AP2000 AutoProbe™

Automated Direct Probe for the Thermo Finnigan™ TRACE DSQ™ or PolarisQ™ MS

Unattended analysis of +200 samples at 3-5 minutes/sample.

Scientific Instrument Services, Inc.

An Automated Direct Exposure MS Probe

SIS AP2000 AutoProbe™

The AutoProbe is a direct exposure probe designed for automated and unattended injection of samples into the Thermo Finnigan TRACE DSQ™ and PolarisQ™ MS. Samples can be analyzed in about three minutes in standard or open access modes of operation.

Features

Capabilities

- **Automated Direct Exposure Probe (DEP or DCI).**
- **For the Thermo Finnigan TRACE DSQ™ and PolarisQ™ Mass Spectrometers.**
- **Unattended, quantitative, reproducible analysis of samples (3 to 5 minutes per sample)**
- **Integrated CTC liquid autosampler (included) for loading samples onto probe tip. Holds four sample trays of +54 samples each.**

Software

- **Fully integrated with Thermo Xcalibur™ software, - optional support for Open Access™ operation.**

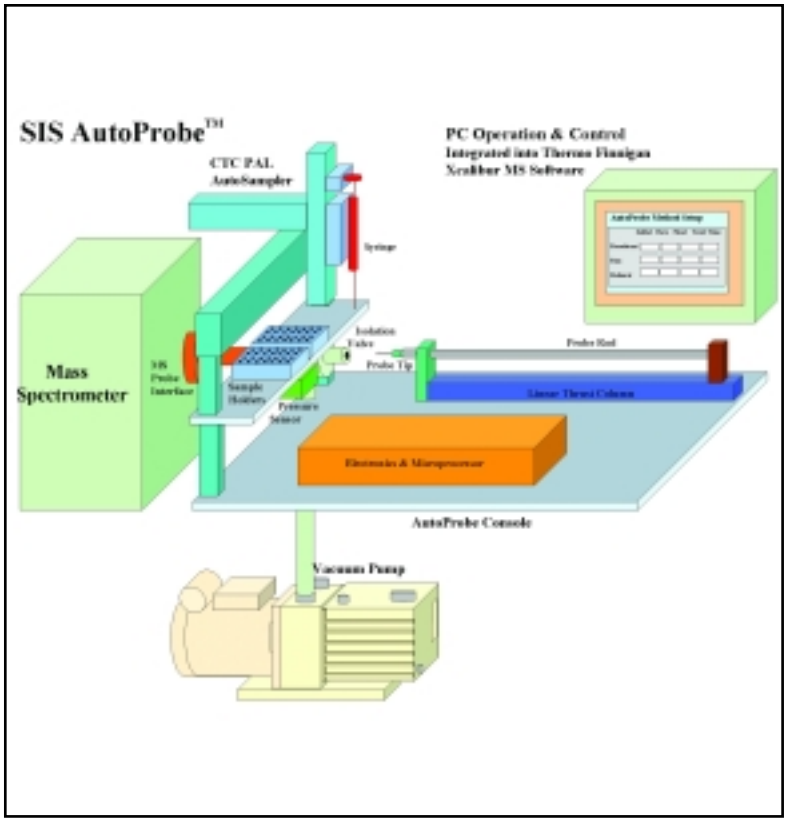
Maintenance and Robustness

- **Plug in replaceable DEP probe filaments (expected life > 1000 samples).**
- **Easily replaceable probe shaft seals (expected life > 1000 injections).**
- **Sequence stops automatically if leaks, broken filaments or other problems occur.**
- **Vacuum gauge in isolation valve prevents insertion of probe into source if seals are leaking.**
- **Automatic repositioning of probe without vacuum leaks if a power outage occurs when the probe is in the MS vacuum.**

The SIS AutoProbe integrates a CTC liquid autosampler with an automated direct exposure probe (DEP) for the automated and unattended injection of dissolved or suspended samples directly into a Thermo Finnigan™ TRACE DSQ™ or PolarisQ™ MS source for direct MS analysis. Using this technique, samples can be analyzed in about three minutes without any chromatographic separation. The AutoProbe is fully integrated with the Thermo Finnigan Xcalibur™ software, including system setup, method setup, system operation and data storage.

The DEP (or DCI) technique is a quick technique useful for the analysis of samples that are not volatile enough to be introduced into the mass spectrometer via a GC and therefore must be directly introduced into the mass spectrometer source. The DEP technique is normally used for single component samples since minimal separation of samples is possible, and samples with two or more components are difficult to interpret. Alternatively DEP sample analysis may be performed utilizing CI (Chemical Ionization) mass spectrometer techniques, which produce less fragmentation and increased abundance of a molecular ion. With the CI techniques, it is possible to analyze and interpret mixtures of two or more compounds in a sample.

