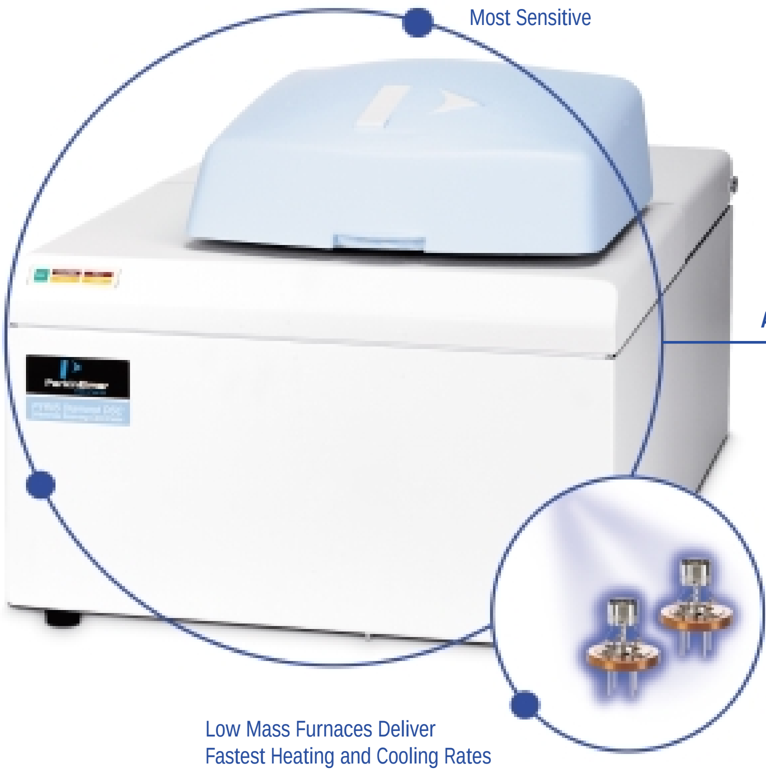
Thermal Analysis

PYRIS Diamond DSC

Most Sensitive



Analytical Instruments

Exclusive Power Compensation Technology

Low Mass Furnaces Deliver Fastest Heating and Cooling Rates

Get the whole story from your sample

PerkinElmer™
instruments.

Unique Capabilities Ensure Clear Identification

When you can't afford to miss something in your DSC analyses, you need the PYRIS™ Diamond Differential Scanning Calorimeter (DSC). Missing important information can make a big difference to your organization's success. It can lead to product failures, additional manufacturing expense and wasted time. With the PerkinElmer exclusive power-compensation technique, you get the whole story about your sample.

PerkinElmer pioneered this proven approach to DSC nearly 40 years ago. Two small, low mass furnaces heat and cool rapidly, providing better resolution and higher sensitivity enabling detection of transitions that are missed in conventional heat-flux systems. And, since the design measures heat flow (energy) you get direct results instead of having to derive them from a temperature difference calculation as in a heat-flux DSC.

The power-compensation technique is well suited for situations where the material being analyzed is not well characterized. Research, development and competitive analysis all benefit from the power-compensation approach. It is also a superior way to simulate production conditions, providing valuable insights into how heating and cooling affects product properties.

What Does a DSC Measure?

It measures the amount of energy (heat) absorbed or released by a sample as it is heated, cooled or held at constant temperature. DSC also performs precise temperature measurements.

