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**AN INTEGRATED CHEMICAL REACTOR-HEAT
EXCHANGER BASED ON AMMONIUM CARBAMATE
(POSTPRINT)**

Douglas Johnson and Jamie Ervin

University of Dayton Research Institute

Soumya Patnaik

**Mechanical and Thermal Systems Branch
Power and Control Division**

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Power and Control Division

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Mechanical and Thermal Systems Branch
Power and Control Division
Aerospace Systems Directorate

//Signature//

JOHN G. NAIRUS, Chief Engineer
Power and Control Division
Aerospace Systems Directorate

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An Integrated Chemical Reactor-heat Exchanger based on Ammonium Carbamate

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Douglas Johnson
University of Dayton Research Institute

Soumya Patnaik
US Air Force Research Laboratory

Jamie Ervin
University of Dayton Research Institute

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ABSTRACT

In this work we present our recent effort in developing a novel heat exchanger based on endothermic chemical reaction (HEX reactor). The proposed HEX reactor is designed to provide additional heat sink capability for aircraft thermal management systems. Ammonium carbamate (AC) which has a decomposition enthalpy of 1.8 MJ/kg is suspended in propylene glycol and used as the heat exchanger working fluid. The decomposition temperature of AC is pressure dependent (60°C at 1 atmosphere; lower temperatures at lower pressures) and as the heat load on the HEX increases and the glycol temperature reaches AC decomposition temperature, AC decomposes and isothermally absorbs energy from the glycol. The reaction, and therefore the heat transfer rate, is controlled by regulating the pressure within the reactor side of the heat exchanger. The experiment is designed to demonstrate continuous replenishment of AC. This requires recovering the depleted glycol while expelling waste gases, dispersing and suspending fresh AC, and injecting the mixture into the heat exchanger. A gasketed plate heat exchanger is used as the reactor for this experiment, and heated water is used to provide the thermal load. The performance of the HEX reactor is characterized as a function of water flow rate and temperature, AC/glycol mixture flow rate, and AC concentration. Varying these parameters permits mapping the performance of the system under different conditions.

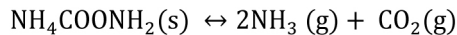
INTRODUCTION

There has been considerable interest in using Heat EXchanger/chemical reactors, or HEX reactors, to improve heat transfer in chemical reactor systems [1]. A HEX reactor is a heat exchanger in which chemical reactants flow through one set of passages where a reaction occurs, while a different heat transfer medium flows through the other set of passages to add or remove thermal energy from the reactor. HEX reactors have received attention recently because they offer improvements over current batch-type chemical reactors such as high throughput, low fouling, high mixing intensity and a narrow residence time distribution (materials enter and exit the reactor uniformly) [2]. Current applications of HEX reactors tend to focus on their ability to quickly remove heat generated by exothermic chemical reactions [5], thereby stabilizing the reaction temperature while permitting higher product throughput. This capability suggests that a HEX reactor can be used as a high-density aircraft thermal management device using an endothermic reaction. The focus of this preliminary paper is on the behavior of the chemical selected for study and the development of the experimental hardware and system configuration to investigate its thermal management capability.

EXPERIMENTAL

The objective of the current research effort is to design, build and demonstrate a system that uses the endothermic decomposition of ammonium carbamate (AC) within a HEX reactor to absorb low quality heat. AC decomposition is well

suitable for this application because the material has a high decomposition enthalpy and exhibits decomposition over a wide range of temperatures. AC decomposition produces ammonia and carbon dioxide gases in the (reversible) reaction



absorbing 1800 kJ/kg thermal energy [6]. To accomplish the project objective, the HEX reactor is connected to two fluid loops, referred to as the reactant loop and the load loop (Figure 1). The reactant loop dispenses a prescribed mass of AC and suspends it in propylene glycol (PG) carrier fluid, which is pumped into the HEX reactor at a prescribed rate. The load loop circulates heated water through the HEX reactor to provide the thermal energy to be removed by AC decomposition. Figure 1 shows the arrangement, with the three sections (load loop, HEX reactor and reactant loop) depicted; each section is discussed in detail below.

