TECHNICAL SPECIFICATION

Rev.0

AMI-300HP and RHP

- **PERFORMS:**
  - **AMBIENT AND HIGH PRESSURE DYNAMIC CHEMISORPTION**
  - **AMBIENT AND HIGH PRESSURE ISOTHERMAL REACTIONS**

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PREPARED BY:

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1. BASIC SYSTEM SPECIFICATIONS

• **System Operating Pressure**	100 bar*
• **Maximum Furnace Temperature**	650ºC*/1200 ºC
• **Maximum Vaporizer and Heat Trace**	220 ºC (if RHP is purchased)
• **Maximum TCD Temperature**	200ºC
• **Materials of Construction**	
○ **Piping**	1/4”, 1/8” and 1/16” stainless steel
○ **Sample Tubes**	
▪ **High Pressure (metal)**	Stainless Steel
▪ **Ambient pressure**	Quartz (if option is selected)
○ **Wetted Parts**	Stainless Steel, Premium Seals
• **Seal Materials**	Premium Seals
○ **Tubing and Fittings**	Stainless Steel
• **Catalyst Charge**	0.1 – 1 g
• **No. of Treatment Ports**	1 (option for 3)
• **No. of Carrier Ports**	1 (option for 3)
• **No. of Blend Gas Feeds**	1 (option for 3)
• **Approximate Dimensions**	
○ **Hardware Cabinet**	128cm W x 64cm H x 64cm D
• **Customer Responsibilities**	
○ **Oil-free, Dry Air**	supply at 80-100 psig
○ **Power Supply**	
• **Hardware Cabinet**	220V/20A; single phase
• **Computer**	220V/15A; single phase
• **Gas Feed System**	
○ **Number of Gases**	4 (1 is a Reference for TCD)
○ **Turn Down**	50:1
○ **Mass Flow Controller Flow Range**	As specified in Section 6
○ **Fittings**	Swagelok Fittings (or equivalent)
○ **Supply Pressure**	105 bar

*316SS reactor 650 ºC (100bar); Quartz reactor to 1200 ºC (ambient pressure)-optional

2. PROCEDURES

The system is designed to perform the following characterization experiments:

- Temperature Programmed Reduction (TPR)
- Temperature Programmed Oxidation (TPO)
- Temperature Programmed Desorption (TPD)
- Temperature Programmed Reaction (TPRx)
- Iso-thermal Reactions with or without vapor flow (RHP allows for dedicated vapor delivery to the reactor)
- Pulse Chemisorption
- Pulse Calibration

3. SYSTEM OPERATION

- The unit is constructed to operate at both ambient and high pressure.
- High-pressure operation is limited to 100 bar at 650 ºC
- Pressure control valves automatically control pressure in the system. This pressure is controlled and monitored from the graphical user interface (GUI) or is set during the design of an experiment.
- The unit uses stainless steel, u-tubes for all high pressure testing.

- If the quartz option is selected, these reactor tubes are only used when the system is at ambient pressure.
- The system utilizes high-precision mass flow controllers for treatment, carrier, blend, and reference gases.
- The system is equipped with the necessary instrumentation on all vital points in order to control, monitor and collect data where necessary.
- If the RHP is selected, an HPLC pump supplies a dedicated liquid feed to a heated vaporizer that consequently allows for delivery of a dedicated vapor stream to the reactor. In addition, there are several valves, which switch the operation of the unit from ambient operation to pressure operation. There is also heat tracing to prevent any condensation.
- The process control design is based on unattended operation, with all necessary measurements and controls available at the computer.