Fastest Heating and Cooling Rates

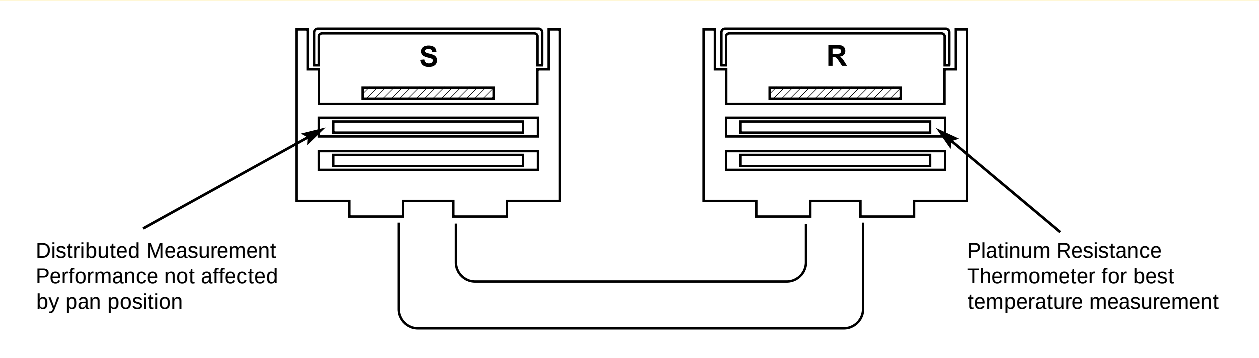
Unlike heat-flux systems utilizing large furnaces that are 30 grams or more, the PYRIS Diamond DSC system features two 1-gram furnaces. This design enables the system to achieve the fastest heating and cooling rates in the industry – up to 500˚C/min. Faster heating translates into higher throughput. Small furnaces also deliver better temperature control providing linearity for better results. Heating and cooling flexibility allows the PYRIS Diamond DSC to more accurately mimic production conditions – facilitating process and property optimization, saving time and reducing production problems.

Better resolution means you see more

Resolution refers to the ability of the DSC to separate or resolve closely occurring thermal transitions. In thermal analysis, small differences can have a huge impact on your success. Ensuring access to all the information is critical to researchers in both the pharmaceutical and polymer industries. In pharmaceutical labs, researchers need to ensure that there are no unknown transitions, which could not only cause undesirable outcomes but may potentially limit patent protection. In polymer work, a small shoulder peak that goes undetected can produce unwanted physical properties, resulting in product failure. Due to the small furnaces, the signal returns to baseline faster providing outstanding resolution. The Diamond DSC delivers industry-leading performance to ensure all transitions are identified.

The Power-Compensation Principle

With power-compensation DSC, the sample and the reference material are placed in separate, self-contained calorimeters. When the temperature rises or falls in the sample material, power (energy) is applied to or removed from the calorimeter to compensate for the sample energy. As a result, the system is maintained at a "thermal null" state at all times. The amount of power required to maintain system equilibrium is directly proportional to the energy changes occurring in the sample. No complex heat-flux equations are necessary with a power-compensation DSC because the system directly measures energy flow to and from the sample.



Higher Sensitivity

Power-compensation yields higher sensitivity, which is the ability of a DSC to detect a weak transition from the background noise. You can even detect phase transitions of low energy, such as in biological or liquid crystalline samples. The PYRIS Diamond DSC has the capability to analyze very small samples if sample availability is limited. The additional capability of fast heating increases sensitivity because the signal is directly proportional to the heating rate.

True Isothermal Measurements

The power-compensation DSC helps to analyze isothermal crystallization processes. By comparison, a heat-flux DSC performs these measurements only with limitations, since the temperature is not kept constant. During a crystallization, the temperature can easily change several degrees celsius, which influences the data obtained and results of any kinetic studies. The PYRIS Diamond DSC maintains the temperature difference at zero and thereby assures high quality results.

Cover Design Maximizes System Baseline Stability

The PYRIS Diamond DSC system includes a unique cover design that improves both ease of use and performance. An innovative rotating sample head cover effortlessly slides in and out of position. The cover is in constant contact with the furnace block, reducing equilibrium times and ensuring baseline stability. The cover and sealing design effectively eliminate furnace frosting.

Size Matters

Heat-flux systems use one large furnace that is between 30 and 200 grams while the PYRIS Diamond DSC system features two 1-gram furnaces. This size difference results in faster heating and cooling rates and allows direct heat flow determination, eliminating the need for complex mathematics.

