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Title: Evaluating the use of PAO (4 cSt polyalphaoelfin) oil instead of DOP (di-octyl phthalate) oil for measuring the aerosol capture of nuclear canister filters

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Evaluating the use of PAO (4 cSt polyalphaolefin) oil
instead of DOP (di-octyl phthalate) oil
for measuring the aerosol capture of nuclear canister filters

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ABSTRACT

This document details the distinction between using PAO (4 cSt polyalphaoelfin) oil instead of DOP (di-octyl phthalate) oil for measuring the aerosol capture of filters. This document is developed to justify the use of PAO rather than DOP for evaluating the performance of filters in the SAVY 4000 and Hagan containers. The design criteria (Anderson et al, 2012) for purchasing SAVY 4000 containers and the Safety Analysis Report for the SAVY 4000 Container Series specified that the filter must "capture greater than 99.97% of 0.45 μm mean diameter dioctyl phthalate (DOP) aerosol at the rated flow with a DOP concentration of 65±15 micrograms per liter." This corresponds to a leakage percent of 0.03% (3.0×10^{-2}).

The density of DOP oil is 985 kg/m^3 and the density of PAO oil is 819 kg/m^3. ATI Test Inc measured the mass mean diameter of aerosol distributions produced by a single Laskin type III-A nozzle operating at a 20 psig air pressure as 0.563 μm for DOP oil and 0.549 μm for PAO oil. (See Appendix A.) For both types of oil in this document, the single fiber method calculated the leakage percent to be 4.4×10^{-5} for DOP oil and 4.7×10^{-5} for PAO oil. Although the percent error between these two quantities is 7.7%, these calculated leakage percent values are more than two orders of magnitude less than the criterion specified in the SAVY canister SAR. As a point of reference, the photometer used to measure the SAVY canister filter performance cannot resolve values for the leakage percent below 1.0×10^{-5}. Additionally, over a range of particle sizes from 0.01 μm to 3.0 μm, there was less than 4.0×10^{-5} error between the calculated filter efficiency for the two types of oil at any particular particle size diameter.

In conclusion, the difference between using DOP and PAO for testing SAVY canister filters is of inconsequential concern.

INTRODUCTION

This document details the distinction between using PAO (4 cSt polyalphaoelfin) oil instead of DOP (di-octyl phthalate) oil for measuring the aerosol capture of filters. This document was developed to justify the use of PAO rather than DOP for evaluating the performance of filters in the SAVY 4000 and Hagan containers. The design criteria (Anderson et al, 2012) for purchasing SAVY 4000 containers and the Safety Analysis Report for the SAVY 4000 Container Series specified that the filter must "capture greater than 99.97% of 0.45 μm mean diameter dioctyl phthalate (DOP) aerosol at the rated flow with a DOP concentration of 65 ±15 micrograms per liter."

ATI (http://atitest.com/index.html) is the manufacturer of aerosol generators and aerosol photometers that are commonly used for measuring the percent leakage of oil droplet aerosol in HEPA filter installations at Los Alamos (Hrbek 2008). The same equipment types (i.e. aerosol generators and photometers) are incorporated into a testing apparatus that measures the performance of filters installed in the lids of canisters used to contain nuclear material (SAVY 4000 and Hagan). The performance testing by the manufacturer (NFT Inc, Golden, CO) currently uses DOP oil. (Klemm 2014)

The quantitative capture of aerosols by fibrous filters is dependent upon the aerodynamic and physical diameter of the aerosol in question (Moore and Veirs 2012).

ATI has published information on their website about the size distribution of both PAO and DOP oil aerosol distributions produced by a Laskin nozzle. A brief summary of their data is presented below, where the mean diameter on a mass/volume normalized basis is selected as the relevant aerosol particle measure. The mass (volume) basis is most relevant for health physics considerations of dose prediction, where the dose is most dependent on the mass of the ingested or inhaled radioactive aerosols that are the primary concern at LANL.

Filter performance is defined in different ways, depending on the application involved. In Table 1, the distinction between "leak and capture" and ""penetration and efficiency" is explained.

Table 1. Filter performance is measured as the ratio between the downstream aerosol concentration, C_D, and the upstream aerosol concentration, C_U. Care must be taken to distinguish "leak and capture" from "penetration and efficiency". Refer to Moore etal 2011.

