

SESSION 17

FILTER TESTING

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Co-Chairmen: W. L. Anderson
J. F. Leonard

NEW PERFORMANCE DATA FOR "EMERY 3002" AND "EMERY 3004," TWO ARMY-APPROVED SAFE MATERIALS TO REPLACE DOP IN MASK AND FILTER TESTING

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NEW PERFORMANCE DATA FOR "EMERY 3002" AND "EMERY 3004," TWO ARMY-APPROVED SAFE MATERIALS TO REPLACE DOP IN MASK AND FILTER TESTING

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Abstract

At the 22nd Conference in Denver, we reported that the U.S. Army Surgeon General (SG) had approved our developmental material "Emery 3004" as a safe replacement for the suspected carcinogen DOP (dioctyl phthalate) in mask and filter testing throughout the Army. Subsequently the SG approved a second, less viscous material, "Emery 3002," for similar applications. We have measured the viscosities and surface tensions of these liquids over a wide range of temperatures, and have initiated liquid breakup studies through Laskin and two-fluid nozzles. New measurements have been carried out with both liquids, e.g. using the ATI, Inc., TDA-4A cold generator to disperse aerosols for which droplet size distributions were measured using the TSI, Inc., Differential Mobility Particle Sizer (DMPS). Among the findings were that Emery 3004 performs very much like DOP in the TDA-4A, with some possible advantages, while Emery 3002 in the TDA-4A produces mean droplet diameters about one-half those of Emery 3004 or DOP. This suggests that Emery 3002 could yield more rigorous filter tests with a smaller consumption of material. New laboratory results will be summarized. Sources of the "Emery" materials will be discussed since the production facility formerly operated by Emery is now run by the Ethyl Corporation and the source products are now known as "Ethylflo 192" (Emery 3002) and "Ethylflo 194" (Emery 3004).

I. Background

The U.S. Army routinely performs 100% quality control testing of filter canisters manufactured for use with field-issue gas masks, and periodic sampling and testing of filters stored in its supply depots. In April, 1986, the U.S. Army's Office of the Surgeon General (OTSG) announced that DOP posed potentially serious health risks to workers, and placed severe restrictions upon testing with it; agencies were also informed that dioctyl sebacate (DOS) would no longer be acceptable as a DOP replacement material, and that similar restrictions would apply for both. These restrictions included occupational exposure monitoring of workers exposed to DOP aerosols and liquid, medical surveillance, issue of personal protective equipment, formal notification to workers of associated risks, and labeling of work areas as "cancer suspect agent areas."

II. Summary of the Successful DOP Replacement Program

The above actions placed severe restrictions upon routine, 100% quality assurance testing of filters and other equipment. For this reason, in 1987 the U.S. Army initiated a detailed study of the problem of finding an acceptable substitute material for DOP that could meet all stan-

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dard military test specifications while itself being a non-carcinogen and, ideally, having other attributes including acceptable acute inhalation toxicity, low cost, ready availability, and the ability to replace DOP directly in machines at test installations without retrofit or other modification of these machines.

The Army had used DOP for many decades in non-destructive servicability testing of respirator canisters and protective filters, and in a variety of aerosol penetration studies including mask leakage and face fit. The program initiated in September 1987 to find a safe replacement material for DOP was sponsored by the Product Assurance Directorate (PAD).^(1,2) A synthetic lubricant named "Emery 3004," from the class of compounds called poly-alpha olefins (PAOs), was approved by the OTSG on 8 January 1992 for use Army-wide as a safe replacement for DOP in "hot smoke" and "cold smoke" testing machines. Emery 3004 was approved after successfully passing three tiers of mutagenicity testing that included the Ames system assay, the sex-linked recessive lethal test in fruit flies, and the rodent bone marrow micronucleus assay performed with rats.

A less-viscous PAO, "Emery 3002," also was also approved by the OTSG on 24 February 1993 for Army-wide use in cold-smoke applications. The Ethyl Corporation⁽³⁾ is the primary manufacturer of these PAOs, under the trade names "Ethylflo 162" (repackaged as Emery 3002), and "Ethylflo 164" (repackaged as Emery 3004). These materials are extremely useful DOP replacements. They perform at least as well as DOP in various testing machines. They can replace DOP directly in existing machines without modification. They are inexpensive, readily available, and should continue to remain so in the future. Ethylflo 162/Emery 3002 and Ethylflo 164/Emery 3004 are readily specifiable, non-corrosive, free of natural impurities, thermally and chemically stable, and safe to work with. They are recommended to replace DOP in Army-wide testing as soon as is practicable.

III. Properties of the Army-Approved Materials

With two safe DOP replacement materials now approved by the OTSG for Army-wide use, a choice is available between them for specific applications. As will be seen, Emery 3004/Ethylflo 164 works very well in both "hot smoke" and "cold smoke" machines, while Emery 3002/Ethylflo 162 is recommended only for cold smokes or machines operating at moderate temperatures. This is because 3004/164 has a flash point of 225°C, well above typical "hot pot" operating temperatures of 170-180°C, while 3002/162 has a flash point of only 164°C. Auto-ignition occurs in either material only at much higher temperatures; nevertheless, we recommend that only 3004/164 be used in "hot smoke" machines.

The PAOs have many unique and interesting properties. They represent a family of synthetic lubricating oils that were unknown a few decades ago. Yet they have replaced natural petroleum products in a great variety of applications, primarily because they work better, they are purer, and they are inexpensive.

Because Ethyl Corp. (and thus Emery) PAOs are produced by combining 10-carbon decene molecules, the resulting products do not contain distributions of sequential carbon chain lengths, like those found in common petroleum products, which are difficult to separate by distillation. Instead, each PAO consists of chain lengths in multiples of 10 which, in principle, should allow easier distillation to obtain samples of a single kind of molecule of high purity. Approximate analyses for the PAOs of interest here are

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listed in Table 1. The last digit of the "3000" or "160" numbering scheme indicates the viscosity in centistokes of that particular mixture at 100°C.

Table 1. Approximate Analyses by Carbon Chain Length for Emery 3002/Ethylflo 162 and Emery 3004/Ethylflo 164.

Ethyl/ Emery Product	Percentages by Carbon Chain Length					
	<u>C20</u>	<u>C30</u>	<u>C40</u>	<u>C50</u>	<u>C60</u>	<u>C70</u>
162/3002	97-99	1.0				
164/3004	0.60	82.1	16.0	1.0	2.0	

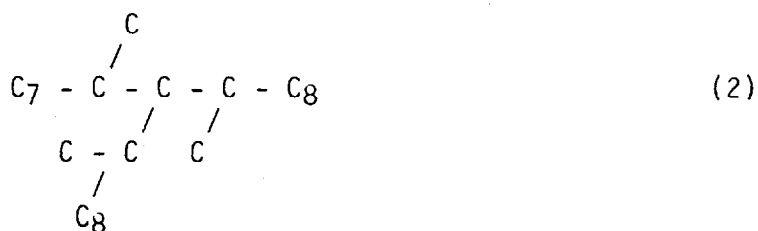
Table 1 shows that Ethylflo 164/Emery 3004 is a mixture of C₃₀ and C₄₀ polyolefins with molecular weights (MW) of 420 and 560, respectively, and traces of other carbon chain lengths. But the Ethylflo 162/Emery 3002 is nearly all "dimer," i.e., 20-carbon chains each formed from the combination of two decene-1 (10 carbon) molecules.

The molecules comprising the PAOs become progressively non-linear with increasing size. Thus, for larger sizes, the molecules increasingly become isomers of one another. The manufacturing process begins with decene-1, a linear hydrocarbon molecule of 10 carbons with one double bond at the first position. The decene-1 is polymerized in the presence of a BF₃ catalyst, hydrogenated to saturate any remaining double bonds, and distilled into fractions (dimer C₂₀, trimer C₃₀, quatramer C₄₀, ...). These are blended to produce desired product mixtures for various applications, as indicated in Table 1. There are many other PAOs of higher viscosities, which are not shown.

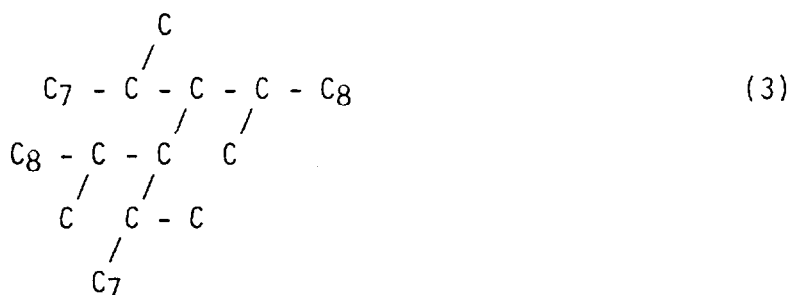
Chemists generally agree that the C₂₀ dimer is nearly straight-chained but, because polymerization occurs at the site of the decene-1 double bond, a "bending" occurs such that the dimer molecule has the form:



Structural theories of the PAOs suggest that as the molecules become larger chain attachment occurs at selected sites such that the C₃₀ trimer has the form:



while the C₄₀ quatramer has a form resembling:



Materials other than decene-1 can be used to make PAOs, but decene-1 gives products with the most desirable range of viscosities for lubricants. To prevent discoloration due to oxidation catalyzed by contact with metals during use, antioxidants can be added to PAOs in concentrations well below one percent.

Some physical properties of the poly-alpha olefins (PAOs) discussed here are given in Table 2.

Table 2. Properties of Poly-Alpha Olefins (PAOs).

Ethyl/ Emery Product	Pour Point, °C	Flash Point, °C	Fire Point, °C	Auto-Ignition Point, °C	Specific Gravity
162/3002	-65	164	178	324	0.80
164/3004	-69	225	250	343	0.82

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The viscosities, in centistokes (cSt), of Ethylflo 162/Emery 3002 and Ethylflo 164/Emery 3004 are plotted versus Centigrade temperature in Figure 1. These data were obtained from manufacturer's specification sheets. At 100°C, the viscosity of 162/3002 is 1.80 cSt, approximating the value "2" indicated by the last digits of the product numbers. For 164/3004 at 100°C, the viscosity is 3.90 cSt, approximating "4" in the product numbers.

Table 3 contains manufacturer's⁽³⁾ viscosity data for the Ethylflo/Emery PAOs, as well as values measured at ERDEC over a limited temperature range for dioctyl phthalate (DOP).

Table 3. Viscosities of PAOs and DOP.

Temperature, °C	Viscosity, Centistokes (cSt)		
	Ethylflo 162/Emery 3002	Ethylflo 164/Emery 3004	DOP
-40	310.0	2460.0	
-18	62.0	341.0	
15			109.80
25			58.36
35			33.90
40	5.54	16.8	
50			17.55
100	1.80	3.90	

Viscosity is an important parameter when liquids are sprayed through two-fluid atomizers to produce fine aerosols such as those used in a variety of laboratory research applications. As temperature is reduced, the ratio of viscosities of 164/162 or 3004/3002 increases. At 100°C this ratio is $u_4/u_2 = 3.90/1.80 = 2.17$. At 40°C it is $16.8/5.54 = 3.03$, and at -18°C the ratio is $341/62.0 = 5.50$.

Thus, at lower temperatures, the viscosity of Ethylflo 162/Emery 3002 increases more slowly than that of Ethylflo 164/Emery 3004. If room temperature is taken as 20°C, the viscosity ratio (Figure 1) becomes approximately $35/10 = 3.5$. When "cold smokes" are generated by spraying at room temperature, these numbers indicate that 162/3002, being much less viscous than 164/3004, should "break up" more readily to form the smaller droplets of the two liquids. Laskin nozzles, which use pressurized gas injected through jets beneath the surface of the liquid being aerosolized, represent a more complex case.