High Caloric Accuracy

The power-compensation principle determines the most accurate caloric data of all available DSCs. With a heat-flux DSC, the results vary with the sample preparation and the contact with the sensor. While precision may be good with heat flux, the result could be off by several percentages from the true value.

With the PYRIS Diamond DSC you minimize the risk of wrong or inaccurate determination – the influence of bad sample preparation on the results is negligible. You know the generated data is correct and feel secure using it as a basis for your decisions.

Confidence Counts

The power-compensation DSC provides the highest confidence in your analytical results. Because the system's technique measures heat flow changes directly, no complex mathematics are required. Heat-flux systems, on the other hand, require extrapolation to get the results, and the more work done after data is collected, the more likely the introduction of error.

The Diamond DSC's small furnaces, combined with the power-compensation technique and the overall system performance, gives you the whole story. More information translates into better decision making.

PYRIS Software Improves Lab Productivity

PYRIS software is the core of PerkinElmer's entire line of thermal analysis products. In combination with the Windows 2000* operating system, it provides new functionality essential for productive operation of all thermal analysis techniques – DSC, TGA, DMA, TMA, and DTA. Whether in a totally automated research laboratory, an automated QA/QC lab, or a stand-alone instrument, you can be sure that the PYRIS software meets your operating requirements. For more information see the PYRIS Software Product Note.

Report Manager
Allows the user to create and define report templates. Reports are created in Microsoft* Word and can include sample information, graphical images, results, text, data, tables, PDF formats – and more.

Calibration Wizard
The integrated Calibration Wizard offers two levels of calibration control. E-Z Cal, based on predefined standards, offers a fast calibration with minimal user interaction. If greater flexibility or accuracy is required, Advanced Cal provides the ability to calibrate using multiple standards, for temperature and heat flow enabling the calibration to cover the entire temperature range of interest. With Advanced Cal customers also benefit from a new algorithm which allows them to use a certified Sapphire standard in accordance with ASTM to calibrate heat flow. In addition to offering two levels of control, the Calibration Wizard utilizes Autotune, which allows automatic baseline optimization.

Valet
Creates pre-set start-up and shut down events. Conditions and equilibrates temperature and environment of sample cells prior to running experiments. Automatically shuts down system after completion of runs.

21 CFR, Part 11 Compliance Tools

Event Log
Records all events, including configuration changes, calibrations, instrument and system errors, run starts, playlist starts, furnace clean, and Valet features. Allows tracking of all events, creating a time-stamped audit trail ensuring proper sequencing of all events.

Secure Save
Saves files to a read-only directory. Altered files cannot be resaved to the secure directory.

Sombrilla™ Web-based Instrument and Data Management System

The Sombrilla system increases productivity by speeding up communication of data and results. It quickly generates reports from anywhere in the world easily by accessing your data using a Web browser and a standard Adobe PDF viewer (Figure 1). The basic configuration works on virtually any type or brand of instrument and unifies laboratory data storage and transfer using a common Web browser. The Sombrilla offering goes beyond reporting and viewing to monitoring instruments and performing data processing functions in a secure Web-based environment.

Sombrilla provides access to many PYRIS software features :

