Aerosol Anatomy - The Aerosol Laboratory

Part I focuses on lab design and construction, safety systems, and basic aerosol charging and testing equipment.

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This series, "Aerosol Anatomy," will dissect and examine various technical topics in aerosol technology, including product development, new technology, components of the aerosol system, and quality control. The material will be presented from a product developers' viewpoint and will begin with "The Aerosol Laboratory." This is the first of a two part discussion of this topic. Part I will explore various aspects of lab design and construction, safety systems, and, basic aerosol charging and testing equipment. In the second installment, "Filling and Safety Procedures," which will appear in a future issue of *Spray Technology & Marketing*, we will cover operational aspects of the lab, as well as safety guidelines. These articles are not meant to be exhaustive treatises on the subject, but rather a practical overview intended to help readers identify potential changes and/or upgrades regarding equipment and safety procedures for their own laboratories. The articles are also intended to provide guidance to anyone who is considering installing laboratory scale aerosol filling capabilities. The aerosol laboratory is a topic near and dear to me–I began my career in aerosol product development nearly 27 years ago, and my first job was as an aerosol pilot lab technician working for the Boyle-Midway Co. While the company no longer exists, its brands live on. Some of the more recognizable are: Wizard Air Fresheners, Black Flag Insecticides, PAM Cooking Spray, Easy Off Oven Cleaner, 3-in-1 Oil, and, Woolite Carpet Cleaners, just to name a few. As you might imagine, given the number of aerosol product lines owned and manufactured by Boyle-Midway, the aerosol pilot lab was a very busy place and was well-equipped for its time. Over the years, there have been many changes and improvements in lab design and equipment technology. We will explore current thinking in aerosol laboratory design, but first, let's lay the groundwork.

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Functions of the aerosol lab

Aerosol laboratories serve a variety of purposes. Some of the primary functions of an aerosol laboratory are:

- *Product Development.* This entails the preparation of a small quantity of samples (<10 sample units at a time) to evaluate new prototypes for development purposes. The cycle of "fill, test, modify, fill, test, etc." continues until a satisfactory result is identified, or until the line of development being pursued is abandoned.

- *Consumer Research Testing.* This is the next step in the development process once an "acceptable" prototype has been identified. The aerosol lab may be asked to provide larger quantities of samples (>50 units) for consumer testing. Examples of this type of testing are:
  - HUT (Home Use Tests);
  - CLT (Central Location Testing)–such as mall intercepts;
  - Focus Group testing;
  - Sensory Panel testing.

- *Product Package Stability Testing.* Samples for this testing may be provided by a production facility (co-packer, or in-house) or, once again, the aerosol lab may be asked to provide samples for testing. Aerosol container corrosion studies and long-term product storage testing are two typical aerosol test programs. Depending upon the formula, product specific testing such as microbiological stability testing may be added to the array of evaluations required for final product approval.

- *Quality Control Support.* Various quantities of filled units may be brought into the aerosol lab from a production line for random or diagnostic testing, typically to audit plant QC operations or to develop and quality new QC test procedures.

Consider your needs: Plan lab capabilities accordingly

Aerosol laboratories span the spectrum from a temporary “simple” set-up, with very basic crimping and charging equipment, typically sharing space within a research or quality lab, to a permanent, fully staffed aerosol formulating lab and pilot lab production facility. In its simplest configuration, the aerosol crimping and filling equipment is temporarily set up to fill a few cans, and then the equipment is dismantled and stored away for future use. In this scenario a person, or two, are selected for training as aerosol technicians and are trained appropriately. The aerosol sample filling duties represent a small portion of the job functions.