	Leak and Capture	**Penetration and Efficiency**
Example areas of usage	LANL SAVY SAR specification, NucFilt Inc testing, and LANL Industrial Hygiene filter leak testing.	HEPA filter definition given in ASME standard AG-1.
Aerosol Measurement Instrument	Single Channel (Photometer)	Multi-Channel (Spectrometer)
Test Aerosol	The aerosol concentration of a polydisperse distribution of sizes is measured by a single channel photometer (e.g. a normal distribution with a mean size of DOP oil droplets of 0.45 µm).	Aerosol concentrations are measured in discrete channels at each individual particle size (i.e. "essentially monodispersed 0.3 µm" as mentioned in ASME AG-1).
	Leak of Aerosol = C_D/C_U	P = Penetration = C_D/C_U
	Capture of Aerosol = $1 - C_D/C_U$	E = Efficiency = $1 - C_D/C_U$

As mentioned in the ATI Test FAQ resource page, past researchers have shown the aerosol size distribution does not vary according to the magnitude of the Laskin nozzle inlet pressure. The ATI Test company publishes DOP and PAO aerosol data from a Laskin nozzle operating at 20 psig, and the LANL FTS (Filter Test System) operates an ATI Laskin nozzle at 1.2 psig. A Laskin nozzle produces aerosol by forcing air through a submerged orifice tube in a pool of oil. The rising air bubbles burst on the liquid oil surface, and the entrained oil droplets in the air bubbles are the source of the submicron-sized aerosol. However, there is a measured difference of 0.014 µm (see Table 2) between the mean aerosol diameter produced from the two different types of oil.

Table 2. Summary of the size difference of aerosol using two different types of oil.

Oil Type	Mean mass/volume size diameter (µm)
PAO	0.549
DOP	0.563

The attached appendix (FAQ "Frequently Asked Questions" section from the ATI Test website) provides information that was used in this document. The aerosol particle size distributions measured by ATI Test were obtained using a TSI Scanning Mobility Particle Sizer (Model 3910 SMPS). This instrument measures aerosol size on a physical diameter basis, in contrast to an aerodynamic diameter basis.

The physical diameter is the independent size variable used for predicting filter leakage (and capture) using the single-fiber calculation method (Hinds 1999). This method was previously used to calculate the filter performance in nuclear material storage canister filters (Moore and Veirs 2012). In the current document, the single fiber method is used to compare the calculated filter capture of DOP aerosol, compared to PAO.

Table 3. Aerodynamic diameter comparison of PAO oil with DOP oil, using the 0.45 μm size diameter for comparison. (See Appendix B for more information.)

Oil type	Density (kg/m^3)	Vapor Pressure (kPa)	Physical (optical) diameter, μm	Slip correction factor C_C(0.45 μm)	Aerodynamic diameter, μm
PAO	819	0.013 kPa at 93.3 °C	0.45	1.49	0.40
DOP	985	0.16 kPa at 93.0 °C	0.45	1.49	0.45
Water	1000	2.4 kPa at 20.0 °C			
PuO	11,500				

