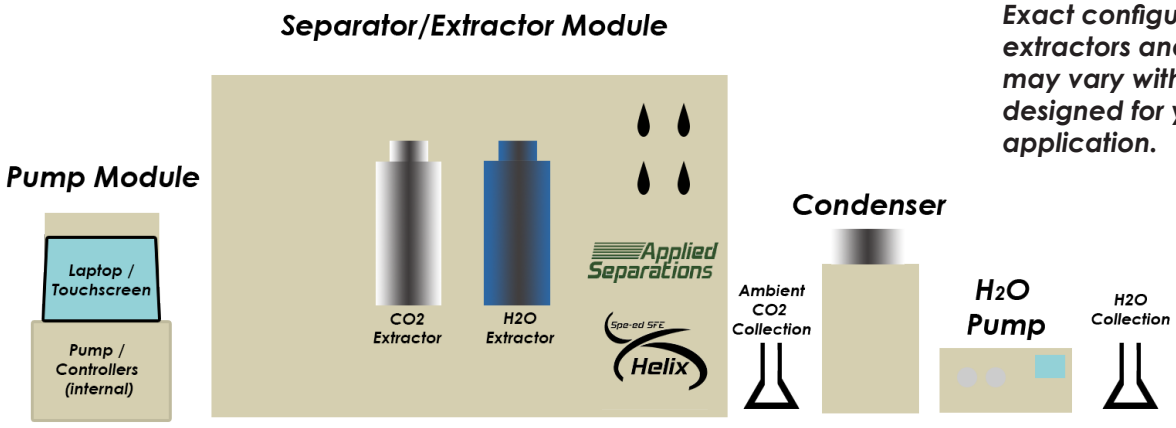
In this Helix configuration with $SCCO_2$ and Subcritical H_2O, there are two extractor vessels. One is dedicated to supercritical CO_2 and the other is used for subcritical water. Although requiring attention, it is possible to operate both techniques simultaneously.

The supercritical CO_2 part of the configuration is the same as the Basic Helix, shown on page 8.

In order to do subcritical water extractions, the Helix employs:

- a separate liquid pump to pump the water into the extractor vessel
- a separate collection/condensation system to safely condense the "superheated steam" and collect the desired extracts
- Applied Separations, Inc. standard vessels configured to subcritical water achieve 240°C. Optionally, vessels can be configured to achieve 300°C.

A potential operation would be to perform a simultaneous supercritical CO_2 extraction while also doing a subcritical extraction of polyphenols and flavonoids from grape pomace.



Exact configurations of extractors and separators may vary with a system designed for your specific application.

Applied Separations

Helix CO_2 /Subcritical H_2O Extraction System with

- ***Dedicated CO_2 Extractor Vessel***
- ***Dedicated H_2O Extractor Vessel***
- ***1 CO_2 Separator***

In this Helix configuration with $SCCO_2$ and Subcritical H_2O, there are three extractor vessels. One is dedicated to supercritical CO_2, one is used for subcritical water, and the other is a CO_2 separator. Although requiring attention, it is possible to operate both techniques simultaneously.

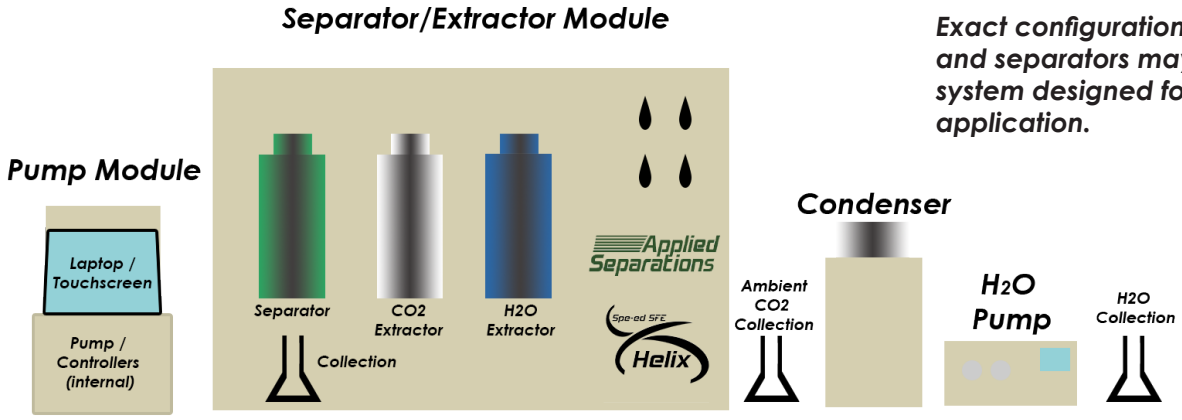
The supercritical CO_2 part of the configuration is the same as the Helix with one separator, shown on page 9.

In order to do subcritical water extractions, the Helix employs:

- a separate liquid pump to pump the water into the extractor vessel
- a separate collection/condensation system to safely condense the "superheated steam" and collect the desired extracts
- Applied Separations, Inc. standard vessels configured to subcritical water achieve 240°C. Optionally, vessels can be configured to achieve 300°C.

A potential operation would be to perform a simultaneous supercritical CO_2 extraction while also doing a subcritical extraction of

- theobromine, caffeine, theophylline,
- epicatechin, catechin, chlorogenic acid and gallic acid from waste cocoa shells including
- valuable degradation products such as 5-hydroxymethylfurfural (5-HMF), furfural, levulinic acid, lactic acid and formic acid.