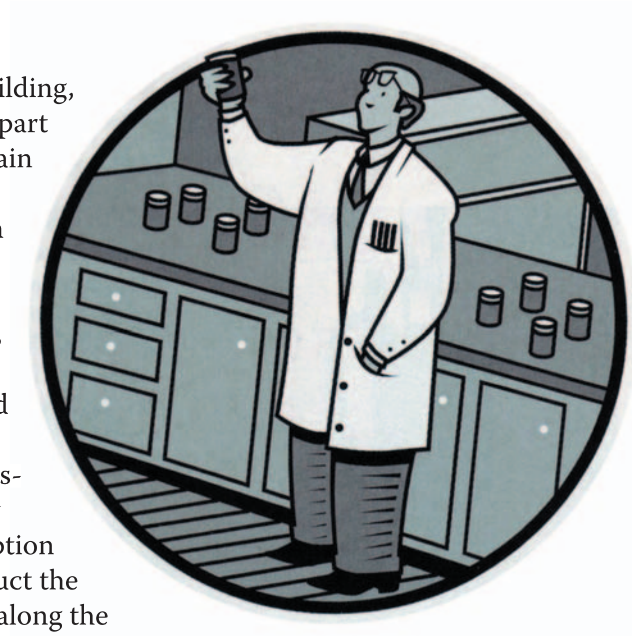
In contrast, the large, permanent aerosol pilot lab represents a laboratory in constant use. At the highest level, the lab services multiple needs, among them; R&D formula development and testing’, a pilot lab to produce small scale (up to 1000 units/day) production runs to supply marketing needs for local, regional and national consumer testing of new aerosol products. The lab may also be utilized by the company’s Quality Assurance Group as a quality control test development lab where QC procedures are designed and refined to suit specific product needs. Typically, this level of capability is used constantly and is found in the largest primary aerosol manufacturers and contract fillers.

Another option, which many manufacturers and suppliers choose, is to convert an existing lab space for part-time aerosol filling. In this scenario, the upgrade cost to the facility minimal. The aerosol filling and gassing is conducted in an explosion-proof fume hood, rather than in a dedicated gas-house (similar to those in production environments). The size and capacity of the lab is dependent upon the specific needs of the individual company.

Location, location, location

Now that we have identified an in-house need for an aerosol lab, let’s start at the beginning. The first step is to identify a suitable location. The first consideration in the placement of an aerosol lab is safety. The nature of aerosol propellant gasses under pressure, which may or may not be flammable, necessitates constructing the lab in a location where the remainder of the facility will be least affected in the event of a problem.

In an ideal world, if safety were the only consideration, the flammable gas filling room might be located in a separate building, physically apart from the main structure. However, in many situations this is impractical, cost-prohibitive, and in my opinion, unnecessary. A very common option is to construct the aerosol lab along the perimeter wall of a building, and ideally, at the corner of a building and away from solvent and propellant bulk storage areas.



The least desirable location is a centralized location within a structure. While a central location may provide access advantages from the remainder of the facility, the disadvantages in terms of decreased facility safety outweigh the advantages of convenient access, especially when filling flammable propellants.

The goal of aerosol lab placement is to minimize the impact to the remainder of the building in the event of an explosion. This is the first layer of safety design considerations. This, along with the other safety related design parameters presented herein, work in concert to produce the high level of employee safety required when working with aerosol products.

A pneumatic vacuum crimper

Building in safety systems

There are many possible layers of safety protection currently available for aerosol filling rooms. The selection and installation of various sub-systems is highly specialized depending upon the specific needs of a particular location and the products to be filled. Furthermore, the

Dual burette pressure filling stations

number of personnel using the lab, the type of work to be conducted, and the estimated number of aerosols produced are all considerations in designing a lab for a specific location.

Dual burette pressure filling stations

There are a number of layers or safety sub-systems available for inclusion in modern aerosol laboratories. The first layer begins with the basic structure of the room. Aerosol labs may be equipped with a "blow-out" wall. Located along the exterior perimeter of the building, this wall is structurally modified to detach in the event of a sudden pressure rise in the aerosol filling room. Typically, this feature consists of one or more panels which are hinged at the bottom. The pressure build-up from an explosion will push the wall outward, pivoting on its hinges. Another construction design feature is the installation of a grounded and/or electrically neutral flooring material. This material does not allow the build-up of a static electric charge.

The second layer of safety systems consists of the mechanical sub-systems. Specialized aerosol room HVAC systems are designed to provide both "normal" and "high-flow" air circulation. The HVAC system is typically linked to a gas detection sub-system. When the gas detection system senses a specified level of flammable gas build-up at a sensor, the HVAC automatically engages the high-speed fans. This is often combined with opening a series of vent panels (or louvers) located near the floor, which vent the gas directly outside. The alarm system is activated simultaneously to alert personnel of a potentially dangerous condition.

The third layer, which some laboratories utilize, is a dedicated explosion suppression system. The system typically consists of a sensitive detector, a high discharge rate suppressant discharge system, and a control module. Explosion sensors have been much improved in recent years. In the past, the systems could be triggered by opening the aerosol room door during a lightning storm or by workers using power tools. False alarms were common and disruptive, not to mention expensive, since the system would need to be recharged and re-armed by the manufacturer after these events. While previous models contained only UV sensors, newer sensors virtually eliminate false alarms by using UV-IR sensors. There have been improvements in the explosion suppressing chemicals used as well. The previous standard for aerosol filling rooms was Halon 1301. Today, water-based systems are gaining popularity.