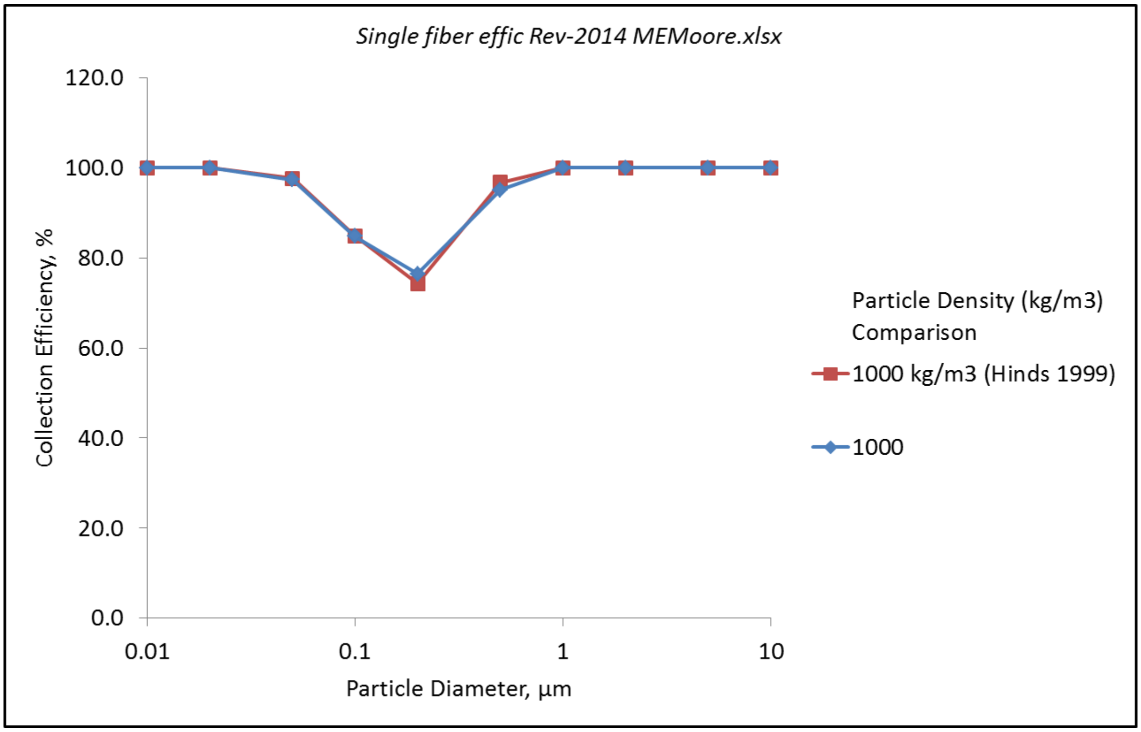

Figure 1. This graph establishes the accuracy of the filter efficiency (and capture) calculations used in this document. Published data (Hinds 1999) is compared to data calculated at a density of 1000 kg/m^3. These conditions are not identical to the Hagan or SAVY parameters. Fiber diameter = 2 µm. Aerosol particle density = 1000 kg/m^3. Filter thickness = 1.0 * 10^{-3} m. Filter fiber volume fraction α = 0.05. Face velocity = 10 cm/sec.

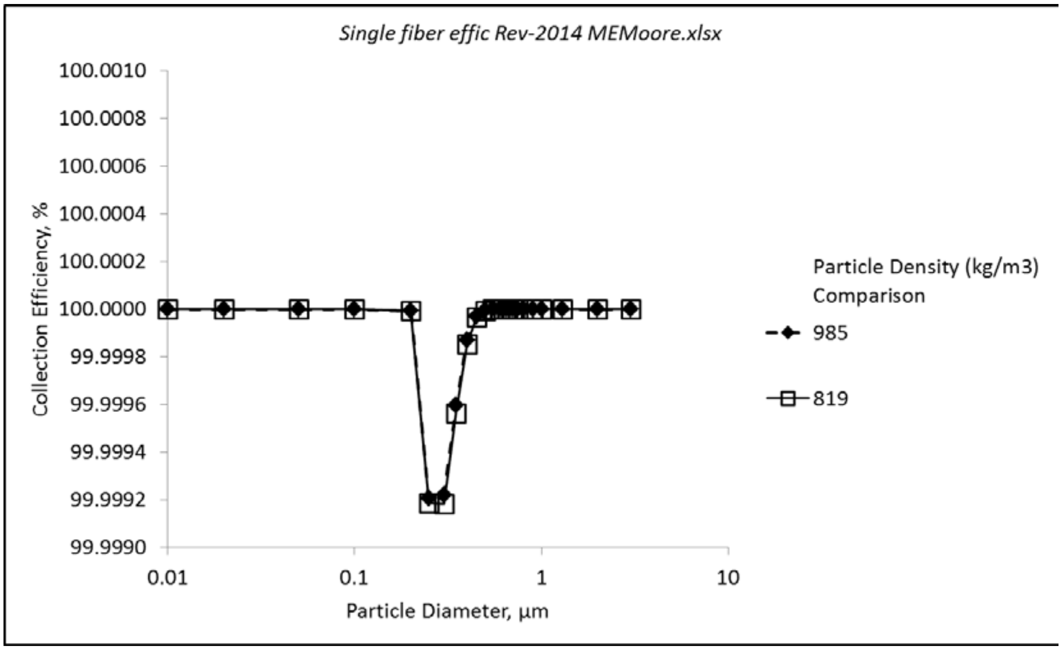

Figure 2. For the physical parameters of the SAVY canister filters, the filter collection efficiency was calculated using the different densities for PAO (819 kg/m3) and DOP oil (985 kg/m3).

Table 4. SAVY canister physical data with DOP oil.

FIRST DATA SET Density of particle (kg/m3)?	985
Filter volume fraction =	0.05
Filter thickness (m) =	3.0E-03
Viscosity of the air? (kg/m*s)	1.85E-05
Air temperature (deg F) =	68
Altitude correction =	1.00
Is the air flow in the same direction as gravity? Yes(+1); No(-1).	1
Fiber diameter (um)=	1.5
SAVY filter flow rate (accm)=	200
SAVY filter area (cm^2)=	1.98
Single Channel Aerosol Leakage Percent =	4.4E-05

Table 5. SAVY canister physical data with PAO oil.

SECOND DATA SET Density of particle (kg/m3)?	819
Filter volume fraction =	0.05
Filter thickness (m) =	0.003
Viscosity of the air? (kg/m*s)	1.85E-05
Air temperature (deg F) =	68
Altitude correction =	1
Is the air flow in the same direction as gravity? Yes(+1); No(-1).	1
Fiber diameter (um)=	1.5
SAVY filter flow rate (accm)=	200
SAVY filter area (cm^2)=	1.98
Single Channel Aerosol Leakage Percent =	4.7E-05