Another important parameter in liquid breakup through nozzles is surface tension (ST). Values of ST over a limited temperature range were measured for the PAOs and for DOP at ERDEC, with the results shown in Table 4.

The data presented in this report for hot and cold smokes produced from Ethylflo 164/Emery 3004, and for cold smokes (only) produced from Ethylflo 162/Emery 3002 are empirical (experimental). Limited progress has been made in the accurate prediction of droplet size distributions from spray nozzles. A well-

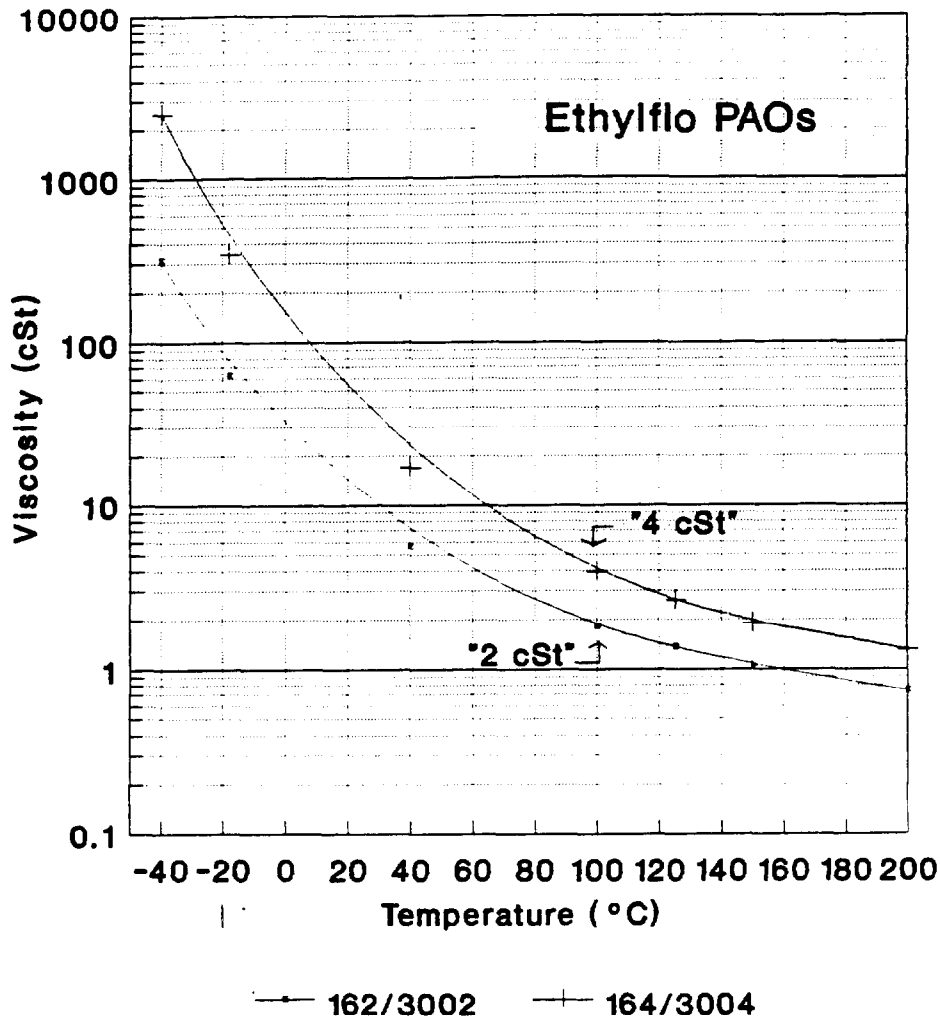


Figure 1. Viscosity (cSt) of PAOs Versus Temperature (°C).

Table 4. Surface Tensions of PAOs and DOP.

Temperature, °C	Surface Tension (dynes/cm)		
	Ethylflo 162/Emery 3002	Ethylflo 164/Emery 3004	DOP
15	28.8	29.9	32.1
25	28.0	29.0	31.3
35	27.1	28.2	30.5
50	26.0	27.0	29.2

known equation for liquid breakup was published in 1939 by Nukiyama and Tanasawa⁽⁴⁾, but this was deficient in that it contains no factors relating to nozzle design. Also, except for sonic flow, there is no dependence upon the physical properties of the gas which is used to disperse the liquid. These factors were pointed out by Gretzinger and Marshall⁽⁵⁾, who proved their importance.

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An empirical equation which answers some of the objections of the Nukiyama-Tanasawa expression, and which appears to fit experimental data more closely, has been given by Kim⁽⁶⁾ for convergent type atomizers. The mixture of English and metric units is Kim's:

$$\bar{x}_m = \frac{249}{A^{0.365} v_{rel}^{1.144}} \left(\frac{\sigma_1}{\rho_a} \right)^{0.572} \left(\frac{\mu_1^2}{\sigma_1 \rho_1} \right)^{0.161} + \frac{1260}{v_{rel}^{0.54}} \left(\frac{\mu_1^2}{\sigma_1 \rho_1} \right)^{0.17} \left(\frac{M_1}{M_a} \right)^m \quad (4)$$

where

\bar{x}_m = mass mean diameter (microns)

μ_1 = liquid viscosity (centipoises)

ρ_1 = liquid density (lb/ft³)

ρ_a = gas density (lb/ft³)

σ_1 = liquid surface tension (dynes/cm)

v_{rel} = difference in gas and liquid velocity (ft/sec)

A = flow area for the gas (in.²)

M_a and M_1 = mass flow rates for gas and liquid respectively

$m = 1$ for $M_1/M_a > 1/3$ and $m = 0.5$ for $M_1/M_a < 1/3$

At large gas-to-liquid flow rates, a limiting small particle size is achieved and the value for this particle size is given by the first term on the right side of the equation. At small gas-to-liquid flow rates, particle size will be appreciably larger and controlled primarily by the second term on the right of the equation. Significantly, factors are present which include the design of the nozzle, namely, its area, as well as the density of the gas stream.

It can be seen in Equation 4 that liquid viscosity and surface tension are both well represented. Droplet diameter increases as the 0.572 power of surface tension. Offsetting this are the terms in parentheses taken to the small powers 0.161-0.17, where the square of viscosity is divided by liquid surface tension and density. Thus at face value, the equation appears to support the view that increasing either viscosity or surface tension, or both, will tend to give larger droplet size distributions from sprayers.

IV. Typical Performance Data

There are two general types of smoke generators: those for "hot" smokes used in mask and filter testing, and those for "cold" smokes used in filter testing as well as for tests such as mask face fit or leakage other than through the filter canister.

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Often a hot smoke is generated by vaporization/recondensation of a liquid, particularly when a "monodispersed" aerosol is required by test directives. Monodispersity implies a very narrow droplet size distribution. For example, U.S. Army mask canisters must be challenged by smokes of 0.2-0.3 μm mean droplet diameter, with a geometric standard deviation equal to or less than 1.3.

There are other designs for hot smoke generators. For example, the Los Alamos National Laboratory (LANL) has built a prototype system⁽¹⁾ called "LAMAPP," an acronym for Los Alamos Monodispersed Aerosol Prototype Penetrometer. This system uses a cold pot to hold the test liquid, which is finely aerosolized by a Laskin nozzle. The aerosol then passes through a heated column and into a mixing chamber. The droplets are vaporized, and recondensation then occurs on very tiny salt (NaCl) nuclei that are much smaller than the resulting, monodispersed, smoke droplets. In our tests, Ethylflo 162/Emery 3002 was found to be the best-performing of all liquids in the LAMAPP system. But in the standard TDA-100 hot pot, hot smoke machine, the best performer was Ethylflo 164/Emery 3004. Due to the vaporization and recondensation temperatures, 162/3002 works poorly in the TDA-100.

Both materials, 162/3002 and 164/3004, can be used in cold smoke applications, and both have attained full approval for use by the U.S. Army's Office of the Surgeon General (OTSG). Thus the choice of one or the other for a given application will include consideration of factors such as operating temperature, size distributions and concentrations obtained from various nozzles, and parameters including flow rates, pressures, and liquid/gas mass flow ratios.

The one restriction has been noted earlier and can be rephrased here:
Ethylflo 162/Emery 3002 should not be used in hot pot machines (above 100°C).

V. Hot Smoke (Vaporization/Recondensation) Applications

Until the recent approval from the OTSG of Ethylflo 162/Emery 3002 for cold smoke testing, the authors had concentrated their efforts upon Ethylflo 164/Emery 3004, which had OTSG approval a year earlier, for both hot and cold smoke applications. Thus, many existing data were taken using 164/3004 to generate cold smokes. 164/3004 was intended primarily for hot smoke generation against stringent military specifications.

Great success in both hot and cold smoke testing with Emery 3004/Ethylflo 164 has been achieved at the Hanford (WA) Nuclear Site, which is now operated by the Westinghouse Hanford Company (WHC) for the U.S. Department of Energy (DOE). WHC scientists reported their own test data, which confirmed ERDEC's conclusions that 3004/164 performs at least as well as DOP, and somewhat better than DOS (dioctyl sebacate, another replacement material), in a variety of machines.

WHC had been using DOS in their Q-76, Q-107, and Q-127 (TDA-100) testers because the use of DOP had been curtailed due to its possible carcinogenicity. DOS was causing gumming and other fouling problems not unlike those encountered with corn oil in other machines. DOE was dealing with the problem of deciding whether to convert to a suitable DOP substitute such as one of

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the authors' materials, or to replace their machines with new units that incorporate completely different "cold smoke" technology. These would generate smokes with much poorer "monodispersity," i.e., wider particle size distributions, than currently are achievable using hot smoke technology.

WHC reported that "Emery 3004 has several advantages over approved performance testing chemicals, including that it is not considered a carcinogen or suspect carcinogen; therefore, respiratory protection is not required during testing." 'Additionally, Emery 3004 does not cause buildup on or plugging of the test equipment like DOP or DOS.' 'By reducing the maintenance required on equipment, use of Emery 3004 increases the efficiency of.... operations." Hazardous waste is greatly reduced using 3004/164 with nuclear dust filters. If DOP is used, the waste is classed as both nuclear and carcinogen contaminated; disposal is much more complex.

As a result of this work, DOE Headquarters has notified WHC that they have approved Emery 3004/Ethylflo 164 as a challenge aerosol for the in-place testing of high-efficiency particulate air filter systems, effective September 1992. This should lead swiftly to approval for similar use throughout DOE.

These results and many additional data taken by independent investigators confirm that Ethylflo 164/Emery 3004 performs extremely well in hot smoke applications^{1,2}. It possesses virtually every attribute that could be desired in a direct replacement material for DOP and DOS. It is also very effective in cold smoke applications, as will be discussed below.

Ethylflo 162/Emery 3002 outperforms 164/3004 only in the "hybrid" of hot smoke applications, mentioned earlier: the LAMAPP machine. In the LAMAPP, because the liquid is first aerosolized using Laskin nozzles in a pot at room temperature, the 162/3002 gives a fine spray of liquid droplets which are then evaporated to allow recondensation upon tiny salt nuclei.

VI. Cold Smoke (Room Temperature) Applications

3004/164 has been more extensively tested in cold smoke applications than has 3002/162, since the former was approved by the U.S. Army's OTSG a year earlier than the latter.

The TSI, Inc., Model 8110 Automated Filter Tester (AFT) is a cold smoke machine that generates an aerosol which is used to challenge a test filter; filter penetration is calculated by a microprocessor. Once a filter or filter canister is installed in the holder, two buttons are pushed and the test runs automatically with results displayed at the conclusion. The AFT has a "low" and a "high" aerosol concentration mode, enabling challenge aerosols to be generated covering the range 15-100 mg/m³.

