TECHNICAL SPECIFICATION

FOR:

AMI-300

DYNAMIC CHEMISORPTION ANALYZER

Revision 0

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PREPARED BY:

ALTAMIRA INSTRUMENTS

149 DELTA DRIVE, SUITE 200

PITTSBURGH, PA 15238

USA

1. BASIC SYSTEM SPECIFICATIONS

System Operating Pressure	ambient
Gas Inlet Pressure Range	40 to 60 psig
Maximum Furnace Temperature	1200ºC
Maximum Valve Oven Temperature	80ºC (option: 150ºC)
Maximum TCD Temperature	200ºC
Mass Flow Controller Flow Range	0-50 sccm (standard); can be modified at sale
Materials of Construction	
▪ **Plumbing**	316SS
▪ **Sample U-tubes**	Quartz
▪ **Wetted Parts**	316SS
▪ **Seal Materials**	Premium, Viton, Buna-N depending on quote
Catalyst Charge	0.1 – 1 g (different U-tubes)
No. of Treatment Ports	4
No. of Carrier Ports	4
No. of Blend Gas Ports	2 (option for 4 with 4 MFCs)
No. of Gas Ports Total	10 (12 if 4th MFC option is selected)
Dimensions	
Hardware Cabinet	64cm W x 64cm H x 64cm D
Utility Requirements	
Oil-free, Dry Air	Air supply at 80 psig
Power Supply	220V 20A; single phase (Cabinet) 220V 15A; single phase (Computer)

2. PROCEDURE

The AMI-300 system is designed to perform the following characterization experiments:

- Temperature Programmed Reduction (TPR)
- Temperature Programmed Oxidation (TPO)
- Temperature Programmed Desorption (TPD)
- Temperature Programmed or Iso-thermal Reaction
- Pulse Chemisorption
- Pulse Calibration
- Flow BET

3. AMI-300 OPERATION:

The unit is constructed to operate at ambient pressure. The unit uses a quartz u-tube in one of several configurations (see Section 8).

The AMI-300 utilizes high-precision mass flow controllers for treatment and carrier gases. This reaction mixture is directed to the reactor and also used as the reference gas for the TCD. The gas flow proceeds to the reactor and is then directed back through the instrument to the TCD for sample gas detection. For auxiliary detection, a mass spectrometer can be attached either to the fitting above the reactor for

sampling directly from the reactor well or to the analytical vent. If purchased at the same time, the masses from the spectrometer can be incorporated into a single Altamira data file.

The unit is equipped with a moveable clamshell furnace, with a maximum temperature of 1200ºC. The furnace is capable of linear temperature ramps up to 50ºC/minute. Air-cooling is used to speed the time between experiments. A single internal temperature-controlled zone containing all the plumbing downstream of the reactor is heated to a selected temperature to prevent condensation in that area.

The system is equipped with the necessary instrumentation on all vital points in order to control, monitor and collect data where necessary. Instrumentation is installed on the two shelves below the plumbing shelf.

The process control design is based on unattended operation; all necessary measurements are 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 a brand name PC, and can be customized upon request. Minimum specifications are:

- PC Processor: 3.00 GHz
- Operating System: Windows
- Hard Drive: 200.0 GB
- Monitor: 19”
- RAM 3 GB

5. PROCESS CONTROL SOFTWARE

The software is based on LabVIEW Real-Time for Windows and is configured by Altamira Instruments to the specific I/O of the AMI system. The configuration for basic operation of the unit includes the following features or more if required:

- Manual operation (operator flow schematic screen)
- Real time data trending
- Historical data trending
- Gas and PID calibrations
- Signal filtering
- Alarm history windows
- Creation, storing, and recalling a set of experimental actions
- Data storage and records of selected parameters, process data, and alarms
- Multitasking capability

6. ANALYSIS SOFTWARE

The Altamira Analysis software provides post-processing of experimental data generated by the control system. Extensive signal processing and report generation features are incorporated.

- Seamlessly import and "zip" archived data generated by control software
- Enhanced report generation capabilities
- "Drag and drop" base-lining, signal smoothing, and numeric integration
- Descriptive calculations including uptake, % dispersion, % consumption and more
- Apply signal transformations including scaling, time-shifts, and dependent offsets
- View pulse data in sequential or overlay mode
- Manually adjustable and automatic peak fitting
- Export signals as text tab-delimited files compatible with other processing and report software

7. GAS FEED

The AMI-300 gas feed system is divided into two main gas sections: treatment and carrier gases. A third mass flow controller (MFC) provides gas blending capability. Each main gas line allows precise control of the gases at the operating pressure and over the following ranges of flow:

Mass Flow Controller	Flow Range *
MFC-T	0-50 sccm
MFC-C	0-50 sccm
MFC-B	0-50 sccm
* - Other ranges available; higher ranges will reduce accuracy at low flows.	

Each gas flow is regulated and monitored using an MFC with 0-5 Vdc output. MFC set point and readout are shown on the PC monitor. Up to three gases can be used for the treatment and carrier gas lines. A set of three-way solenoid valves makes up the manifold to supply the system.

