

PHYSICAL AND CHEMICAL CHARACTERISTICS OF SFE FIRE SUPPRESSANT ATMOSPHERES: COMPARISON OF SMALL WITH LARGE SCALE LABORATORY ATMOSPHERES.

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INTRODUCTION

SFE is a fine, dry powder aerosol fire extinguishant which is currently undergoing evaluation as a possible replacement for halon 1301 total flooding agent used in some Naval systems. The results of an investigation conducted at the Naval Research Laboratory / Chesapeake Bay Detachment (NRL/CBD) of SFE fire extinguishment efficacy have been reported (1). However, comprehensive evaluation of any possible halon replacement candidate, including SFE, requires assessment of it's potential inhalation toxicity. Findings of a pilot investigation of **SFE** (SFE formulation A) inhalation toxicity (2) conducted at the Naval Medical Research Institute Detachment: Toxicology (NMRI/TD) demonstrated a need for further investigation of potential toxicity, as well as, a more complete characterization of SFE atmospheres as deployed in field testing conditions.

SFE aerosols are pyrotechnically generated by ignition of the bulk **SFE**. SFE pyrolysis is self perpetuating and produces transient pressure and thermal pulses which could feasibly obfuscate inhalation toxicity evaluations. Hence, for toxicity evaluations a system was designed in which the SFE could be ignited in one portion of the system for rapid dissipation of the pressure and thermal pulse prior to transport of the atmosphere produced into a standard inhalation exposure chamber (3). This procedure differs from the fire extinguishment testing scenario used at NRL/CBD in which **SFE** is ignited within the test chamber itself and which more closely simulates present deployment methods and conditions. Although the basic pyrotechnical methods of SFE aerosol generation are ~~the same~~ between the two test conditions, some fundamental system differences suggested the need for a more extensive characterization of the aerosol and gas/vapor phases of the both test atmospheres. The present investigation was undertaken to facilitate extrapolation of results between toxicity testing and fire extinguishment testing atmospheres.

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METHODS

SFE

The SFE (Formulation A) tested in both systems was obtained from the Spectrex, Inc., the U.S. subsidiary of an Israeli company Spectronix, Ltd. The composition of SFE is proprietary. Experiments in both systems were conducted at two nominal concentrations in which either 50 or 80 g of test material per m³ of chamber volume was ignited. The volume of the NRL/CBD facility has been reported at 56 m³ and the volume of the inhalation exposure chamber and system used at NMRI/TD was 0.7 m³.

Atmosphere (aerosol) Generation

The aerosol generators used in the 56 m³ chamber test system have been previously described in detail (1). The aerosol generator used in the 0.7 m³ test system was manufactured out of two sections of 3 in. diameter schedule 80 stainless steel pipe [Figure 1 - (4)]. The lower section (h \cong 7.6 cm) was flanged and bolted to an upper flanged section of pipe (h \cong 20.3 cm). A stainless steel fritted metal plate (0.3 cm thick) was fitted between the flanges. The lower section included an 1/8 in. compression fitting through which metered air flow was introduced to facilitate transport the SFE combustion atmosphere to the exposure chamber. The upper section of the generator was fitted with 1/16th in. and 1 in. compression fittings through which a thermocouple and an insulator/ electrode assembly were secured. A 5 cm diameter ceramic boat was placed in the bottom of the upper plenum (on the fritted plate) in which nichrome wire (connected to the electrodes) was coiled. Weighed portions of 100 g SFE pellets were placed directly on the coils. The top of the generator/igniter assembly was a portion of 3 in. flange coupler welded to the pipe to accept a threaded adapter to 3 in. diameter aluminum duct for transport to the exposure chamber (total length \cong 2.5 m). The SFE was by application of an 18 v, 6 amp current through a length of 26 AWG nichrome with 3 ohm resistance (\cong 28 cm) which produced the \cong 500°C temperature required to start pyrolysis of the SFE. The 3 in. duct (\cong 2.5 m) also served to dissipate the thermal energy. Generator temperatures reached 1100 \pm 100°C yet chamber temperatures remained at 22 to 24 °C.