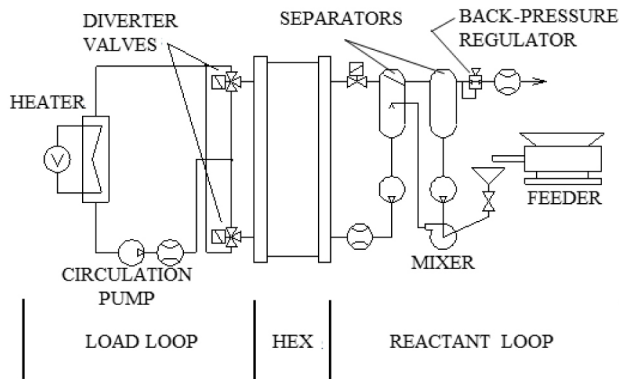


Figure 1. Experiment Block Diagram, Showing Major Subassemblies

Load Loop

The load loop consists of a heater, a power supply for the heater, a circulation pump, and the HEX reactor load side. The heat load is supplied by circulating water through a 1500 W inline immersion heater (TruHeat STFT-1500-120) powered by a digital DC power supply (Agilent N5770A). The power supply output is controlled using a PID loop embedded in the LabVIEW control program. Using this control scheme, it is possible to measure the heat removed by AC decomposition in the HEX reactor by directly measuring the electric power required to reheat the water to the load-side inlet temperature, typically 70°C. In addition to using the power supply to measure heat removal, calorimetry of the load loop is performed by measuring the load loop inlet-to-outlet temperature difference and the water flow rate. The hot water is plumbed into the HEX reactor through two three-way diverter valves, which permit testing the HEX reactor in either a parallel or a counter-flow arrangement. The heater power supply and a variable-speed positive-displacement circulation pump provide complete control of the load conditions.

Hex Reactor Assembly

The HEX reactor for this project is a modified Alfa Laval M3-F2 gasketed plate heat exchanger (HX). A gasketed HEX reactor is chosen for this effort because it offers several features advantageous to a research project. For example, the HX can be disassembled, permitting internal sensors to be installed; plates can be added or removed, providing adjustable heat transfer area. A further experimental capability is added by replacing the rear pressure plate with a transparent plate onto which the HX plate chevron pattern had been cast. This permits visual observation of the reaction in one channel. Figure 2 (left) shows the rear face of the HEX reactor, with transparent pressure plate and associated instrumentation ports, and in Fig. 2 (right), the port face of the reactor with the diverter and shutoff valves and liquid/gas separator first stage.

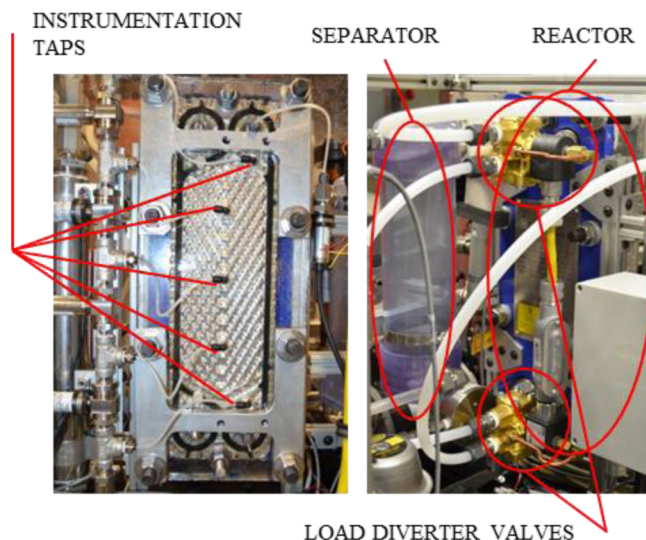


Figure 2. (left) HEX Reactor Rear Face, showing Transparent Pressure Plate with Instrumentation Taps; (right) Port Face of HEX Reactor showing locations of Waste Gas/Liquid Separator and Load Loop Diverter Valves

Reactant Loop

The reactant loop consists of the gravimetric powder feeder, an isolator valve, the mixer, a charge pump, an injector pump, two liquid/gas separators, and a back-pressure regulator valve (BPRV). The feeder (Schenck Accurate Mechatron) meters the powdered AC through the isolator valve into the mixer (IKA Werke MagicLAB with attached MHD generator module). The isolator valve acts to positively feed dry powdered AC into the mixer during operation without affecting the feeder's mass measurements, to minimize air ingestion into the mixer, and to close the mixer's powder inlet when the mixer is off. The feeder feedrate and mixer speed can be adjusted to vary the concentration of AC suspended in PG. The AC/PG suspension is mixed with depleted PG in the

first separator, which acts as a reservoir and pumped into the HEX reactor at approximately five liters/minute.