The third MFC (the gas blending MFC) can be used on either treatment or carrier gas sides. In addition, there are options for:

- Fourth MFC to provide additional gas blending capabilities
- Enhanced blending which allows for all of the gas-streams to be mixed together, this is particularly useful if the AMI is to be used for gas-phase iso-thermal reaction experiments with an MS or GC.

8. FURNACE AND SAMPLING REACTOR

The AMI-300 furnace is a single zone clam-shell design. Its nominal maximum temperature is 1200ºC. The furnace temperature is controlled through a software-driven independent PID loop based on the furnace or bed temperature readings. Sample u-tubes can be installed and removed easily by opening the furnace.

The sample station is located near the center front of the instrument. Quartz sample u-tubes are held in place by Ultra-torr compression fittings. The fittings are sealed with 1/4" O-rings to ensure optimal sealing.

Several types of sample u-tubes are available:

- **Standard u-tube**
- **3/8" bubble u-tube:** incorporates a three inch long, 3/8" diameter bubble on one leg;
- **1/2" bubble u-tube:** incorporates a three inch long, ½" diameter bubble on one leg;
- **BET u-tube:** rectangular u-tube to permit rapid evaporation of the adsorbate from the sample;
- **Sub-ambient u-tube:** special tube-within-a-tube design for cryogenic cooling of sample and carrier gas;
- **Core monolith sample holder:** incorporates a 1" ID tube with an o-ring seal.

A 1/16" sample thermocouple may be inserted into the sample tube. Alternatively, a 1/8" quartz sheath may be used to isolate the thermocouple. When using the quartz sheath, an 1/8'' to 1/8" graphite ferrule is used.

9. THERMOCOUPLES

Two type-K thermocouples are fixed to the furnace wall. The bottom thermocouple feeds the furnace over-temperature control. The top thermocouple or the bed thermocouple can serve as the indicator for the furnace PID control loop.

10. HEATED VALVES AND LINES

All plumbing downstream of the gas saturator is heated to prevent condensation and retention of the adsorbate in valves and lines prior to the detector.

11. THERMAL CONDUCTIVITY DETECTOR

All AMI-300 instruments use a high quality 4-filament TCD, with high resolution, linearity, accuracy and stability. Standard filament material is tungsten. Other filament materials (Gold-plated, Nickel, etc…) are available.

12. ANALYSIS LINK

The system can be used with any analytical instrumentation, such as MS, GC, and/or an FID which provides its own independent control and data collection system. Data integration can be provided if the analytical instrument provides DDE communications capabilities or if the instrumentation gives an analog output (voltage) signal.

13. STANDARD OPTIONAL ACCESSORIES

13.1 **Sub-ambient Option -** Supports operation at –130ºC: The subambient option consists of a liquid nitrogen dewar, a flexible stainless steel hose that connects to the sample holder, and a Teflon hose that connects to a solenoid valve inside the AMI instrument. Nitrogen gas is supplied to the

solenoid and through the dewar filled with liquid nitrogen. This cooled gas is then fed to a special subambient sample holder, which provides a jacket around the reactor tube. When the AMI-300 software is set for a temperature below the standard room temperature of 25ºC, the valve supplies nitrogen to the dewar. This cooled nitrogen proceeds to the jacket to achieve the desired temperature. Temperature ramping is linear from –130ºC to 1000ºC

13.2 **Mass Spectrometer Option**: The mass spectrometer with an enclosed ion source provides supplementary analytical capability, aiding in the identification of materials generated while performing analysis in the AMI-300. A dry pumping system prevents any additional hydrocarbons originating from pump oils from getting into the system and providing false hydrocarbon indications. The mass spectrometer can have mass ranges of up to 100, 200, and 300 AMU's and a capillary heater is provided to prevent condensation. The software displays the plots of mass numbers along with the TCD data to allow better interpretation of results.

13.3 **Fourth MFC Option**: The fourth MFC option consists of an additional mass flow controller, a three-way valve, a filter, and a check valve. The option permits two different blending gases to be on either on the carrier or treatment side. There are two gas inlet ports connected to the MFC. Please note, only one port can be selected at a time. If this option is selected there are a total of 12 inlet gas ports on the AMI.

13.4 **Saturator Option**: The saturator options includes a heating mantle and a Pyrex vessel that allows for the delivery of a vapor (via sparging) to the solid sample in the reactor u-tube in treatment or pulse steps. The saturator can be heated to 75°C and all lines downstream are contained in a heated box. This option is able to be added in the field after the sale.

13.5 **High Temperature Valve Oven:** The high temperature valve oven allows for temperatures downstream of the reactor up to 150°C.

13.6 **IR Option**: Consists of an IR transmission cell, heaters, and FTIR for real-time observation of the catalyst surface. Maximum temperature (in argon-rich atmosphere) is 500°C. The IR option aids in the determination of the mode/type of adsorption.