In other tests, the TSI Model 3932 Differential Mobility Particle Sizing System (DMPS), which prints a bar chart of particle size distribution versus mobility channel, was utilized. Emery 3004/Ethylflo 164 was found to give an aerosol mass concentration from the Model 8110 that was greater than the DOP yield by 52 percent in the "high" mode and 60 percent in the "low" mode.

Other tests were conducted in the Edgewood Area of Aberdeen Proving Ground using a new prototype machine developed by the Los Alamos National

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Laboratory (LANL) which is known as the High Flow Alternative Test System, ATS. Aerosol is generated in a cold pot through a selectable number of Laskin nozzles (to control mass concentration), and is analyzed before (with dilution) and after filter penetration using a Laser Aerosol Spectrometer (LAS). Tests were run with Emersol 875 (isostearic acid, another candidate DOP replacement material), and with Emery 3004/Ethylflo 164 at 1500 CFM air flow rate under various conditions.

The ATS operates on a new principle, in that a broad array of particle (droplet) sizes is generated, but only those size ranges that are of interest need to be monitored to determine filter penetration. The system does, however, print out the full range of penetrations in each size interval or "bin."

The final cold smoke generator that was utilized and will be discussed here is the ATI, Inc., Model TDA-4A. This generator features a total of eight Laskin nozzles, which are selectable by opening or closing a pair of valves so that, e.g., one, three, six, or eight nozzles can be used. The liquid to be aerosolized is placed in the bottom of the housing, submerging the nozzle outlets. Compressed air creates the liquid aerosol by shearing the liquid.

Most cold smoke generators use some configuration of Laskin nozzles, which produce rather wide particle size distributions. To limit the upper size range, thus conserving most of the liquid which is sprayed in droplets too large to be useful for test purposes, some sort of crude pre-sizing is used. All of the generators discussed in this section use a system of pads, filters, impactors, or baffles to limit mean particle sizes of their output aerosols to values typically near 0.7 μm .

These resulting size distributions are still much broader than those from hot smoke, "monodispersed" aerosol machines, which produce aerosols having geometric standard deviations (GSDs) of 1.30 or less; these GSDs can be smaller than 1.20 in well-tuned machines. By contrast, cold smoke machines typically produce aerosols with GSDs of 1.60 - 1.90, or even larger values.

Experiments were conducted in our laboratory, using the Model TDA-4A cold smoke aerosol generator, to allow direct comparisons of the performances of Emery 3002/Ethylflo 162, Emery 3004/Ethylflo 164, and DOP with each other in true cold smoke applications. We have shown⁽²⁾ that 3004/164 is superior in hot smoke, monodispersed machines, and that 3002/162 works best in the hybrid (cold pot, hot smoke) LAMAPP machine.

The TDA-4A output was connected through a pair of aerosol diluters to a TSI, Inc., Differential Mobility Particle Sizer (DMPS) with a Condensation Nucleus Counter (CNC). Air pressure was 20 psi. The liquids tested were DOP, Ethylflo 162/Emery 3002, and Ethylflo 164/Emery 3004. One Laskin nozzle was operated to limit TDA-4A output and conserve liquids. The air flow from one nozzle at 20 psi was 0.391 liters per minute. Mass concentrations and other data for the three liquids are shown in Table 5.

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Table 5. Data for Three Liquids in the Model TDA-4A Aerosol Generator, Operating with One Nozzle at 20 psi Pressurization.

Liquid	Mass Concentration,	Geometric Mean Droplet Diameter, μm		
	g/m^3	Count	Surface	Volume
Ethylflo 162/Emery 3002	5.03	0.202	0.287	0.333
Ethylflo 164/Emery 3004	5.61	0.374	0.482	0.532
Diocetyl Phthalate (DOP)	4.79	0.384	0.503	0.548

Before looking at the droplet size distributions for aerosols of these three liquids, some interesting observations can be made concerning the data in Table 5. First, note that the mass concentration yield for DOP is the smallest

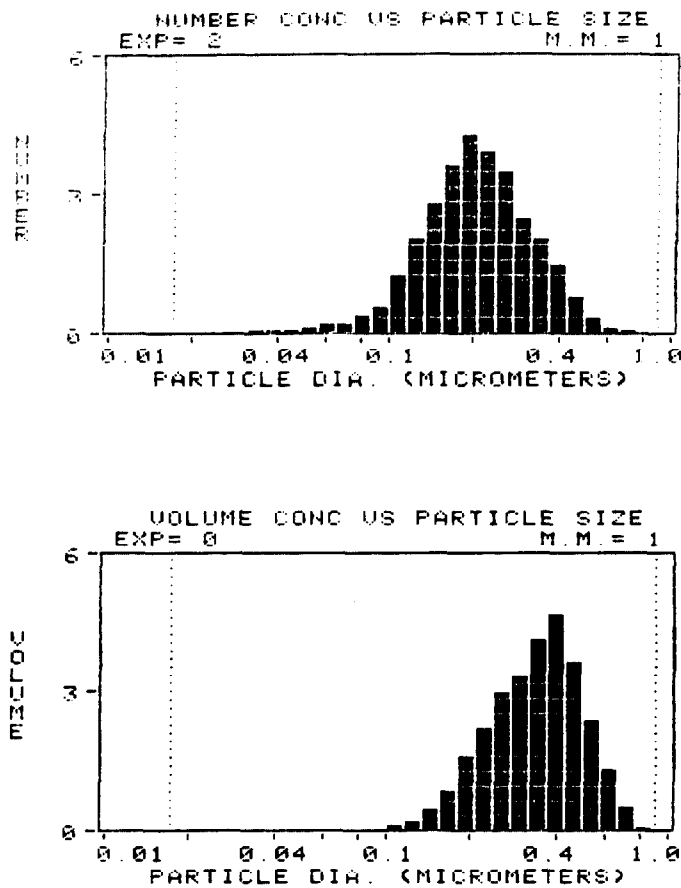


Figure 2. DMPS Particle Size Distributions for Ethylflo 162/Emery 3002.

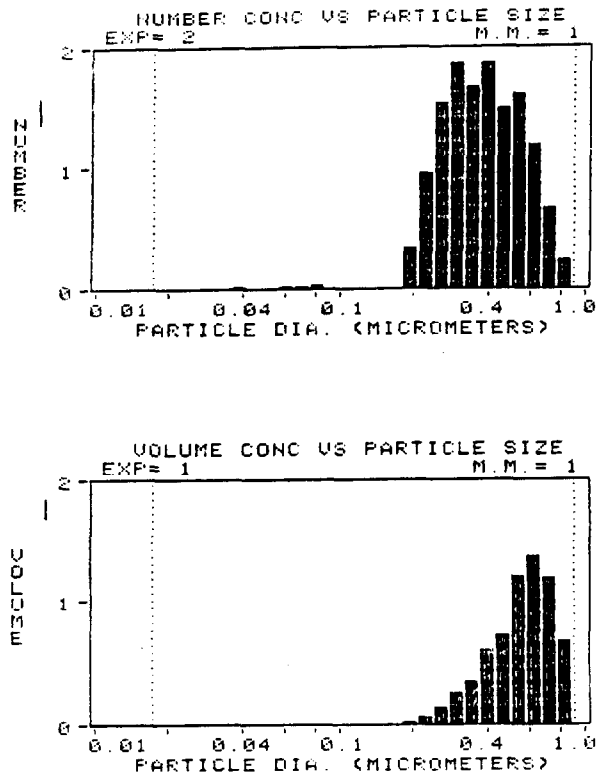


Figure 3. DMS Particle Size Distributions for Ethylflo 164/Emery 3004.

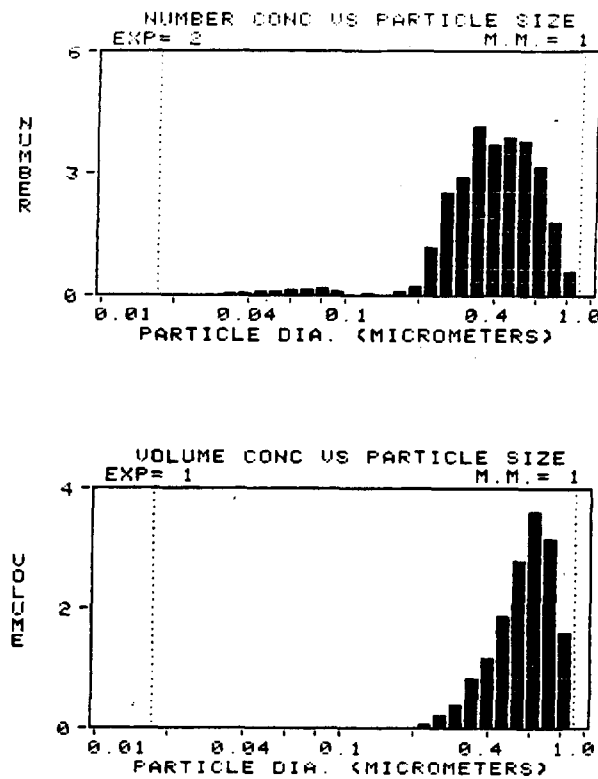


Figure 4. DMS Particle Size Distributions for Dioctyl Phthalate (DOP).

of the three liquids. It is about 15 percent smaller than that of 164/3004. Second, it is seen that the mean droplet diameters of DOP and 164/3004 are quite similar, with the DOP droplets being the slightly larger ones. But the 162/3002 droplets are by far the smallest of any, and lie in a size range that is nearly optimum for filter penetration. Thus Ethylflo 162/Emery 3002 seems capable of producing much higher counts of more penetrating particles with similar mass flow rates or concentrations to those of Ethylflo 164/Emery 3004 and/or DOP.

Droplet size distributions and other data for aerosols of these three liquids in the TDA-4A are shown in Figures 2-4. Number or count distributions (top) and volume or mass distributions (bottom) are shown in each figure. In general, the number distributions are quite symmetrical, while the volume distributions are skewed toward the right. In Figure 2, both the number and volume peaks are seen to lie far to the left of those in Figures 3 and 4 for 164/3004 and DOP, respectively.

VII. Conclusions

The information and discussions presented in this technical report lead to the following conclusions:

- There are now two liquids approved by the U.S. Army's Office of the Surgeon General (OTSG) for use in mask and filter testing and in general aerosol testing to replace the suspected carcinogen DOP; these are Emery 3004/Ethylflo 164 (approved by OTSG on 8 January 1992), and Emery 3002/Ethylflo 162 (approved by OTSG on 24 February 1993);
- Emery 3002/Ethylflo 162 should be used only in cold pot machines since its flash point of 164 °C is close to that of temperatures used in "hot pot" machines;
- The physical and chemical properties of both liquids are well known (Tables 1,4, Figure 1);
- The molecular structures of the two liquids are completely different (Table 1); Emery 3002/Ethylflo 162 is almost pure "dimer," its molecules contain 20 carbon atoms; Emery 3004/Ethylflo 162 is a mixture of mostly "trimer" (30 carbons) and some "tetramer" (40 carbons);
- Liquid breakup behavior by submerged Laskin nozzles cannot be expected to be the same as that for two-fluid pneumatic nozzles in gas or air; Equation 4 can be validated only by experiment;
- Emery 3004/Ethylflo 164 has been found to be highly useful by the Westinghouse Hanford Company (WHC), in many kinds of testing and in cost savings realized.

