

SAA[®] 8100

SELECTIVE ADSORPTION ANALYZER



micromeritics[®]

**Effective Solutions for
Material Characterization**

OPERATOR MANUAL

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CORPORATE PROFILE

Micromeritics Instrument Corporation is a leading global provider of solutions for material characterization with best-in-class instrumentation and application expertise in five core areas: density; surface area and porosity; particle size and shape; powder characterization; and catalyst characterization and process development. Founded in 1962, the company is headquartered in Norcross, Georgia, USA and has more than 400 employees worldwide. With a fully integrated operation that extends from a world class scientific knowledge base through to in-house manufacture, Micromeritics delivers an extensive range of high-performance products for academic research and industrial problem-solving. The implementation of tactical partnerships to incubate and deliver valuable new technologies exemplifies the company's holistic, customer-centric approach which extends to a cost-efficient contract testing laboratory – the Particle Testing Authority (PTA). The strategic acquisitions of Freeman Technology Ltd and Process Integral Development S.L. (PID Eng & Tech) reflect an ongoing commitment to optimized, integrated solutions in the industrially vital areas of powders and catalysis.

Freeman Technology (Tewkesbury, UK) brings market-leading powder characterization technology to Micromeritics' existing portfolio of particle characterization techniques. The result is a suite of products that directly supports efforts to understand and engineer particle properties to meet powder performance targets. With over 15 years of experience in powder testing, Freeman Technology specializes in systems for measuring the flow properties of powders. In combination with detailed application know-how these systems deliver unrivalled insight into powder behavior supporting development, formulation, scale-up, processing and manufacture across a wide range of industrial sectors.

PID Eng & Tech (Madrid, Spain) complements Micromeritics' renowned offering for catalyst characterization with technology for the measurement and optimization of catalytic activity, with a product range that extends to both standard and bespoke pilot scale equipment. Launched in 2003, PID Eng & Tech is a leading provider of automated, modular microreactor systems for the detailed investigation of reaction kinetics and yield. These products are supported by a highly skilled multidisciplinary team of engineers with in-depth expertise in the design, construction and operation of laboratory units and process scale-up.

The Particle Testing Authority (PTA) provides material characterization services for fine powders and solid materials using Micromeritics' instrumentation alongside complementary solutions from other vendors. With the certification and expertise to operate across a wide range of industries the PTA offering runs from single sample analysis to complex method development, method validation, new product assessment, and the analytical support required for large scale manufacturing projects. An experienced, highly trained team of scientists, engineers, and lab technicians works closely with every client to ensure that all analytical requirements are rapidly and responsively addressed.

Micromeritics has a strong global network with offices across the Americas, Asia, and Europe complemented by a dedicated team of distributors in additional locations. This ensures that local, knowledgeable support is available for every customer, in academia or industry. Micromeritics works across a truly diverse range of industries from oil processing, petrochemicals and catalysts, to food and pharmaceuticals, and at the forefront of characterization technology for next generation materials such as graphene, metal-organic-frameworks, nanocatalysts, and zeolites. Engineering solutions that work optimally for every user is a defining characteristic of the company.

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Table of Contents

Corporate Profile	i
Contact Us	ii
SAA 8100 and MKS Cirrus 3 Interface	1
Overview	1
Cirrus 3 Mass Spectrometer Connection	1
Network Configuration	2
Create a Recipe	3
Load a Sample	4
SAA Data and the MicroActive Application	6

SAA 8100 AND MKS CIRRUS 3 INTERFACE

This manual provides guidelines for setting up experiments and how to load data into the Micromeritics MicroActive application. Other manuals and documents to be used in conjunction with this SAA 8100 Operator Manual:

- Micromeritics MicroActive Operator Manual
- MKS Cirrus 3 Manual
- MKS Process Eye Professional and EasyView User Manual
- PID Process@ User's Manual

OVERVIEW

The 8100 Selective Adsorption Analyzer (SAA) is a dynamic adsorption analyzer based upon the vapor-solid equilibrium for reversible systems. Both transient and equilibrium adsorption data can be collected and analyzed. A carrier gas flows continuously through the sample column. Adsorptives of interest are blended in with automatic valves. After exiting the column, the composition of the gas stream is analyzed by a mass spectrometer. Gas flow rate is controlled by mass flow controllers (MFCs). Gas blending is done with pneumatically controlled, zero-dead-volume rotary valves. Thermocouples measure the gas temperature above and below the sample. The thermocouple positions are adjustable to accommodate a range of sample volumes.

