Supercritical Fluid Extraction/Chromatograph System

Nexera UC
Unified Chromatography…
Just another chromatographic technique… or the only technique you’ll need?

Conventional LC/MS and GC/MS face these challenges…

- Time-consuming sample preparation
- Degradation of labile compounds during sample preparation
- Low abundant peaks hidden in noise

**Nexera UC** provides uncompromising solutions to these problems.

**Fully automated on-line sample preparation and analysis**

Target compounds are automatically extracted and analyzed.

**Even labile compounds can be analyzed without degradation**

Sample extraction is performed under light-shielding and anaerobic conditions, preventing the degradation of labile analytes.

**Unified speed of analysis, sensitivity, and resolution**

Supercritical fluid enables highly efficient sample extraction and high resolution chromatographic analysis. The result; improved sensitivity and throughput for multi-analyte analyses.

Supercritical fluid is a fluid over its critical point. It has unique properties like liquid and gas. Low viscosity, high diffusion coefficient, liquid-like dissolving power. CO₂ is most popular for use.
Unified Chromatography

Nexera UC improves your analytical workflow by utilizing a completely new separation technology, Unified Chromatography, which unites sample separation, analysis with various separation modes, and high-sensitivity detection.

SFE (Supercritical Fluid Extraction) : An extraction method using supercritical fluid. It is available as a pretreatment method for solid sample analyses.

SFC (Supercritical Fluid Chromatography) : The chromatographic technique using supercritical fluids as mobile phases. With its unique properties, it enables high-speed, high-resolution analyses.
Fully automated on-line sample preparation and analysis of target compounds

Nexera UC on-line SFE-SFC is a revolutionary system that combines on-line SFE and SFC in a single flow path. Target compounds are extracted from solid samples and then automatically transferred to SFC/MS so that no human intervention is required. The Nexera UC on-line SFE-SFC system reduces the time for pretreatment of samples and acquires highly accurate data.

Comparison of QuEChERS sample preparation and Nexera UC in the analysis of residual pesticides

A typical sample preparation takes 35 minutes and requires several manual steps. With Nexera UC, the same sample can be ready for on-line SFE/SFC analysis in as little as five minutes with only a few simple sample preparation steps.

QuEChERS — Requires **35** minutes of sample preparation

**Nexera UC** — As little as **5** minutes to be ready for analysis

* “Miyazaki Hydro-Protect”, Patented in Japan No. 3645552
Hundreds of compounds are simultaneously analyzed with Nexera UC. These include pesticides that are usually analyzed with LC and LC/MS/MS or GC and GC/MS/MS. Nexera UC enables analysis of compounds over a wide polarity range.

- Pesticide compound analysis with GC or GC/MS/MS
  1. Diazinon
  2. Metalaxyl
  3. Tolclofos-methyl
  4. Lenacil
  5. Mepronil
  6. Dioxathion
  7. Cypermethrin

- Pesticide compound analysis with LC or LC/MS/MS
  8. Aramite
  9. Isonuron
  10. Acephate
  11. Aminocarb
  12. Cyazofamid
  13. Diquat
  14. Chromafenozide
  15. Imidacloprid
Prevent degradation of labile compounds

With conventional solvent extraction, labile compounds may react with extraction solvents or could be oxidized and/or degraded.

Analysis of biomarkers from dried blood spots (DBS)

Nexera UC can extract a trace amount of liquid samples. For biomarker validation, the preparation requires simply enclosing a blood spot in the 0.2 mL special extraction vessel.
Only a few simple preparation steps for cleaning validation

Nexera UC can be applied to cleaning validation which is performed in the pharmaceutical industry to confirm that manufacturing equipment has been properly cleaned. Nexera UC automatically runs a series of steps from extraction to analysis, by only putting the sample swab in the extraction vessel. In conventional cleaning validation, the sample swab needs to be extracted with water, and then the extraction is analyzed by TOC. However, when a target compound is hydrophobic, swab extraction is performed with ethanol and TOC is not applicable. Nexera UC is capable of performing both types of cleaning validation.

Polymer additives are widely used to prevent optical or thermal degradation, or to enhance functionalities. An example, Irganox 1010, is insoluble in THF or Chloroform, and hard to sublime. Therefore, analysis by GPC or GC is difficult. By simply homogenizing a sample and enclosing it in the extraction vessel, Nexera UC can detect labile or trace quantities of additives.

Supercritical fluid extraction of trace additives in polymers

Polymer additives are widely used to prevent optical or thermal degradation, or to enhance functionalities. An example, Irganox 1010, is insoluble in THF or Chloroform, and hard to sublime. Therefore, analysis by GPC or GC is difficult. By simply homogenizing a sample and enclosing it in the extraction vessel, Nexera UC can detect labile or trace quantities of additives.
Unified speed of analysis, sensitivity, and resolution

Solutions provided by Nexera UC

- Very fast separation speed due to the relatively low viscosity of supercritical fluid
- Improved peak capacity and chromatographic resolution
- Efficient separation of analogues and/or chiral compounds by high penetration mobile phase
- Different separation mode leads to high sensitivity
- Improved sensitivity resulting from split-less introduction into detector
- Reduction of environmental impacts and costs by reducing amount of organic solvents needed

Higher resolution

Improved separation and detection capabilities result from the low viscosity and high diffusion coefficient of supercritical fluid. As shown below, Nexera UC demonstrates high-separation selectivity for isomeric compounds that are difficult to separate by conventional LC.

