Aerosol Anatomy - The Aerosol Laboratory Part 2

Part 2 encompasses lab design and construction, safety systems, as well as basic aerosol charging and testing equipment.

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This article continues the Aerosol Anatomy series, which dissects and examines various technical topics in aerosol technology, including product development, new technology, components of the aerosol system, and quality control of aerosol products.

The series began in April 2006 with Part 1, which covered basic concepts relating to the installation of an in-house aerosol laboratory, including: the functions and advantages of the on-site lab; factors to consider in selecting a suitable location; various safety systems built into the infrastructure; and, the basic equipment required to crimp and charge single aerosol units. Part 2 addresses outfitting the lab with basic and optional equipment, and, also explores routine quality control testing of samples made in the aerosol tab.

The array of possible lab equipment options–and quality testing methodology–can vary widely, depending on your needs, location, and budget.

Basic aerosol lab equipment

Once a suitable location for the lab is determined–and the "infrastructure" is in place, and the type and capacity of aerosol charging equipment has been decided upon (single action manual crimper/filler or an automated higher capacity system)–then we can focus on additional "support" equipment. This equipment is specific to the aerosol lab and is in addition to the array of lab glassware, stir plates, mixers, balances, pH meters, viscometers, etc., which may be found in most typical cosmetic, food or household product development laboratories.

At a minimum, the charging of flammable propellants should occur in an explosion proof (XP) hood. This hood, as well as the surrounding area, should be equipped with propellant leakage monitoring equipment installed and calibrated for the flammable propellants to be used in the lab. A new XP hood unit may be installed in an existing lab space, or, an existing hood may be upgraded to the XP requirement by replacing the electrical components, lights and fan motor with the appropriate XP counterparts. Note, however, that appropriate safety procedures must be followed when filling any aerosol product.

The gas monitoring-detection-alarm system typically consists of a control unit and one or more gas detection heads located in the hood and at floor level around the hood. Flammable hydrocarbon gas is heavier than air, and the system is designed to monitor gas build-up at the floor level. The system includes alarms (both audible and visual), which are activated when a relatively low level of explosive gas is detected.

This first stage activation alerts personnel to shut down the equipment immediately and look for the source of the leak, possibly a loose pressure hose connection, poor can crimps, or a faulty gas valve.

MSA and Bacharach are two suppliers of gas monitoring and alarm systems. System design and pricing are quite variable depending on the options selected and the specific location to be monitored. An on-site visit by the supplier's sales representative can be arranged to ascertain specific needs. (An additional description of the operation of these systems can be found in the previous article in this series.)

Another "typical" fixture in the aerosol lab is a spray booth. Regardless of the type of aerosol

Figure 1: *After 30 seconds of shaking, the formula solids remain in suspension and neither dip tube is visible in these glass aerosol bottles. Each contains a different formula, with a different rate of settling for solid ingredients.*

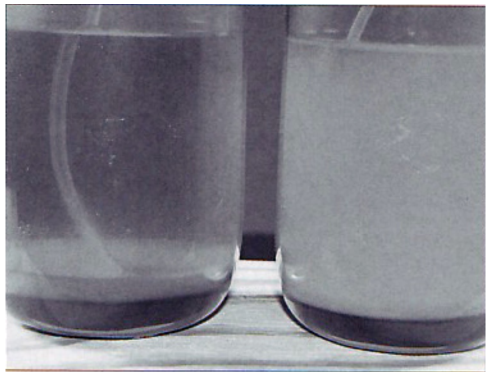


Figure 2: *Over time, the glass bottle aerosols clearly indicate which formula is more stable. The bottle on the left, with the dip tube clearly showing, indicates formula instability, with a loss of suspension and the "hard-pack" on the bottom of the vessel. Preferred is the formula that is slow settling, with formula solids still in suspension (and the dip tube obscured).*

product, all spraying should be done inside a vented enclosure to contain and safely exhaust aerosol fumes. If propellant charging is conducted in an XP hood, and the hood is large enough, this component may be able to function as both a gassing area, as well as a spray booth.

The advantage in this scenario is that the gas detection system and the XP rated components of the hood will service both functions. An additional utility requirement is a cold water feed for clean-up of the spray collection system, which deposits product run-off into accumulation containers for later disposal. Depending on the products being evaluated, several accumulation containers may be required, such as individual receptacles for water-based products, oil-based products, paints, insecticides, etc.