VIII. Recommendations

We recommend that:

- Emery 3004/Ethylflo 164 be used in all hot-pot, hot smoke machines that require replacements for DOP or other materials to meet safety criteria;

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- Emery 3002/Ethylflo 162 be used in hybrid cold-pot, hot smoke machines such as the LANL LAMAPP (see text) to meet safety criteria;
- Emery 3004/Ethylflo 164 or Emery 3002/Ethylflo 162 be used in cold-pot, cold smoke machines such as the TSI Model 8110, the LANL HFATS, or the ATI TDA-4A, to meet safety criteria;
- Further studies are needed of the performance of both liquids in a variety of Laskin nozzle and two-fluid pneumatic nozzle systems;
- A mathematical modeling capability needs to be validated to predict droplet size distributions expected from a given nozzle under given operating parameters (e.g., Equation 4). this will require in-house measurements of surface tension versus temperature for Emery 3002/Ethylflo 162 and Emery 3004/Ethylflo 164.

References

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DISCUSSION

FRANKLIN: In filter testing we know that something like 0.1 μm is much more challenging to a HEPA filter than 0.3 μm . I am wondering if that size would not be preferred as a challenge. I do not know what size particles you are really trying to collect at DOE, whether it is more toward the smaller size particles.

BERGMAN: To answer the question about whether or not a smaller particle size would be better, all of the tests reported here were conducted with cold DOP, which is typically about 0.7-0.8 μm median diameter. None of the tests are valid for filter efficiency certification. These aerosols are used for leak testing. What you are asking is, how do Emery oils perform as substitutes for the hot DOP aerosol. I have no data to be able to say anything about that. My impression is that this report should be viewed as preliminary since there are no filter test data to answer that question.

COMPARISON OF EMERY 3004 AND 3006 CHARACTERISTICS WITH
DOP FOR POSSIBLE USE IN HEPA FILTER LEAK TESTS

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Abstract

The particle size distribution, concentration, liquid to aerosol conversion rate and ignition properties of DOP, Emery 3004 and Emery 3006 aerosols generated by the NUCON Aerosol Generators Models SN-10 and DG-F were obtained. Results demonstrate the Emery products are acceptable replacements for DOP in performing leak testing of HEPA filters.

I. Introduction

After DOP (di-octyl-phthalate) was identified by the National Cancer Institute's National Toxicology Program as a carcinogen, as stated in the MSDS⁽³⁾ various groups initiated a search for an acceptable aerosol substitute^(1,2). Two of the frequently suggested substitutes are polyalphaolefins, or (PAO); trade-names "Emery 3004"⁽⁴⁾ and "Emery 3006"⁽⁵⁾. Emery 3004 [polyalphaolefin 4 centiStokes (cSt) viscosity (PAO 4 cSt)] and Emery 3006 (PAO 6 cSt) are non mutagenic, inexpensive and thermally stable liquids. The US. Army's Office of the Surgeon General (OTSG) has approved Emery 3004 for aerosol leak testing of filters and respirators. According to Carlon and Guelta⁽²⁾, the performance characteristic of the PAOs, as aerosol challenge agents are similar to, or better than DOP. This suggests the possibility of these compounds being used with the NUCON F-1000 series aerosol instruments.

Various NUCON testing procedures were followed using DOP, Emery 3004, and Emery 3006 respectively as aerosol challenge agents. Testing included particle size distribution measurements, aerosol concentration measurements, ignition properties and liquid-to-aerosol conversion rates.

II. Description of Materials and Equipment

DOP was obtained from the Ashland Chemical Company; Emery 3004 and Emery 3006 were obtained from the Henkel Corporation. Aerosols were generated with standard production NUCON Aerosol Generators models SN-10⁽⁶⁾ and DG-F⁽⁷⁾. The SN-10 is an air operated "pneumatic" aerosol generator, and the DG-F is a large capacity electrically heated "thermal" aerosol generator. For particle size and distribution analysis a CLIMET

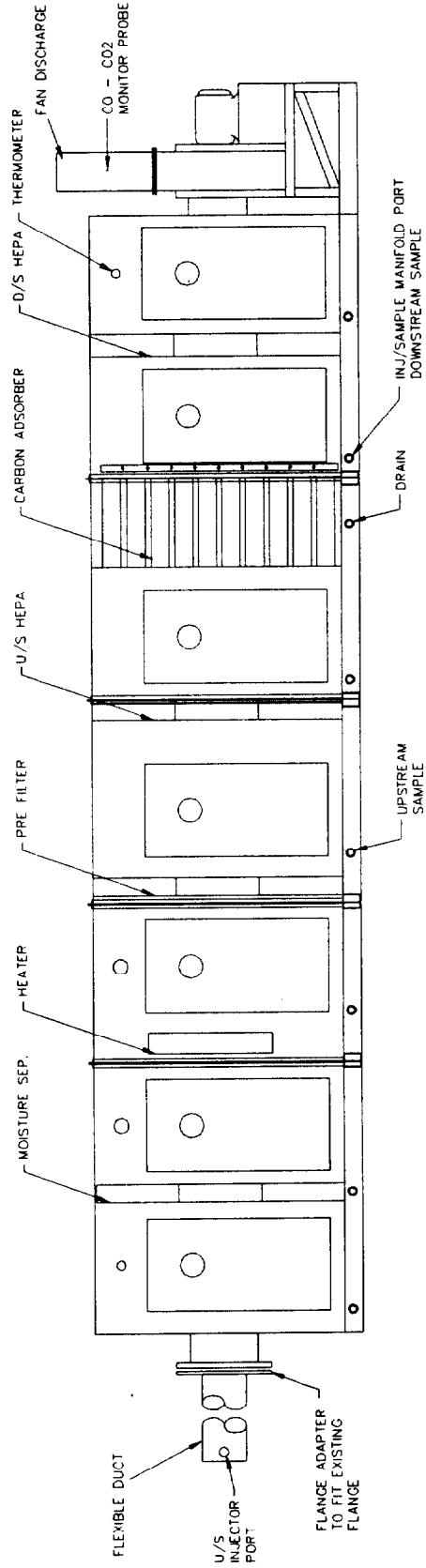


Figure 1. NUCON Air Cleaning Unit (ACU)

model CI-225 detector with a CI-8040 analyzer was used. Tests were performed on a typical air cleaning unit (ACU) consisting of a 6 filter HEPA bank (Figure 1) operating at 500 SCFM (2.36 m³/s). Output capacity of the aerosols generated by the SN-10 were measured using a NUCON F-1000-DD-F Aerosol Detector⁽⁸⁾.

III. Procedure And Methods

Three identical Model SN-10 Aerosol Generators were filled with DOP, Emery 3004, and Emery 3006 respectively. The generated aerosol was injected into the inlet of the ACU (Figure 1). To provide a diluted sample and prevent the saturation of the CLIMET detection equipment, a bypass around the HEPA filter bank was created using a 1/4 in. (6.35 mm) diameter hole. The bypassed mixture of air and aerosol was then analyzed for particle size distribution and particle count by the CLIMET detection equipment. The test was first performed using DOP as the challenge agent, then repeated using the Emery products. The operating air pressure of the SN-10 generators was maintained at 20 psi for all three tests.

The performance of the thermal aerosol generator (DG-F) was tested using the same ACU arrangement. A consistent aerosol output was maintained by adjusting the generator's built in flow meter to the same value for the three tests. Only one generator was used for all three challenge agents to assure like comparisons. The generator was thoroughly cleaned after each test series.

The aerosol output capacity of NUCON aerosol generator SN-10 was measured according to NUCON procedure 12-67⁽⁹⁾ using a NUCON aerosol detector, model DD-F.

Ignition tests were performed only on the thermally generated aerosol produced by the NUCON F-1000-DG generator. With compressed air as the carrier gas, and operating according to the instrument manual, the generated aerosol stream was exposed to an open propane flame placed approximately twelve inches from the generator's aerosol outlet. A spark ignition test was done using a welders' electronic igniter (such as used to ignite oxyacetylene torches). All ignition tests were repeated using bottled nitrogen as the carrier gas.

Liquid to aerosol conversion was performed by using the weight loss method. Each liquid was put into an SN-10 aerosol generator operated for two hours. The beginning weight and the ending weight were compared, and the liquid to aerosol conversion rate was calculated in grams/minute units.

IV. Results

Particle size distribution

The CLIMET aerosol detection/measurement equipment were arranged to sample the air aerosol stream bypassing the first HEPA bank. Before beginning injection, background count readings were obtained. A total of 5 sample counts were made and averaged which then established the average background count.

The CLIMET was operated so the six channels were analyzed simultaneously. The data presented in Table 1 represents the total average count, minus the average background count, per cubic foot of air. A total of 5 samples were taken and averaged.

Table 1 : Particle Size Distribution

Channel number	Particle size micrometer	SN-10						DG-F					
		DOP	%	Emery 3004	%	Emery 3006	%	DOP	%	Emery 3004	%	Emery 3006	%
	Total	131875	100	244419	100	250392	100	138539	100	144071	100	284215	100
1	0.3-0.4	93710	71.1	174600	71.4	172400	68.9	136700	98.7	140800	97.7	273000	96.1
2	0.4-0.5	26340	20.0	47910	19.6	51620	20.6	1765	1.3	3231	2.2	11040	3.9
3	0.5-0.6	7818	5.9	14160	5.8	16430	6.6	46	.030	39	.02	172	.060
4	0.6-0.8	4001	3.0	7733	3.2	9896	2.0	27	.010	1		2	
5	0.8-1.0	6	.004	16	.006	45	.020	1		0		1	
6	1.0-2.0	0		0		1		0		0		0	

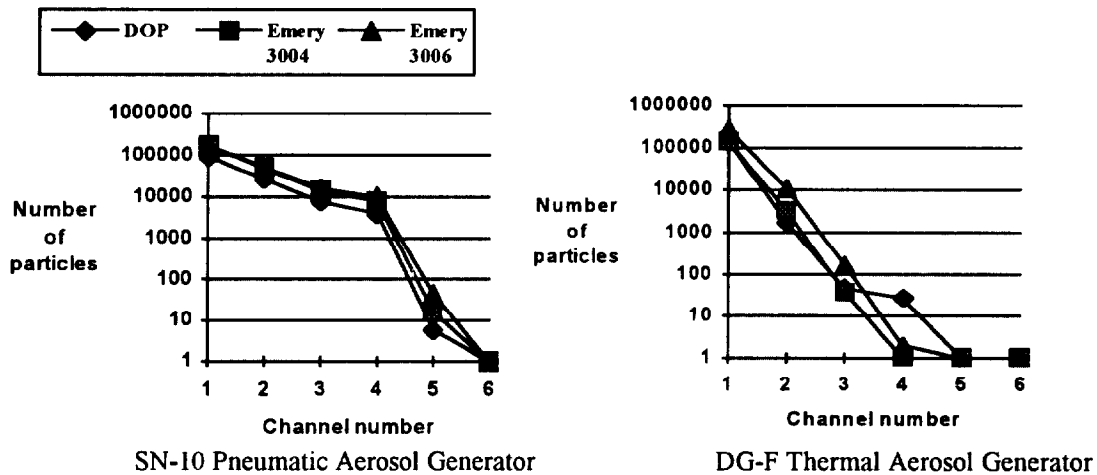


Figure 2. Aerosol Particle Size Distribution

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NUCON F-1000-DD-F Aerosol Detector response to DOP and Emery 3004 aerosols

A comparison test was performed to determine if the NUCON aerosol detector equipment possessed similar sensitivity characteristics to the Emery products as compared to DOP. The upstream injection port was used for aerosol injection (using NUCON SN-10 generator) - while the NUCON DD-F aerosol detector sampled the airstream through the upstream and downstream sample ports respectively (see Figure 1). DOP aerosol measurements were recorded first to establish a baseline. With the SN-10 generator operating at 20 psi, the calibrated DD-F detector was operated to read the upstream sample then the downstream sample respectively.

This method was performed using Emery 3004. The generator was operated at 20 psi and no adjustments were made to the aerosol detector. The data collected are given in Table 2.