Comparison of retention time and separation acquired by Conventional LC and SFC (sample: α-tocopherol)
Supercritical fluid has unique properties different from liquid.

Using SFC in front of a mass spectrometer offers greater sensitivity than achieved with LC/MS/MS.

**Sensitivity results from different separation modes in HPLC vs SFC**

Comparison of peak intensity detected by the same MS detector.
(Sample: Prostagrandin D2 10 pg)

**Enhanced sensitivity using splitless transfer to MS**

Low dead volume back pressure regulator suppresses diffusion of peaks and can transfer the total eluate directly to a mass spectrometer to achieve higher sensitivity.

Comparison of intensity of two peaks detected by the same MS detector.
(Sample: Reserpine 10 pg)
Easy and efficient method scouting for separating chiral compounds

Automatically performs a variety of method scouting processes

The high-speed performance of SFC can shorten the time required for method scouting. It automatically generates a large number of methods by utilizing combinations of up to 12 columns, four modifiers, and different ratios of modifiers to mobile phase.

Chiral analysis with “Nexera UC Chiral Screening System”

CHIRALPAK® Series and CHIRALCEL® Series columns (Daicel Corporation) for chiral analysis are capable of resolving a wide variety of compounds by showing complementary separation targets. The combination of the Nexera UC Chiral Screening System and these columns simplifies method scouting for chiral analysis.
Supercritical Fluid Extraction/Chromatograph System

Supercritical Fluid Extraction Unit SFE-30A
This unit operates at a maximum temperature of 80°C to allow faster and more complete extractions. Two sizes of extraction vessels (5 mL and 0.2 mL) can be chosen based on the sample amount. This unit has the internal capacity to run up to four samples automatically; with the addition of an optional rack changer, it can accommodate unattended operation for up to 48 samples.

Rack changer: max. 48 samples
Two sizes of extraction vessels

CO₂ Solvent Delivery Unit LC-30ADsr / Back Pressure Regulator Unit SFC-30A
Stable baseline with low pulsation is realized by LC-30ADsr with a built-in cooler for pump heads, which delivers mobile phases up to 5 mL/min at pressures up to 66 MPa. The low dead volume of SFC-30A (0.7 µL) allows a mass spectrometer to be directly connected to the SFC system without splitting so that higher sensitivity can be achieved.

Nexera UC reduces environmental impact
Comparison of costs and consumption of organic solvent for a single analysis by conventional normal-phase LC vs. SFC is shown below. By using SFC, the total cost of analysis is reduced by 87.6% and the consumption of organic solvent is reduced by 94.2%.
System configuration examples

**Nexera UC On-line SFE-SFC-MS System**

In this system, solid samples are extracted by supercritical fluid and introduced to SFC on-line. The time for pretreatment of samples is drastically shortened. In addition, samples are extracted under light-shielding and anaerobic conditions in order to protect labile analytes from degradation.

**Simultaneous analysis of pesticides over a wide range of polarity**

With conventional methods, both LC (LC/MS/MS) and GC (GC/MS/MS) are needed to analyze pesticides. In contrast, the Nexera UC On-line SFE-SFC system can extract and analyze the full range of pesticides automatically. Pretreatment requires only homogenization and dehydration, which takes 1/7 the time of the QuECHERS method. Adding the optional Rack Changer enables automated analysis of up to 48 samples.

**Extraction and analysis of labile samples without degradation**

Samples are extracted under light-shielding and anaerobic conditions so that labile compounds can be analyzed without degradation.

**Quick start of cleaning validation in pharmaceutical industry**

Regardless of the sample polarity, the only required preparation step is enclosing the sample swab in an extraction vessel. The Nexera UC On-line SFE-SFC-MS system automatically starts extraction for analysis.

**Analysis of polymer additives where other analytical instruments struggle**

Polymer additives are insoluble in THF or chloroform and hard to sublime; therefore, GPC and GC are not applicable. The Nexera UC On-line SFE-SFC system easily extracts and analyzes them.
Nexera UC UFMS System

This system realizes high speed and high resolution by using supercritical fluid. The low volume of the SFC-30A back pressure regulator unit transfers all eluate from the column to a mass spectrometer in order to achieve high-sensitivity analysis.

Off-line simultaneous analysis of trace pesticides in foods
The Nexera UC UFMS system is able to reduce analysis times while maintaining high sensitivity. Combining the low volume of the SFC-30A back pressure regulator unit with supercritical fluid provides sensitivity six times higher than conventional LC-MS and requires 1/3 the time.

