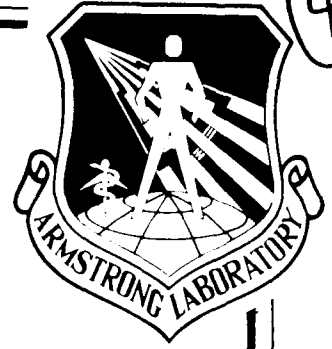


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**A HIGH PRESSURE AEROSOL GENERATOR
FOR VISCOUS FLUIDS**

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SEPTEMBER 1991

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TECHNICAL REVIEW AND APPROVAL

AL-TR-1991-0148

The experiments reported herein were conducted according to the "Guide for the Care and Use of Laboratory Animals," Institute of Laboratory Animal Resources, National Research Council.

This report has been reviewed by the Office of Public Affairs (PA) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.

FOR THE COMMANDER



JAMES N. McDOUGAL, Maj, USAF, BSC
Deputy Director, Toxic Hazards Division
Armstrong Laboratory

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PREFACE

This is one of a series of technical reports describing the results of the experimental programs conducted at the Toxic Hazards Research Unit, ManTech Environmental Technology, Inc. This document serves as an initial report on the development and use of a high pressure aerosol generator for viscous fluids, with specific reference to a candidate oil-in-water emulsion hydraulic fluid for Naval use. A less detailed letter report describing the initial development of the generator was submitted to the Contract Technical Monitor on 18 May 1990. The research described herein began in January of 1990 and includes data obtained through September of 1990 under U.S. Air Force Contract No. F33615-85-C-0532. Lt Col Michael B. Ballinger, USAF, BSC, served as Contract Technical Monitor. This report was written under U.S. Air Force Contract No. F33615-90-C-0532 (Study No. N04). Maj James N. McDougal, USAF, BSC, served as Contract Technical Monitor for the U.S. Air Force, Armstrong Laboratory (AL/OET).

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TABLE OF CONTENTS

SECTION	PAGE
PREFACE	1
LIST OF TABLES	3
LIST OF FIGURES	4
ABBREVIATIONS	5
1 INTRODUCTION	7
2 MATERIALS AND METHODS	8
Test Material	8
Aerosol Generator and Test Apparatus	8
Inhalation Exposures	11
Statistics	12
3 RESULTS	13
Generator Development	13
Inhalation Exposures	18
4 DISCUSSION	25
5 CONCLUSION	30
6 REFERENCES	31
QUALITY ASSURANCE STATEMENT	33

LIST OF TABLES

TABLE	PAGE
1 Physical and Chemical Characteristics of HS 5047F Hydraulic Fluid	8
2 Effect of Carrier Air Flow on the High Pressure Aerosol Generator Performance	14
3 Effect of Hydraulic Fluid Pressure on the High Pressure Aerosol Generator Performance	14
4 Comparison of Four Different Impingement Nozzles of Identical Manufacture on the High Pressure Aerosol Generator Performance	14
5 Exposure Aerosol Concentrations and Size Distribution	19

LIST OF FIGURES

FIGURE		PAGE
1	Schematic Diagram of the High Pressure Aerosol Generator	9
2	Schematic Diagram of the High Pressure Aerosol Generator Test System	10
3	IR Spectrum of Aerosol Sampled from the Test Chamber Midpoint	15
4	IR Spectrum of HS 5047F	16
5	IR Spectrum of Mineral Oil	16
6	IR Spectrum of Ethylene Glycol	17
7	IR Spectrum of Recycled HS 5047F	18
8	IR Spectrum of Aerosol Sampled from the High Concentration Exposure Chamber	19
9 a, b	Mean Aerosol Concentration, by Day of the Week, of the Low and High Concentration Exposure Atmospheres	21
10 a, b	Mean Aerosol Mass Median Aerodynamic Diameter, by Day of the Week, of the Low and High Concentration Exposure Atmospheres	22
11 a, b	Mean Aerosol Particle Count per cm ³ , by Day of the Week, of the Low and High Concentration Exposure Atmospheres	23
12	Weekly Mean Ethylene Glycol Concentration of the High Concentration Exposure Atmosphere	24
13	Mean Ethylene Glycol Concentration, by Day of the Week, of the High Concentration Exposure Atmosphere	24
14	Material Loss Due to Evaporation of Pure Mineral Oil, Neat HS 5047F, HS 5047F Used to Generate Low Concentration Atmospheres, and HS 5047F Used to Generate High Concentration Exposure Atmospheres	28

ABBREVIATIONS

°C	Degrees celsius
CV	Coefficient of variation
cm	Centimeter
D _o	Original droplet diameter
D _{mmd}	Droplet mass median diameter
D _j	Jet diameter
d	Diameter
D ₁	Droplet diameter at pressure 1 (initial pressure)
D ₂	Droplet diameter at pressure 2
g	Grams
h	Hour
HS 5047F	Houghto-Safe oil-in-water hydraulic fluid
IR	Infrared
id	Inside diameter
kg	Kilogram
L	Liter
MMAD	Mass median aerodynamic diameter
mg	Milligrams
min	Minutes
mL	Milliliters
mm	Millimeters
mm Hg	Millimeters of mercury
m ²	Square meters
m ³	Cubic meters
N	Newtons

ABBREVIATIONS CONTINUED

P_1	Initial nozzle pressure
P_2	Increased (decreased) nozzle pressure
p	Probabability
ppmv	Parts per million volume
psi	Pounds per square inch
RH	Relative humidity
SD	Standard deviation
s	Seconds
THRU	Toxic Hazards Research Unit
U	Relative liquid to gas velocity
v_g	Gas velocity
v_l	Liquid velocity
wk	Week (s)
σ	Surface tension
σ_g	Geometric standard deviation
μg	Micrograms
μm	Micrometer
μ_l	Viscosity (liquid)
ρ_g	Density (gas)
ρ_l	Density (liquid)

SECTION 1

INTRODUCTION

Naval operations include, on provisional status, the use of a relatively viscous, noncompressible, oil-in-water hydraulic fluid for which additional health risk assessment data are required. As part of these requirements to evaluate the potential inhalation toxicity of this and other candidate hydraulic fluids, it was necessary to develop an aerosol generator with which both physicochemically and toxicologically relevant studies could be conducted. One of the requirements was that the test aerosol be generated under conditions simulating those in which the hydraulic fluids are used (i.e., under high pressure). Operating pressures of the hydraulic systems of concern may vary, reaching pressures up to 3000 psi, and the system operating pressure may impact the physicochemical (hence toxicological) characteristics of the materials released from a microscopic breach (or breaches) in the system. Thus, another specific requirement of the generator was the ability to generate respirable size aerosols at variable hydraulic fluid pressures, without extraordinary alteration (beyond that which occurs during a system breach) of the fluid's physical properties or chemical composition; primarily, without excessive evaporation of volatile components of the hydraulic fluid. Furthermore, the generator needed to produce sufficient aerosol mass to conduct subchronic inhalation studies at target concentrations up to 1 mg/L in very large ($\approx 22 \text{ m}^3$) exposure chambers such as Thomas Domes (Carpenter et al., 1987).

Available conventional aerosol generators (see reviews - Fuchs and Sutugin, 1966; Mercer et al., 1968; May 1973; Berglund and Liu, 1973; Kerker, 1975; Grassel, 1976; Raabe, 1976; Willeke, 1980; and Hinds, 1982) were not suitable for this investigation for either one or all of several reasons: (1) low aerosol mass output; (2) limitations on fluid operating pressure; or (3) excessive evaporation of test material volatile components through the use of compressed gas to provide the atomization energy. Previous attempts to use nebulization methods to generate aerosols of hydraulic fluids (specifically the material used in this investigation - see below) did not satisfy the requirements of the present investigation (Kinkead et al., 1987). Therefore it was necessary to design and develop a unique generation system to satisfy study requirements.

SECTION 2
MATERIALS AND METHODS

TEST MATERIAL

The oil-in-water hydraulic fluid used for characterization of the generator performance and as the test material for subsequent inhalation studies was a mineral oil-water-ethylene glycol emulsion (HS 5047F, E.F. Houghton Co., Valley Forge, PA) provided by NMRI/TD. The physical and chemical characteristics of HS 5047-F, as provided by the manufacturer, are given in Table 1.

TABLE 1. PHYSICAL AND CHEMICAL CHARACTERISTICS OF HS 5047F HYDRAULIC FLUID

Content/Parameter	% by wt./value	CAS No.
Mineral Oil	60% ^a	64741-89-5
Water	30% ^a	-----
Ethylene glycol	10% ^a	107-21-1
Boiling point	101.7 °C	
Vapor pressure	like water (17.6 mm Hg at 20 °C) ^b	
Vapor density	like water (7.5×10^{-4} g/cm ³) ^b	
pH neat	9.0	
Density	920 kg/m ³	
Absolute viscosity	8.3×10^{-2} (N*s) /m ² @ 38 °C	
Surface tension	35 dyne/cm	

^a Values are approximate
^b Values are those of water

AEROSOL GENERATOR AND TEST APPARATUS

The high pressure generator system described herein (Figure 1) consisted of two major components; a variable pressure hydraulic pump assembly and a high pressure descaling/impingement nozzle. A compressed air driven hydraulic pump (Model 27740 - 1/3 hp M-V36-CF5L, Haskel, Inc., Burbank, CA) was selected to provide the hydraulic fluid pressures required. The hydraulic fluid pressure generated by this pump was directly proportional to the applied air pressure (1:41 psi, air to fluid), thus affording controlled variable fluid pressure. The maximum fluid delivery rating of 655 mL hydraulic fluid/min at 3000 psi with a driving pressure of 100 psi at 283.2 L/min air consumption rate for this pump was well within existing laboratory compressed air delivery

capabilities, and the fluid output rate was deemed more than adequate for experimental requirements. For atomization of the test material several commercially available simple pressure and ultrasonic nozzles were evaluated. The nozzle of choice was an impingement nozzle normally used for industrial descaling processes (Model PJ-8, Bete Fog Nozzle, Inc., Greenfield, ME), which provided a well dispersed fog from which respirable size droplets could be entrained in carrier gas flow for transport to an exposure or test apparatus. The typical PJ-8 consisted of a simple single, 2.03×10^{-2} cm d orifice pressure nozzle equipped with a tapered styus (2.03×10^{-2} cm, smallest d) positioned concentric to and directly above (7.62×10^{-3} cm) the orifice to serve as an impingement surface for the nozzle fluid jet. An absolute filter (Model 15705, Haskel Inc., Burbank, CA) was located in the hydraulic pressure line just prior to the atomizing nozzle.