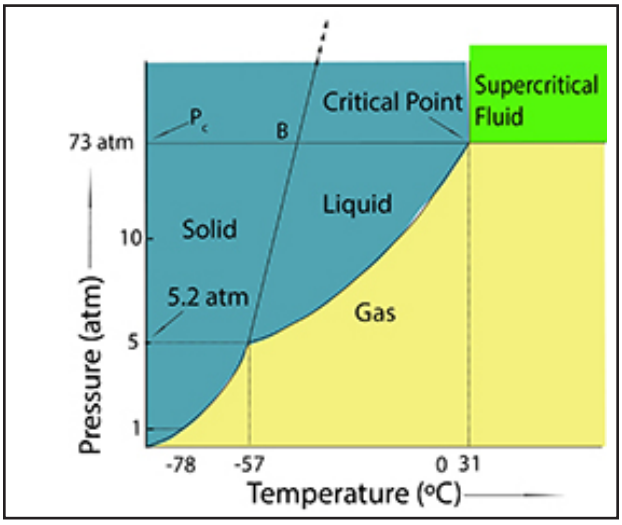


Exact configurations of extractors and separators may vary with a system designed for your specific application.

Applied Separations

Why Supercritical Fluids?

Carbon dioxide is in its supercritical fluid state when both the temperature and pressure equal or exceed the critical point of 31°C and 73 atm (see diagram). In its supercritical state, CO_2 has both gas-like and liquid-like qualities, and it is this dual characteristic of supercritical fluids that provides the ideal conditions for extracting compounds with a high degree of recovery in a short period of time.



Supercritical Fluids "Green" Revolutionize Your Processes

No longer an exotic laboratory curiosity, but now a cost-effective tool to improve your process development.

Use "green" existing carbon dioxide and no solvents

By controlling or regulating pressure and temperature, the density, or solvent strength, of supercritical fluids can be altered to simulate organic solvents ranging from chloroform to methylene chloride to hexane.

This dissolving power can be applied to purify, extract, fractionate, infuse, and recrystallize a wide array of materials.

Because CO_2 is non-polar, a polar organic co-solvent (or modifier) can be added to the supercritical fluid for processing polar compounds. By controlling the level of pressure/temperature/modifier, supercritical CO_2 can dissolve a broad range of compounds, both polar and non-polar.

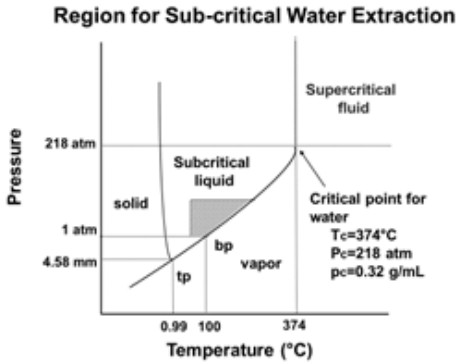
Not only can the supercritical fluid act as a solvent, it also has the characteristics of a gas. One can apply changes in pressure to effective foaming, diffusion, and many other additional physical parameters. The applications are endless, limited only to one's imagination.

Why Subcritical Water?

Water can be physically altered to dissolve non polar compounds

There is a great demand for environmentally friendly extraction procedures that eliminate the need for toxic organic solvents. Supercritical carbon dioxide is one and has been highlighted here. However, one can also use water in some innovative ways.

Water can be held in a liquid state above its normal boiling temperature (100°C) by increasing the pressure. This process is demonstrated using a simple pressure cooker. Water above its normal boing point but below its supercritical temperature (374°C), is "subcritical" water.



Water can be used as an alternative to toxic organic solvents because the dissolving power of water can be changed by changing its temperature. Water can be physically altered to become "subcritical" water

Polar

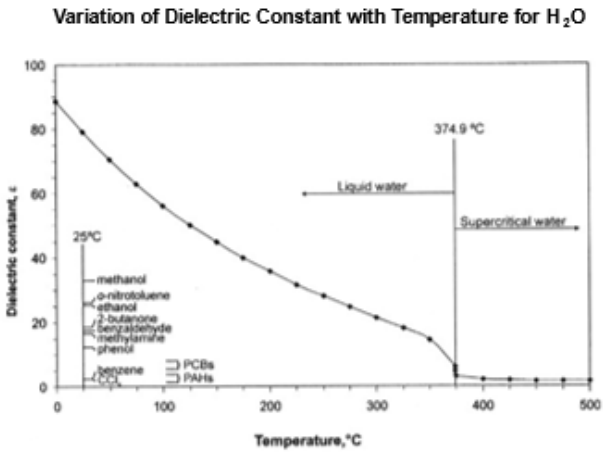
- Water at room temperature has a high dielectric constant - dissolving polar and ionic compounds.

More Non Polar

- Water at high temperatures has a lower dielectric constant - dissolving non-polar organic compounds

Viscosity and surface tension decrease

- Viscosity and surface tension decrease with increasing temperature
- Solubility of solutes increases
- Solute diffusion increases, decreasing time to solubilize



Replace alcohol with subcritical water

- Since the dielectric constant of water decreases significantly as it is heated, it can behave like alcohol

Subcritical water may be used to extract many organic molecules

- Eliminate the use of conventional organic solvents.

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