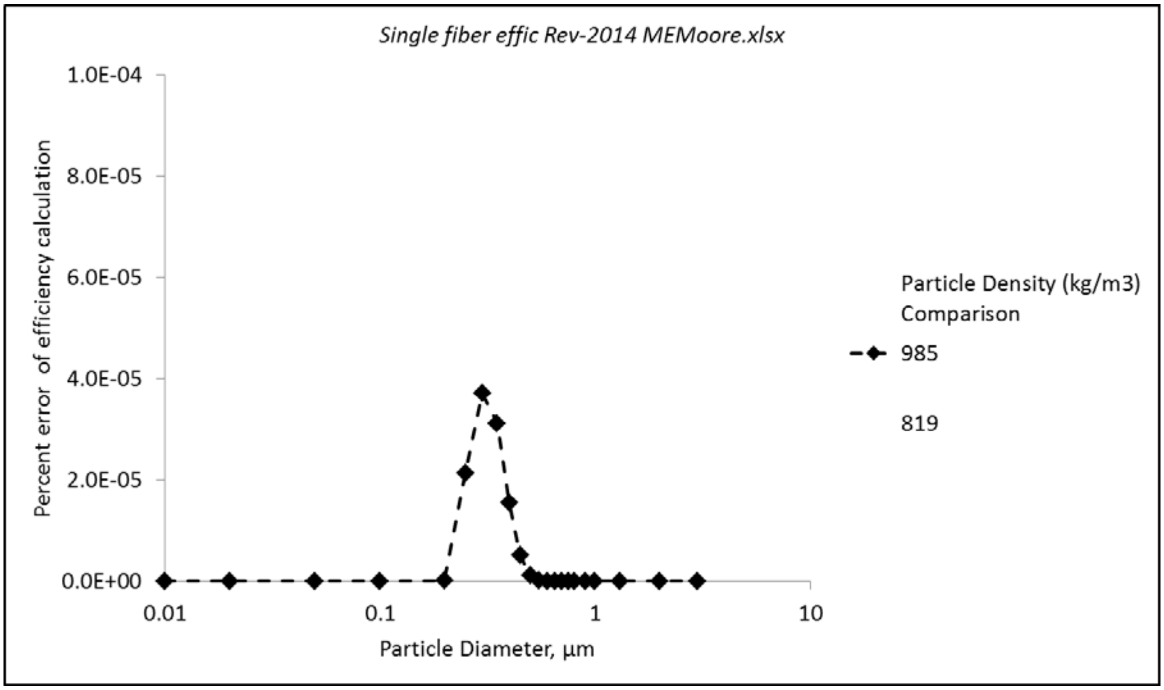

Figure 3. For the calculated filter collection efficiency for LANL SAVY canisters, there is less than a 4.0E-5% error in calculating the collection efficiency for any given diameter.

The Los Alamos FTS (Filter Test System) device is used to measure the aerosol capture percent in nuclear material canister filters. The FTS system uses a single Type III-A Laskin nozzle (ATI Test Inc. Model TDA-6C aerosol generator). The Laskin nozzle generates aerosol by forcing air through a small nozzle that is submerged in a pool of oil. The air bubbles in the liquid oil rise to the surface, then they burst, and entrained oil droplets become a cloud of oil aerosol. Due to the physics of the oil droplet formation, since the aerosol is created by bursting bubbles of air that have risen through a pool of liquid oil, the produced aerosol size distribution is independent of the air pressure used to operate the Laskin nozzle aerosol generator (Hinds et al 1983). The LANL FTS aerosol generator operates at a 1.2 psig pressure of air to the Laskin nozzle.

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APPENDIX A. FAQ (Frequently Asked Questions_ Section from the ATI Test website.
http://atitest.com/index.html