Test Chambers

The 0.7 m³ inhalation exposure chamber was not operated in the mode routinely employed for toxicity experiments. Inhalation exposure chambers normally are run as dynamic (continuous flow) systems with the test atmosphere being generated and mixed continuously with the chamber flow. However, for single event generation such as combustion of SFE a static (non-flowing) operation mode was used. The SFE atmospheres were generated and piped into the exposure chamber during dynamic mode operation; once transport of the test atmosphere was complete the chamber was switched to the static mode via automatic shut off valves at the inlet and exhaust ports. A sight glass fitted into the chamber inlet duct enabled visual determination of cessation SFE pyrolysis. Automated control of system flow and pressure differential assisted chamber filling with no or inconsequential loss of test atmosphere through the chamber exhaust during the dynamic operation/generation phase, while providing control of the generation pressure pulse as

well. Generator and chamber flow during ignition and filling was 30 - 31 LPM and chamber filling time ranged from 0.75 to 1.0 min. at which point static operation was initiated (Figure 1).

Aerosol Analysis

Analytical methods for characterization of the aerosol component of the SFE atmospheres were identical for both test systems. Aerosol mass concentration was determined by gravimetric analysis of particles collected on 37 mm diameter glass fiber filters placed in holders specifically designed for aerosol sampling (Intox Products, Albuquerque, NM) through which a known volume of test atmosphere was drawn. Sample flow was measured and controlled with variable area rotameters calibrated to NIST traceable standards. Sample volume was controlled by electronic timing of solenoid valves in the sample lines. Flows and subsequently sample volumes were adjusted for changes in pressure differential across the sampling device. In the 0.7 m³ chamber samples were drawn through short sample probes (1.2 cm dia) penetrating the chamber wall. Sample velocity and probe design eliminated sample probe aerosol deposition. In the large chamber, the filter holders were located directly within the chamber at the end of separate vacuum lines. Rotameters, solenoids, and electronic timers were used for precision control of sample flow and volume. Filter samples were collected at 1, 15, 30, and 60 min. in the 0.7 m³ chamber and at 1.5, 15, 30 and 60 min. in the 56 m³ chamber. In the small chamber volumes of air equivalent to the sample volume were injected in the chamber exhaust plenum to prevent depressurization of the exposure chamber. Reported concentrations have been adjusted for loss of mass concentration due to previous sample collection.

Mass Median Aerodynamic Diameter (MMAD) and the geometric standard deviation (σ_g) of the aerosol distribution were determined by two methods. Multistage (8 stages), multijet cascade impactors (Intox Products, Albuquerque, NM) were used to collect samples for gravimetric analysis at time points corresponding to filter sample collections. Sample flows and pressure differentials were controlled in a manner similar to filter samples. In the large chamber the impactors also were located with the chamber at the terminus of individual vacuum lines. Aerosol size distribution characteristics also were determined using a laser, time of flight, individual particle analyzer (Model 3300B Aerodynamic Particle Sizer, TSI, Inc., St. Paul, MN). The APS was capable of separating the particle distribution into 58 individual size ranges from 0.47 to 30.0 μm diameter particles, thus allowing for a much greater resolution of the particle size distribution. APS samples were taken at 1 or 1.5, 15, 30, 45, and 60 min. MMAD and σ_g determinations were based on impactor samples whereas changes in aerosol distribution shape and modality were based on APS samples.

Gas Analysis

CO and CO₂ concentrations were determined in both the 0.7 m³ and 56 m³ chambers. Chromatographic methods were used to analyze for CO, at the NRL/CBD test site (56 m³ chamber) and have been described previously (1). Carbon monoxide concentration in the 56 m³ chamber was measured continuously by wavelength-specific, non-dispersive IR spectrometry (Enviromax 3000, Liston Scientific, Irvine, CA). Wave-length-specific, non-dispersive IR spectrometry was used for both CO and CO₂ in the 0.7 m³ chamber (Model 865 Beckman

Industries, La Habra, CA). The latter samples were collected at times corresponding to APS samples. Electro-chemical measurement of O_3 concentration in the 56 m^3 chamber was continuous (Enviromax 3000) and on a **grab** sample basis in the 0.7 m^3 chamber (Model 326RA, Teledyne Analytical Instruments, City of Industry, CA).

Particle morphology

Electrostatic precipitation was used to collect particles for examination by scanning and transmission electron microscopy to determine aerosol particle morphology and composition by x-ray diffraction.