A furnace is available for activating samples or maintaining an elevated temperature through the analysis. Temperatures over 450 °C are not recommended for the stainless steel sample column.

Pressure in the system is controlled with servo valve. Pressures over 10 bar are not recommended.

The mass spectrometer connection is as close to the bottom of the sample column as possible to minimize delay and spreading in the signal. For analyses at elevated pressure, an alternative mass spec port is provided after the pressure valve.

The outlet flow rate is measured by a mass flow meter.

Furnace temperature, valves, MFCs, and pressure are controlled through the PID Process@ software. Automatic operation is achieved by defining sessions. Refer to the PID Process@ User's Manual for further details.

CIRRUS 3 MASS SPECTROMETER CONNECTION

The mass spectrometer capillary attaches to a bulkhead connector on the left side of the instrument. An adapter kit [*part number 810-33010-00*] is provided for connecting an MKS Cirrus 3 mass spectrometer to the instrument. See Micro-Cirrus 3 Interface Installation Instructions [*part number 004-42805-00*] for information on connecting the SAA 8100 and the Cirrus 3 Mass Spectrometer.

NETWORK CONFIGURATION

The SAA 8100 and Cirrus 3 require separate Ethernet connections to a computer. A switch can be used to connect both instruments to one Ethernet port on the computer, however, they must both be on the same subnet.

Initially, the 8100 is configured to use an address that starts with 168.12 and the Cirrus 3 is set to automatically configure the PC's IP address. Once a connection to the Cirrus 3 is established, it can be configured to use a static IP address that begins with 168.12. Refer to the Micro-Cirrus 3 Interface Installation Instructions [*part number 004-42805-00*] for information on connecting the SAA 8100 and the Cirrus 3 Mass Spectrometer.

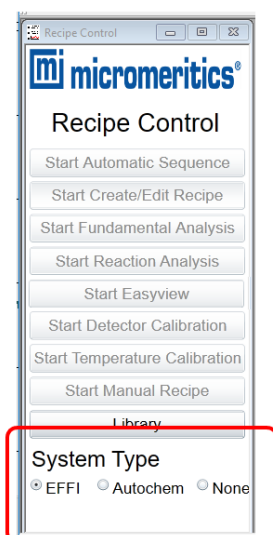
CREATE A RECIPE

Prepare a session in the MKS Process Eye Professional software to set flow rates, operate valves, and trigger the mass spec. Refer to the *MKS Process Eye Professional and EasyView User Manual*.

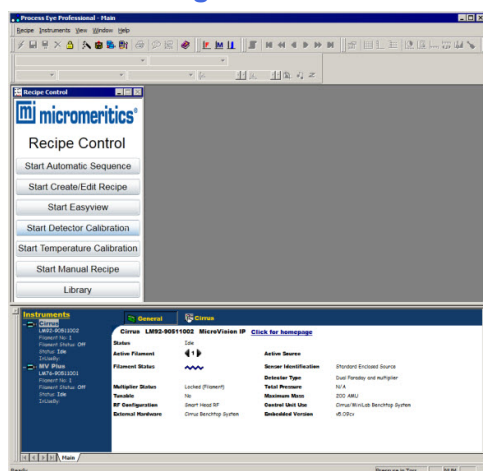
Setting GC01 to 1 triggers the mass spectrometer. Setting GC01 to 1 in a subsequent step stops data collection on the mass spectrometer.

In the MKS Process Eye Professional application:

1. Select *EFFI* as the system type on the Recipe Control panel.



2. Create a recipe to measure the masses of interest. Refer to the *MKS Process Eye Professional and EasyView User Manual* for detailed instructions.
3. Load the sample in the SAA 8100 sample column. See [Load a Sample on the next page](#).
4. Click **Start Automatic Analysis** and select the recipe.
5. Click **Begin**. Data collection will start when the trigger signal is received from the SAA 8100.