- View Data Files
- Rescale Curves
- Analyze Data
- Generate Reports

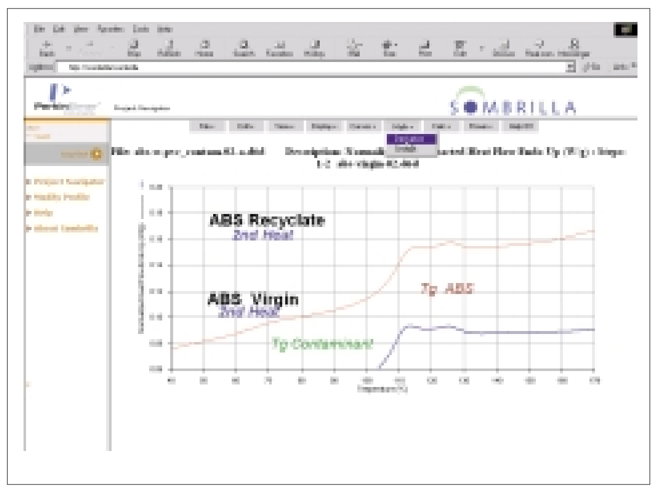


Figure 1. Sombrilla WebEnabler.

Accessories Provide Limitless Flexibility

The PYRIS Diamond DSC accommodates a wide range of accessories and options from Modulated Temperature DSC and automatic gas switching accessories to cooling devices and the widest variety of sample pans. Access to the DSC cell is quick and easy, simplifying accessory changeover.

Autosampler

The Autosampler Accessory permits unattended operation of the instrument. It automatically loads the sample and initiates the experiment. Using the PYRIS Player Software, a queue of samples can be automatically loaded and unloaded, run and the results analyzed without user intervention.

Enhanced Cooling Capabilities

The PYRIS Diamond DSC provides a variety of cooling options, including water circulation, the completely closed Intracooler system and liquid nitrogen, allowing experiments that require subambient temperatures.

StepScan DSC

StepScan DSC is a modulated temperature DSC technique that operates in conjunction with the power-compensation DSC. The approach applies a series of short interval heating and isothermal hold steps to cover the temperature range of interest. StepScan DSC is only possible with a power-compensation DSC. This approach requires a DSC with very fast responsiveness to achieve short interval linear heating and isothermal steps. The use of the ultra-low mass furnaces (1 g) with the power-compensation DSC ensures the fastest response time of any DSC instrument.

StepScan DSC offers many advantages over conventional DSC and other MTDSC heat-flux DSC approaches:

- Straightforward – pure linear heating ramps and isothermal steps
- No sine waves and possibilities of distortions and other experimental artifacts
- No mathematical deconvolution (Fourier transforms) required with associated complexities
- Highly quantitative
- Eliminates need to perform heat-cool-reheat experiments
- Greatly helps in data interpretation since reversible and irreversible effects are separated out
- Provides much clearer identification of the glass transition event (Tg)
- Yields enhanced sensitivity for detection of difficult transitions
- Provides more accurate heat capacity results since Cp measurements are generated over short interval temperature segments

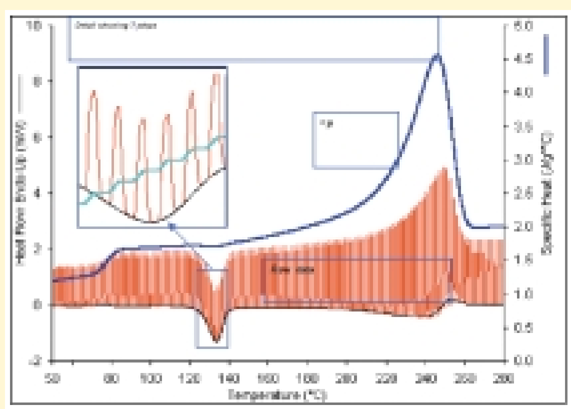
StepScan: A Pure Approach to MTDSC Applications

With the StepScan DSC approach, two signals are obtained. Thermodynamic Cp signal represents the reversible aspects of the material, while the Iso K signal reflects the irreversible nature of the sample during heating. The following basic equation mathematically describes the StepScan DSC approach:

Heat Flow = Cp(dT/dt) + f(T,t)

In this equation, Cp is the sample's heat capacity, dT/dt is the applied heating rate and f(T,t) is the kinetic response. The first Cp term represents the reversible aspects of the sample and the power-compensation DSC applies a purely linear heating ramp for the best results rather than a sine wave where the first item is continuously varying. When the sample is held under isothermal conditions, the heating rate becomes 0 and the sample's heat flow is purely described by the kinetic term.