Table 2: Output capacity of SN-10

	Upstream concentration		Downstream concentration	
	DOP	Emery 3004	DOP	Emery 3004
Concentration	60	200	0.02	0.06

Ignition test of DOP, Emery 3004 and Emery 3006

This test revealed that no possibility exists of self ignition under normal operating conditions, however; by exposing the generated aerosols of the thermal aerosol generator to an open flame it is possible to ignite the DOP aerosol as well as Emery 3004 and Emery 3006.

To demonstrate this, compressed air was connected to the DG-F thermal generator to perform as the carrier gas. When an aerosol plume was generated it was exposed to an open flame. The source of the fuel for this flame was propane gas generated from a hand held gas torch. The flame produced from the ignition of the aerosol propagated back to approximately one inch from the aerosol outlet of the generator. From that point it expanded outward approximately eighteen inches and engulfed the entire aerosol plume. Past this point the flame was no longer self-supporting. Performing this test and using an electronic spark igniter instead of the open flame did not result in the ignition of the aerosol plume.

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The flame size and closeness to the generator's aerosol outlet could be changed (within limits) along this axis by varying the liquid or carrier gas outputs. Identical conditions were observed when this test was repeated using nitrogen as the carrier gas.

Liquid to aerosol conversion rate, using an SN-10 aerosol generator

A comparison test was performed to compare the liquid to aerosol conversion rate of DOP, Emery 3004 and Emery 3006, to establish a baseline. An SN-10 generator was filled with DOP. The filled generator was weighed initially and then operated at 20 psi for 2 hours. At the end of the 2 hours the generator was re weighed. The difference between the beginning and the ending weight was determined as the liquid-aerosol conversion rate, and is reported in units grams/minute. (Table 3). This method was then repeated using Emery 3004 and Emery 3006.

Table 3.: Liquid to aerosol conversion rate of the SN-10 aerosol generator

Conversion rate	DOP	Emery 3004	Emery 3006
grams/minute	0.533	1.921	1.678

V. Evaluation Of Results

Particle size distribution and aerosol output

The data (Figure 2, Table 1) indicate that the particle size distribution curves for the aerosols generated by using Emery 3004 and Emery 3006 are essentially the same as the data generated by using DOP and are well within the ANSI/ASME-N510-75, 80, 89 size distribution requirements (Table 4).

Table 4.: Particle size comparison, by count

ANSI-N510 Particle Size Requirements	SN-10			DG-F		
	DOP	3004	3006	DOP	3004	3006
99% less than 3.0 microns	100%	100%	100%	100%	100%	100%
50% less than 0.7 microns	97%	97%	96%	>99%	>99%	>99%
10% less than 0.4 microns	71%	71%	69%	99%	98%	96%

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The output capacity test performed by the SN-10 aerosol generator with Emery 3004 and the DD-F aerosol detector showed an increased amount for generated aerosols of approximately 3:1 compared to DOP under the same operating conditions. This increase is due in part to the lower viscosity of Emery 3004. This was also confirmed with the liquid to aerosol conversion test.

Ignition test of DOP, Emery 3004 and Emery 3006

The ignition temperature of Emery 3006 is higher than that of Emery 3004, therefore it is somewhat harder to ignite. If an ignition occurs, the flame can be extinguished simply by turning off the liquid flow. Tests performed during the original design process of the DG-F, demonstrated that the 1/4 inch outlet tube acts as a flame arrester, therefore it is not necessary to use nitrogen as propelling gas ⁽¹⁰⁾.

VI. Conclusions

The test results indicate that Emery 3004 and Emery 3006 are acceptable substitutes for DOP in aerosol generation with NUCON F-1000 instrumentation. The particle size distribution is almost identical for either method of aerosol generation.

Results of the ignition test demonstrated that there is no advantage of the use of nitrogen gas as propellant instead of compressed air with the DG-F.

The thermally generated aerosols have a much larger population (in excess of 96%) of the particles generated at 0.4 micrometer or smaller. The increase in aerosol output of the SN-10 for the Emery products means that a larger air system can be tested without thermally generated aerosols.

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6. Bulletin 9B11-3.91 Model SN-10 Aerosol Generator
7. Bulletin 9B9-12.91 Model F Thermal Aerosol Generator
8. Bulletin 9B7-6.89 Model F Aerosol Detector
9. NUCON Procedure 12-67: In-place Leak Test HEPA Stage
10. Original design for DG-F

DISCUSSION

CROSBY: Why did you use a detector with a cut off of 0.3 μm diameter when a sizeable amount of the aerosol is $<0.3 \mu\text{m}$ in size? It seems to make your aerosol distribution data incomplete.

KOVACH, B.: Correct - it is incomplete. We had no detector for a particle size distribution below 0.3 μm . I wish I had had one at the time of the experiments. However, it makes little or no difference for leak testing.

SIGLI: The in-place DOP test is a leak test. As far as a leak test is concerned, the size distribution obtained with a substitute can be slightly different than with DOP. You will not see any difference if properly measured. If you want to look to efficiency, it is another problem, the size distribution having a direct influence on the result. Have you a comment about your experience using Emery vs. DOP?

KOVACH, B.: I thank you for confirming that the leak test and the efficiency test do not require an identical particle size distribution to accomplish the ANSI-N510 requirement.

**REVIEW OF IN-PLACE HEPA FILTER TESTING
AT SEVERAL DOE FACILITIES¹**

by

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ABSTRACT

The Office of Nuclear Energy Self-Assessment recently sponsored reviews of HEPA filter systems at several DOE facilities. One aspect emphasized in these reviews was in-place filter testing practices. Although in-place testing was generally performed as required in facility specifications, we noted several areas in which improvements were possible. Examples of some common problems and approaches to their solution will be presented. Areas of suggested improvement include: 1) ensuring the validity of test results; 2) recognizing and quantifying the uncertainty in penetration measurements; 3) expanding the analysis and reporting of test results to provide more than pass/fail information; 4) addressing the special problems of multiple stage systems; and 5) increasing the technical support and training provided in-place testing personnel. Ensuring the validity of test results, for example, requires more careful attention to the operation of test equipment, checking test measurements and system operating parameters for internal consistency, and more attention to documentation of system geometry and operation. Some issues will require additional study before the results can be incorporated into decision making on filter bank testing requirements and performance specifications.

I. INTRODUCTION AND BACKGROUND

In-place testing of HEPA filter installations has been part of the routine operating procedures at facilities supported by the US Atomic Energy Commission and its successor organizations for approximately 30 years. The importance of these tests in ensuring the performance of HEPA filter systems is recognized world-wide. The proper performance of these systems has become increasingly important as allowable emissions and exposures have been progressively lowered. For example, many DOE nuclear air cleaning systems were designed and built when the public dose limit was 500 mrem/year. This limit was reduced to 10 mrem/year in 1989 when DOE facilities came under the jurisdiction of the EPA and may be reduced even further in the future. These changes have not been addressed by comparable changes in in-place testing practices or requirements.

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² Environmental Health Sciences, Inc., contractor to Los Alamos National Laboratory.

Partly as a response to the reductions in allowable public exposures, the DOE Office of Nuclear Energy Self-Assessment (formerly NE-80 now NE-1.2, Quality Assurance Staff) initiated a series of selected environment, safety, and health reviews in late FY 1992. These reviews considered air emission control systems at several NE sites and facilities. One aspect of this effort was a survey of HEPA filter installations and testing procedures initiated as a DOE Safety and Health Issue (1). Another aspect was on-site reviews of specific HEPA filtration systems. The emphasis during these reviews was generally on the largest or most critical systems at each NE-supported facility. Consultants from Los Alamos and Sandia National Laboratories and The University of Michigan participated in those reviews concerned with air emission controls and associated monitoring. The reviews were not intended to be comprehensive audits of facilities or programs. Rather, they were to help describe program elements, bring attention to those elements that might benefit from changes or additional effort, and provide information to site personnel. The reviews have been summarized in a formal Los Alamos report (2).

The types and designs of air emission control systems, the extent of in-place filter testing, and testing practices varied widely between the sites. Within a given site, however, policies and practices were generally applied to all facilities regardless of the DOE office providing support and/or direct oversight for a specific facility. Similar observations at many of the sites implied common problems concerning technical and information or policy aspects of in-place filter testing. Many of the filter systems were designed and installed several decades ago and reflect outdated approaches and practices. To the extent the facilities and systems considered in these reviews are representative of those supported by other DOE offices, the observations presented here may also apply to other DOE facilities.

II. HISTORICAL NOTE

In-place testing of HEPA filters has been discussed at almost every Air Cleaning Conference since 1960. The first presentations on the technique and results of in-place testing were made at the Seventh Conference in 1961. The original work drew heavily on the expertise of the Naval Research Laboratory. Since those initial efforts, there has been considerable discussion on both the technical aspects of in-place testing and the role of in-place testing in ensuring that air emission control systems perform adequately. Many of these previous presentations were concerned with the problems of providing a uniform challenge aerosol concentration at the upstream face of a filter bank and obtaining downstream samples that correctly represent the average concentration of aerosol in the filtered air. These problems are still very much with us.

In reviewing the proceedings of previous conferences for papers concerned with in-place filter testing, we realized that many papers of interest were not found when using the key word (subject) index published as a volume of the Proceedings of the 20th Conference (3). We therefore compiled a list of conference papers we believe are of interest to individuals concerned with in-place HEPA filter testing. The subjects of the listed papers are primarily in the areas of testing methodology, field experience and observations, and discussions of the role of in-place testing. The papers are listed, by conference, in the Appendix. The listing does not include papers concerned primarily with HEPA filter quality, quality assurance testing, or the development of ANSI standards.

III. OBSERVATIONS AND RECOMMENDATIONS

In this paper we will depart from normal technical writing practice and present both the major problems observed during the reviews and our associated recommendations or solutions in a single section. The problems have been divided into two broad categories -- those that are essentially technical in nature and those that can be described roughly as "administrative" or organizational. As is often typical with such categorization, there is some overlap in the details of the problems and their solutions.

A. Technical Problems

1. Injection and Sampling Locations

The air emission control systems included in these reviews were not new. Most of them had been designed and built before publication of the first editions of current design and testing documents. Not surprisingly, these systems do not conform to currently recommended and accepted design and construction practices. In fact, recognition of the problems posed by these systems was part of the impetus behind development of the current recommended practices.

A frequent problem in these older systems is the location of ports for test aerosol injection and sampling. An example of such a system is shown in Figure 1. The test aerosol inlet (not shown in the figure) is more than 20 diameters upstream of the filter plenum inlet. The test aerosol is

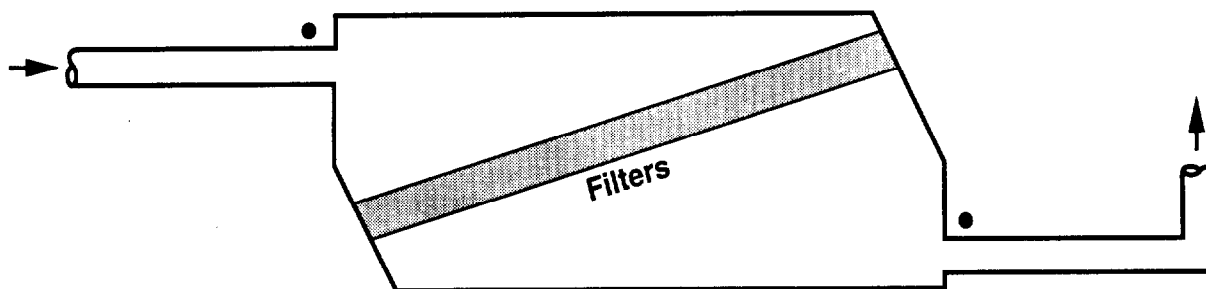


Figure 1. Schematic illustration of a HEPA filter housing with one bank of several filters. The solid dots indicate the aerosol sampling locations for in-place testing.

expected to be well mixed both at the upstream sampling point and over the upstream face of the filter bank. The downstream sampling point is on the duct center line approximately one duct diameter from the abrupt transition from the plenum to the duct. A sample taken at this point is unlikely to be representative of the average aerosol concentration in the discharge. In addition to the length of duct being too short to provide good mixing, the plenum itself appears to have been carefully designed to provide smooth, approximately constant velocity flow. Thus, aerosol penetrating through a leak at the periphery of the bank will not be uniformly mixed into the flow until it has passed through a considerable length of exhaust duct beyond the sampling point. The primary problem with this system is that it is very unlikely the present sampling approach can provide a representative downstream sample.