Figure 3.

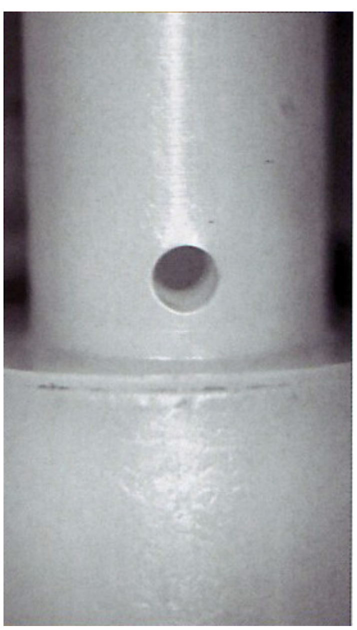


Figure 4.

Using various tools, lab technicians can expose the internal surfaces of the aerosol, identifying problem areas, including container or valve corrosion. Figure 3 is a normal control stem, with the orifice opening clearly visible. Figure 4 is a non-functioning valve, showing a visibly clogged stem orifice.

Glass aerosols

In addition to the two most common aerosol containers–three-piece tinplate and aluminum aerosol containers–glass aerosols can also be filled in the aerosol laboratory. A special crimper is used to crimp 20mm glass bottle valves onto stock plastic coated glass bottles.

Glass aerosols are quite useful for research purposes. They provide the ability to "see into the can," which I have found especially valuable when working with formulas that incorporate solid components that are in powder form. Being able to observe the settling rate of the formula solids–and to see whether they "hard-pack" on the bottom of the vessel over time–can save numerous headaches down the road (see Figures 1 and 2).

Aerosol container evaluations

If "failure analysis" is part of the function of the lab, then the tools required to safely disassemble aerosol cans will also be required. Conventional metal cutting hand tools are used to cut open the aerosol container to expose the internal surface for observation.

In addition, a good quality stereomicroscope will aid in the identification of container and

If failure analysis is part of the function of the lab, then the tools required to safely disassemble aerosol cans will also be required.

valve corrosion, as well as in the determination of root causes of returned (defective) aerosol products from the field.

After dissection of the valve system, the individual components can be examined. The cause of the spray defect can also be determined–possibly a valve stem orifice which is totally clogged, preventing the unit from functioning and resulting in a dissatisfied customer. (See Figures 3, where the orifice opening is clear versus Figure 4, where the orifice has a clogged stem.) Note also that appropriate safety procedures must be followed when de-gassing pressurized aerosol containers.

Laboratory quality assurance testing

At a minimum, there are several routine procedures which may be conducted prior to using aerosol samples prepared under lab conditions. An overview of basic testing of individual units includes:

Valve Crimp Specification: After the sample can is crimped and charged, the valve crimp should be checked to ensure compliance with valve supplier's recommended depth and diameter specifications. If the valve crimp is found to be out of specification, the unit must be quarantined for disposal, and the crimper should be adjusted as necessary before proceeding.

This adjustment cycle is repeated until a valve supplier's approved crimp specification is attained. Any units which are out of the specified crimp range should be considered un-safe and should be immediately de-gassed and disposed of properly. Crimp depth and diameter gauges, and a gauge calibration block are shown in Figures 5, 6 and 7 respectively.

Water Bath Testing: Aerosol samples may be immersed in a hot water bath, of sufficient temperature and for a sufficient time, to ensure the integrity of the package. The water bath is designed to subject the filled aerosol can to a pressure equivalent to the pressure that the contents of the can reach at an equilibrated temperature of 130° F. Any signs of leakage Or can deformation (dome or bottom end buckling) are unacceptable, and the can should be cooled and disposed of immediately. Also, a second function of a water bath is to equilibrate filled aerosol samples to 70° F for spray testing. This is typically how lab filled samples, or production samples, are conditioned for the evaluation of spray geometry, spray rate, and particle size analysis.

A suitable water bath for 70° F conditioning can be a fairly simple arrangement consisting of a sink, or any suitable plastic or stainless steel vessel, a thermostat-controlled water heater, and a temperature probe. Alternatively, the entire water bath set-up with auto- mated temperature controls can be purchased as a package from a vendor.