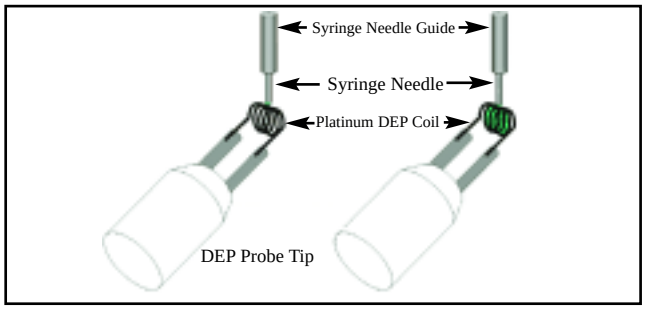
Theory of Operation



The AutoProbe components are shown at right. A CTC liquid autosampler loads samples onto the replaceable probe tip via a syringe. The probe tip is attached to a probe rod attached to a servo linear actuator that automatically drives the probe through an isolation valve and into the mass spectrometer source.

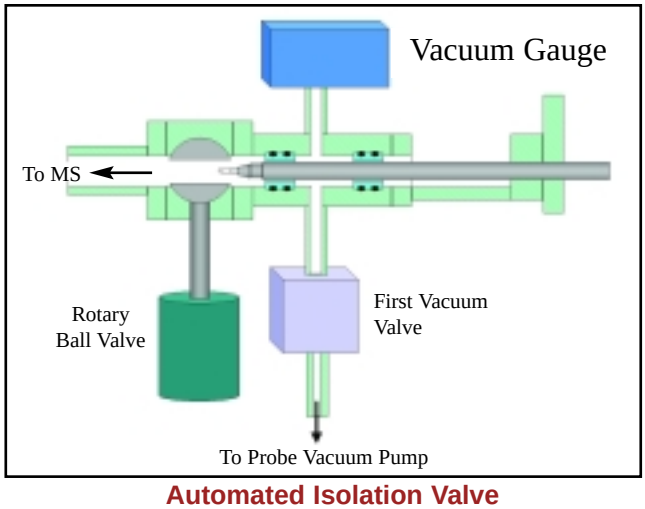
Before (or during) analysis, the operators prepare individual sample vials containing samples dissolved in suitable solvent and place these on the sample tray.

For each sample in an analysis sequence, the autosampler starts by loading 0.125 to 1.0 µl of sample and accurately injects it onto the platinum DEP probe wire filament.



DEP Probe Tip Wire Coil

A small initial current is then passed through the DEP filament wire to evaporate the solvent from the sample.



Automated Isolation Valve

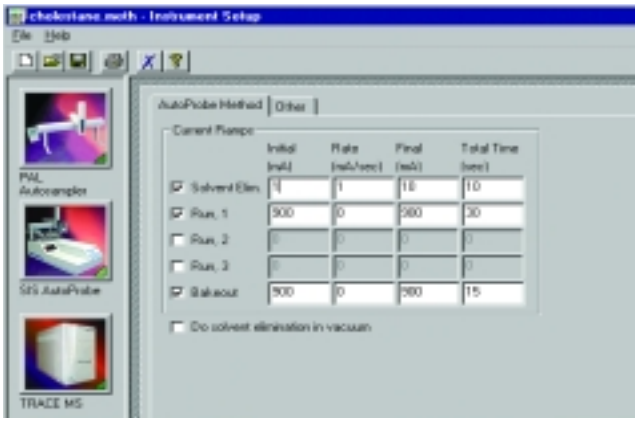
An isolation valve (bottom left) permits the introduction of the probe into the mass spectrometer without compromising the MS vacuum. When the probe is inserted into the first probe seal of the isolation valve, a vacuum valve opens to allow the accessory vacuum pump to evacuate the probe inlet. A vacuum gauge measures this vacuum, and when the vacuum reaches approximately 200 millitorr, the probe is advanced to a second seal. This vacuum gauge acts as a safety feature to prevent the probe from being inserted into the MS source should a vacuum problem occur anywhere in the system. Afterward, a pneumatically controlled ball valve opens to permit the probe to be inserted through the isolation valve and into the mass spectrometer.

Once the probe has been inserted into the mass spec source, a small current (typically 10 to 1000 mA) is passed through the platinum wire to heat the wire and desorb the sample for analysis. This DEP probe filament can be set to a constant current, or the current can be programmed in up to three ramp steps to obtain the optimum results depending on the sample being analyzed.

After the sample analysis is complete, the probe tip is retracted slightly from the MS source, and a high filament current is passed through the wire coil to burn off any sample remaining on the filament wire coil. This assures that the filament wire is clean and ready for the next sample.