13.7 **Enhanced Gas-Blending**: Allows for the blending of three gases inside the AMI. This is ideal for isothermal reaction tests couples with a mass spectrometer.

14. SAFETY

A number of features have been incorporated into the design of the AMI-300 Catalyst characterization System to ensure safe operation:

- Hardware over-temperature limit switch with independent thermocouple on the furnace that can be adjusted for customer specifications;
- Pressure relief valves are set at 19 psig in line with the saturator and quartz reactor to prevent overpressure of the glassware.
- All process equipment operated by a power source are equipped with fuses
- Software-coded safety backups monitor temperature for possible excursions. These alarms are mandated by the equipment safety limitations, and are configured by Altamira Instruments.

15. INSTALLATION AND TRAINING

15.1 **Site acceptance testing:** Site acceptance testing is performed to ensure the following:

- Flow paths are correctly plumbed
- All valves work properly
- TCD operates properly
- Mass flow controllers work properly
- Furnace exhibits linear temperature ramp and maintains a stable hold at ramp completion
- A single Temperature Programmed Reduction (TPR) of Altamira Instrument's standard catalyst obtains predicted results

15.2 **Training:** Site training and installation includes the following:

- Set up the AMI-300 and perform a few quick tests to ensure that all components arrived in good operating condition
- Give users a description of instrument components
- Walk users through a thorough explanation of the AMI-300 software
- Instruct users on sample loading and unloading
- Perform a temperature programmed reduction with standard Altamira Instruments catalyst

15.3 **Utilities and Consumable Requirements:** Customers must provide the following:

1. A 220V/20A power source in close proximity to the AMI-300
2. A 1/4" source of compressed air at 80 psig in close proximity to the AMI-300
3. Sufficient ¼" tubing and fittings to supply air to the AMI-300
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
7. Sufficient 1/8" tubing and fittings to connect the regulators to the back panel of the AMI-300 and provide four vent lines. Stainless steel tubing is suggested. Please ensure that all gases supplied to the instrument are ultra-high purity and that any residual moisture, oxygen, and hydrocarbons are trapped before the AMI ports.

NOTE: Altamira Instruments recommends the use of ultra-high purity gases (99.999%) for all analyses.

16. ALTAMIRA INSTRUMENTS INSTRUMENT ACCEPTANCE TESTING

The characterization functionality of the AMI-300 is tested in accordance with Altamira Instruments' standard testing criteria. A temperature-programmed reduction is performed using Altamira Instruments' standard catalyst. Additional testing for the reactor functionality is as follows:

After completion of the mechanical construction, the unit is tested for proper operation of the various components at the operating pressure, flow and temperature. Altamira Instruments will not be responsible for acceptance testing concerning customer reactions.

- **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.

- **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.

- **Leak Test:** Apply bubble detection fluid to all joints and connections. There should be no visible leaks at the design pressure of the unit.

- **Functional Test:** In order to check the performance of the equipment and related instruments, in relation to the design specifications, functional testing will be performed. This will include a TPR on our standard reduction catalyst, a pulse chemisorption on our standard chemisorption catalyst, and a BET surface area analysis on our standard BET support.

- **Control Test:** The proper functioning of control loops, e.g. control valves, heaters, etc. will be checked during the same period as mentioned above and checked against the design specifications. All PID control loops will be checked for proper tuning to deliver optimum control.

- **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.

- **Documents:** Check that all documents, the system and manufacturers manuals are in the "as built" version and complete.

17. 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
  - Manufacturers' manuals

18. 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
Nitrous Oxides			
Pyridine	Do Not Use	Do Not Use	Excellent/Good
Sulfur Materials			
Hydrogen Sulfide	Do Not Use	Do Not Use	Excellent/Good
Sulfates (SOx)	Excellent	Do Not Use	Excellent
Water			
Steam	Do Not Use	Excellent	Excellent

Premium seals include parts constructed of the following materials: Perlast, Kalrez, Kel-F, PEEK, Teflon, Tefzel, and EPDM.

19. DETECTOR MATERIALS

Substance	Tungsten (W/WX)	Nickel (Ni)	Gold-sheathed Tungsten (AuW)
Air/Oxygen	Good	Good	Very Good
Water	Good	Good	Good
Steam	Good below 700 °C	Good	Good
Ammonia/Amines	Good	Poor in presence of H_2O	Poor
CO/CO_2	Good	Good	Good
Hydrogen	Good	Good	Good
Nitrogen	Good	Good	Fair
Fluorine	Poor (fluoride) forms at 20 °C	Good	Poor
Chlorine	Fair	Good	Fair
Bromine	Fair	Good	Fair
Iodine	Fair	Good	Fair
Sulfur	Fair	Poor	Good
H_2S/SO_2 (sulfuric acid)	Fair	Poor	Good
HC	Fair	Good	Fair
Aqua Regia	Fair	Poor	Poor
HF	Fair	Good	Fair
HF/HNO_3	Poor	Good	Poor