After AC decomposition, the waste gases and depleted PG flow from the HEX reactor into a baffle-type separator which separates heavy liquid from lighter gases and liquid droplets. The liquid from this stage is collected in the reservoir. The wet gases are piped into a centrifugal-type liquid/gas separator, where the liquid droplets are separated from the waste gas stream. The waste gases are then piped through the BPRV (Equilibar GS4) and out of the system. Liquid collected in the second separator is used as the liquid charge for the mixer. A gate valve is located between the HEX reactor and the first separator to provide a second method of controlling the reaction and to isolate the HEX reactor from the BPRV between cycles.

Instrumentation

Instrumentation consists of pressure, temperature, and flow sensors. Five pressure taps are placed along the flow path through the transparent pressure plate to a reactant channel in the HEX reactor, to permit measurement of both absolute and differential pressures along the channel during a reaction. Thermocouples are mounted in line with the pressure taps, but along a different plate. The internal thermocouples are placed to measure the temperature difference across a plate from the load side to the reactant side. Dual-bead thermocouples are mounted in the load side inlet and outlet ports to measure load loop inlet and outlet temperatures, and to provide feedback for the heater power supply. Flow meters are installed in the fluid lines. A turbine flow meter is placed in the load loop (Omega FTB-1440). The reactant loop has three flow meters: a turbine flow meter in the charge pump line, between the charge pump and the mixer (Omega FTB-2001); a positive-displacement gear-type flow meter between the mixer/scavenge tee and the HEX reactor inlet (Omega FPD-2022); and a turbine gas flow meter in the waste gas exhaust line (Omega FTB-931). The amount of AC remaining in the depleted PG is measured using the luminance concentration technique [4].

Communications & Control

Operator safety is the primary consideration for the experimental installation due to toxic gas (ammonia) generation during operation. Therefore, the experiment is intended to be remotely operated. A secondary control system that can shut down the experiment independent of operator or computer input is installed. An AutomationDirect Productivity 3000 programmable automation controller (PAC) is used to monitor the safety sensors installed in the test cell and on the experiment. Should any of these sensors detect an unsafe condition, the experiment is disabled. The PAC provides 16 digital input channels, 32 digital output channels, four analog input, and four analog output channels. For remote communication, an internal network is installed to

provide PC access to various components of the system. An Ethernet hub (E-Linx EIR205) and a USB-IP adapter (Digi AW-USB-5) connected the National Instruments SCXI data acquisition unit, P3000 PAC secondary control unit, feeder and mixer to a control-room computer running LabVIEW. The feeder and mixer drives were connected to the network through serial-to-USB adapters. Through the network, the operator has real-time access to the SCXI, PAC, feeder and dispersion pump drives.

Data acquisition and primary control of devices directly related to the experiment are accomplished through a National Instruments SCXI signal conditioning unit. Data are collected through either an analog voltage module (thermocouples, pressure transducers) or a frequency module (flow meters). Signals for speed control of the pumps and position control of the BPRV and reactor shutoff valves are generated by an analog voltage output module.

Safety & Waste Handling

As described previously, AC decomposition produces ammonia gas. It is required that the experimental installation include some method of sequestering ammonia for later disposal, and the installation is also required to have safety systems to reduce operator exposure to ammonia. For sequestration, an ammonia scrubber (Advanced Air Technologies Apollo 50) is installed in the test cell, and the exhaust from the experiment is ducted to it. Additionally, the scrubber requires dilution air for cooling the ammonia liquor (ammonia absorption in water is exothermic), and this air is also taken from the experiment cart to remove any gases generated by spontaneous decomposition of stored AC. The scrubber is rated at 95% efficiency and is capable of absorbing up to 13 kg NH₃ on a single 208-liter water charge. The pH of the ammonia liquor is tested prior to disposal; if the liquor has a pH above 12 it is reduced with 20% sulfuric acid before disposal.