Because the sample is either linearly heated or held isothermally (true isothermal), the StepScan DSC approach is straightforward and provides the purest approach to MTDSC measurements.



StepScan DSC Results for PET

Power-Compensation Technique Yields
Confidence-Building Results

Unique Power-Compensation

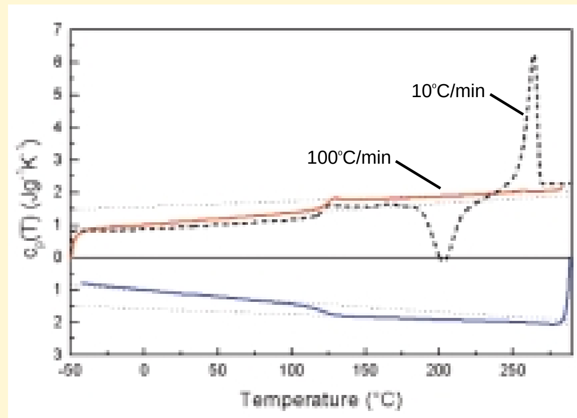
High Speed DSC Helps Mimic Processing Conditions

The plastics industry has a high interest in having a clear understanding of how materials behave under process conditions. In injection molding, blow-molding and extrusion processes, high cooling and heating rates play the dominant role. Product development requires lab-scale experiments in which these conditions can be simulated. High speed DSC provides that additional information that you don't capture with standard DSC. In addition, high heating and cooling rates minimize noise, thereby enabling highly quantitative measurements.

The analysis of a PET sample demonstrates the influence of the scanning rate on the results.

The slow heating at 10ºC/min after cooling the PET with 100ºC/min shows extensive cold crystallization followed by a melting of the formed crystals (dashed line). This heating run performed at 10ºC/min is definitely not representative of the thermal history (blue line) in which no crystallization event occurs. Heating with 100ºC/min (red curve) after cooling with 100ºC/min shows hardly any cold crystallization on the time scale of the experiment. With increasing heating rate, recrystallization is suppressed and the resulting heating curve reflects the characteristic of the material at room temperature obtained by a fast cooling.

Note: Acknowledgement to Vincent Mathot, Thijs Pijpers, Eric van der Vegte, DSM Research, The Netherlands for their initiative and work in this application field.



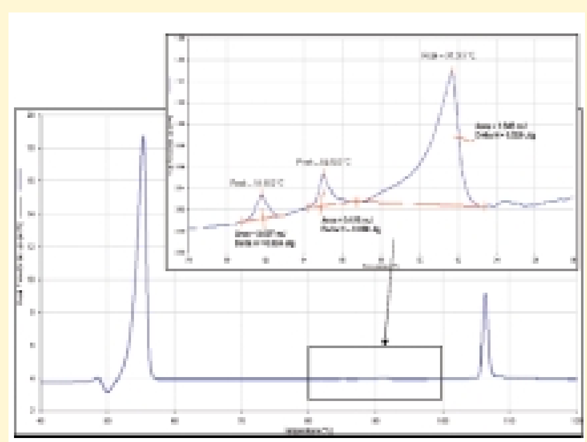
High Speed DSC PET Analysis:
No Cold Crystallization During Fast Scanning

PYRIS Diamond DSC Tells the Whole Story

Effective Competitive Analysis Requires High Resolution and Sensitivity

Liquid crystalline materials can exhibit a wide range of liquid crystalline phases with varying degrees of molecular order. These changes can be seen by DSC studies but the energies involved in these transitions can be very small, making their identification difficult. In the past, most of these small energy transitions have been identified by optical microscopy alone. In this example, we show that it is possible to see these small energy transitions plainly with the new PYRIS Diamond DSC.