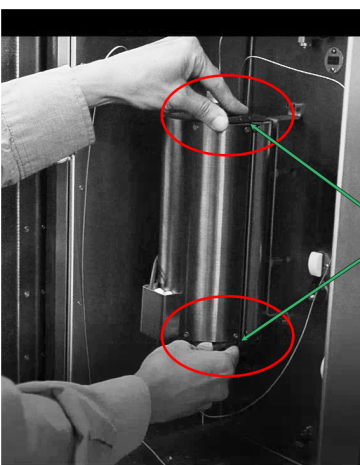


LOAD A SAMPLE

Required tools:

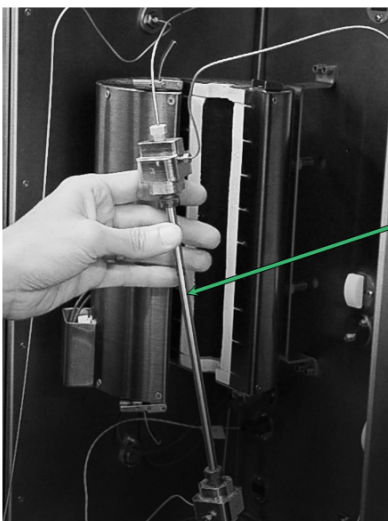
- 7/8 in. wrench
- 1/2 in. wrench
- Glass packing rods
- Funnel

1. Open the furnace compartment door.
2. Open the latches on the top and bottom of the furnace.



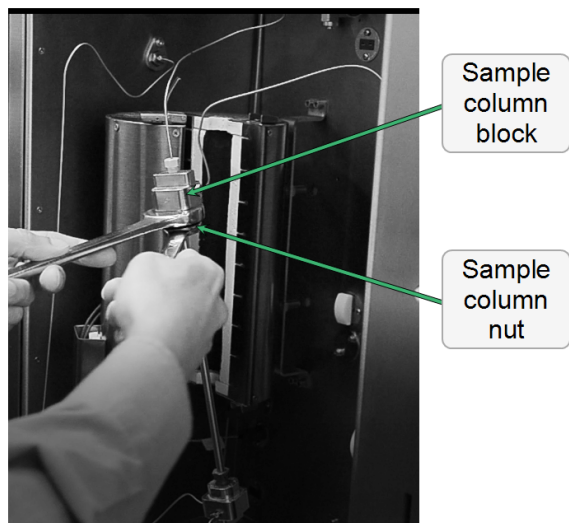
Open latches at top
and bottom of
furnace

3. Open the furnace and gently pull the sample column clear of the furnace.



Sample
column

4. Place a 7/8 in. wrench on the bottom of the top sample block and a 1/2 in. wrench on the sample column nut. Loosen the nut just enough so it can be turned by hand. Repeat the process for the bottom nut.



5. Turn the top nut with your fingers until it slides down the sample column. Hold the column and lift the top block until the thermocouple is clear.
6. Turn the bottom nut until it is free and lift the sample column clear of the bottom thermocouple.
7. Remove (or load) the sample. Quartz or glass wool is packed into the tube to provide a platform for the sample. Glass packing rods and a funnel for loading the sample are provided in the accessories kit.
8. Reverse the removal process to install the column. The position of the thermocouples may be adjusted after loosening the 3/8 in. PEEK nuts on the sample blocks. Ensure the sample column nuts and thermocouple nuts are snug to avoid leaks.

SAA DATA AND THE MICROACTIVE APPLICATION

Data recorded by the SAA 8100 can be exported from the data viewer as a text file. (Refer to the Process@ User's Manual.) The MKS Process Eye Professional application saves the mass spectrometer data as a text file. These two files are combined with mass spectrometer files in the Micromeritics MicroActive application to produce a sample file. In the MicroActive application, integration limits can be changed interactively, summary and plot reports can be produced, and advanced reports are available. (Refer to the MicroActive Operator Manual [*part number 202-42827-01*].)