4. COMPUTER CONTROL

- The reactor system is controlled by means of the LabVIEW process control software with direct control from the PC. The computer control is able to control and monitor process parameters, acquire data in real time, monitor alarms and take proper actions, and trend both real time and historical data and generate reports and graphs.
- The computer is fully installed with Windows.
- Minimum specifications are:
  - Processor: 3.0 GHz
  - Operating System: Windows
  - Hard Drive: 200.0 GB

5. PROCESS CONTROL SOFTWARE

- The software is based on LabVIEW for Windows and will be configured by the supplier to the specific I/O of the reactor system. The configuration is for basic operation of the unit and will include the following features:
  - Manual operation (operator flow schematic screen)
  - Real time data trending
  - Historical data trending
  - Gas and PID calibrations
  - Signal filtering
  - Alarm history windows
  - Capability to create, store, and recall a set of experimental actions
  - Data storage and records of selected parameters, process data, and alarms
  - Multitasking capability
  - Data files in ASCII format to facilitate exchange with worksheet format
  - Data handling
  - Integration and overlay of experimental data

6. GAS FEED

- The gas feed system is divided into two main gas sections: Treatment and Carrier gases. The third mass flow controller provides gas-blending capability. MFCs are calibrated in nitrogen at an inlet pressure of 105 bar.

Mass Flow Controller	Flow range
MFC-T (one port)	0 - 200 SCCM
MFC-C (one port)	0 - 200 SCCM
MFC-B1 (one port)	0 - 100 SCCM
MFC-Ref (two ports)	0 – 250 SCCM

NOTE: Other flow ranges or calibrations are available and can specified at the time the instrument is ordered.

- Each gas flow is regulated and monitored using a mass flow controller, 0-5 Vdc output. MFC setpoint and readout are shown on the PC monitor.

7 FURNACE AND SAMPLING REACTOR

- The system furnace is a single zone clam-shell design. The furnace is capable of temperatures up to 1200ºC (reactor material dependent). The furnace temperature is controlled through a software-driven independent PID loop. Sample tubes can be installed and removed easily by opening the furnace.
- The sample station is located near the center front of the instrument.

8. THERMOCOUPLES

- Two type-K thermocouples are fixed to the furnace wall. The bottom thermocouple feeds the furnace over-temperature control. The top thermocouple serves as the indicator for the furnace PID control loop.
- An additional type-K thermocouple is used for temperature measurement of the sample.

9. THERMAL CONDUCTIVITY DETECTOR

- All instruments use a high quality 4-filament TCD, with high resolution, linearity, accuracy and stability. Standard filament material is tungsten; gold-plated tungsten filaments are available as an option for oxidative services, with some sacrifice in resolution.

10. ANALYSIS LINK

- The system can be used with any analytical instrumentation, such as an MS, GC or FID that provides its own independent control and data collection system. Data integration can be provided if the analytical instrument provides DDE communications capabilities.

11. SAFETY

- A number of features have been incorporated into the design of the system to ensure safe operation:
    - Check valves on blend MFC lines to prevent back-flow
    - Hardware over-temperature limit switch for the furnace is in the control cabinet and can be adjusted for customer specifications;
    - Pressure relief valves are in line with the reactor tube to prevent overpressure.
    - All process equipment operated by a power source are equipped with fuses
    - Software-coded safety backups monitor temperature and pressure for possible excursions. These alarms are mandated by the equipment safety limitations, and are configured by the system supplier.
    - GFIs for heat tracing.

12. INSTALLATION AND TRAINING

12.1 Site acceptance testing Site acceptance testing is performed to ensure the following:

1. Flow paths are correctly plumbed.
2. All valves work properly.
3. TCD operates properly.
4. Mass flow controllers work properly.
5. Furnace exhibits linear temperature ramp and maintains a stable hold at ramp completion
6. A Temperature Programmed Reduction (TPR) of standard catalyst at ambient pressure obtains predicted results.

12.2 Training Site training and installation includes the following:

1. Set up the system and perform a few quick tests to the computer to ensure that all components arrived in good operating condition.
2. Give users a description of instrument components.
3. Walk users through a thorough explanation of the control software.
4. Instruct users on sample loading and unloading.
5. Perform a temperature programmed reduction with standard catalyst.
6. Vendor personnel do not run customer samples as part of the installation and training session. However, if the customer requests, we will help set up a run at the conclusion of the installation and training session.