If the DSC trace is zoomed into the area of 80-95ºC, it is possible to identify some small endothermic events that occur in this range. The energies involved in these transitions are very small (0.024J/g) and demonstrate the extremely high sensitivity of the Diamond DSC.



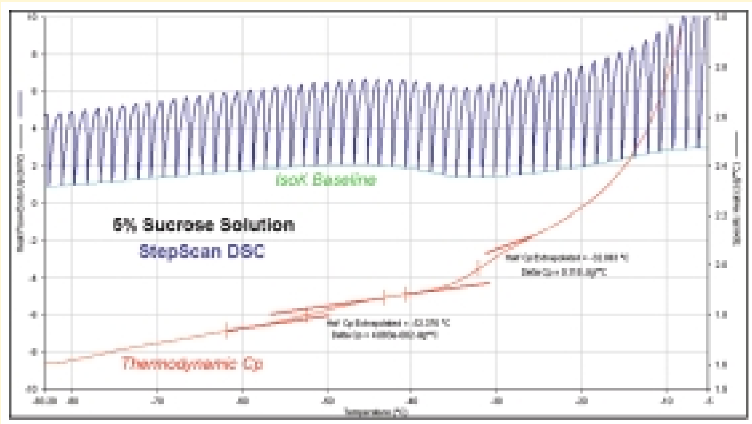
Liquid Crystal Phase Change Transitions

Pharmaceutical Application

Modulated Temperature DSC Helps to Understand the Lyophilization Process Better - StepScan DSC

The production of many new drugs requires that the active material first be dissolved in water and then freeze-dried or lyophilized in order to generate a compound which is easily ingested. Due to energy consumption considerations, this can be expensive and it is desirable to know the maximum acceptable temperature to which the solution can be cooled before a vacuum is applied. One key parameter that has been identified as crucial to understanding the lyophilization process is the subambient glass transition temperature, or Tg, of the given formulation. Detection of glass transition events in frozen formulations can be challenging and difficult for a standard DSC measurement due to the inherently weak nature of these transitions.

StepScan shows that for a 5% sucrose formulation, the Thermodynamic Cp data set reveals two Tg's occuring in the subambient regions. This is very useful information for freeze-drying purposes as it helps to identify the key transitions for lyophilization optimization.



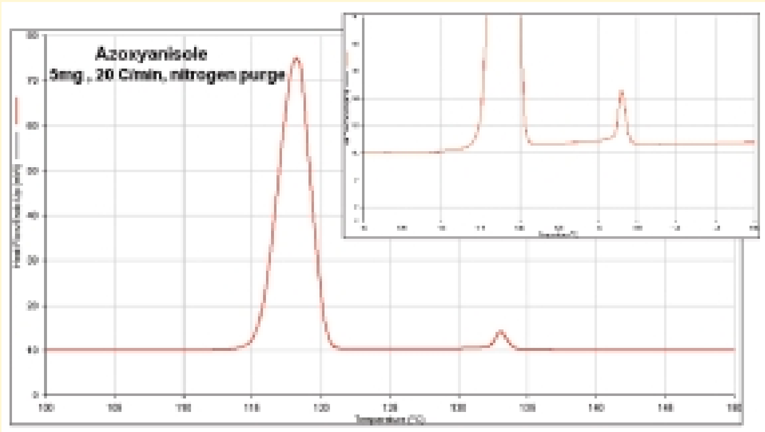
StepScan DSC Subambient Results on 5% Sucrose Formulation

Proof of Performance

Oftentimes, instrument companies provide specifications as to sensitivity and resolution without describing how they were generated.

The use of 4, 4' azoxyanisole has been recommended as both a resolution and sensitivity standard for assessing DSC performance by the Netherlands Society for Thermal Analysis (TAWN). This substance has two closely occurring endothermic transitions at 117ºC and 134°C. The resolution of the DSC instrument can be defined as how well the heat flow response returns to a linear baseline in the region between the two transitions when the sample (5 mg) is heated at a rate of 20°C/min using a nitrogen purge gas. The better the resolution of the instrument, the better the heat flow response returns to the baseline. An instrument with a high inherent resolution will yield a linear heat flow response between the two transitions and a better return to the baseline.