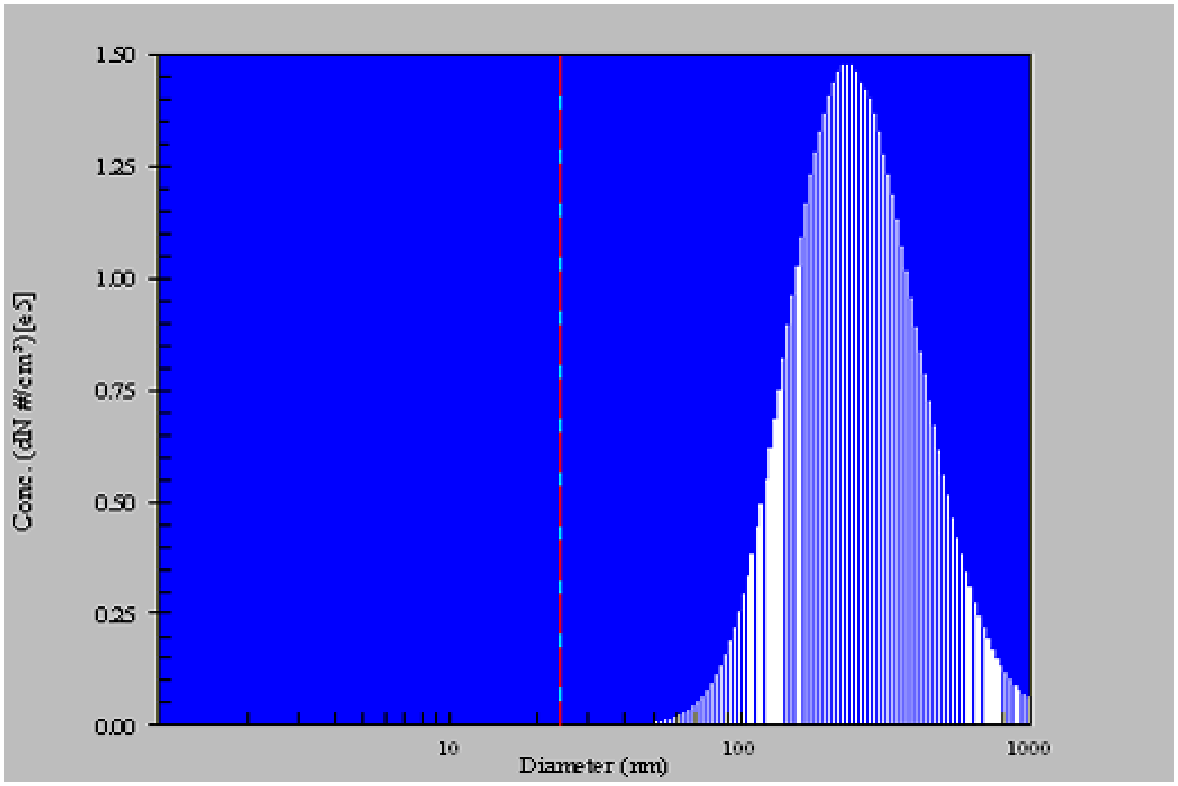
5) How much compressed air does a Type III-A Laskin nozzle aerosol generator need?

Each Type III-A Laskin nozzle consumes approximately 2.64 cfm (75 Liters) of air at 20 psi (1.4 bars) and total air consumption is a factor of the number of nozzles in use. Therefore the maximum theoretical compressed air requirement at 20 psi is 7.92 cfm for a three nozzle generator and 15.84 for a six nozzle generator.
The applied pressure of 20 psi should be measured at the nozzle inlet and must remain constant to allow calculation of the aerosol generator output. Please note, ATI only reccommends calculations when using fewer than 3 nozzles due to interaction of the nozzles in a confined space.

Air compressor performance specifications that will show output volume (cfm) versus pressure (psi) are available from most vendors and will make selection of an appropriate compressor less difficult.

6) What is the particle size distribution of a Laskin nozzle generator using PAO (4 cSt polyalphaoelfin)?

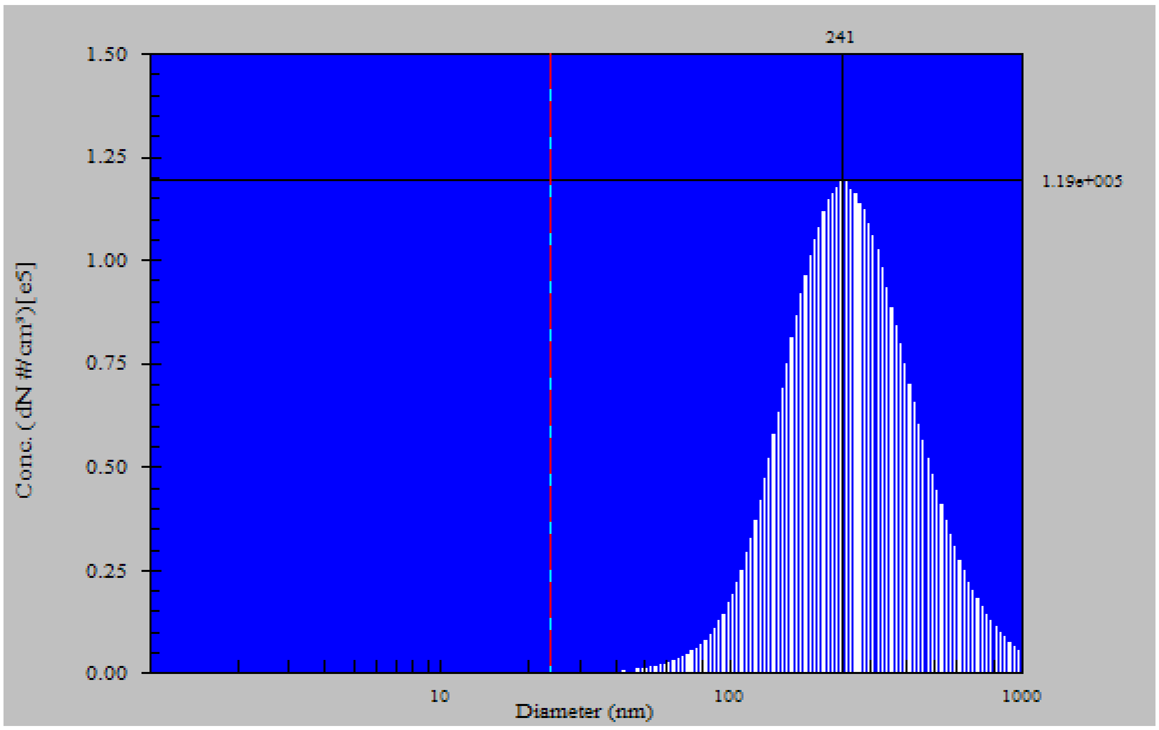
TDA-4Blite (operating a single Type III-A Laskin nozzle) @ 20 psi using PAO-4