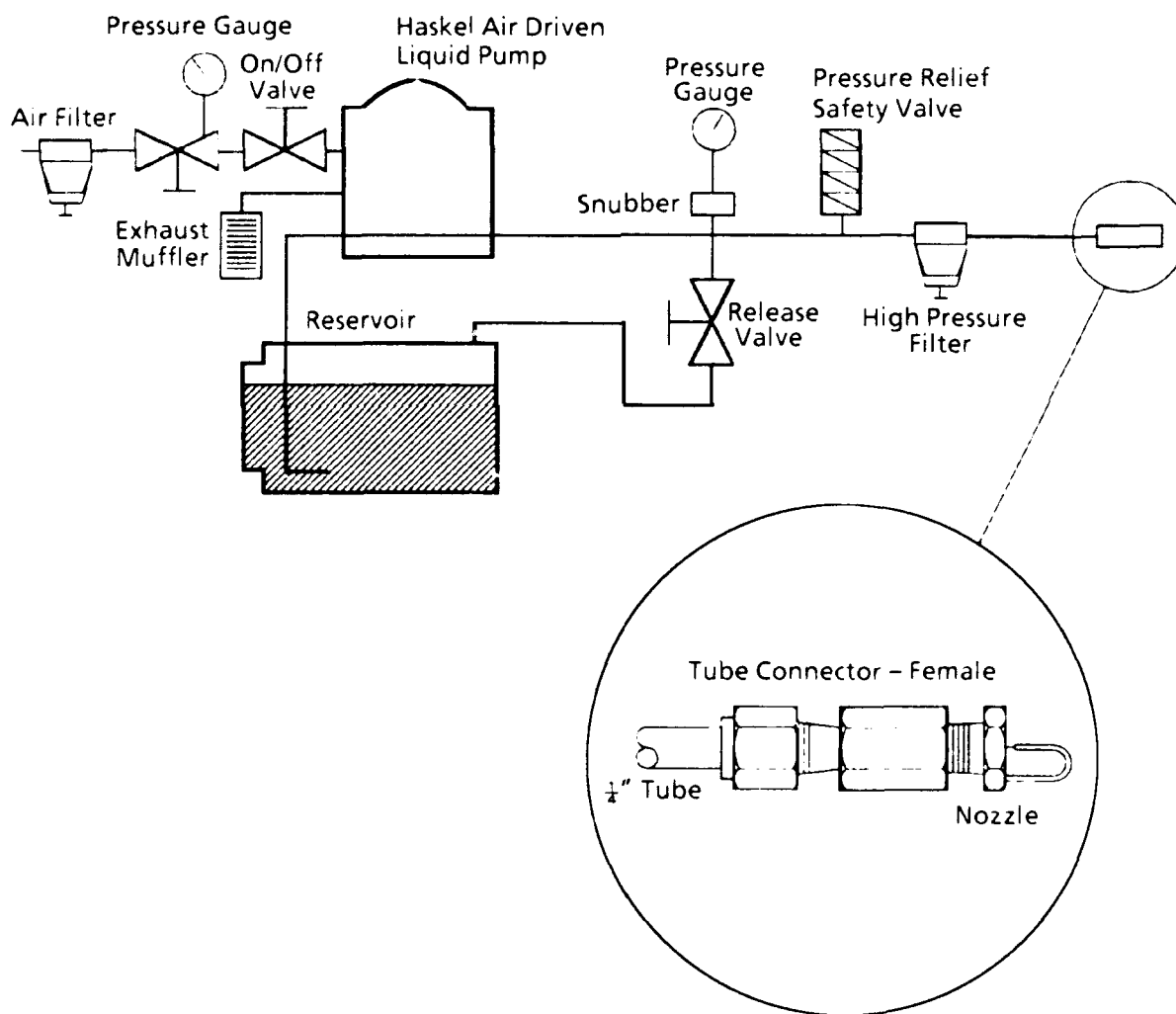


Figure 1. Schematic Diagram of the High Pressure Aerosol Generator.

In the complete test system (Figure 2) the atomizing assembly was sealed midline in a polypropylene cylinder (28 cm id x 38.5 cm, = 19 L carboy), which served to confine the high volume 25.4 cm d spray cone produced by the nozzle during fluid atomization. In addition, the cylinder functioned as a vertical elutriator in which larger droplets in the fog were removed either by impaction on the side walls or gravitational force. The cylinder also served as a reservoir for collection of test material not entrained in the carrier air flow and which then was gravity-fed back to the generator (pump) supply reservoir. Carrier air flow, as shown in Figure 2, was metered through tubing that extended from the bottom of the elutriator well into the nozzle fog production zone. The carrier gas exited the elutriator through a 1.1 cm id port located at the top and side of the cylinder. Entrained aerosol droplets were delivered to a prefiltered (HEPA, Airpure Model 007-C-08-3, Flanders Filters Inc., Washington, NC) 283 L/min air flow which then was delivered to a 222.4 L (61 cm, id x 76.2 cm) cylindrical test chamber fitted with two axially opposing, triangular (equilateral, circumscribed) mixing baffles which trisected the chamber volume. Air entered the test chamber at top-center through a 3.8 cm id duct and was exhausted through a 2.5 cm id tube positioned approximately 13 cm from chamber bottom and at 0.7 radial distance from the center, vertical axis.

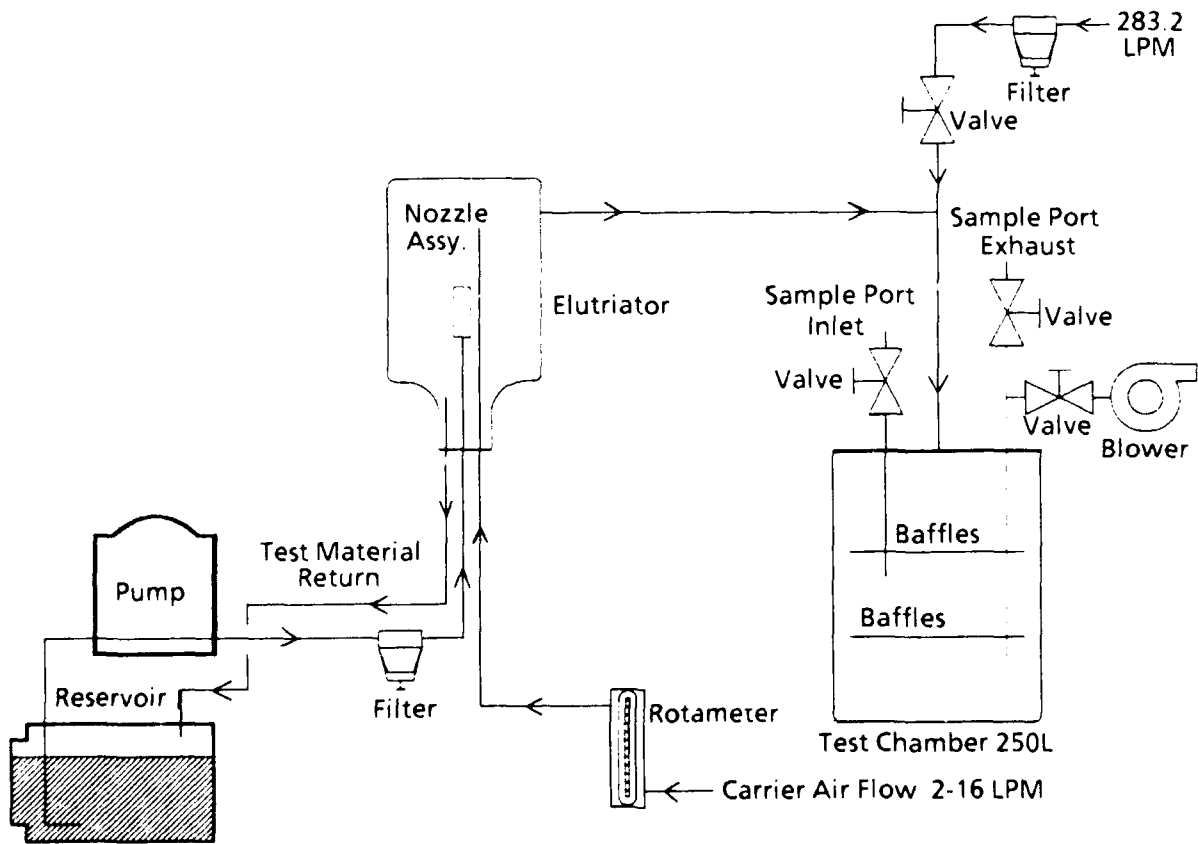


Figure 2. Schematic Diagram of the High Pressure Aerosol Generator Test System.

The test chamber was fitted with sampling ports for determination of aerosol mass concentration and size distribution at mid chamber (port located at 0.7 radial distance from chamber vertical axis - opposing exhaust - chamber penetration 38 cm from top) and at the exhaust point. The test chamber flow rate (283 L/min) and configuration were held constant for all generator assembly test conditions.

Filter (37 mm glass fiber, Gelman Sciences Inc., Ann Arbor, MI) and cascade impactor (8 stage, 28 L/min multijet, fabricated at the THRU - following Marple's criteria, Marple, 1970) samples were taken from both sampling ports to determine the test chamber aerosol mass concentration and size distribution. Chemical composition of the aerosol droplets were determined via thin layer infrared (IR) spectrophotometry (AccuLab 4, Beckman Instruments, Fullerton, CA) of pooled samples collected on uncoated impactor substrates.

The concentrations of airborne volatile components of HS 5047F, viz. ethylene glycol and water vapor, in the test chamber were determined by IR spectrophotometry (Miran 1A, Wilks Foxboro, S. Norwalk, CT) and dewpoint hygrometry (Model 1500/1211 H/T General Eastern Instruments, Watertown, MA), respectively.

INHALATION EXPOSURES

When initial development and testing of the generator was completed, two generators (one per exposure concentration) were employed to conduct a 13-week (92 days) continuous (24 h/day, 7 days/week) inhalation exposure of rats to HS 5047F (Kinkead et al., 1991). The Thomas Dome exposure systems were used and the target exposure concentrations were 0.2 mg/m³ and 1.0 mg/m³, concentrations well below the development target concentration of 1.0 mg/L. Fluid pressure in each generator varied from 1000 to 1200 psi, whereas carrier air flow through the low concentration generator ranged from 0.5 to 2.0 L/min, and the carrier air flow for the high concentration generator ranged from 3 to 8 L/min (depending on the specific nozzle used; all nozzles were of the same type and manufacture).

Continuous operation of the generators during the exposure study required periodic replenishment of HS 5047F in the generator reservoirs, therefore the generators were loaded with fresh HS 5047F every Monday, Wednesday, and Friday morning of each week during the study.

Concentration of the aerosol component of the exposure atmospheres was determined gravimetrically by periodic collection of filter samples, as well as continuously by near-forward angle light scattering aerosol photometry (RAM-S, MIE, Inc., Bedford, MA). Exposure atmosphere aerosol size distribution was determined using both cascade impaction (weekly) as described above and by laser aerosol photometry (Model APS 33B, TSI, Inc., St. Paul, MN) twice daily. Concentration of the volatile ethylene glycol component of HS 5047F in the exposure chambers was measured continuously

by IR spectrometry as described above. Baseline data were collected at 30 min intervals by switching spectrophotometer flow from the exposure chamber to room air. Water vapor concentration in the exposure chambers was monitored continuously using aspirated wet/dry bulb telethermocouples located within each exposure chamber. Water vapor concentration in the control chamber, which shared a common pretreated air supply with the exposure chambers but in which aerosols were not generated, was considered the baseline water vapor concentration.