Ammonia gas level monitoring is performed using a refrigerant leak detection system (Thermal Gas Systems HaloGuard IR). Two alarm thresholds are programmed: 25 ppm, corresponding to the AIHA 1-hour exposure limit, and 300 ppm, corresponding to the OSHA IDLH limit. The alarm circuit is tied to the experiment's PAC so that an ammonia concentration exceeding the first limit disables the experiment and exceeding the second limit triggers a laboratory evacuation signal. The alarm system activates both visual and audio signals (strobe and horn) if a threshold is exceeded. A remote alarm for the HaloGuard system is installed in the control room.

The experiment and scrubber are located in an explosion-proof test cell with an exhaust flow rate of 57 air changes per hour (ACH). The air change rate is such that complete decomposition of the AC stored in the feeder hopper would

result in a maximum concentration of 48 ppm without the scrubber operating. With the scrubber and exhaust operating, the test cell ammonia concentration remains below 5 ppm. To further reduce NH_3 release into the test cell, the experiment is enclosed in a secondary containment, which is evacuated by the scrubber air dilution line. Operator access to the test cell is not controlled, but the test cell does have door interlock switches installed to prevent experiment operation should a door be left open. The experiment secondary containment lid is likewise switched. The test cell is monitored through closed-circuit video.

TEST PROCEDURE

As stated previously, the primary objective of the project is to demonstrate heat absorption using AC decomposition. The experiment is designed to permit the experimenter to control both the heat load and the heat absorption capacity of the system.

The primary input parameters are AC feedrate and heat load. AC feedrate control involves balancing the amount of fresh AC supplied by the feeder with the amount of AC remaining in the depleted PG in the scavenge sump, so that the amount of AC flowing into the HEX reactor is known. The AC concentration and mixture flow rate can be changed to accommodate a desired AC feedrate. The heat load control consists of developing a heat load temperature profile and programming it into the computer. The power supply output is driven according to this profile. A typical test lasts approximately three minutes, but tests as long as ten minutes can be performed.

Flow Visualization Study

To characterize the hydrodynamics of the HEX reactor, a flow visualization study was conducted at room temperature using surrogate materials before the safety and ammonia removal systems were installed. This study involved injecting a sodium bicarbonate/water/PG mixture (comparable to the viscosity expected of PG at 60°C) into the HEX reactor, while simultaneously injecting dilute acetic acid through a separate port. The NaHCO_3 reacted with the CH_3COOH within the HEX reactor. High-speed videos of the reaction at different points along the flow path were recorded. Pressure taps along the flow path permitted recording the reactor pressures. The results were compared to pressure drop predictions.

Mixer Characterization Study

A mixer characterization experiment, consisting of the MagicLAB mixer, the charge pump, and the isolator valve was conducted. Special attention was given to the preparation, handling and dispensing of AC into the mixer. AC flowability is strongly affected by ambient humidity, where high humidity leads to spontaneous powder caking

while quiescent. AC must be positively dispensed, because it will not flow through a tube due to gravity. Therefore the isolator valve incorporates a feed auger to force AC through the valve into the mixer powder chamber. Other characteristics investigated were the mixer/charge pump flow rate ratio, mixer powder inlet vacuum level, post-suspension AC particle size, particle settling, and mixer computer control and feedback. Suspended AC was also decomposed under controlled conditions, and the average evolved gas bubble size and the pressure/temperature relation were investigated.