	Number	Surface	Mass	Volume
	Particle Size	**Particle Size**	**Particle Size**	**Particle Size**
median (nm)	245	415	528	528
mean (nm)	281	454	549	549
geo. mean (nm)	248	407	503	503
mode (nm)	233	429	594	594
geo. st. dev.	1.65	1.62	1.55	1.55

7) What is the particle size distribution of a Laskin nozzle generator using DOP (DEHP)?

TDA-4Blite (operating a single Type III-A Laskin nozzle) @ 20 psi using DOP (DEHP)



	Number	Surface	Mass	Volume
	Particle Size	Particle Size	Particle Size	Particle Size
median (nm)	254	430	546	546
mean (nm)	291	468	563	563
geo. mean (nm)	256	420	517	517
mode (nm)	241	429	685	685
geo. st. dev.	1.66	1.61	1.54	1.54

11) What is the recommended shelf life of 4 cSt PAO (polyalphaolefin)?

The shelf life for 4 cSt PAO (ATI PAO-4) is ten (10) years in the original, sealed and unopened container from date of shipment. The shelf life of an open container, that is tightly sealed between uses, is one (1) year. The product should be stored in a temperature controlled, dry, environment between 15 and 30 °C while avoiding exposure to UV or direct sunlight.

22) What are acceptable substitutes for DOP (DEHP)?

Customers concerned with potential health problems to people working with Di [2-ethyhexyl] phthalate (aka DOP or DEHP) have inquired about acceptable substitute liquids for DOP. As a manufacturer of test equipment, ATI does not control which substitutes can be used. However, ATI recommends, in order of preference, the following substitute liquids:

(1) PAO (ATI-PAO 4)
(2) Ondina EL
(3) White Mineral Oil
(4) DOS / DEHS (Dioctyl-sebacate)
(5) PEG400/Polyethylene Glycol
(6) Peanut Oil
(7) Sunflower Oil
(8) Cottonseed Oil
(9) Corn Oil

In January 1992 the Army Surgeon General's office gave its approval for the use of 4 cSt PAO in lieu of DOP for U.S. Army filter test equipment, followed by the Food and Drug Administration in December 1996 for FDA regulated facilities. Because of these approvals, ATI has placed 4cSt PAO at the top of its recommended list of substitutes.

ATI has performed several studies on the various substitute liquids and found no significant difference in aerosol distribution. However, tests revealed that for the same generator pressures, *different concentration values where achieved depending on the substance used. This should be taken into account if a photometer's reference feature is calibrated for a liquid other than the one in being used.*

Many ATI customers who selected corn oil as an alternate liquid consequently encountered maintenance difficulties. Many of these customers are now switching to PAO which, in ATI's estimation, is the best substitute from a maintenance and operational point of view. The second most popular choice has been mineral oil and customer feedback indicates few maintenance problems.

31) Laskin Nozzle Aerosol Concentration

The purpose of this bulletin is to summarize the basics of concentration expected from a Laskin Nozzle aerosol generator, which includes the ATI Models TDA-4B, 4Blite, 6C & ATI 6D.

The standard Laskin III-A Nozzle has four jets located beneath four entraining holes. The volume of compressed air required to produce a given amount of aerosol depends on the pressure of compressed air applied to the nozzle.

The original design research was conducted by Echols and Young of the Naval Research Laboratory. This information is covered in Naval Research Laboratory Report #5929, dated 26 July 1963. Using this report and it's Appendix, the concentration versus pressure of the Laskin Nozzle can be extrapolated. Using the report information, it can be shown that when 20 psig is applied to the nozzle and diluted with 135 cfm of air, the output of one Laskin Nozzle provides a concentration of 100 micrograms per liter. The data also indicates that each nozzle requires 2.67 cfm of compressed air to maintain the 20 psig pressure drop.

Over the years, the impression has been that the only way the Laskin Nozzle Generator can be used is by applying 20 psig to the nozzle. According to the Echols & Young Report, as you increase the pressure, the concentration increases and as you decrease the pressure, the concentration decreases. Further studies by Dr. Melvin First at the Harvard School of Public Health and Wendell Anderson at *the Naval Research Laboratory have shown that by varying the pressure up and down on the Laskin Nozzle, the aerosol size distribution is not significantly affected. Wendell Anderson has also concluded that two of the jet holes may be plugged to obtain half of the standard concentration. Also, if three jets are plugged the concentration drops to one-fourth of the standard output concentration. This information is useful for customers who want to test air filtration systems that operate at lower than 135 cfm and other special low flow applications.*

Detailed information on the Laskin Nozzle and its use is available in Section 8 of the U.S. Department of Energy, Nuclear Air Cleaning Handbook. Dr. First's reference is available in the American Industrial Hygiene Association's 1983 Journal, Pages 495 - 500.

