TECHNICAL SPECIFICATION

FOR:

AMI-300*ip* (Integrated Pre-treatment Station)
High-throughput Chemisorption Analyzer
Revision 0

12 February 2020

PREPARED BY:

ALTAMIRA INSTRUMENTS

149 DELTA DRIVE, SUITE 200

PITTSBURGH, PA 15238

USA

1. BASIC SYSTEM SPECIFICATIONS

No. of Total Stations	2 (1 for characterization; 1 for pretreatment)
No. of Pretreatment Stations	1
System Operating Pressure	ambient
Gas Inlet Pressure Range	40 to 60 psig
Maximum Furnace Temperatures	1200ºC
Maximum TCD Temperature	200ºC
Mass Flow Controller Flow Range	0-50 sccm (standard); can be modified
Materials of Construction	
Plumbing	316SS
Sample U-tubes	Quartz
Wetted Parts	Stainless Steel, Quartz, Viton is standard
Catalyst Charge	0.1 – 1 g (with different sample tubes)
Type of MFCs	4 MFCs
No. of Treatment Ports	4
No. of Carrier Ports	4
No. of Blend Ports	2
No. of Pretreatment Gas Ports	2
Dimensions	
Hardware Cabinet	128 cm W x 64cm H x 64cm D
Customer Responsibilities	
Oil-free, Dry Air	supply at 80 psig
Power Supply: Standard	
Hardware Cabinet	220V 25A
Computer	220V 15A

2. PROCEDURES

The AMI-300*ip* 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)
- Gas-phase Isothermal Reaction
- Treatment
- Pulse Chemisorption
- Pulse Calibration
- Flow BET
- Automatic switching between the pre-treatment and the analysis station

3. AMI-300*ip* OPERATION:

The unit is contained within a modified AMI-300 framework, the unit is constructed to operate at ambient pressure and will use quartz u-tubes identical to the original AMI-300.

The AMI-300*ip* utilizes high-precision mass flow controllers for treatment, carrier, blend, and pretreatment gases. The reaction mixture is used as the reference gas for the TCD. The gas flow proceeds to the reactor and then be directed back through the instrument to the TCD for sample gas detection.

The unit is equipped with a moveable clamshell furnace, which can heat the sample to 1200ºC and is capable of linear temperature ramps up to 50ºC/minute. Air-cooling is used to decrease the time between experiments. A single internal temperature-controlled zone containing all the plumbing downstream of the reactor is heated to prevent condensation and retention of any adsorbates in valves and lines upstream of the detector.

The pretreatment station of the AMI-300ip performs catalytic activation and/or treatment steps up to 1200 ºC. Automatic switching between the pre-treatment and the analysis station allows the user to conduct an analytical step on a sample without the need to move it, thus preventing contamination from the atmosphere.

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: 17” LCD
- 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 reactor 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*ip* gas feed system is divided into: Treatment, Carrier, Blend, Pretreatment gases, each with a turndown ration of 50:1. Each gas line allows accurate control (within 1.0% of full scale) of the gas 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
MFC-P	0-50 sccm
* - Other ranges available; higher ranges will reduce accuracy at low flows.	

MFC setpoint and readout are shown on the PC monitor. Up to four 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 pretreatment MFC (MFC-P) is used exclusively for the pre-treatment station.

8. FURNACE, SAMPLING REACTOR, AND PRETREATMENT

The AMI-300*ip* 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. Sample u-tubes

can be installed and removed easily by opening the furnace. Air cooling is used to lessen the time between experiments.

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.

The pretreatment furnace has a maximum nominal temperature of 1200ºC.

9. THERMOCOUPLES

Two type-K thermocouples are fixed to each furnace wall. The bottom thermocouple feeds the furnace over-temperature control. The top thermocouple serves as the indicator for the furnace PID control loop. A sample thermocouple is also placed inside of the reactor. This thermocouple can be used to control or monitor the temperature of the reaction. In addition there is a thermocouple for the valve oven, TCD, and saturator heaters.

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. Maximum temperature is 80ºC.

11. THERMAL CONDUCTIVITY DETECTOR

All AMI-300*ip* instruments use a high quality 4-filament TCD, with high resolution, linearity, accuracy and stability. Standard filament material is tungsten; gold-plated tungsten is available for oxidative services.

12. ANALYSIS LINK

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

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. For the AMI-300ip, each station is equipped with the sub-ambient valve, but only one dewar is supplied.

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. 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. There is one capillary for the MS, it can be connected to either station.

13.3 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.

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

13.5 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*ip* Catalyst Characterization System to ensure safe operation:

- Hardware over-temperature limit switches for all furnaces are in the control drawer and can be adjusted for customer specifications;
- Pressure relief valves are set at 17 psig in line with the saturator and quartz reactor to prevent overpressure of the glassware.
- Power switches control power output to the instrument in case of an emergency.
- 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 Altamira Instruments.

15. INSTALLATION AND TRAINING

15.1 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 single Temperature Programmed Reduction (TPR) of Altamira Instrument's standard catalyst obtains predicted results

15.2 Training

Site training and installation includes the following:

1. Set up the AMI-300*ip* and perform a few quick tests 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 AMI-300*ip* software

4. Instruct users on sample loading and unloading
5. Perform a temperature programmed reduction with standard Altamira Instruments catalyst

NOTE: Altamira Instruments 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.

15.3 Customer Requirements

Customers must provide the following:

1. A 220V power source in close proximity to the AMI-300*ip*
2. A ¼" source of compressed air at 80 psig in close proximity to the AMI-300*ip*
3. Sufficient ¼" tubing and fittings to supply air to the AMI-300*ip*
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 set at 40 psig
7. Sufficient 1/8" tubing and fittings to connect the regulators to the back panel of the AMI-300*ip* and provide four vent lines.

NOTE: Altamira Instruments recommends the use of ultra-high purity gases (99.999%) with traps for water, oxygen, and hydrocarbons for all analyses.

16. ALTAMIRA INSTRUMENTS INSTRUMENT FACTORY ACCEPTANCE TESTING

The characterization functionality of the modified AMI-300*ip* will be tested per Altamira Instruments' standard testing criteria. A temperature-programmed reduction will be performed using Altamira Instruments' standard catalyst. Any additional testing for any reactor functionality is as listed in 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. Altamira Instruments will not be responsible for acceptance testing concerning customer reactions.

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:

- **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, a so-called 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 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;
Process and electrical bill of materials;
Mechanical drawings;
Electrical wiring diagrams;

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

18. SEAL MATERIALS

The chemical compatibility chart below can be used to determine, which seal material will work best with your applications. Viton is the standard seal for the AMI-300ip.

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
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 Kalrez, Perlast, Kel-F, Teflon, PEEK, and Tefzel.