RESULTS

Flow Visualization Study

The flow visualization study had three primary objectives: measure the pressure drop along the HEX reactor reactant channel while evolving gas; record high-speed video of gas evolution with the intent of observing internal flow patterns and regions of bubble coalescing; and to test the HEX reactor modifications (instrumentation and transparent window). The materials used for the flow visualization study were the surrogate chemicals sodium bicarbonate and acetic acid, and propylene glycol diluted with water to reduce the viscosity to that expected at 60°C. The target for the study was to evolve as much gas as would be expected in a single channel assuming AC decomposition. Different acid/base concentrations were tested to determine their effect on HEX reactor total pressure and channel pressure drop. To evolve the gas, the sodium bicarbonate was dissolved in the PG/water mixture which flowed into the HEX reactor at an average flow rate of 3.5 liters/minute. Acetic acid solution at a near-stoichiometric ratio was injected into the HEX reactor channel once the bicarbonate solution flow had stabilized. The reaction began immediately, and continued as long as acid flowed (typically ten seconds). High-speed video was recorded at three locations along the HEX reactor flow path. The pressure drop measurements indicated a total pressure drop from HEX reactor inlet to outlet of 2.77 kPa at an average bicarbonate solution flow rate of 3.7 liters/minute and acid flow rate of 1 liter/minute. This value was consistent with predictions. The evolving gas volume produced little back pressure, averaging 115 kPa. [Figure 3](#) shows the average pressures at five points along the reactor channel for the 3.7 liter/minute test series. The average differential pressure drop was 1 kPa.

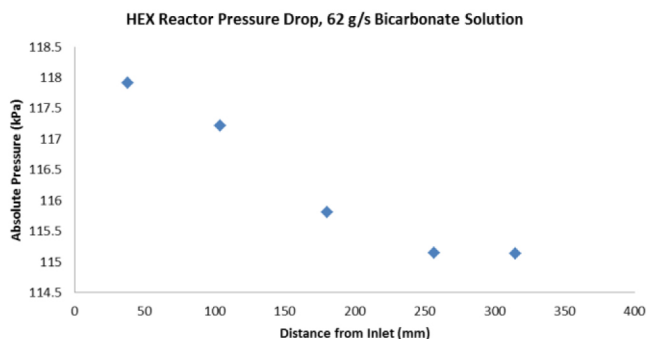


Figure 3. Pressure Drop in HEX Reactor at a Total Fluid Flow Rate of 4.7 Liters/Minute

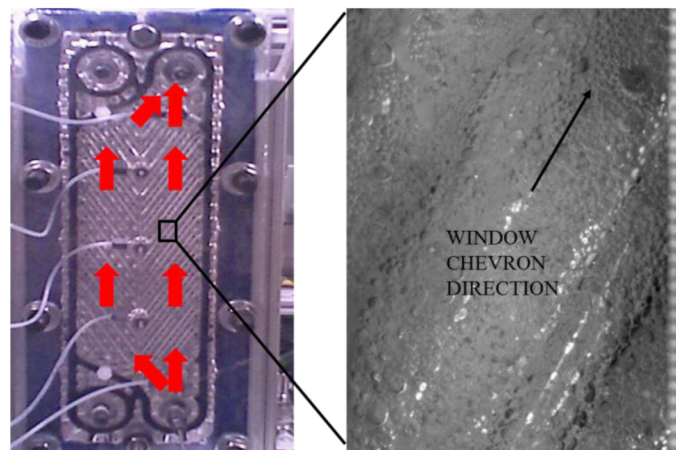


Figure 4. HEX Reactor Transparent Window with Indicated Flow Pattern (Red Arrows), and Screen Shot of Flow Visualization Experiment High-Speed Video Showing Gas Bubble Formation in HEX Reactor. Index Marks along Right Edge of Screen Shot are 1/32"

High-speed video indicated very intense mixing during the reaction. Several flow patterns were evident: a rotational flow pattern in each chevron could be seen, with the material in each chevron rotating and flowing along the chevrons simultaneously. A core flow between the plates and following the chevron pattern made up the bulk (approximately 90%) of the flow. Due to the chevron pattern on the HEX reactor plates, the flow divided between the two halves of the pattern as indicated by the red arrows in [Figure 4](#), and little mixing between halves was observed. The multiple flow patterns along the plates produced regions of mixing between the patterns, with the rotational chevron flows flowing along the chevrons and the core flow flowing directly along the channel. Gas bubbles were continuously flushed from the chevrons, and there was no indication of bubbles adhering to the stainless steel reactor plates (a few bubbles did adhere in the chevrons of the acrylic transparent window, presumably due to imperfections in the casting). Bubbles did not coalesce, bubble size remained constant, and there were no regions where the gas volume forced liquid from the plates. The high-speed video indicated that gas evolution within the HEX reactor should significantly enhance heat transfer due to vigorous mixing, even at low flow rates. Visual observation of the tests showed that the HEX reactor exhibited plug flow, in that the reaction front could be seen moving along the channel without slowing anywhere along the path. [Figure 4](#) is a screen shot of a high-speed video recording of the center of the flow path during a test, about four seconds after acid injection. Bubbles are generally small, less than one millimeter in diameter. The larger bubbles are approximately 1.5 mm diameter. A chevron in the transparent window is indicated; a row of bubbles (dark bands) can be seen on either side of this chevron. The scale along the right side of the figure is graduated in 1/32" (0.8 mm).