Pressure Testing: A finished product pressure test gauge may be used to check the can pressure. Typically, the can is allowed to equilibrate to 70° F before taking a pressure reading. Note that the appropriate gauge-to-valve adapter must be used to ensure a secure pressure seal and an accurate pressure reading. These adapters are available from the valve companies or through aerosol equipment supply companies.

Product Specific Testing: Depending on the type of aerosol

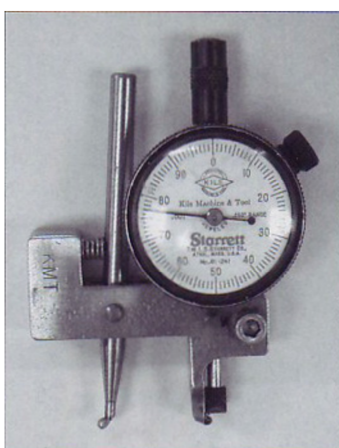
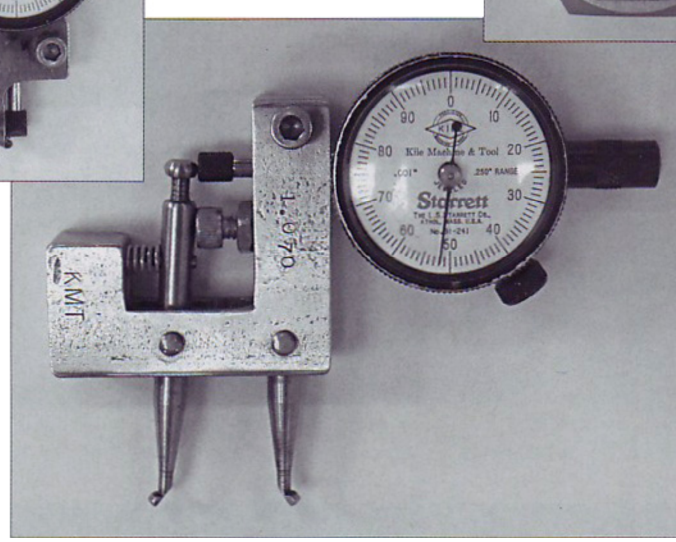


Figure 5.

Figure 7.



Among the tools used in an aerosol laboratory are those for measuring crimp dimension, including (from left to right): a crimp depth gauge; crimp diameter gauge; and a calibration block.

Figure 6.

product filled, there may be additional–product specific–quality control testing conducted to assure conformance to pre-established product performance parameters. Basic aerosol functionality testing may include the following:

- **Spray Rate.** This is the most basic and probably the most often used functionality test for finished product aerosols. The spray rate is determined by using a stop watch and a lab balance. A temperature-conditioned aerosol sample is weighed, sprayed for a period of time, normally 10 seconds; then the can is re-weighed. The difference in weight is determined, and the result is reported in grams/second.

  Out-of-spec readings can point to: problems with the valve system, such as improper orifice sizes, or blockages in the valve, or the wrong propellant, or too much/too little propellant. Also, while less likely, it is possible that an out-of-spec spray rate may be attributed to a mistake in the preparation of the formula concentrate.

- **Pattern Geometry.** Typically, the geometry of the spray pattern is evaluated via a side-by-side comparison of a test unit to a "control" aerosol which is filled on-line or in the lab and verified as acceptable.

  A second mode of evaluating spray geometry is to compare the spray of the test sample to a written set of acceptance specifications. In this scenario, the test can is sprayed onto a flat surface, which is oriented perpendicular to the line of spray.

  Parameters–such as distance to target and the amount of time that the product is sprayed–are defined beforehand. The resultant pattern on the target surface is against pre-defined acceptance criteria for pattern diameter and shape.

- **Sample Evacuation.** This test is referred to as an "extrusion test" or a "label weight conformance test." A test unit is selected it is then agitated and sprayed for a period of time, then allowed to rest for a specified time. This process is repeated until the container is empty. The final weight is recorded and a calculation is performed to determine whether the can expelled the minimum amount of contents required–a value equal to, or slightly exceed, the declared net label weight.

The array of possible lab equipment options-and quality testing methodology vary widely, depending on your needs, location, and budget. While a full dissertation on the topic is beyond the scope of this article, I offer the above as an overview of the basic necessities in terms of aerosol lab equipment and routine lab sample test procedures.

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