AutoProbe Software and PC Control

The AutoProbe is controlled by PC software that is fully integrated within the Thermo Finnigan™ **Xcalibur™ MS software**. The AutoProbe operates as any other standard Xcalibur virtual instrument, which means that the AutoProbe is configured, started, stopped and monitored entirely within the Xcalibur user interface. AutoProbe method settings, such as filament current ramps within the three heating steps, are stored within the MS method. (See figure below.)



AutoProbe Method Setup Screen

The AutoProbe may optionally be used in the Xcalibur "**Open Access™**" mode of operation. This permits the chemist or technician to log samples into the system using a very simple and locked-down interface. With no subsequent user interaction, the samples are analyzed automatically when the system becomes available, and the results are computed and reported back to the chemist in a printout or e-mail. This results in increased productivity from the mass spectrometer lab.

Xcalibur "Open Access" Log In Screen

Reproducibility, Quantitation, Sensitivity and Low Memory Effects

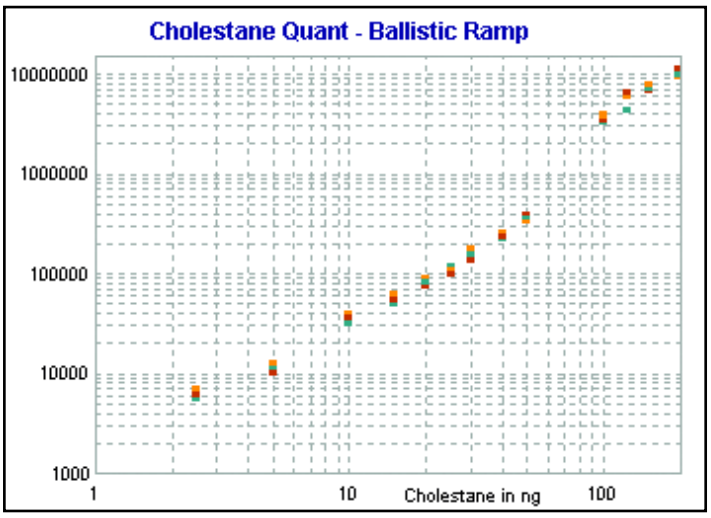
Reproducibility

Historically the direct probe techniques are not known for their reproducibility. When manually injecting a sample onto a DEP probe, it is quite difficult to reproducibly apply a sample to a DEP coil, and the coil is easily repositioned. The AutoProbe eliminates these problems with its automated sample handling. As a result when the same sample was analyzed more than 150 times as shown below, the areas of the resultant total ion chromatograms were reproducible within 10% or better of each other.

Reproducibility of the DEP Technique

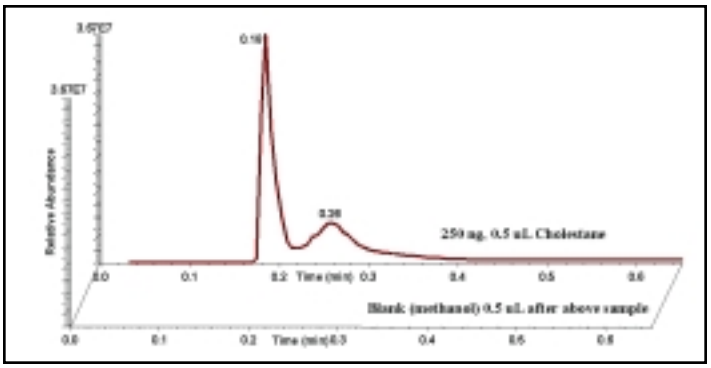
Quantitation

Quantitation techniques have not been previously accomplished with the DEP probe techniques. This is due to the same reasons discussed under "reproducibility" above. However the AutoProbe overcomes these shortcomings, and the technique has been found to be quantitative as shown in the graph on the following page. In this study, as well as several others reported in an application note on our web site, linear calibration curves of pure analytes were achieved with the AutoProbe technique. This reproducibility and quantitation capability is due to the automation of the system with the very reproducible conditions being applied to each sample and the unchanging geometry of the DEP filament. Since the filament coil is never touched or moved, the sample is injected onto the DEP coil in precisely the same point for every sample. This opens up many new applications for the AutoProbe technique.



