TECHNICAL SPECIFICATION

FOR:

Custom

AMI-300S plus

CATALYST CHARACTERIZATION INSTRUMENT FOR HARSH ENVIRONMENTS

Revision 0

PREPARED BY:

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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	150ºC
Maximum TCD Temperature	200ºC
Number of Mass Flow Controllers	4 (Carrier, Treatment, Blend, H2S)*
Mass Flow Controller Flow Range	0-50 sccm (standard); can be modified
Materials of Construction	
▪ **Plumbing**	316SS/Sulfinert/glass-lined/Dursan
▪ **Sample U-tubes**	Quartz
▪ **Seal Materials**	Premium
Catalyst Charge	0.1 – 1 g (different U-tube)
No. of Treatment Ports	4
No. of Carrier Ports	4
No. of Blend Gas Ports	2
No. of H2S Gas Ports	1
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)

***MFC Notes:**

-Carrier [C]-MFC is used for the analytical gas to the reactor. The MFC has premium seals. All tubing from the inlet to the instrument to the reactor is 316SS. All seals in valves and/or fittings is of premium type. H2S should not be used in this inlet line.
-Treatment [T]-MFC is used for non-corrosive gases in order to treat the sample. The MFC has premium seals. All tubing from the inlet to the instrument to the reactor is 316SS. All seals in valves and/or fittings are of premium type. H2S should not be used in this inlet line.
-Blend [B]-MFC is used for additional flow allowances on either the Carrier or Treatment side of the instrument. The MFC has have premium seals. All tubing from the inlet to the instrument to the reactor is 316SS. All seals in valves and/or fittings are of premium type. H2S should not be used in this inlet line.
-H2S MFC is used in-place of the Carrier-MFC for TPS experiments to the TCD (up to 15%). The MFC has premium seals and Au/Ni Braze. All tubing from the inlet to the instrument to the reactor is passivated. This provides a passivation layer for the H2S. An alternate flow-path to the TCD is used to mitigate cross-contamination. Although the usage of any fittings is minimized, the fittings that are needed are coated with Dursan. Usage of valves, check valves, and filters is also minimized. Spares for these components (valves, check valves, filters) should be kept since H2S is extremely corrosive and can shorten component lifetime.

2. PROCEDURES

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

- Temperature Programmed Sulphidation (TPS)
- Temperature Programmed Reduction (TPR)
- Temperature Programmed Oxidation (TPO)
- Temperature Programmed Desorption (TPD)
- Gas Phase Isothermal Reaction
- Treatment with H2S
- Pulse Chemisorption
- Pulse Calibration

3. AMI-300S plus 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 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.

The unit is equipped with a moveable clamshell furnace, with a maximum operating 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. 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-300S plus gas feed system is divided into two main gas sections: treatment and carrier gases. A third mass flow controller (MFC) provides gas blending capability. The H2S MFC is a dedicated MFC that allows for TCD experiments with H2S. 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
MFC-H2S	0-50 sccm

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 Blend MFC (the gas blending MFC) can be used on either treatment or carrier gas sides.

8. FURNACE AND SAMPLING REACTOR

The AMI-300S plus 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 6mm (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;

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, a 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. GAS SATURATOR/VAPOR GENERATOR

The gas saturator is connected to the right-most connections in the front cavity. The saturator is used to saturate a treatment gas with a volatile organic species, and can be heated to 75ºC.

11. 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.

12. THERMAL CONDUCTIVITY DETECTOR

The AMI-300S plus instrument uses a high quality 4-filament TCD with Gold filaments and Monel block for high resolution, linearity, accuracy and stability.

13. 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.

14. SAFETY

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

- Hardware over-temperature limit switch for the furnace in the control cabinet and 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.
- E-stop button on front panel
- 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.