STATISTICS

Comparison of control and exposure chamber relative humidity (RH) was performed using an analysis of variance with Bonferonni's test (Dixon, 1990). Unless specified otherwise, numerical data are given as mean \pm standard deviation, and error bars on graphical data are \pm standard error of the mean

SECTION 3

RESULTS

GENERATOR DEVELOPMENT

Generator performance with regard to aerosol mass output and size distribution was evaluated using three sets of test conditions. These included: (1) maintaining a constant hydraulic fluid pressure (1000 psi) while varying carrier air flow through the elutriator (2 to 16 L/min); (2) maintaining constant carrier air flow (8 L/min) while varying hydraulic fluid pressure (500 to 1500 psi); and (3) maintaining constant hydraulic fluid pressure (1000 psi) and constant carrier air flow (8 L/min) while using four different impingement nozzles of the same type and manufacture. The effects of varying carrier air flow on aerosol mass concentration and size distribution in the test chamber are shown in Table 2. Briefly, an eightfold increase in carrier air flow resulted in a sixfold increase in aerosol concentration at both the chamber midline and exhaust sampling ports, whereas aerosol size distribution remained unchanged. However, it was noted that with a progressive increase of carrier air flow there also was a progressive 2.7- to 4.0-fold decrease in aerosol concentration difference between the chamber midline and exhaust sample ports, suggesting increased entrainment and delivery of aerosol droplets of all sizes as a function of increased carrier air flow velocity. At all carrier air flows there was a 1.0 μm decrease in aerosol mass median aerodynamic diameter (MMAD) accompanied by insignificant changes of aerosol size distribution geometric standard deviation (σ_g) between the midline and exhaust sample ports. The effects of varying hydraulic fluid pressure on test chamber concentration are shown in Table 3. Doubling hydraulic fluid pressure from 500 to 1000 psi resulted in a nearly twofold increase in aerosol concentration at both sampling ports. At both pressures there was a 5.2- to 5.5-fold difference in aerosol concentration between the two sampling points. Interestingly, increasing the hydraulic fluid pressure to 1500 psi did not result in a significant increase in aerosol concentration at the test condition carrier flow rate. Although at all pressures there was a 1.0 to 1.1 μm difference in aerosol MMAD between the sampling points, varying hydraulic fluid pressure had no apparent effect on the σ_g . The effect of changing atomizer nozzles at fixed carrier flow (8 L/min) and hydraulic fluid pressure (1000 psi) was rather marked with regard to aerosol concentration delivered to the midline sample point of the test chamber (Table 4). There was nearly a twofold difference in aerosol concentration produced between the least and most proficient nozzles tested. The MMAD of the highest concentration aerosol was 0.4 μm larger than that of the lowest concentration aerosol (2.9 vs. 2.5 μm). However, there was no apparent differences between the σ_g s of the aerosols delivered by the four nozzles tested.

TABLE 2. EFFECT OF CARRIER AIR FLOW ON THE HIGH PRESSURE AEROSOL GENERATOR PERFORMANCE^a

	Carrier Air Flow					
	2 L/min		8 L/min		16L/min.	
Sample Port ^b	Mid	Exh	Mid	Exh	Mid	Exh
Concentration (mg/m ³)	26	9	77	22	163	41
MMAD ^c (μm)	3.9	2.8	3.7	2.8	3.9	2.8
σg ^d	1.7	1.6	1.7	1.7	1.7	1.7

^aSystem flow = 283.2 L/min, hydraulic fluid pressure 1000 psi
^bMid and Exh = midline and exhaust sample ports respectively
^cMMAD = Mass median aerodynamic diameter
^dσg = Geometric standard deviation

TABLE 3. EFFECT OF HYDRAULIC FLUID PRESSURE ON THE HIGH PRESSURE AEROSOL GENERATOR PERFORMANCE^a

	Hydraulic Pressure					
	500 psi		1000 psi		1500 psi	
Sample Port ^b	Mid	Exh	Mid	Exh	Mid	Exh
Concentration (mg/m ³)	49	10	98	18	86	20
MMAD ^c	4.2	3.1	3.9	2.8	3.8	2.8
σg ^d	1.6	1.7	1.7	1.7	1.7	1.6

^aSystem flow = 283.2 L/min, carrier flow 8 L/min
^bMid and Exh = midline and exhaust sample ports respectively
^cMMAD = Mass median aerodynamic diameter
^dσg = Geometric standard deviation

TABLE 4. COMPARISON OF FOUR DIFFERENT IMPINGEMENT NOZZLES OF IDENTICAL MANUFACTURE ON THE HIGH PRESSURE AEROSOL GENERATOR PERFORMANCE^a

	Nozzle			
	1	2	3	4
Concentration (mg/m ³)	83	44	63	57
MMAD ^b (μm)	2.9	2.5	2.6	2.6
σg ^c	1.6	1.7	1.7	1.7

^aSystem flow 283.2 L/min, carrier air flow 8 L/min, hydraulic fluid pressure 1000 psi. Impactor samples from exhaust port, filter samples from midline port
^bMMAD = Mass median aerodynamic diameter
^cσg = Geometric standard deviation

Comparison of the IR spectrum of aerosol droplets (Figure 3) collected from the test chamber midpoint (8 L/min carrier flow and 1000 psi fluid pressure) and the IR spectrum of HS 5047F (Figure 4) demonstrated remarkable differences between the composition of the aerosol droplets and the parent material. Large absorbance bands found between the 2.6 and 3.3 μm wavelengths and between the 5.6 and 6.7 μm wavelengths, as well as smaller bands found between the 9.0 and 10.0 μm wavelengths in the spectrum of HS 5047F, were not present in the aerosol droplet IR spectrum. Furthermore, the IR spectra of pure mineral oil (Figure 5) aerosol droplet samples were nearly identical, indicating that the aerosol droplets resulting from the atomization of HS 5047F were virtually mineral oil aerosol droplets. The IR spectrum of pure ethylene glycol (Figure 6) had a large absorbance band between the 2.6 and 3.3 μm wavelengths, similar to that found in HS 5047F, and two other large absorbance bands between the 8.7 and 10.5 and the 10.5 and 12.3 μm wavelengths, none of which were observed in the aerosol droplet spectrum. The large absorbance band between the 5.6 and 6.7 μm wavelengths found in the HS 5047F IR spectrum, but not in the aerosol droplet IR spectrum, also was not present in the IR spectra of ethylene glycol and mineral oil. Differences in the IR spectra of these materials suggest that the aerosol droplets produced by the generator and delivered to the test chamber did not contain appreciable amounts of the more volatile components of HS 5047F, namely ethylene glycol and water, both of which were found in the vapor phase of the test chamber atmosphere (see below).

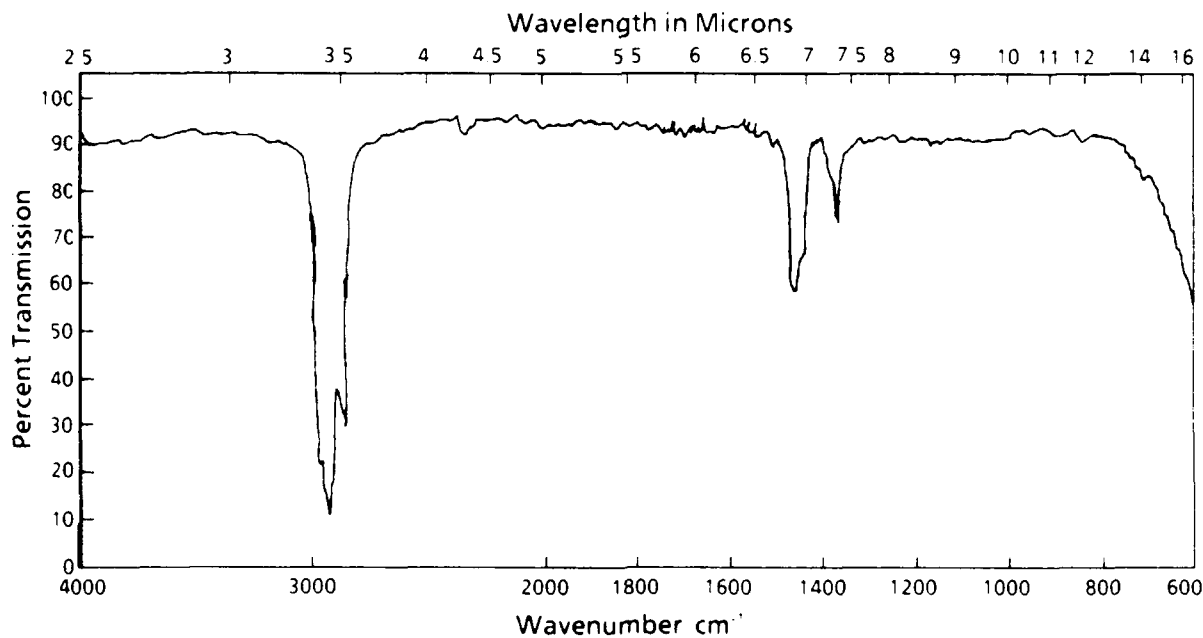


Figure 3. IR Spectrum of Aerosol Sampled from the Test Chamber Midpoint.

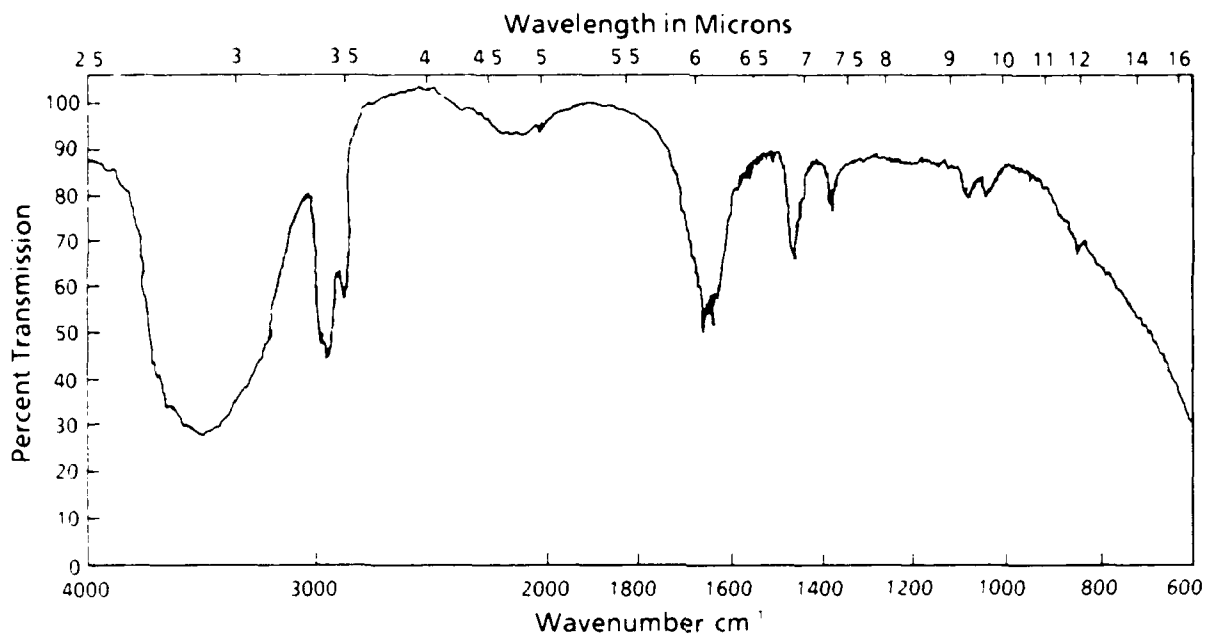


Figure 4. IR Spectrum of HS 5047F.