ATI presented a paper to the Institute of Environmental Sciences (IEST) in 1993 which confirmed that the liquid level did not have significant effect on aerosol concentration. This data begins on Page 559 of the 1993 IEST Proceedings, Volume I.

32) Laskin Nozzle Generator Conversion from DOP to PAO

What is necessary to convert my TDA-4 series or TDA-6 series Laskin Nozzle Aerosol Generator to Poly-Alpha Olefin (PAO)? Most Government agencies and departments are changing from DOP to PAO because DOP has listed on the suspected carcinogen list.

Since Dr. Hugh Carlon of the Army Chemical Center has completed research on Emery 3004, the Surgeon General has accepted PAO as an acceptable substitute for DOP in the U.S. Army. The Department of Energy and their various test sites, along with the Food and Drug Administration, are also accepting PAO as a replacement for DOP. Research conducted by the Army Chemical Center and Dr. Hugh Carlon has also shown that PAO is safe.

To convert TDA-4 series or TDA-6 series Generators from DOP to PAO:

(1) Drain the existing DOP liquid from the unit.
(2) Fill the reservoir to approximately half way on the sight gauge with PAO liquid.

(3) Drain the reservoir to eliminate any residue DOP remaining in the reservoir.

(4) Refill the reservoir to approximately half way on the sight gauge with PAO.

(5) Operate the unit as normal.

34) Aerosol Photometer Calibration with DOP/PAO

A photometer can be used regardless of aerosol liquid substitution because by setting 100% by sampling actual upstream aerosol, the photometer is "calibrated" to the system under test. In normal situations, a sample of the aerosol-air mixture upstream of the filter under test is sampled and the gain level is adjusted to obtain a 100% baseline. The photometer is then set to the CLEAR mode and an air sample is drawn through a reference filter with all particulate removed from the air stream.

At this point on analog photometers, the unit can be switched to normal operating range, usually 0.1% or 0.01%, and the straylight control used to adjust the 0.000% baseline. Digital units require pressing the Zero key to set the 0.000% baseline. Establishing the 100% and 0.000% baselines constitutes calibrating the photometer to the filtration system under test (ratiometric mode).

There are some instances (i.e., biological safety cabinet certification) where an upstream sample of the aerosol air mixture cannot be obtained. In this situation, the concentration is calculated and the sensitivity of the photometer adjusted to the correct level using the Internal Reference feature of the photometer. When the Internal Reference feature is used, the photometer must be calibrated to the specific aerosol reagent in use or the result will not be accurate. This is because DOP and various substitutes used in the field have different refractive indexes and they give different concentrations and photometric responses for the same weight per volume.

For additional technical information regarding photometric responses and particle size distribution, please refer to "Concentrations Produced By A Laskin Nozzle Generator" written by

David W. Crosby of ATI and "Characteristics Of Laskin Nozzle Generated Aerosols" written by Dr. Mel First at the Harvard Air Cleaning Laboratory, Page 109 through Page 125 in the 1990 21st DOE/NRC Nuclear Air Cleaning Conference Proceedings.

David W. Crosby has also written an article which was published in Performance Review, the technical journal of the Controlled Environment Testing Association, which details various factors and correct adjustments to compensate for substitute liquids. A copy of this article can be obtained by calling CETA, telephone (202) 737-0204.

The ATI Service Department will calibrate an older photometer to either DOP or PAO-4. TDA-2GA and later analog photometers, as well as all digital models, have PAO-4 and DOP Internal Refernce values stored from the Factory. A customer returning a pre TDA-2GA analog photometer for recalibration should specify the desired liquid.

39) PAO-4 (Emery 3004) Liquid

ATI frequently receives many different questions from customers regarding the use of Emery 3004. This Technical Bulletin addresses the most frequently asked questions.

EMERY 3004 was a trade name for 4 cSt polyalphaolefin (PAO-4), CAS #68649-12-7. The Henkel Company originally manufactured Emery 3004 PAO which was used in the initial research performed by Dr. Hugh Carlon et al. ATI carries 4 cSt polyalphaolefin, sold under the name PAO-4, in stock for the convenience of its customers. It is sold in 1 or 5-gallon containers and can be shipped via UPS or Federal Express. The 5-gallon container has a built in pour spout to aid customers in filling the aerosol generators.