Analysis of fatty acids and phospholipids that have many analogues
Phospholipids have many analogues. Since their UV spectra are very similar, MS detection is more suitable for analysis. The Nexera UC UFMS system can analyze these compounds accurately and with high separation.

Analysis of synthetic compounds
Finding a suitable analytical method for the analysis of synthetic compounds is often a time consuming task, requiring the user to manually change columns and mobile phases to test different combinations. Nexera UC UFMS simplifies this task by automatically screening different combinations of columns and mobile phases to find the most suitable conditions. Additionally, the polarity of the mobile phase can be adjusted by the addition of modifiers, allowing a greater number of possibilities to be evaluated.
System configuration examples

**Nexera UC Chiral Screening System**

This system is best for developing methods to separate chiral compounds. It automatically generates a large number of methods by utilizing combinations of up to 12 columns, four modifiers, and a different ratio of modifiers to mobile phase.

**Nexera UC SFC-UV System**

This is the minimum setup of Nexera UC and is suitable to replace both normal phase and reverse phase LCs. A wide range of analyte polarity can be covered by the combination of supercritical fluid and modifiers (for example, MeOH). Hazardous organic solvents such as hexane or chloroform are eliminated. This system reduces environmental impact by utilizing low-toxicity mobile phases and completing analyses in a shorter time.
### Nexera UC Customized Units

#### Supercritical Fluid Extraction Unit SFE-30A

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of processable samples</td>
<td>4</td>
</tr>
<tr>
<td>Extraction vessel volume</td>
<td>0.2 mL, 5.0 mL</td>
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<tr>
<td>Maximum operation pressure</td>
<td>40 MPa</td>
</tr>
<tr>
<td>Temperature control range</td>
<td>10°C above room temperature to 80°C</td>
</tr>
<tr>
<td>Operating temperature range</td>
<td>15 to 28°C</td>
</tr>
<tr>
<td>Dimensions, weight</td>
<td>W260 × D500 × H415 mm, 30.4 kg</td>
</tr>
<tr>
<td>Power requirements</td>
<td>AC 120 V, 230 V, 450 VA, 50/60 Hz</td>
</tr>
</tbody>
</table>

#### CO2 Solvent Delivery Unit LC-30ADSF

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solvent delivery method</td>
<td>Micro-volume double plunger pump</td>
</tr>
<tr>
<td>Plunger capacity</td>
<td>10 µL</td>
</tr>
<tr>
<td>Flow-rate setting range</td>
<td>0.0001 to 5.0000 mL/min</td>
</tr>
<tr>
<td>Maximum pumping pressure</td>
<td>66 MPa (0.0001 to 3.0000 mL/min), 44 MPa (3.0001 to 5.0000 mL/min)</td>
</tr>
<tr>
<td>Flow-rate accuracy</td>
<td>Less than ±1% or ±2 µL/min, whichever is greater (1 to 40 MPa, under specified conditions)</td>
</tr>
<tr>
<td>Flow-rate precision</td>
<td>Less than ±2% or ±2 µL/min, whichever is greater (40 to 60 MPa, under specified conditions)</td>
</tr>
<tr>
<td>Pressure limits function</td>
<td>Upper and lower limits</td>
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<tr>
<td>Plunger rinsing mechanism</td>
<td>Equipped with an automatic rinsing kit</td>
</tr>
<tr>
<td>Operating temperature range</td>
<td>15 to 28°C</td>
</tr>
<tr>
<td>Dimensions, weight</td>
<td>W 260 × D500 × H280 mm, 25.5 kg</td>
</tr>
<tr>
<td>Power requirements</td>
<td>AC 100 V, 230 V, 600 VA, 50/60 Hz</td>
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</table>

#### Supercritical Fluid Back Pressure Regulator Unit SFC-30A

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pressure setting range</td>
<td>10 to 40 MPa (0.01 MPa step)</td>
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<tr>
<td>Pressure control accuracy</td>
<td>0.10 MPa (100% CO2)</td>
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<tr>
<td>Maximum setting pressure</td>
<td>40 MPa</td>
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<tr>
<td>Temperature control range</td>
<td>40 to 70°C (1°C step)</td>
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<tr>
<td>Safety measures</td>
<td>Thermo sensor, Thermal fuse, Leak sensor, BPR cover sensor</td>
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<td>Operating temperature range</td>
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<td>Dimensions, weight</td>
<td>W260 × D500 × H140 mm, 12 kg</td>
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<tr>
<td>Power requirements</td>
<td>AC 120 V, 230 V, 300 VA, 50/60 Hz</td>
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</table>
This product was co-developed with Osaka University, Kobe University, and Miyazaki Agricultural Research Institute in the program "JST-SENTAN" (Development of Systems and Technology for Advanced Measurement and Analysis) by Japan Science and Technology Agency (JST).