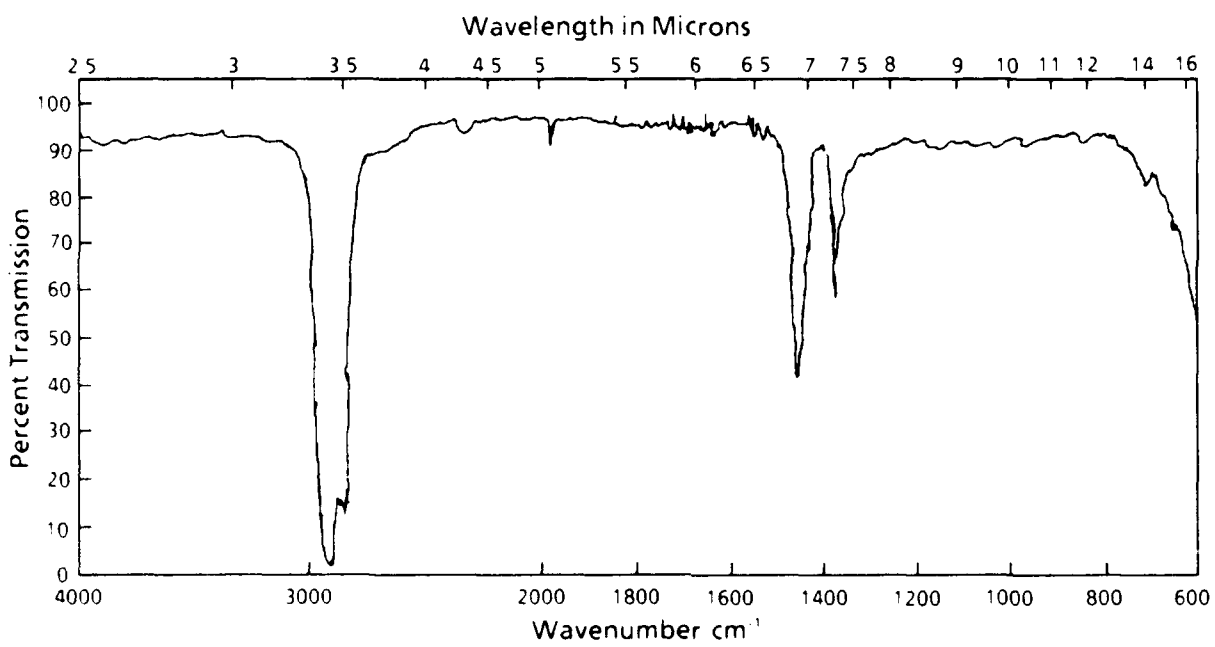


Figure 5. IR Spectrum of Mineral Oil.

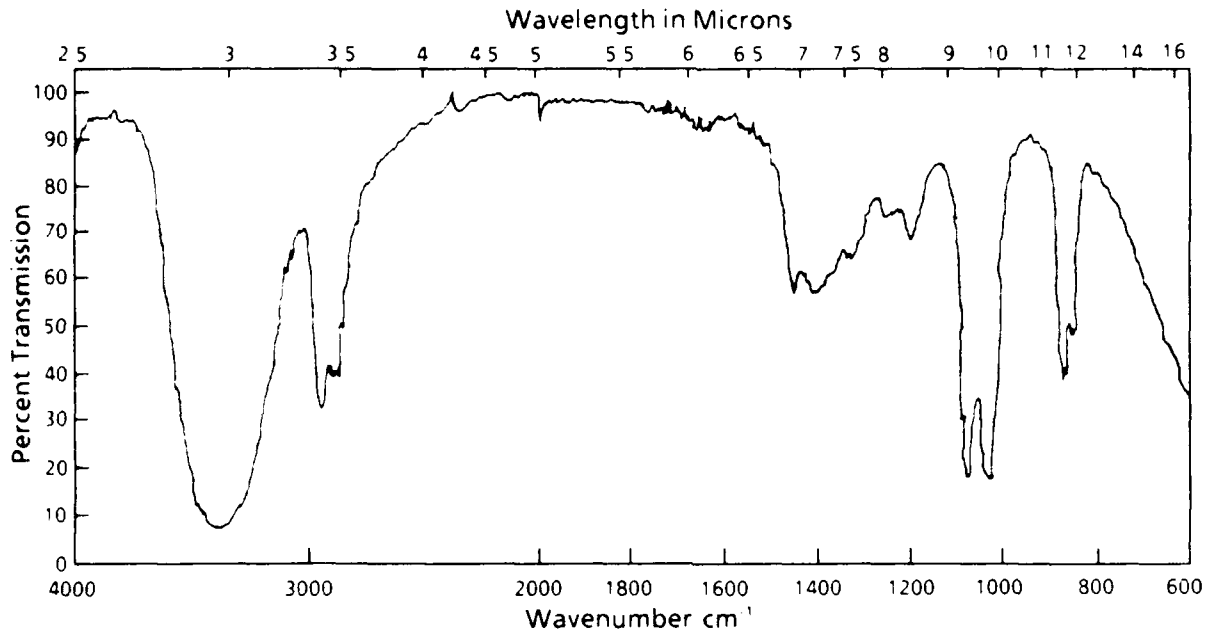


Figure 6. IR Spectrum of Ethylene Glycol.

The IR spectra of fresh HS 5047F and recycled HS 5047F (Figure 7) which had been passed through the generator nozzle between 20 and 30 times, although quite similar, show that during the atomization process some alteration of the chemical composition of HS 5047F occurs. A reduction in the relative magnitude of the absorbance bands found between both the 2.7 and 3.3 μm and the 5.6 and 6.7 μm wavelengths and other absorbance bands in the spectrum was observed in the IR spectrum of recycled HS 5047F. The changes in the composition of HS 5047F was most likely due to the disproportionate production and removal from the generator of mineral oil aerosol droplets by the carrier air and the removal of the more volatile components carried off as vapors.

A small fraction, 1.3%, of the total water vapor present in the test chamber atmosphere was attributable to the atomization of HS 5047F. An 8 L/min carrier air flow through the aerosol generator, without atomization of HS 5047F, resulted in a water vapor concentration of $1.50 \times 10^4 \pm 0.0$ ppmv (parts per million volume) measured at the test chamber exhaust port. Under identical system operating conditions with HS 5047F atomization, the test chamber water vapor concentration was $1.52 \times 10^4 \pm 46$ ppmv. Increasing the carrier air flow through the generator to 16 L/min increased test chamber aerosol concentration by a factor of 0.86 raising concentration from 22 to 41 mg/m^3 . However, the concomitant increase in water vapor density in the test chamber was not as great. Water vapor concentration attributable to HS 5047F atomization increased by a factor of 0.5 from 2.0×10^2 ppmv at 8 L/min to 3.0×10^2 ppmv at 16 L/min ($1.54 \times 10^4 \pm 117$ ppmv without atomization

to $1.57 \times 10^4 \pm 41$ ppmv with atomization). Thus, 1.9% of the total moisture content in the test chamber came as a result of the atomization process.

Ethylene glycol concentration in the test chamber during a typical test run (8 L/min carrier air flow and 1000 psi fluid pressure) was 4.5 mg/m^3 . Doubling the carrier air flow increased the ethylene glycol concentration by a factor of 0.49 to 6.7 mg/m^3 .

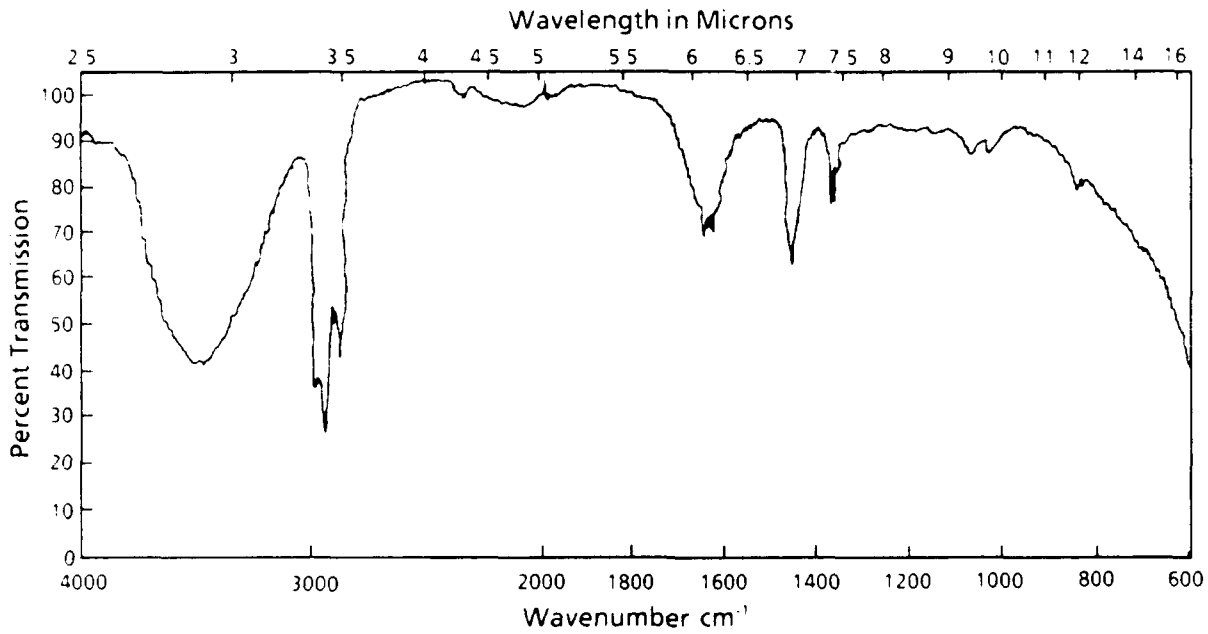


Figure 7. IR Spectrum of Recycled HS 5047F.

INHALATION EXPOSURES

Mean weekly exposure chamber aerosol concentration and size distribution data for the inhalation exposure study are presented in Table 5. Exposure aerosols with MMADs ranging from 2.8 to 3.1 μm and σgs of 1.49 and 1.47 were slightly smaller and less polydisperse than test chamber aerosols with MMADs ranging from 2.8 to 4.2 μm and σgs of 1.6 and 1.7 (Tables 2 and 3). These small size distribution differences were not due to compositional differences between the aerosols; the IR spectrum of aerosol droplets collected from the high concentration exposure chamber (Figure 8) demonstrated that the exposure aerosols were virtually mineral oil droplets (compare Figures 5 and 8), just as were test chamber aerosols.