Poorly located injection and sampling points were encountered in nearly all the emission control systems reviewed. This observation reflects, at least partially, the old design of these systems. It was not clear in any of these cases, however, that consideration had been given to making changes that would correct the deficiencies. The prevailing attitude appeared to be that the injection or

sampling points had been specified and, no matter whether they were recognized as inadequate or not, they would be used without further consideration of their effect on the validity of performance measurements. It seems likely that in some cases the test points are so poorly located that the test data cannot represent the average performance of the filter bank. If the conditions are such that it is unlikely that accurate aerosol measurements can be made, it would be reasonable to consider such systems untestable unless modified.

Correction of these problems will require engineering review of injection and sampling locations and assessment of the degree to which they permit good mixing and representative sampling. If adequate injection and sampling locations are not possible, it will be necessary to assess the influence of test conditions on in-place test data. This assessment should include consideration of the measurement uncertainty introduced by the locations of the available injection and sampling points and other system characteristics. In some cases it may also be necessary to consider alternative approaches for establishing filter bank integrity.

2. Testing Practices

In-place filter testing is a deceptively simple appearing task --even if the problems of uniform challenge aerosol distribution and representative sampling have been recognized. We believe three changes in testing procedures would enhance the reliability of test results. A fourth change is essential if the uncertainty of test results is to be quantified and considered in system evaluation and the establishment of system performance criteria.

The first change in testing practice focuses on the magnitude of the challenge aerosol concentration. During the on-site reviews we found that tests are often performed with an essentially arbitrary challenge concentration. This is unfortunate because it means the concentration may be outside the optimum range of the photometer response characteristics. If the challenge aerosol concentration is too low and the common practice of adjusting the photometer gain to give a full scale reading is followed, excessive gain will unnecessarily increase electronic noise in the downstream reading. On the other hand, too high a challenge concentration will force the photometer beyond its linear range for the challenge concentration and result in an overestimation of the penetration. Aerosol generators should be operated so that challenge concentrations are in the recommended range of 20 to 100 micrograms per liter of a DOP (or suitable alternative material) aerosol with a size distribution that has a count median diameter of approximately 0.25 μm and a geometric standard deviation of approximately 1.5. The generator operating conditions to reach the desired challenge concentration should be a part of the test record for each filtration system.

The second change concerns photometer operation. Modern photometers are equipped with an internal reference or "calibration" light. The manufacturers recommend its use for setting instrument gain rather than the older practice of adjusting gain so that a full scale reading corresponds to the challenge concentration. The recommended practice ensures operation of the photometer electronics at a useful sensitivity without introducing excessive electronic noise. Setting the gain in this way also allows the photometer to be used as an approximately direct reading aerosol concentration instrument. Although operating in this manner will require calculation of filter penetration, the practice provides a simple method of verifying the reasonableness of test results as discussed in the next paragraph.

The third suggested change involves comparison of the photometer measurement of challenge aerosol concentration with the concentration calculated from system air flow rate and generator output. One of the shortcomings of the present approach of only comparing upstream and downstream concentrations is that there is no check on system operation or instrument performance. Using the photometer as a direct reading instrument, however, provides a method of verifying test equipment and system operation. From the air flow rate through a filter system and generator output rate, one can calculate an expected challenge aerosol concentration. This should agree, within some tolerance, with the photometer measured concentration. The tolerance will need to be determined after gaining field experience with the method but initially it seems reasonable to expect the two values to agree within a factor of two. On the other hand, it seems unreasonable to expect routine agreement to better than 10 - 20 per cent. Exact agreement between the observed and calculated concentrations is not the goal. The important point is that it is quite easy to determine that system and test instrument operation are consistent with each other. Some of the examples of inaccurate tests encountered during our reviews would have been avoided if this testing procedure had been in use.

Although it is generally assumed that an air emission control system will be operating normally during an in-place filter test, it is important to verify this. In this way testing personnel will be more certain that no changes have been made since previous tests and that flow and pressure conditions are within the expected range. Configuration and operating conditions, both expected and actual, should be part of the record from all tests. These should be verified on-site before a test is started. The field testing aid described later would be very useful for this purpose and would avoid reliance on the tester's memory of past test results.

The fourth suggested change involves how concentrations are read. Current practice is to observe an analog (or sometimes a digital) display for a short period of time and estimate the "average" reading. There is no attempt to or means of quantifying the variability of the meter readings. Penetration is therefore reported as single number with no indication of the uncertainty in the measurements used in the calculation. A more technically sound approach would be to record several (10 to 15) readings and calculate their mean and standard deviation. Although difficult and time consuming if done manually, the process is simple if done electronically. A device to make the readings and calculations and display the results would not be expensive and could be added to current photometers, many of which already have output connections suitable for use with a data logger or other external recording or display device. The initial use of such statistical information would be to describe temporal uncertainty in reported penetrations. The information may also be useful for monitoring changes in a system or comparing different systems.

3. Knowledge of Systems Being Tested

The depth of knowledge in-place testing personnel have of the systems they test is an example of a problem with both technical and organizational aspects and solutions. Testers frequently appear to simply follow management-provided-test procedures with little concern for understanding or evaluating the applicability of the procedures. This strict conformance to procedures appears, at least in part, to be a consequence of a major emphasis on quickly completing tests according to a tight schedule. In some cases this adherence to procedures had no impact on the testing but this was not always the case. For example, major problems were discovered with the procedures for a filter housing similar to the illustration in Figure 2. This housing is approximately 7 feet tall and 9 feet wide. The inlet duct, entering from the left of the figure, is 12 inches in diameter and is used to introduce the

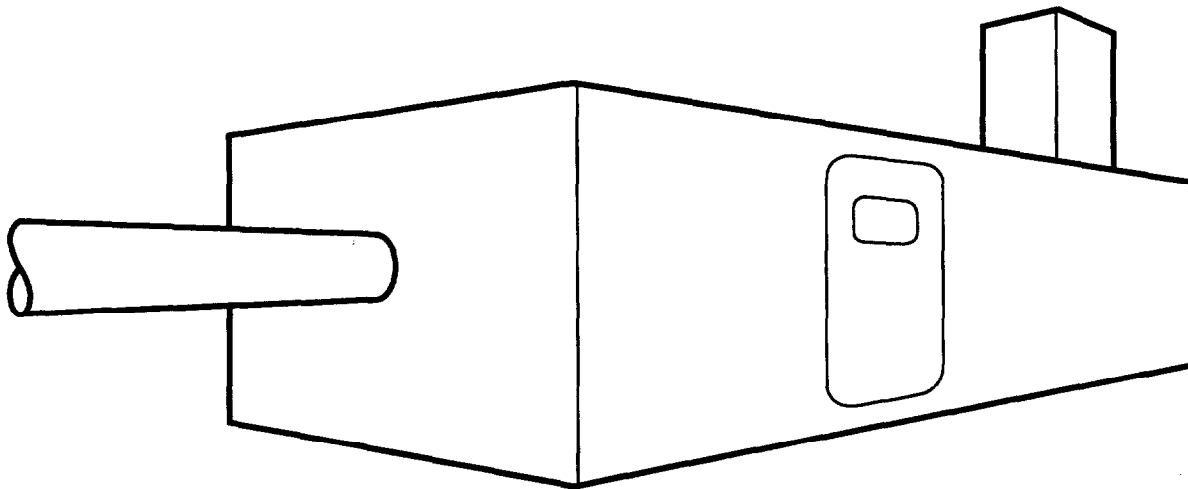


Figure 2. View of a medium size filter HEPA filter housing.

test aerosol. The upstream aerosol sample is taken near the entry to the filter housing. The downstream sample is taken near the top of the short duct on the fan discharge. Both samples are well mixed **BUT** the penetration calculated from them does not represent the performance of the HEPA filters in the housing. Two factors, as shown in the system schematic in Figure 3, cause this. First, the primary inlet, which is approximately 30 inches in diameter, is not apparent to anyone working around the filter housing. Second, there is a bank of prefilters that, in the case encountered during the review, testing personnel were not aware of. One way to ensure that test personnel know about all relevant system features would be to provide them a schematic of the exhaust system. The schematic should also include information on anticipated system operating conditions and test point locations.

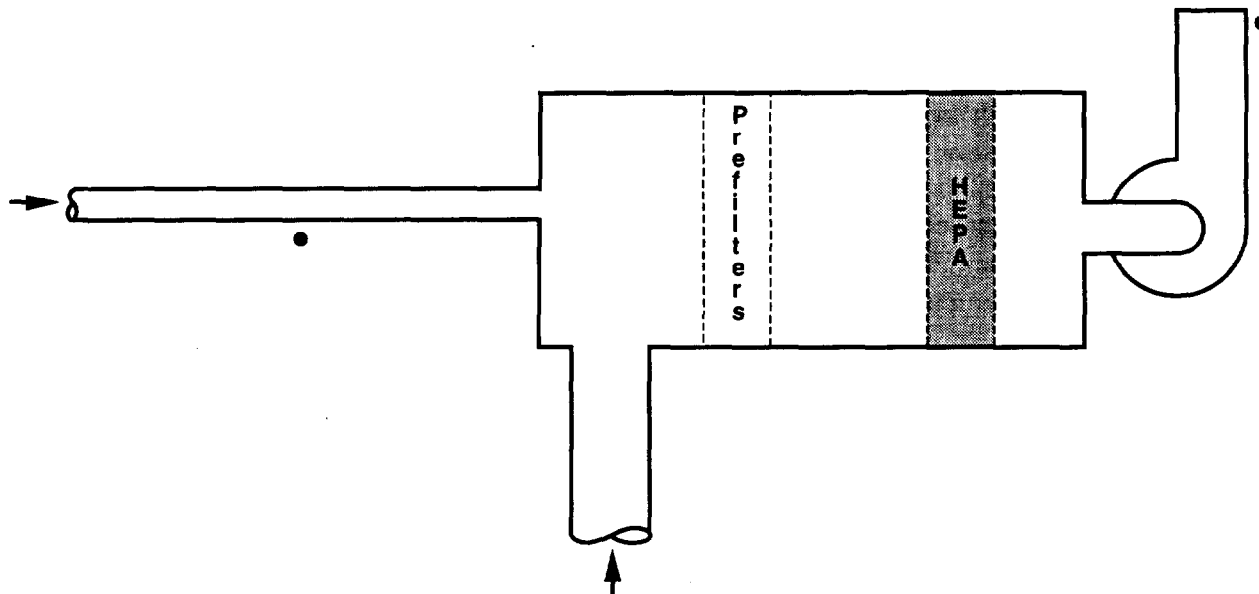


Figure 3. Schematic illustration of the HEPA filter housing shown in Figure 2. The solid dots indicate the aerosol sampling locations for in-place testing.

What is the extent of the engineering background and knowledge it is reasonable to expect of in-place testing personnel? In the situation described above, technical common sense would have shown that the obvious inlet duct is much too small for the filter housing served. Often the job of testing personnel is to follow established procedures and conduct the tests as rapidly as possible. Meeting the testing schedule is considered a primary goal. Under these circumstances, there is little opportunity or expectation that the suitability of the measurement points will be evaluated. This is a reasonable expectation in that such an evaluation should be carried out before routine in-place testing is initiated. This is one example of the need for the application of engineering judgment to the in-place testing of HEPA filter systems.