Mixer Characterization Study

A study was conducted to investigate the operation and performance characteristics of the IKA Werke MagicLAB dispersion pump (mixer) selected for the experiment. The MHD generator module was chosen for the mixer because it is designed to handle solids and liquids in separate inlet streams [3]. [Figure 5](#) shows the mixer with attached isolator valve mounted on the mixer characterization rig.

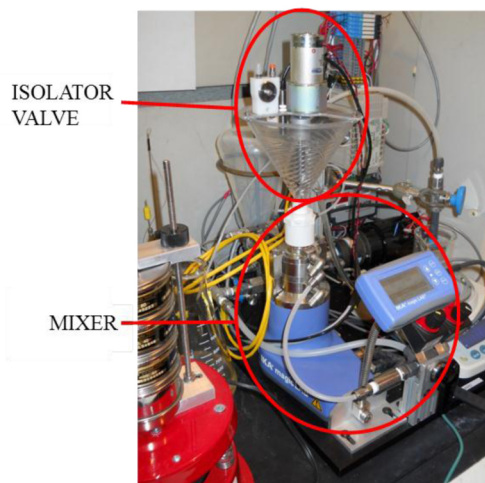


Figure 5. Mixer Characterization Rig, Showing IKA MagicLAB Mixer with Attached Isolator Valve

Several performance characteristics were investigated: the powder inlet pressure, the charge pump flow rate required for a desired mixer output, isolator valve operation and overall component sequencing. In addition to the performance characteristics, AC preparation, handling and dispensing, as well as AC decomposition characteristics were also investigated.

Early tests indicated that AC powder cakes under experimental conditions, with finer particles (smaller than 60 mesh) caking more readily than larger particles. To regulate consistency in dispensing, the isolator valve positively feeds AC powder into the mixer independent of particle size.

Powder suspension tests were conducted with various particle sizes. It was found that larger particle sizes-40- and 60-mesh-were too large to remain suspended in PG for extended periods, settling out within five minutes. Finer particles remained suspended after one week.

Decomposition tests were performed on the suspensions produced, primarily to observe decomposition at ambient pressure and elevated temperatures. It was observed that visible decomposition began at 32°C with approximately ten bubbles per second being formed. Typical bubble size during this period was approximately 1 mm. This rate continued until the suspension reached 57°C, when vigorous decomposition began. The PG in the flask appeared as if it were boiling; bubbles were as large as 3 mm diameter. Bubbles formed at the bottom of the flask because the AC had settled out (the AC in this suspension was 60-mesh). The temperature of the suspension climbed slowly, approximately 1°C/minute, during vigorous decomposition, but as material was depleted, the temperature rose faster, at approximately 4°C/minute.

SUMMARY

The experiment described in this paper is designed to demonstrate a heat exchanger based on the endothermic reaction of ammonium carbamate. The multiple test objectives include developing and demonstrating controlled operation of the HEX along with the investigation of the environmental and handling characteristics of AC. Initial tests performed on the HEX reactor and mixer subsystems indicate that the AC reaction can be effectively used for heat absorption and that the heat transfer within the reactor is enhanced by the turbulence caused by AC decomposition.

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CONTACT INFORMATION

Douglas J. Johnson
douglas.johnson.ctr@wpafb.af.mil
University of Dayton Research Institute
300 College Park Avenue
Dayton, OH 45469

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DEFINITIONS/ABBREVIATIONS

AC - ammonium carbamate
ACH - air changes per hour
AIHA - American Industrial Hygiene Association
IDLH - Immediately dangerous to life and health
OSHA - Occupational Safety and Health Administration
PAC - programmable automation controller
PG - propylene glycol
ppm - parts per million