TABLE 5. EXPOSURE AEROSOL CONCENTRATIONS AND SIZE DISTRIBUTION

	Low Exposure			High Exposure		
	Concentration ^a	Size ^b		Concentration ^a	Size ^b	
	(mg/m ³)	MMAD ^c	og ^d	(mg/m ³)	MMAD ^c	og ^d
Week 1	0.195	2.81	1.39	1.06	3.32	1.41
Week 2	0.203	2.69	1.39	1.05	3.37	1.40
Week 3	0.199	2.76	1.42	1.03	3.19	1.45
Week 4	0.203	2.91	1.42	1.04	3.09	1.44
Week 5	0.208	2.96	1.39	1.06	3.14	1.43
Week 6	1.999	2.86	1.39	1.04	3.08	1.45
Week 7	0.202	2.65	1.41	1.03	3.01	1.46
Week 8	0.214	2.52	1.42	1.03	3.02	1.48
Week 9	0.205	2.67	1.42	1.04	2.98	1.47
Week 10	0.207	3.05	1.40	1.02	3.09	1.45
Week 11	0.203	2.76	1.45	1.03	2.91	1.48
Week 12	0.204	2.79	1.48	1.01	3.03	1.49
Week 13	0.204	2.90	1.47	1.02	3.05	1.49
Mean	0.204	2.79	1.42	1.04	3.10	1.45
SD ^e	0.0046	0.145	0.026	0.015	0.130	0.029
CV ^f	2.3%	5.1%	2.1%	1.4%	4.2%	1.9%

^aValues are weekly mean of continuous observations (n = 1425 to 1450/day)

^bValues are mean of twice daily observations

^cMMAD = mass median aerodynamic diameter (μm)

^dog = geometric standard deviation

^eSD = standard deviation

^fCV = coefficient of variation

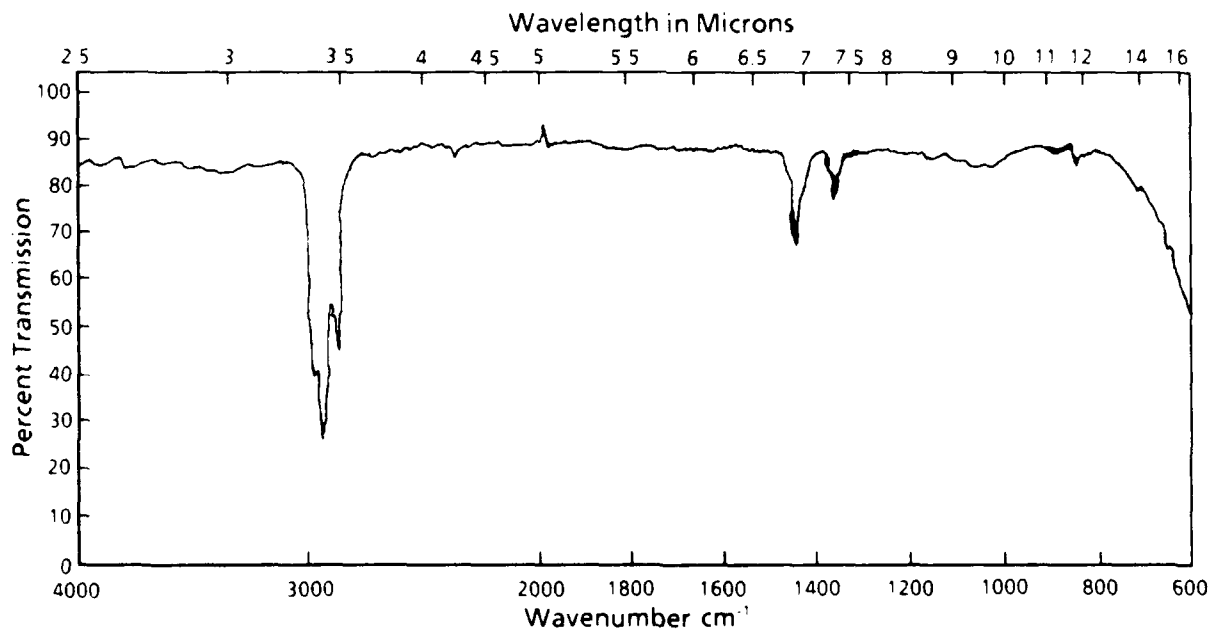


Figure 8. IR Spectrum of Aerosol Sampled from the High Concentration Exposure Chamber.

Continuous monitoring (1425 to 1450 samples per day) and periodic adjustment of generator carrier air flow minimized the variability of weekly mean aerosol concentrations. The coefficient of variation (CV) for the aerosol concentrations in the low and high level exposures were 2.3 and 1.4%, respectively. Aerosol size distributions varied slightly over the duration of the study. The CVs of the MMADs and σ_g s of the low and high concentration exposures were 5.1 and 4.2% (MMAD) and 2.1 and 1.9% (σ_g), respectively. Despite the low aerosol concentration variability over the course of the exposures, and periodic adjustments of generator output toward target concentrations, distinct cyclic generator performance patterns were disclosed when aerosol concentration and size distribution were examined as a function of time after replacement of the generator reservoir fluid with fresh HS 5047F. As noted previously, during the exposure studies virgin HS 5047F was placed into the generator reservoirs every Monday, Wednesday, and Friday morning. Aerosol concentrations for both the low and high level exposures peaked on these mornings and were followed by gradual decay of chamber concentration until the next reservoir fluid replacement (Figures 9a and 9b). Regardless of repeated daily adjustments of generator output, the effect of replacing generator fluid increased average exposure chamber concentration from the 12-h period prior to fluid replacement to the period immediately following replacement by as much as 32% (average concentrations for these periods over the 13 weeks of exposure). Accompanying these concentration peaks were corresponding increases in aerosol MMAD for both the low and high concentrations (Figures 10a and 10b, respectively) by as much as 0.4 μm and corresponding decreases in the number of aerosol droplets per cm^3 of sample (Figures 11a and 11b) by as much as 42%.

Ethylene glycol concentration in the low concentration exposure chamber was below detectable levels, whereas that in the high concentration exposure chamber was slightly above the minimum detectable limit. The average (by week) ethylene glycol concentration in the high concentration exposure chamber ranged from 217 to 334 $\mu\text{g}/\text{m}^3$ for an overall average concentration of $266 \pm 41.9 \mu\text{g}/\text{m}^3$. Ethylene glycol concentration steadily diminished over the first seven weeks of exposure before stabilizing at the lower end of the concentration range (Figure 12). Ethylene glycol concentration in the exposure chamber also was cyclic with peak concentrations occurring on days when fresh material was placed in the generators (Figure 13).

Based on wet/dry bulb determination of RH, there was a significant contribution ($p \leq 0.05$) to water vapor content in the high concentration exposure chamber as a result of aerosolization of HS 5047F. Daily mean RH (based on 1425 to 1450 measurements) in the high concentration, low concentration, and control exposure chambers were 62.0 ± 3.8 , 57.1 ± 7.3 , and $55.9 \pm 6.3\%$, respectively.

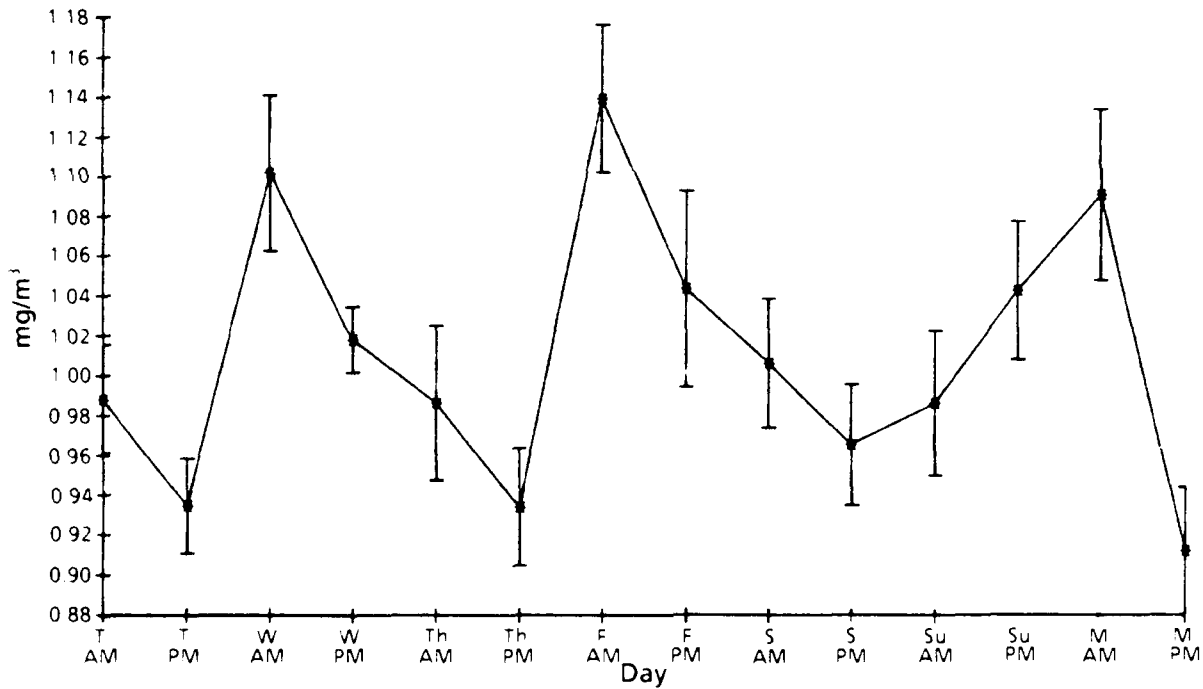
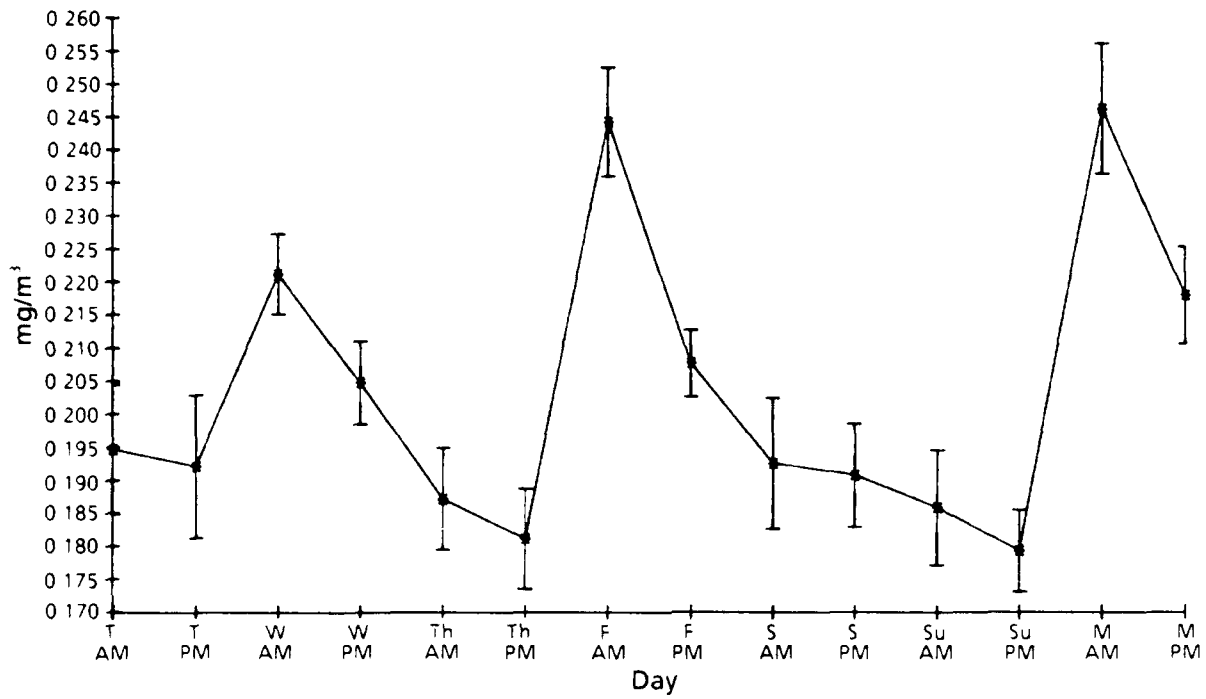


Figure 9 a, b. Mean Aerosol Concentration, by Day of the Week, of the Low and High Concentration Exposure Atmospheres.