The resolution index, or R value, can be used to compare DSC performance. This is done by taking the heat flow value at the minimum point between the two peaks and then dividing this by the value of the heat flow at the smaller peak maximum.



DSC Results on Azoxyanisole Test

Solutions for Material Property Analysis

In addition to the PYRIS Diamond DSC, PerkinElmer offers a wide range of thermal analysis products, providing solutions for a vast array of material property analyses.

PYRIS TGA

The PYRIS Thermogravimetric Analyzers measure the change in weight of a sample as it is heated, cooled or held at constant temperature. Our instruments provide robustness and reliability for quality control and the answers researchers need to solve even the toughest problems.

PYRIS DMA

The PYRIS Dynamic Mechanical Analyzer (DMA) delivers performance and flexibility to help you mechanically characterize a broad range of materials – from soft samples, such as elastomers, to hard samples, like composites.

Whatever you're looking for, we've got it

PerkinElmer Instruments is a world leader in chemical analysis and imaging. Our Analytical Instruments technologies serve the fast-evolving pharmaceutical, chemical, environmental and semiconductor industries, providing integrated solutions – from sample handling to interpretation and communication of test results.

As one of the best known brands in research, analysis and testing, ours was probably the first analytical instrument you ever used. In addition to our Thermal Analysis product line, we offer a broad range of solutions in Luminescence, AA, UV-Vis, NIR, GC, GC/MS, HPLC, MALDI-TOF MS, ICP, ICP-MS, or FTIR.

There are over 60 years of experience built into every product we make. So, for leading edge R&D and demanding QA/QC, you get the speed, accuracy and reliability you seek – for the productivity you need. Our service and support people are located in 125 countries throughout the world and are factory-trained. Compliance doesn't get any easier than with our software, including 21 CFR Part 11 technical compliance on many products. And online consumables and accessories ordering lets you get your hands on what you need fast.

In addition to Analytical Instruments, PerkinElmer Instruments includes Princeton Applied Research, with instrumentation for materials and energy source characterization, electrochemistry and sensor research, and Detection Systems, the world's leading supplier of X-ray security screening systems and metal detectors. PerkinElmer Instruments is part of the PerkinElmer, Inc. family, which also includes Fluid Sciences, Optoelectronics and Life Sciences.

Getting the most from your system

In today's lab, productivity and performance are essential. That's why no matter where in the world you are, we provide service and support to you from PerkinElmer-trained professionals. Maintenance support from PerkinElmer ensures optimum system performance and minimizes downtime.

Validation Services

Adhering to the strict requirements of internal and external regulations is a challenge facing many laboratories. PerkinElmer makes it easier by offering a variety of validation tools and services. Plus, many of our products are already 21 CFR Part 11 compliant. And for others, compliance is just around the corner.

Preventive Maintenance Agreement

This option is highly recommended to maintain the integrity of your system and extend its useful life.

Through our basic maintenance agreement, our service personnel are at your disposal, scheduled on a priority basis. Factory-trained service technicians can be dispatched to your site, or we can service your instrument at one of our Authorized Service Centers. Plans cover the cost of all labor, travel and any non-consumable parts.

On-demand Services

Without a maintenance agreement, factory-authorized services are available on demand. All work is guaranteed and priced at competitive labor rates.

Technical Support

We give you the help you need, when you need it, through a remarkable support system that's unparalleled in the industry. If you have a technical, application or software question or problem, you can consult with one of our trained technical representatives over the phone, which can often resolve a problem and get your instruments working without a service call.

Access our Technical Support Document Library, search for self-help tips, and get software updates and revisions at www.perkinelmer.com. We welcome product feedback and will respond promptly.

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