Explosion anatomy

If there is an undetected / uncontrolled buildup of flammable gas beyond the LEL (lower explosive limit) and there is an ignition source, the UV-IR sensors of the explosion suppression system "see" the fireball forming from microsecond to microsecond.* The control module reacts–sending a signal to explosively discharge the fire suppressing agent. This all occurs before the developing explosion fireball is large enough to cause damage. If, for whatever unlikely reason, the explosion suppression system falls, then the structural "blow-out" wall is deployed, which prevents widespread damage to the facility. On that gloomy note, let me add that, to my

*Note: for additional information on LEL, refer to Monty Johnsen's article–ST&M, June 2005, p. 26.

**Note: this is by no means a thorough dissertation on the subject of aerosol lab safety. I highly recommend exploring the following reference resources: the CSMA Aerosol Guide, and the Aerosol Propellant Handbook by Dr. Jiang Guomin, Dr. Montfort Johnsen and Dr. Bernd V. Braune. I also suggest consulting with aerosol safety specialists to discuss your specific needs and design parameters.

knowledge, an aerosol lab explosion suppression system, as previously described, has never failed to deploy properly in a laboratory environment.**

Aerosol lab equipment

The equipment found in an aerosol lab falls into two broad categories: the equipment needed to aerosolize products and the equipment needed to perform basic aerosol product testing post-aerosolization. The aerosol lab may or may not include the equipment and supplies needed to prepare the product concentrates prior to aerosolization and specific equipment to perform efficacy testing of finished (filled) aerosol samples. This category of equipment is very product specific. For example, if cooking sprays are being developed, then the lab will typically have the capability of blending food grade raw materials. Along with the typical array of beakers, balances and assorted general lab ware, this lab might have a cooking area to test the efficacy of no-stick aerosol pan sprays. This was incorporated into the design of the American Home Foods (now ConAgra) PAM Cooking Spray Laboratory in Milton, Pennsylvania. For our purposes here, we will confine ourselves to the former category–the lab equipment dedicated specifically to the preparation of aerosolized samples and basic aerosol testing equipment. This equipment may be found in most aerosol labs, regardless of the product type being filled.

Once the concentrate is blended and filled into the aerosol container, propellant is added in one of two ways: either "under the cup," where the propellant is injected under the valve cup and then the valve is crimped; or "TTV" ("through the valve"), where the valve is crimped to the can and then the propellant is charged through the valve system. The most common propellant filling setup is the TTV arrangement. For this method, the can is crimped in one of two ways: either with a manual crimper or a pneumatic crimper.

Propellant charging is accomplished by using a pressure burette setup. Liquid propellant is conveyed via a hard or flexible piping to the pressure burette. A series of valves is used to control the flow of the propellant and to vent the burette (externally). A filling adapter (or filling pin) is installed at the base of the burette. This adapter is designed to mate with the valve used on the aerosol can (or glass bottle). Also, an inert gas, typically nitrogen, is used to supply "head" pressure to force the liquid propellant out of the burette and through the valve system of the sample can.

To charge the can, first the aerosol valve is aligned with the filling adapter and the can is forced upward to overcome the spring force of the valve, thereby opening the aerosol valve. Once the valve is open to the burette, the pressure differential (assisted by the head pressure in the burette) forces the liquefied propellant to flow through the filling adapter, through the aerosol valve, and into the can or bottle. Once the calculated volume (measured visually on the graduated burette) of liquid propellant is charged into the can, the operator releases upward pressure on the can, thereby closing the can's aerosol valve and sealing the propellant inside the can.

Non-liquefied propellants, such as C02, N2, N20 and air, may be filled directly into the can, through the valve, without a burette. The pressurization is accomplished using a suitable filling adapter, which is connected to a flexible pressure hose. The hose is connected directly to a suitable pressure regulator. Whereas liquid propellant filling through pressure burettes is done on a volume basis (using the graduations on the wall of the pressure burette), filling of non-liquefied propellants is done on a weight basis. For example, the sample can/bottle is placed on a balance; the balance is tared to zero, and the gas is added incrementally until the target fill weight is achieved. Caution–careful pressure monitoring and a suitable "step-down" tank regulator are necessary to safely fill compressed gasses in this manner.

This discussion on aerosol lab equipment will be continued in the next installment of this article (The Aerosol Lab-Part 2: Filling and Safety Procedures).