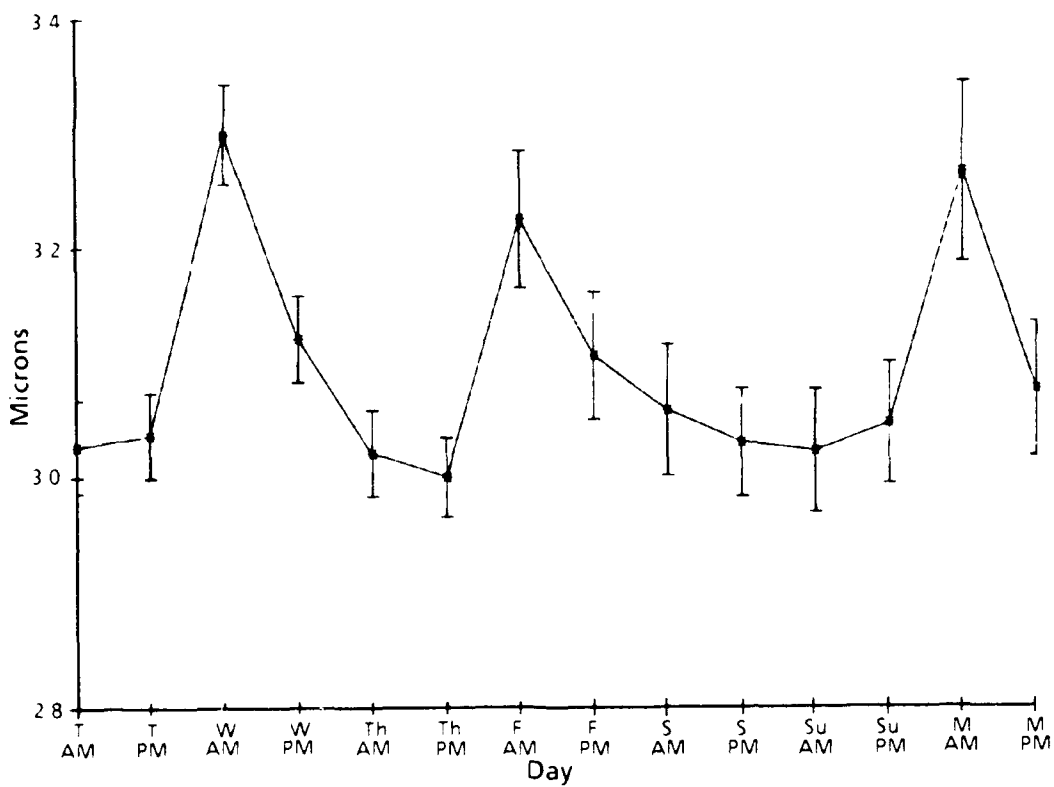
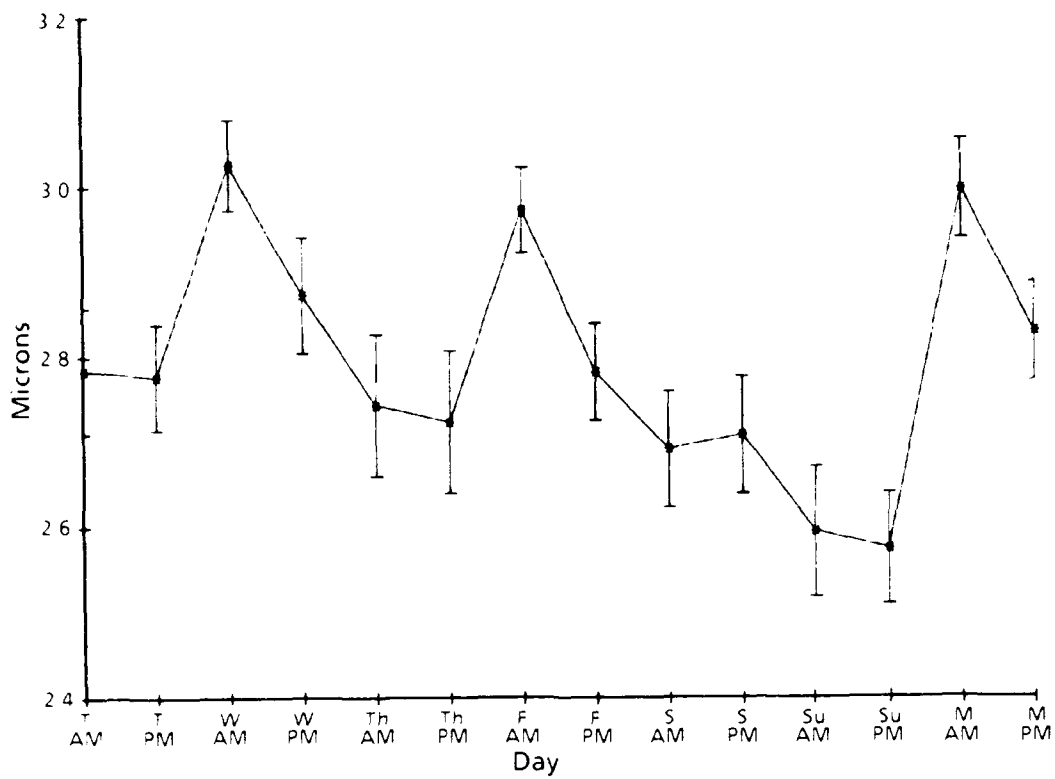


Figure 10 a, b. Mean Aerosol Mass Median Aerodynamic Diameter, by Day of the Week, of the Low and High Concentration Exposure Atmospheres.

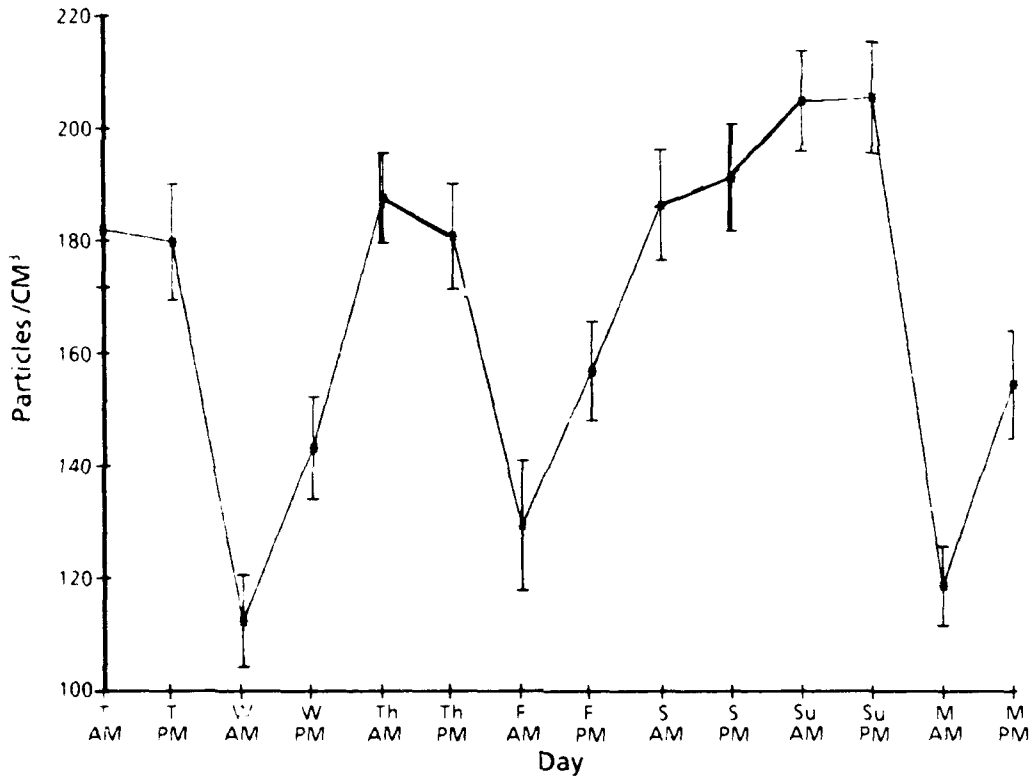
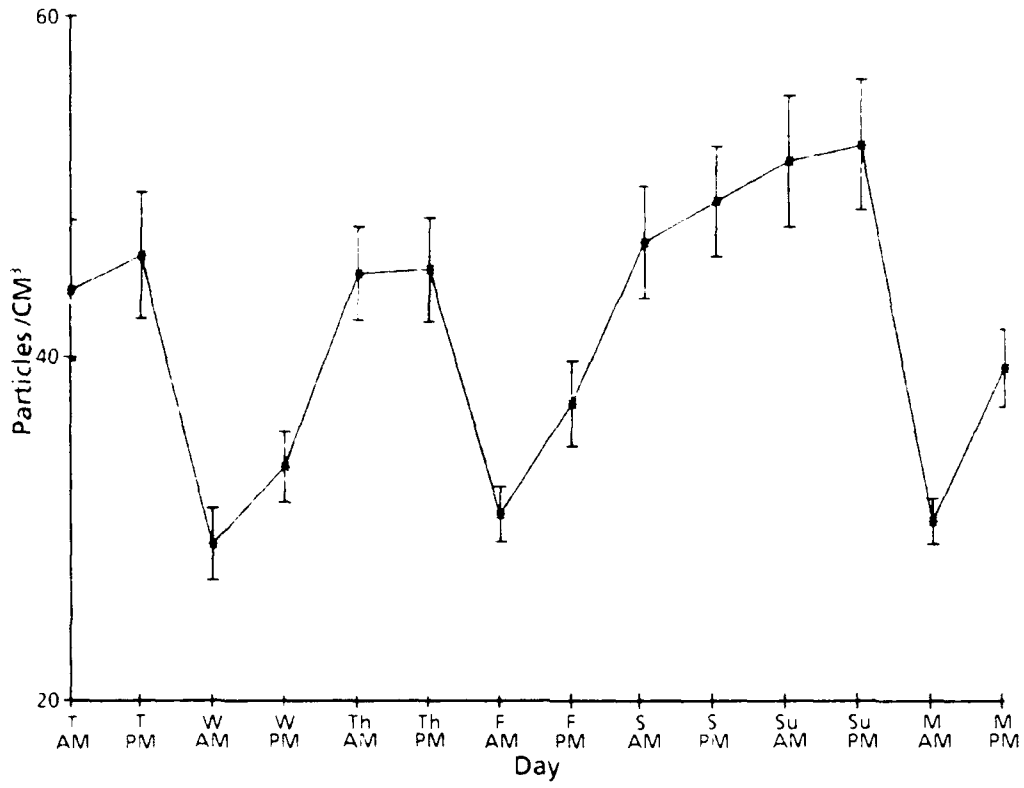


Figure 11 a, b. Mean Aerosol Particle Count per cm³, by Day of the Week, of the Low and High Concentration Exposure Atmospheres.