The US Army Surgeon General has approved 4cSt PAO as the official replacement liquid for Di (2-ethylhexyl) phthalate (DOP, DEHP), CAS #117-81-7. Please note that it is not up to the liquid manufacturer to approve it for a specific use. Most government agencies or departments do not approve anything. For example, 4 cSt PAO is not approved by the Department of Energy. However the Department of Energy accepts the use of PAO as a replacement for DOP. In a letter from Robert L. Sorensen *of the Food and Drug Administration to the Director of Quality Assurance of Eli Lilly and Company he states that based on submitted documentation, research work, and papers, the FDA concurs with the military that 4cSt PAO is an acceptable replacement for DOP.*

There have recently been rumors that PAO manufactured at different sites are not suitable replacements for DOP/DEHP.

The following is an excerpt from the Food & Drug Administrations Human Drug CGMP Notes (Vol. 4, Number 4), December 1996.

"The original manufacturing site which produced the Emery 3004 (PAO) for the data submitted has changed since the study and Emery 3004 (PAO) is now manufactured at a different site. Discussions with the Army and the companies involved in the original studies indicate the product remains the same from the new site of manufacturing. CDER has also compared the original specifications and the new site specifications along with data from the Material Safety Data Sheets and agrees that there is no significant difference in the product from either site."

"The Chemical Abstracts Service (CAS) number which identifies this product also remained as 68649-12-7."

"Other reported alternatives used in the industry include DOS (Di (2-ethylhexyl) sebacate) and Ondina Oil. However, no manufacturer has yet submitted all the necessary data to evaluate these alternatives."

"As such, Emery 3004 PAO with the CAS number 68649-12-7 still remains an acceptable replacement for DOP."

Contact for further information:
Michael J. Verdi, HFD-322
Phone: 301-594-0095
E-mail: verdim@cder.fda.gov

The full text of the referenced document is available at: http://www.fda.gov/downloads/Drugs/DevelopmentApprovalProcess/Manufacturing/UCM193376.pdf.

Other relevant developments ATI feels its customers should be aware follow:

Beginning in the mid-2000's some manufacturer's of 4 cSt polyalphaolefin began using dual CAS#'s to define the product they were producing. 4 cSt polyalphaolefin previously identified as CAS# 68649-12-7, began including CAS# 68037-01-4, even when supplied by the same manufacturer under the same product number. The explanation was that the "New" CAS#, 68037-01-4, more accurately defined the product than the older number.

ATI has performed comparison testing on aerosol characteristics relavent to both filter leakage and efficiency testing and found no reportable difference between the two CAS identified products. The aerosol particle size distribution produced, aerosolization properties and photometric response were the same.

The U.S. Army, in conjunction with some Government attorneys, has acquired patents on the use of this liquid to generate aerosols. The first patent number 5,059,349 was issued on 22 October 1991. This patent is very specific in nature and covers the use of PAO and its use as a liquid to generate a monodispersed aerosol in the ATI TDA-100 Aerosol Penetrometer that is used for measuring the efficiency of respirator filters. The second patent 5,059,352 was issued on the same date. This particular patent covers the liquid PAO and its use in a prototype aerosol penetrometer that is no longer used for performing aerosol filter testing. The third patent 5,076,965 was issued on 31 December 1991 and covers the use of PAO liquid as the substance in a TSI model 8110-filter tester. These three patents really have no application in the normal in-place filter certification work that is carried out world wide in the pharmaceutical, electronic, and aerospace industries, although it is believed that one pharmaceutical company did pay the Army for a license agreement.

APPENDIX B. Calculation of aerosol slip correction factors and aerodynamic diameters.

Compare PAO to DOP oil.xlsx								
Oil type	Density (g/cm^3)	Physical (optical) diameter, D_P(μm)	Slip correction factor, C_C(D_P,μm) at 7300 ft	Aerodynamic diameter, D_A(μm) - first iteration	Slip correction factor, C_C(D_A,μm) at 7300 ft - first iteration	Aerodynamic diameter, D_A(μm) - second iteration	Slip correction factor, C_C(D_A,μm) at 7300 ft - second iteration	Aerodynamic diameter, D_A(μm) - third iteration
PAO	0.819	0.45	1.49	0.41	1.54	0.40	1.55	0.40
DOP	0.985	0.45	1.49	0.45	1.49	0.45	1.49	0.45
Note	*Values from the MSDS documents*	*Input value based on LANL SAR document.*	*"Aerosol Technology" Hinds WC, 2nd Ed*	$D_A = D_P * (\rho/\rho_0)^{1/2}$		$D_A = D_P * [C_C(D_P)/C_C(D_A)]^{1/2} * (\rho/\rho_0)^{1/2}$		The 3rd and 2nd iteration agree within 1%.
			Hinds Eq. 3.19; pg 49	*Hinds Eq. 3.28; pg 54*		*Hinds Eq. 3.28A; pg 54*		
			$C_C = 1 + 2.52\lambda/D$					Input parameters
			λ=air mean free path					Calculation cells
			D = diameter, physical or aerodynamic					Calculation and comparison cells