We believe it would be useful to have a simplified schematic diagram for every system that is to be in-place tested. This schematic should be in a form that can easily be taken into the field for each test. In addition to showing the arrangement of the system, including all inlets and how the different flows combine before entering the filter housing, information on expected operating conditions (flow rates and pressure drops) should be included. Such a diagram will allow testers to verify that system operation is in the expected range before starting a test. The schematic could also serve as the data sheet for recording test results as well as test equipment operating conditions.

4. Test Result Reporting and Analysis

In-place filter testing results appear to be considered only as indicators of the current acceptability of an emission control system. Test results are typically reported to system "owners" in a memo specifying the filter banks tested, the efficiencies (or occasionally the penetrations), and the test date. Although records of test results are maintained, we did not find any indication during these reviews that new data are considered in the context of previous results. It would be a relatively simple additional step to prepare and distribute test results in graphical form as an aid to comparing new results to earlier data and more readily noting trends or sudden changes in performance before specifications are exceeded.

While it seems appropriate to expect those relying on a filter system to control emissions from their facility to watch for trends in test data, we believe the responsibility should rest with the in-place filter testing group. There are three principal reasons for this. First, the testing group is specifically -- and sometime exclusively -- concerned with HEPA filter system performance. Second, what appears to be a change in system performance may be the result of a change in test method or instrument performance. Comparison of current data to previous results provides a further check on test validity beyond that described earlier. Third, in-place filter testing personnel should have a broader perspective on systems and their performance than those operating the processes that rely on the filter systems for control of emissions. This broader perspective gives the testing group the ability to interpret specific test results in the general context of the capabilities and limitations of in-place testing and the influence of air cleaning system design features on test results.

Another aspect of test result reporting and analysis we believe requires attention is that of the reliability of results. The technical aspect of the issue of measurement uncertainty was discussed briefly earlier in the section on testing practices. The influence of different sources of uncertainty is discussed in detail in our recent report (2). System design, construction, and operation can also affect the reliability of penetration measurements. In some instances we found that results were reported that we cannot be certain are representative of the filter banks tested. System designs that exclude provisions for in-place testing can make it impossible to obtain a uniform challenge aerosol concentration or representative samples. The reports for such systems, however, did not qualify or limit the results in any way or distinguish them from those obtained for systems with proper provisions for in-place testing.

5. Multiple-Stage Systems

Filter systems that incorporate more than one stage of HEPA filtration represent a particular problem for in-place testing. The most obvious problem is that many of the older systems were not designed for in-place testing of each filtration stage. Sometimes it is possible to extract a sample after the first stage of filtration but even this may not represent the average concentration of penetrant aerosol. In other cases it is impossible to properly test any of the stages.

One of the less obvious forms of multiple-stage filtration was found during some of the reviews of older systems. The system on which Figures 2 and 3 are based was only one of several systems encountered during the reviews in which prefilters were included in the test measurements for a single stage of HEPA filtration. Such testing is often forced by a filter mounting design that makes it impracticable to properly introduce and sample the challenge aerosol between the pre- and HEPA filters. When this was done, however, we did not observe any recognition in either the penetration requirements or test reports that the filters were being tested in this manner.

The problems of testing multiple-stages of HEPA filters have been discussed by several authors at previous conferences. The discussions and proposed solutions have been primarily technical in nature. Although some of the solutions appear quite practical, during these reviews we encountered several older multiple-stage systems in which the individual stages could not be tested. One obstacle is that of whether the anticipated remaining useful life of a facility warrants the expenditure to install and verify the performance of improved in-place testing features. A second is that of how performance of multiple-stage systems is specified. The specifications often seem to ignore the issue of whether it is even possible to perform a reliable in-place test.

B. Administrative/Organizational Problems

1. Technical Support for In-Place Testing

The numbers and backgrounds of in-place filter testing personnel, as well as the continuity of their testing work, varied widely between the sites visited during these reviews. In-place testing staff sizes range from one or two people performing filter tests less than 10% of the working year to groups with several full-time testers. The educational backgrounds of the testers span a similarly broad range from nondegreed maintenance and laboratory technicians to industrial hygienists with undergraduate technical degrees. Preparation of in-place testing personnel to perform their work generally emphasizes on-the-job training supplemented by attendance at one of the available specialized courses. In all cases the testing personnel made a serious effort to perform their work efficiently and according to the procedures they had been directed to use.

The variety of group sizes and backgrounds also lead to a wide range of capabilities to recognize and deal with unusual or nonideal conditions at the different sites. This range points to one of the shortcomings of most in-place filter testing programs. Although test personnel generally followed specified test procedures, we found that testing was often done without consideration of the reasonableness of the data obtained. This reflects, in part, the tendency to treat measurements as point values and evaluate them only in comparison to a specific pass/fail criterion. It is also indicative of the general emphasis on completing tests quickly and with minimal professional technical involvement with the details of routine in-place testing. Increased professional involvement should help reassert the importance of in-place testing and the need to ensure the validity of test data.

In-place filter testing groups do not always have the staff capability to perform an engineering review and evaluation of the systems they must test. At least one of the larger groups has recently added a professional engineer to its staff but this appears an exception rather than a normal practice. It is clearly out of the question for the smallest groups to have someone with such a background routinely available. Regardless of whether done by a permanent member of the in-place testing group or by an engineer temporarily assigned to the task, review and evaluation of HEPA filter systems is essential for three reasons. First, the review will provide the information necessary to prepare the schematics and basic information needed for the testing aid recommended previously. Second, there is a need to establish whether current testing locations and practices are providing accurate measurements of filter system performance. Third, the evaluation is an essential step in quantifying the influence of system design on measurement uncertainty.

In addition to more emphasis on engineering support for in-place testing personnel, they also should have their own "laboratory" system for testing, calibration, and instrument comparison. This is necessary both as a training aid and as a tool for monitoring instrument performance. This attention to ensuring correct functioning of test instruments will enhance the accuracy of filter system measurements. Availability and use of a system for this type of quality assurance effort was not common at the sites reviewed.

2. Communication/Coordination

The topics considered in this section provide a reminder of the many possible interactions and relations between all the groups specifying, working with, and benefiting from HEPA filters. Each of the topics was encountered during these reviews and, regardless of the positive or negative aspects encountered, emphasizes the need for communication between all those involved with HEPA filters.

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Exhaust systems that include HEPA filters are normally used to control a recognized potential hazard identified during an engineering and safety analysis. In one instance, however, we found HEPA filters being used inappropriately. An exhaust system had been installed over a hot work area in a large room to provide local control of the heat and the fumes and vapors that were sometimes released in this area. Sampling showed the exposures of unprotected workers at the hot location were within applicable occupational exposure limits but it was decided to filter the exhaust and "just to be on the safe side" HEPA filters were specified. The filters, of course, have no effect on vapors, perhaps the most serious of the materials entering this exhaust system. There were also no criteria established for filter performance based on the need to control process releases. Nevertheless, the filters, because they are HEPA filters, must be tested annually to satisfy facility requirements. The common penetration limit of 0.05% is applied. Here the lack of communication and coordination has led to the use of HEPA filters to "control" -- sometimes -- an exhaust that is not truly hazardous and the commitment of in-place testing resources to verify the performance of a system without a rational performance specification.

Use of HEPA filters to control laboratory or process exhaust emissions normally includes consideration of the chemical nature of the exhaust stream. The susceptibility of standard HEPA filters to damage by acid aerosols, gases, and vapors is well known to those who routinely work with HEPA filters. The knowledge is not always possessed by those performing experiments or operating processes in areas served by a HEPA filtered exhaust. Thus, we have encountered systems that showed signs of corrosion because of changes in the chemical nature of the emissions being controlled. The changes were made without discussion with those responsible for the exhaust system. Fortunately the problem was observed before there was a serious failure of the filters. The problem will be corrected by the replacement of the present filters with acid resistant ones. Better coordination between the operating groups served by the exhaust system and those knowledgeable about HEPA filters would have prevented this incompatibility and the damage to the filters that might have led to an unacceptable discharge of dangerous materials.

Problems may also arise as the usage of an exhaust system changes and total flow rates are changed or the number, size, and flow rates of specific inlets are changed. Although it is likely that those responsible for maintaining the performance of the HEPA filters will be aware of these changes, it should not be assumed. Perhaps the most serious problem is the influence of changes in flow rate and pattern on the ability to conduct in-place tests that provide a realistic evaluation of filter performance.

Coordination of exhaust system operation -- or nonoperation -- with maintenance and construction activities is also important. Here the problem is the damaging effect of dusts, fumes, and vapors generated during these activities on filter or adsorber capacity. Most operators appear to be aware of the potential problems and some have formal procedures to minimize the effects.

Establishing the necessary schedule for in-place testing appears to have been principally an arbitrary choice of a convenient interval. Ideally testing frequency would be based on the likelihood of HEPA filter failure and the severity of the consequences of such a failure. (Note that failure here refers to leakage and not a catastrophic failure such as fire or blow out.) The possibility of developing a formal approach for converting this ideal to a practical in-place testing schedule is not known. A practical testing schedule must also recognize factors other than physical or risk related ones. Annual testing is a practical minimum frequency, for example, based primarily on the administrative and record keeping convenience of annual tasks. Near the other conceptual extreme, daily testing would

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probably present too severe a burden both in terms of testing personnel effort and the loading of the filter medium with test aerosol material. The issue is the need to have a rational, documented assessment for setting the testing frequency that includes consideration of the potential hazard and the effects of testing on filter service life and operation of the facility or process served by the filters. Guidelines for performing such assessments should be developed.

Although it might appear that exhaust air monitoring should be considered in establishing an in-place testing schedule, we do not believe this is appropriate. The two efforts have very different purposes and philosophies. The fundamental purpose of emissions monitoring is to document that emissions are below specified limits. In some respects the monitoring serves as proof to others that a HEPA filter equipped system is controlling emissions. Emission levels above planned limits are certainly cause to reevaluate the controls. However, other independent programs such as in-place testing should also be the first line of defense to ensure air cleaning system performance and minimize the likelihood of excess releases. The effectiveness of air emission controls cannot be determined by air monitoring and must be demonstrated by an independent method. This strategy should ensure that routine emissions are always below specified limits.

3. Recognition of Uncertainty in Performance and Testing Specifications

The issue of uncertainty in penetration measurements has already been mentioned as a technical problem. It is, however, also an administrative one in the sense that developing performance criteria and setting standards involve administrative action. The present approach fails to incorporate recognition of either the uncertainty inherent in the measurement process or the influence of departures from ideal aerosol mixing and sampling conditions. The nearly universal application of a 0.03% or 0.05% measured penetration limit is inappropriate given the wide range of measurement uncertainty that exists for penetration measurements at DOE facilities. At the minimum, it seems reasonable to rewrite filter performance requirements to require that the measured penetration and its experimental uncertainty be such that there is a 95% probability that the actual penetration is less than a particular value. Whether this value is 0.05% or some other value should be based on an analysis of the contributions of system design and construction limitations to the uncertainty of test results.

IV. CONCLUSION

Although in-place testing has been practiced for more than 30 years, we still face many of the same problems encountered three decades ago. Because of the need to demonstrate that air emission control systems can meet increasingly stringent emission restrictions, we need to improve our test procedures and how we use the test results. We must recognize the uncertainty in our measurements in both our procedures and our performance criteria. Many filter systems, especially the older ones, are at best difficult to test and the reliability of test data should be considered in establishing filtration performance requirements.