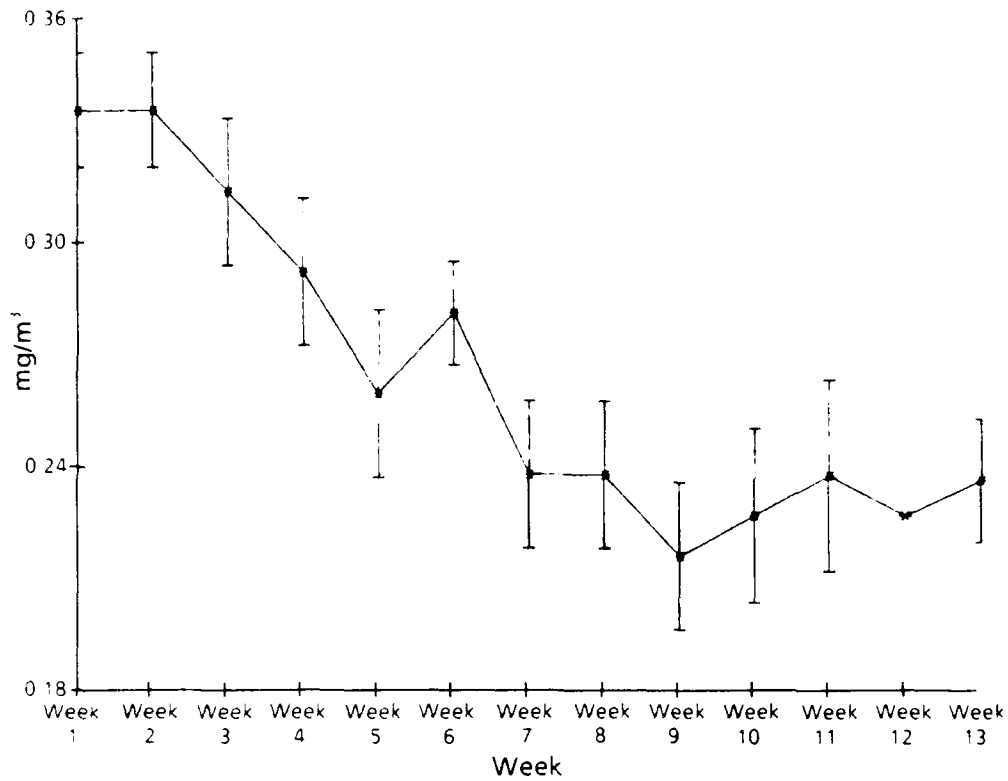


Figure 12. Weekly Mean Ethylene Glycol Concentration of the High Concentration Exposure Atmosphere.

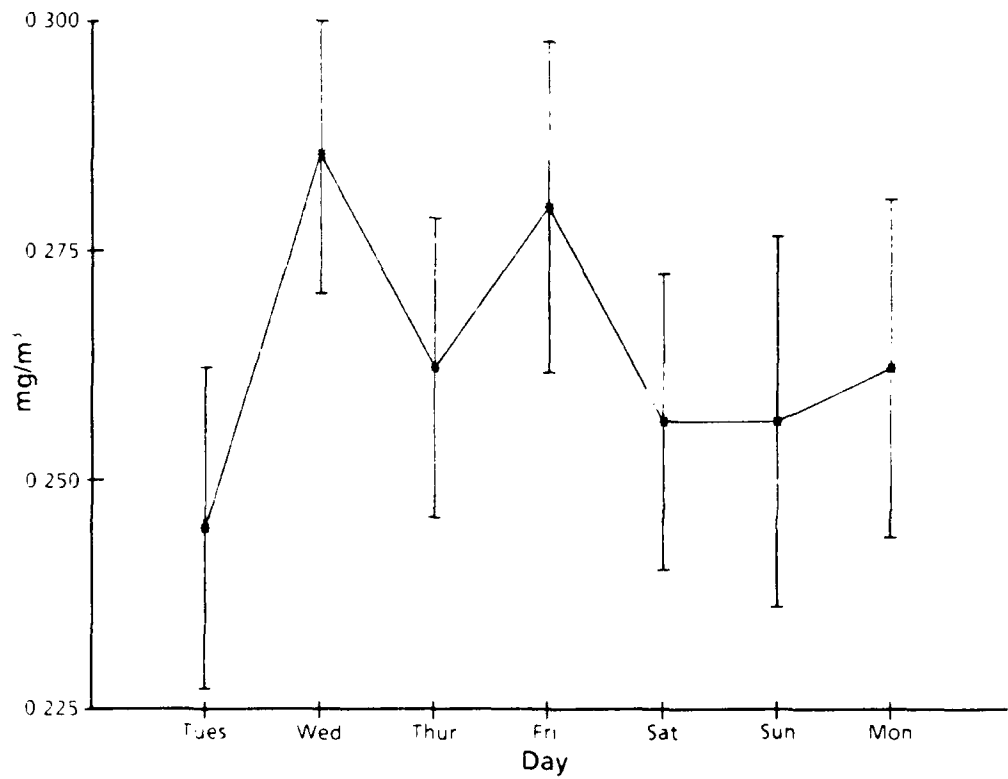


Figure 13. Mean Ethylene Glycol Concentration, by Day of the Week, of the High Concentration Exposure Atmosphere.

SECTION 4

DISCUSSION

Comparable to conventional compressed air nebulizers currently in use to generate respirable aerosols for inhalation studies, several factors govern the operating and performance characteristics of pressure nozzle atomizers, including the high pressure aerosol generator described herein. One of the most, if not the most, critical of these factors is nozzle orifice diameter. Although not all the dynamics of droplet formation from orifices are well understood, some mechanisms of droplet formation have been described mathematically, and these descriptions have been applied to the gross prediction of droplet size as a function of orifice diameter. The equations also serve as a basis for assessing nozzle performance for use in novel applications and with developmental materials. Simple Rayleigh fractionation of a fluid jet from an orifice has been shown to occur at nodes in the jet stream spaced at intervals which are 4.5 times the jet diameter. Separation of the jet stream at these nodes results in the formation of droplets with diameters 1.89 times the initial jet (orifice) diameter. However, the reliability of this simple description of the relationship between droplet size and orifice diameter diminishes with increasing viscosity of the fluid. More viscous materials form larger droplets than Rayleigh fractionation predicts. Thus, for high viscosity fluids, Weber's equation (given below) which accounts for the physical properties of the fluid, is used to calculate the diameter of droplets formed by simple orifices.

$$D_{mmd} = 1.89D_j [1 + 3\mu_l/(\sigma\rho_l D_j)]$$

where: D_{mmd} = droplet mass median diameter

D_j = jet (orifice) diameter

μ_l = viscosity of the liquid

σ = surface tension of the liquid

ρ_l = density of the liquid.

Given a 2.03×10^{-4} m orifice diameter of a typical impingement nozzle used in the present generator and the physical characteristics of HS 5047F (see Table 1), the D_{mmd} was calculated to be 419 μm , which was well beyond respirable size range. However, individual droplets formed from a liquid jet undergo additional fractionation when suspended in a gas stream proportionate to the relative velocities of the gas and liquid streams. This droplet breakup leads to a large reduction in D_{mmd} accompanied by an increase in the polydispersity of the aerosol. The Wolfe-Andersen

equation, shown below, quantitates so called "bag" and "stripping" mechanisms, by which droplet breakup occurs (Steinmeyer, 1984).

$$D_{mmd} = [(136\mu_l\sigma^{3/2}D_o^{1/2})/(\rho_g^2\rho_l^{1/2}U^4)]^{1/3}$$

where: D_{mmd} , μ_l , σ , and ρ_l are previously defined

D_o = original droplet diameter

ρ_g = density of the gas (air = 1.29 kg/m³)

U = relative velocity of the liquid and gas flows,

or v_l/v_g .

v_l = liquid velocity

v_g = gas velocity.

Given a typical nozzle liquid throughput of 240 mL/min (at 1000 psi) and a typical nozzle diameter, v_l was calculated to be 1.23×10^4 cm/s. Carrier air flow through the elutriator cylinder accelerated upon approaching discharge through the exit port, thus resulting in variable flow velocity in the generator elutriator volume. Thus, v_g in the generator was calculated as a mean velocity in the elutriator based on the flow (8 L/min) and the mean of the cross-sectional areas of the elutriator cylinder body and the exit port; and was determined to be 1.01×10^1 cm/s. For the values of v_l and v_g given above, U of the system was calculated to be 1.22×10^3 . Thus, for a D_o of 419 μ m, the new D_{mmd} was 75.4 μ m. Further reduction in droplet size resulted from impingement on surfaces. Although no quantitative guidelines exist for impingement breakup, reduction of droplet size by factors of 10 and greater after impingement have been demonstrated (Steinmeyer, 1984). Therefore, based on a conservative estimate of a factor of 10 reduction due to impingement, the theoretical D_{mmd} of HS 5047F aerosols produced by a typical nozzle in the generator system described was 7.5 μ m. A 7.5 μ m D_{mmd} corresponds to a MMAD 7.8 μ m for HS 5047F aerosols or roughly twice that observed during generator development tests. The twofold difference between predicted and observed MMAD could not readily be accounted for by any single factor. Overestimation of predicted droplet size because of an underestimation of impingement fractionation of the droplets was likely due to the highly empirical nature of impingement breakup factors. Secondary impingement on the elutriator walls of droplets previously shattered by the nozzle impinger also may have further reduced droplet size. Underestimation of the size of droplets produced by the nozzle because of disproportional entrainment of smaller droplets in the generator carrier air stream was possible, but not likely a principal contributing factor to the discrepancy between observed and predicted MMAD. An eightfold increase in generator carrier air flow did not result in a corresponding increase in aerosol size or significant change in aerosol distribution (Table 2). Regardless of the causes of the

discrepancy, a twofold difference between the observed and predicted droplet size was not considered inordinately large considering the empirical determination of impingement fractionation of droplets.