12.3 Customer Requirements Customers must provide the following:

1. A 220V/20A power source in close proximity to the instrument.
2. A ¼” continuous source of compressed air in close proximity to the instrument, regulated to 80 psig and a 1/4 ‘’ 1550 psig source of compressed air for the PCV.
3. Sufficient ¼” tubing and fittings to supply air to the system.
4. A gas cylinder of 10% hydrogen in argon (mixtures of 5-10% are also acceptable)
5. A gas cylinder of pure argon (99.999% recommended).
6. Regulators for all process gas cylinders, capable of regulating to 105 bar at the outlet of the regulators going into the AMI-300HP unit.
7. Sufficient 1/8” tubing and fittings to connect the regulators to the back panel of the instrument and provide four vent lines. Stainless steel tubing is acceptable.
8. Altamira Instruments recommends the use of ultra-high purity gases (99.999%) for all analyses.

13. FACTORY ACCEPTANCE TESTING

- The functionality of the instrument will be tested per Altamira Instrument’s standard testing criteria. A temperature-programmed reduction will be performed using standard cobalt on alumina catalyst. Additional testing will include the following:
- After completion of the mechanical construction, the unit will be tested for proper operation of the various components at the operating pressure, flow and temperature.
- Factory acceptance testing on the reactor functions will be performed with nitrogen only. The unit will be deemed accepted if it meets, and passes, the following criteria and/or tests during the acceptance testing:
    1. **Completeness:** Check that all process, electrical, instrumentation, computer materials and components included in the engineering design and in the bill of materials have been installed properly.
    2. **Mechanical Test:** Check all automatic valves and other devices for on/off operation. e.g. valves, pumps and heaters. Check all control loops for full open and full close position. Check all heaters for on/off operation.
    3. **Leak Test:** A pressure drop leak test will be performed. Bubble detection fluid will be used locate leaks. There should be no visible leaks at the design pressure of the unit. There should be less than a 4 % drop over 2 hours.

4. **Functional Test:** In order to check the performance of the equipment and related instruments, in relation to the design specifications, a so-called functional testing will be performed. This will include a TPR on our standard reduction catalyst and a pulse chemisorption on our standard chemisorption catalyst.
5. **Control Test:** The proper functioning of control loops, e.g. control valves, heaters, etc. will be checked during the same period as mentioned under “e”, and checked against the design specifications. All PID control loops will be checked for proper tuning to deliver optimum control.
6. **Alarm Tests:** Check alarm actions at the alarm conditions in the process design specifications. This will include checking the proper functioning of the temperature safety switches.
7. **Documents:** Check that all documents, the system and manufacturers manuals are in the “as built” version and complete.

14. DOCUMENTATION

- The final documentation sets provided with the system unit will consist of the following items:
    - Software Manual including: User manual for process and peripherals
    - Hardware Manual including:
    - Process and instrumentation diagram;
    - Mechanical drawings;
    - Electrical wiring diagrams;

- All manufacturers’ manuals and documentation received with buy-out materials.

15. SEAL MATERIALS

The chemical compatibility chart below can be used to determine, which seal material will work best with your applications.

Chemicals	Viton	Buna-N	Premium
Acetone	Do Not Use	Do Not Use	Excellent
Acids			
Chromic	Excellent	Do Not Use	Excellent
Hydrochloric	Excellent	Do Not Use	Excellent
Hydrofluoric	Excellent	Do Not Use	Excellent
Nitric	Excellent	Do Not Use	Excellent
Phosphoric	Excellent	Do Not Use	Excellent
Sulfuric	Excellent	Do Not Use	Excellent
Amines	Do Not Use	Do Not Use	Excellent
Diethylamine	Do Not Use	Poor	Excellent
Ammonia			
10%	Do Not Use	Excellent	Excellent
Anhydrous	Do Not Use	Good	Excellent
Benzene	Good	Do Not Use	Excellent
Carbon Dioxide	Good	Excellent	Excellent
Hydrocarbons			
Aromatic	Excellent	Poor	Excellent
Naphtha	Excellent	Poor	Excellent
Pyridine	Do Not Use	Do Not Use	Excellent
Sulfur Materials			
Hydrogen Sulfide	Do Not Use	Do Not Use	Excellent
Sulfates (SOx)	Excellent	Do Not Use	Excellent
Water			
Steam	Do Not Use	Excellent	Excellent

* Premium seals include those made of Kalrez, Perlast, Kel-f, Tefzel, Teflon, and PEEK.