REFERENCES

- (1) O.W. Lowe, Safety and Health Issue No. ISS-92-6, October 22, 1992 and responses from various facilities through January, 1993.
- (2) B.V. Mokler and R.C. Scripsick, "High Efficiency Filter Systems - General Observations, 1992-1993," Los Alamos National Laboratory report LA-12763-SR, 1994.

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(3) S.D. Jennings (Preparer), "An Index to the 1st through 20th AEC/ERDA/DOE and DOE/NRC Nuclear Air Cleaning Conferences," Volume III of CONF-880822.

APPENDIX

Compilation of Air Cleaning Conference presentations concerned with in-place testing of HEPA filters. The publication number for each proceeding is shown in parentheses; publication was generally in the year following the conference.

Seventh Air Cleaning Conference, Brookhaven National Lab, Oct 10- 12, 1961 (TID-7627)

A.L. Breslin, "Simple Apparatus for the In-Place Testing of Unit Filters" (Title not shown), pp 56-57

J.A. Young, "Evaluation of High-Efficiency Air Filter Systems," pp 84-97

R.W. Schneider, "Procedures for Testing High Performance Filters at ORNL," pp 187-203

B.J. Held, "High Efficiency Filter Program - National Reactor Testing Station," pp 215-219

H.W. Heacock and C.E. Jones, "The Hanford Reactor Confinement Program," pp 284-306

Eighth AEC Air Cleaning Conference, Oak Ridge National Laboratory, Oct 22-25, 1963 (TID-7677)

Round Table -- In-Place Filter Testing, pp 426-438

E.C. Parrish and R.W. Schneider, "In-Place Testing of High- Efficiency Filters at ORNL," pp 484-493

Ninth AEC Air Cleaning Conference, Boston, MA, Sept 13-16, 1966 (CONF-660904)

R.H. Knuth, "An Evaluation of Two Portable Thermal Aerosol Generators for In-Place Filter Testing," pp 763-780

F. Panchuk and W. Rachuk, "Filter Testing Program Atomic Energy of Canada Limited," pp 979-1020

F. Billard and J. Brion, "Testing of Air Cleaning Systems - Testing of the Components - In-Place Tests," pp 1021-1031

H. Flyger and H.C. Rosenbaum, "On-Site Testing of Filters with Special Reference to Solid Aerosols," pp 1032-1062

IAEA Symposium - Treatment of Airborne Radioactive Wastes, New York City, NY, Aug 26-30, 1968

E.C. Parrish and R.W. Schneider, "Review of Inspection and Testing of Installed High-Efficiency Particulate Air Filters at ORNL," pp 243-264

Twelfth Air Cleaning Conference, Oak Ridge, TN, Aug 28-31, 1972 (CONF-720823)

B.J. Grady, Jr and K.H. Henry, "In-Place Efficiency Tests of a Large Scale Ventilation Exhaust Filter," pp 646-654

A.G. Rolie and F.E. Adley, "High Capacity DOP Aerosol Generator," pp 718-730

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Thirteenth Air Cleaning Conference, San Francisco, CA, Aug 12-15, 1974 (CONF-740807)

A. O'Nan, R.P. Williams, and J.M. Goldsmith, "Reliability and Testing Considerations in the Design of Nuclear Reactor Filtration Systems," pp 30-40

F.J. Linck and J.A. Geer, "In-Place Testing of Multiple Stage HEPA Filter Plenums," pp 526-539

Fifteenth Air Cleaning Conference, Boston, MA, Aug 7-10, 1978 (CONF-780819)

B.G. Schuster and D.J. Osetek, "In Situ Testing of Tandem HEPA Filter Installations with a Laser Single Particle Spectrometer System," pp 838-847

L.G. Musen, "A Remote Sampling System for HEPA (1500 cfm) In- Place Filters with a Fluid (Channel) Seal System," pp 987-997

Sixteenth Air Cleaning Conference, San Diego, CA, Oct 20-23, 1980 (CONF-801038)

J. Dupoux, "In Situ Measurement of the Efficiency of Filtration Installation in the Nuclear Industry by the Soda-Fluorescein (Uranin) Aerosol Method - AFNOR Standard NFX 44.011," pp 17-34

M. Marshall and D.C. Stevens, "A Comparative Study of In-Situ Filter Test Methods," pp 35-55

L.P. Murphy, S.J. Fernandez, and B.G. Motes, "Comparison of HEPA Filter Test Methods in Corrosive Environments," pp 67-85

J.C. Elder, T.G. Kyle, M.I. Tillery, and H.J. Ettinger, "In-Place Testing of Tandem HEPA Filter Stages Using Fluorescent Aerosols," pp 86-103

M.W. First and H. Gilbert, "Aerosol Filtration," pp 638-666

Seventeenth Air Cleaning Conference, Denver, CO, Aug 2-5, 1982 (CONF-820806)

J. Furrer and A. Linek, "A New Method of Determining the Overall Particle Decontamination Factor for Multiple Offgas Cleaning Components in Reprocessing Plants," pp 576-590

M. Dupoux, "In-Situ Control of Filtration Systems in France: 5 Years Experience," pp 591-601

R.L. Herman, "In-Place HEPA Filter Aerosol Test System," pp 847- 866

F.A. Hohorst and S.J. Fernandez, "In-Place Realtime HEPA Filter Test Method," pp 867-881

W.D. Hanson, "DOP Testing HEPA Filter Banks in Series," pp 882- 894

Eighteenth Air Cleaning Conference, Baltimore, MD, Aug 12-16, 1984 (CONF-840806)

K.N. Kirchner, C.M. Johnson, J.J. Lucerna, and R.L. Barnett, "In- Situ Continuous Scanning High Efficiency Particulate Air (HEPA) Filter Monitoring System," pp 277-298

J.P. Ortiz, "In-Place Testing of Multiple Stage Filter Systems without Disruption of Plant Operation in the Plutonium Facility at Los Alamos," pp 299-310

L. Dauber, J. Barnes, and W. Appel, "Two-Detector Dioctylphthalate (DOP) Filter Testing Method and Statistical Interpretation of Data," pp 1399-1416

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Nineteenth Air Cleaning Conference, Seattle, WA, Aug 18-21, 1986 (CONF-860820)

J.W. Jacox, "Review of Current Halide and Aerosol Leak Testing Methods of Nuclear Air Treatment Systems, Components and Banks," pp 486-510

Twentieth Air Cleaning Conference, Boston, MA, Aug 22-25, 1988 (CONF-880822)

J.R. Edwards, "In-Place Testing of Close-Coupled Redundant Series Filters," pp 1094-1098

G.Q. Kirk, C.E. Childress, and D.D. Schmoyer, "Methods of Testing HEPA Filters with Short Upstream Approaches," pp 1210-1215

D. Loughborough, "Development of Filter Systems: Injection Systems and Mixing Device Evaluation," pp 1216-1230

Twenty First Air Cleaning Conference, San Diego, CA, Aug 13-16, 1990 (CONF-900813)

Ph. Cassette, N. Dupoux, and J.C. Laborde, "New Injection System for a Short Mixing of Test Aerosols and Tracers Inside Ventilation Ducts," pp 80-94

D. Loughborough, D. Morris, and S. Capon, "Development of Filter Systems - Part 2 -Injection Systems and Multi-Point Sampler Evaluation," pp 139-154

Panel: How to Use N510 Testing Methods and Acceptance Criteria for Air Treatment Systems Not Constructed According to N509, pp 317-342

A.E. Dunbar, Jr, J.R. Edwards, and K.W. Heffley, "Progress Report on the In-Place Acceptance Tests of Walk-In Plenum Filter Housings with Close Couple Redundant Series Filters," pp 360-365

Twenty Second Air Cleaning Conference, Denver, CO, Aug 24-27, 1992 (CONF-920823)

R.C. Parker, M. Marshall, and R.B. Bosley, "A New Method of In-Situ Filter Testing Using Pulses of Aerosol and Photometric Detection with Computer Control," pp 154-162

C. Costigan and D Loughborough, "Development of the Quickmix Injector for In-Situ Filter Testing," pp 163-176

J.D. Paul, "Effects of Filter Housing and Ductwork Configuration on Air Flow Uniformity Inside Air Cleaning Filter Housings," pp 205-210

DISCUSSION

ANON: You mentioned the need to consider test equipment accuracy. Is there a method to incorporate inaccuracy into results which is commonly used?

MOKLER: Uncertainty - or inaccuracy - of the results is presently not considered in making or performing HEPA filter penetration measurements. As noted in the paper, obtaining the average and standard deviation of a series of concentration measurements would require a simple addition to or modification of existing photometers. Standard error propagation calculations could then be used to quantify the uncertainty of the penetration. A more complete description of the effect of measurement uncertainty and nonuniform concentration and flow distributions on the uncertainty of calculated penetrations is available in the Los Alamos report referenced in the paper.

BERGMAN: Is there a technical report available that describes the result of the in-place filter test survey? The present report is too general and does not provide the tester with useful information. How do the different groups compare with the N510 requirements? Do the sites comply and how far off are they? Also, it is important that an in-place test accommodate production schedules and not turn into a research project. A users group should be formed with research support, and not have a research technique forced on the users.

MOKLER: The report summarizing the survey observations is referenced in the paper. Facility specific observations and recommendations, which would be excess detail here, were included in the reports to each site surveyed. As noted in the paper, the majority of the reviewed systems predate N509-1076 and compliance with any version of N510 is inappropriate and often impossible. An in-place testing users group has been considered as a potentially useful way of sharing and disseminating the experience of the different testing groups.

HOLTORP: What are your views on including the prefilter in with the HEPA filter test provided this is done consistently within a particular system?

MOKLER: No problem, as long as the procedures and test report do not give the impression that just the HEPA bank is being tested.

HOLTORP: Can you give a general example of an "unnecessary" HEPA filter installation?

MOKLER: Exhaust from a room with a low and occupationally acceptable exposure level that passes through a HEPA filter is an example of an unnecessary installation.

DYMENT: Do the regulatory/licensing authorities need to satisfy themselves of the technical viability of test procedures in order to issue or maintain a license?

MOKLER: Because my experience is all with DOE, I haven't had to deal with licensing situations.

CLOSING COMMENTS OF SESSION CO-CHAIRMAN ANDERSON

The first two papers in our session this morning dealt with the replacement of di-octyl phthalate (DOP) with a poly alpha olifin (PAO) in the QA/QC area and in-place testing. This action was deemed necessary when safety factors questioned the use of DOP in the plant environments. The reported efforts have determined that PAO can be substituted for DOP and is acceptable from the aerosol generation and safety standpoint. Because of viscosity similarities, Emery 3004 (Ethylflo 194) appears to be the PAO of choice. It should be pointed out, however, that the PAO selected is not a direct drop-in replacement for DOP. Because of vapor pressure differences, a temperature adjustment for the "hot" generators must be made to maintain challenge concentration levels. Since the densities of the two materials are different, one can expect differences in the penetration/velocity and penetration/size profiles. At identical test conditions, a difference in absolute penetration and maximum penetrating size will be observed for standard media. The refractive index of the materials is different, thus influencing the light scattering response of the photometer; it is surmised that separate photometer calibrations will be required for the challenge aerosol concentration. Of primary concern is that to preserve the 50 years of DOP performance data, some relationship of the new PAO data must be found so that the PAO information can be used as an extension to the existing DOP database.

The third paper reviewed in-place HEPA filter testing procedures at several DOE sites. Areas of potential concern were identified and possible changes/improvements suggested. This paper provides field support to a paper presented by Mr. Leonard at an earlier session of this conference where he addressed numerous problems in the filtration area and gave action items for their solution.

We would like to thank the presenters for their excellent papers and the attendees for your interest as reflected by your rapt attention and inquisitive questions.