During development testing the aerosol mass output of the generator was found to be directly proportional to both the hydraulic fluid pressure and the carrier air flow through the elutriator; the latter having relatively more influence on the aerosol mass delivered to the test chamber. The effect of increasing fluid pressure on a simple pressure nozzle should reduce droplet size by the following relationship (Steinmeyer, 1984).

$$D_1/D_2 = (P_2/P_1)^{1/3}$$

where: D_1 = droplet diameter at P_1

D_2 = droplet diameter at P_2

P_1 = original nozzle pressure

P_2 = increased nozzle pressure.

Theoretically, an increase in nozzle pressure from 500 to 1500 psi should decrease droplet size from 4.2 to 2.9 μm , however, a decrease in droplet size this large was not observed in the present experiment. The effect of increasing nozzle pressure from 500 to 1500 psi was a reduction of droplet size from 4.2 to 3.8 μm , about 36% of the predicted reduction. However, as fluid extruded through the nozzle there was a concurrent proportional drop in nozzle fluid pressure initiating a pump cycle to return nozzle fluid pressure to maximum. Therefore, fluid pressure in the nozzle was not constant but cyclical with maximum nozzle pressure occurring intermittently at regular intervals. Because nozzle pressure was not constant, the effect of increasing maximum nozzle pressure on droplet size was diminished.

The periodic oscillation of aerosol droplet size and number concentration observed in the inhalation exposure atmospheres was related to reloading of the generator reservoir with fresh HS 5047F. When loaded with fresh material the generator initially produced larger and fewer aerosol droplets (Figures 10a, 10b, 11a, and 11b). Typically, the generator nozzle atomized 240 mL/min HS 5047F, of which 56.5% (see below) was mineral oil (aerosol droplets were found to be virtually pure mineral oil - density = 0.875). If all of this material had been delivered to an average exposure chamber flow of 1.42 m^3/min , chamber aerosol concentration would have been $8.36 \times 10^4 \text{ mg}/\text{m}^3$. The excess aerosol mass not delivered to the chamber was collected in the generator elutriator, returned to the generator reservoir, and recycled through the nozzle. The recycling process resulted in the disproportionate removal (via carrier flow) of the more volatile constituents of atomized HS 5047F which gradually altered the proportion of HS 5047F emulsion constituents in the generator

reservoir. Figure 14 illustrates the change in the proportion of volatile HS 5047F constituents after they had been cycled through the generator for 48 h. Comparison of time course curves of material loss, due to evaporation from freestanding fresh and recycled HS 5047F, demonstrated that evaporative loss from recycled HS 5047F was 10% less than evaporative loss from fresh HS 5047F. This indicated that the recycled material initially had a lower fraction of volatile constituents due to disproportionate loss of these constituents during the aerosol generation process. Comparison of material loss from both fresh and recycled HS 5047F with that from pure mineral oil demonstrated that virtually all bulk loss from both types of HS 5047F was due to evaporation of the ethylene glycol and water constituents. Alteration of the proportion of volatile and nonvolatile constituents led to a gradual change of the physical characteristics of HS 5047F in the generator reservoir, resulting in a progressive production of a larger number concentration of smaller aerosol droplets. Either a decrease of the μ_1 , a decrease of the σ , an increase of the ρ of reservoir HS 5047F, or a combination of all three phenomena may have caused the production of smaller aerosol droplets.

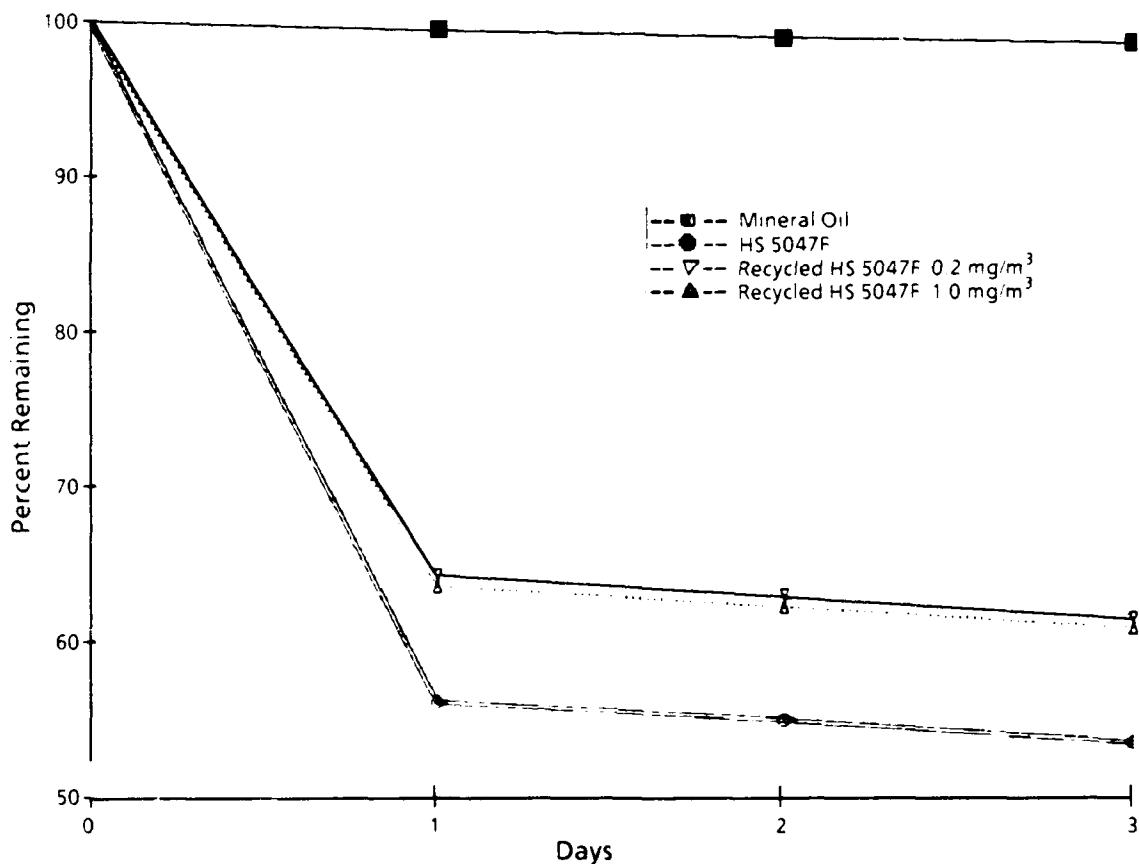


Figure 14. Material Loss Due to Evaporation of Pure Mineral Oil, Neat HS 5047F, HS 5047F used to Generate Low Concentration Atmospheres, and HS 5047F used to Generate High Concentration Exposure Atmospheres.

Exposure chamber concentrations of the volatile constituents of HS 5047F were relatively low. In the low concentration chamber ethylene glycol was not detectable and there was not a significant difference between the water vapor concentration in the low and control chambers. Therefore, the amount of water vapor in the low concentration chamber attributable to HS 5047F aerosol generation could not be determined. Very low levels of ethylene glycol were detected in the high concentration chamber and approximately 10% (6.1 of 62% RH) of the total water vapor density in the chamber could be attributed to HS 5047F aerosol generation. The average ethylene glycol concentration in the chamber was 0.27 mg/m³. Reasons for the gradual decay of the exposure atmosphere ethylene glycol concentration over the course of the exposures are unknown (all HS 5047F used had the same manufacturer's lot number and was stored under identical conditions). A significant difference in mean RH of 6.1% between the control and high concentration exposure atmospheres was attributed to water vapor from the aerosol generation process. At an average local barometric pressure of 740 mmHg and chamber temperature of 23.9 °C, 6.1% RH corresponds to a water vapor density of 1.42 mg/m³ (Weiderhold, 1981; Bagnoli, 1984). Based on comparison of the evaporation rate constants of pure mineral oil and HS 5047F the apparent mineral oil fraction of fresh HS 5047F used was ≈ 56.5%, by weight (see Figure 14). Ethylene glycol content of HS 5047F was ≈ 3.2% by weight, therefore, the constituent proportion of mineral oil:water:ethylene glycol in the HS 5047F used was ≈ 56.5:40.3:3.2. Given nozzle fluid flow, 96.7 g/min water was atomized. At 23.9 °C the vapor pressure of water is 22.4 mmHg, thus the mole fraction of water in the vapor phase was 2.85 g/min. At an average chamber air flow of 1.42 m³/min the water vapor density in the chamber atmosphere attributable to HS 5047F aerosol generation was predicted to be 2.01 g/m³, which was 1.4 times the measured water vapor density in the chamber atmosphere. Given identical generation system operating conditions, chamber temperature and barometric pressure, density of 1.109 and a calculated vapor pressure of 0.11 mmHg (Clausius-Claperyon method - Levine, 1988) for ethylene glycol, the predicted vapor phase mole fraction of ethylene glycol transported to the chamber was 1.27 mg/m³. Consequently, the actual concentration in the exposure atmosphere was 4.7 times less than predicted. The tendency towards decay of chamber concentration of ethylene glycol concentration in the exposure atmosphere over time demonstrates that the discrepancy between measured and predicted atmospheric ethylene glycol concentration was not due to passivation of the exposure system.

SECTION 5

CONCLUSION

Simple pressure nozzle technology can be applied to the generation of respirable size aerosols for inhalation studies of relatively viscous materials when coupled with a device which creates high fluid pressure drops across the nozzle orifice. High aerosol mass concentrations in very large exposure chambers can readily be obtained and maintained on a continuous basis for long periods of time. Forcing viscous fluids under high pressure through small orifices to create unstable fluid jets that break up due to intrinsic physical forces, as opposed to using extrinsic forces, such as high velocity gas jets, as an external force to create aerosol droplets from fluid streams minimizes excessive volatilization of test material fluids as may occur with conventional aerosol generation techniques such as nebulization. This is demonstrated by the finding that the aerosol generation process in the present investigation did not produce vapor concentrations of the volatile components of HS 5047F in excess of those expected from passive, static ambient evaporation. As with nebulization processes, care must be taken to assure that reflux of test material into the generation fluid reservoir does not significantly alter the composition of the material in the reservoir. This investigation suggests that empirical descriptions of droplet formation from nozzles may be useful for predicting, within limits, the size of the droplets formed by a simple nozzle when a few physicochemical characteristics of the fluid under examination are known and if a better understanding of shattering of droplets due to impingement can be developed.

SECTION 6

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QUALITY ASSURANCE

The study, "A High Pressure Aerosol Generator for Viscous Fluids," was conducted by the ManTech Environmental Technology, Inc., Toxic Hazards Research Unit under the guidance of the Environmental Protection Agency's Good Laboratory Practices Guidelines, 40CFR PART 792. No claim will be made that this was a "GLP" study as no attempt was made to adhere to the strict requirements of these guidelines. The various phases of this study were inspected by members of the Quality Assurance Unit. Results of these inspections were reported directly to the Study Director at the close of each inspection.

DATE OF INSPECTION:

June 3-7, 1991

ITEM INSPECTED:

Final report data audit

The Quality Assurance Unit has determined by review process that this report accurately describes those methods and standard operating procedures required by the protocol and that the reported results accurately reflect the raw data obtained during the course of the study. No discrepancies were found that would alter the interpretation presented in this Final Report.

M. G. Schneider

M. G. Schneider
QA Coordinator
Toxic Hazards Research Unit

Date *July 9, 1991*