Typical Calibration Curve for an Analyte Using the AutoProbe

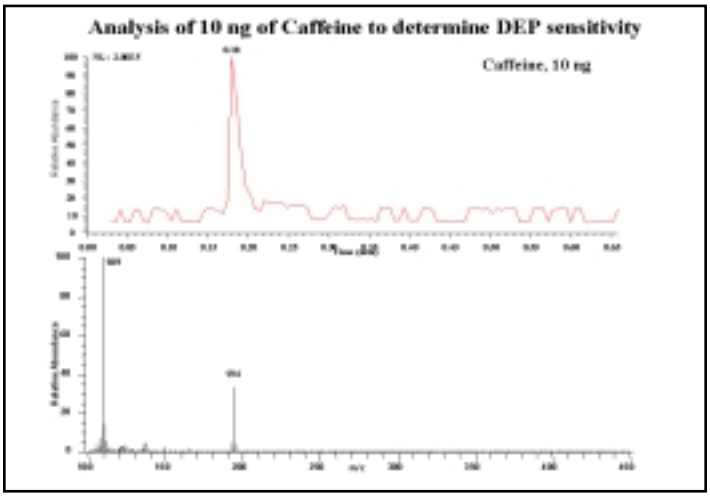
A technique is of little use if "cross contamination" or "memory effects" occur during sample analysis. This problem does not occur in the AutoProbe because the DEP wire is "baked out" at high temperatures between samples. The first chromatogram below shows the analysis of a 250 ng Cholestane sample dissolved in methanol. This analysis was repeated 5 times. The DEP coil was baked out at 900 mA for 15 seconds after each sample and then a blank clean methanol sample was analyzed. The blank injection produced a flat baseline (second chromatogram) with no carryover from the previous samples.



Background Check After Running Strong Samples

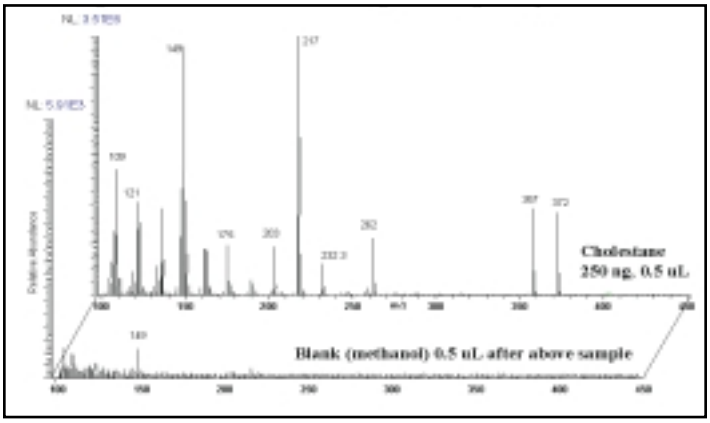
Sensitivity

Direct probe techniques are less sensitive than GC techniques because the probe peaks in the total ion chromatograph are comparatively much wider and therefore less intense. However, by utilizing the rapid temperature ramp capabilities of the AutoProbe, sample sizes less than one nanogram have been detected and identified.



A Typical Sample Analysis is Fast and Sensitive

The chart below shows the corresponding MS data at 0.18 minutes for the 250 ng of Cholestane and the blank methanol sample. Note that the scale on the blank methanol spectrum is 1000 times the scale of the 250 nanogram of Cholestane. No peaks were found in the blank sample that could be attributed to the Cholestane. In this study, as well as many others, it was confirmed that "memory effects" or "cross contamination" problems do not occur with the AutoProbe technique. Therefore, although it is recommended that the high temperature "bake out" be used in all analysis, running a blank sample between analysis is optional.



Background MS After Running 5 Consecutive Strong Samples

For additional information call Scientific Instrument Services, Inc. at 908-788-5550 or visit our web site at http://www.sisweb.com/autoprobe

Features:

- Automated Direct Probe MS Analysis
- Interfaces to Thermo Finnigan™ TRACE DSQ™ or PolarisQ™ MS
- Utilizes DEP Probe Technique
- Mass Spec Probe Analysis in ~3 minutes
- Fully Integrated with Xcalibur™ Software
- Can be used in "Open Access" operation
- Multi-Step Probe Tip Heating
  - Low Temperature Solvent Removal
  - Programmable Current Heating of Samples
  - High Temperature Ramp for Cleaning

Applications:

- Drug Discovery Confirmation
- Quality Control Analysis
- Open Access MS Analysis
- Sample Purity Verification
- Detection of Sample Contamination
- Sample Profiling

AutoProbe System Specifications & Requirements

Voltages

AutoProbe Console Voltage: 110 or 220 VAC
Edwards Vacuum Pump: 110 or 220 VAC

Compressed Air: minimum 60 psi

Mass Spectrometer System Requirements

Thermo Finnigan™ TRACE DSQ™ or PolarisQ™ MS System with Xcalibur™Software
(TRACE MS™ supported on our AP1000 AutoProbe)

Dimensions

Console without CTC PAL Autosampler:
17" wide x 42" Deep x 16" High
Console with CTC PAL Autosampler:
23" wide x 42" Deep x 37" High

Weight

Console Weight: 100 pounds

Home Page: http://www.sisweb.com
E-mail: http://www.sisweb.com/contact.htm

SIS

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