Experimental Design

All test periods were 60 minutes. The test schedule was **as** follows:

0.7 m^3 chamber @ 50 g/m^3 - 3 trials
 0.7 m^3 chamber @ 80 g/m^3 - 4 trials
 56 m^3 chamber @ 50 g/m^3 - 3 trials
 56 m^3 chamber @ 80 g/m^3 - 2 trials

RESULTS

Spatial Distribution - 56 m^3 chamber

The 0.7 m^3 inhalation exposure chamber is specifically designed for mixing and even distribution of aerosols within the chamber exposure volume. In the large chamber, mixing and dispersal of the aerosol was accomplished by using a fan during first **1.5 min.** after ignition of the SFE. A single trial was made at 50 g/m^3 to determine spatial variation within the chamber of aerosol concentration, size distribution and CO_2 concentration. Measurements were made at 1.5 and 60 min. after ignition of SFE at heights in the chamber of 1, 3, 6 and 11 R. (Figure 2). A maximum difference in initial actual aerosol **mass** concentration of 10.1 to 11.4 g/m^3 at the 1 and 11 ft levels respectively was observed with virtually no difference between any of the levels at 60 min. Initial particle MMAD's were 2.9 or $3.0\text{ }\mu\text{m}$ at **all** levels. At 60 min. particle size ranged **from** a low of $4.4\text{ }\mu\text{m}$ MMAD at the 6 ft level a high of $5.2\text{ }\mu\text{m}$ MMAD at 1 R.. Both the initial and final CO_2 concentration varied by **as** much **as** 3 g/m^3 between levels, from 26.5 to 23.4 g/m^3 initially to 11.2 to 13.9 g/m^3 after 60 min.

Aerosol Mass Concentration

Initial, actual aerosol **mass** concentration for both target loads, 50 and 80 g/m^3 , was higher in 56 m^3 chamber at 10.2 and 14 g/m^3 respectively vs. 6.3 and 10.0 g/m^3 respectively for the 0.7 m^3 chamber. Gravitational settling of particles throughout the test period resulted in final concentrations of 1.7 and 1.2 g/m^3 in the 50 and 80 g/m^3 loads in the 56 m^3 chamber. Final aerosol concentrations in the 0.7 m^3 chamber were 0.7 g/m^3 for both loads (Figure 3). In both chambers and for both target loads decay of aerosol concentration was exponential. Shorter

half-times ($T_{1/2}$) of 15.5 and 16.1 min. were found for the higher target load for the 0.7 and 56 m³ chambers respectively. $T_{1/2}$ for the low target load was 18.6 and 22.4 min. respectively.

Aerosol Size Distribution and Particle Growth

The initial MMAD's ranged from 2.4 to 3.0 μm for the 56 m³ chamber - 80 g/m³ load and 0.7 m³ chamber - 80 g/m³ load respectively. The σ_g 's for the particle distributions ranged from 1.42 to 2.01 at various times, however average σ_g 's were 1.66 to 1.88. Particle growth occurred in both chambers with maximum MMAD being reached at the following times:

- 0.7 m³ - 50 g/m³ - 30 min.
- 0.7 m³ - 80 g/m³ - 15 min.
- 56 m³ - 50 g/m³ - 60 min.
- 56 m³ - 80 g/m³ - 30 min.

Growth was more rapid for the higher target load in both chambers and apparently much more rapid in the 56 m³ chamber (Figure 4). Initially the particle distributions were bimodal with a major mode occurring near the MMAD and minor mode ranging from 4 to 17 μm . However, due to gravitational settling the minor mode disappeared after 15 min. in nearly all distributions (Figure 5a and 5b).

CO and CO₂ Concentration

CO₂ concentrations in the 56 m³ chamber were much higher at both target loads than corresponding concentrations in the 0.7 m³ chamber. The large chamber was not a sealed containment vessel, as was the inhalation exposure chamber, thus gas concentration decayed over time due to chamber leak. Initial CO₂ concentrations for the 50 and 80 g/m³ loads were 14,475 and 22,200 ppm (26.5 and 40.6 g/m³) respectively. Final CO₂ concentrations for the large chamber were 7,100 and 9,500 (13 and 17.4 g/m³) respectively (Figure 6). CO₂ concentrations in the 0.7 m³ chamber were steady state at 10,230 to 10,105 ppm for the 50 g/m³ target load. CO₂ concentrations for the 80 g/m³ load in the 0.7 m³ chamber were 9,457 ppm initially to 9,376 ppm at 60 min. However, two distinct CO₂ concentration levels were noted in the small chamber trials at the 80 g/m³ load, with two trials averaging 12,548 to 12,033 ppm CO₂, and two trials averaging 6,366 to 6,720 ppm CO₂, (Figure 6). A corresponding difference in CO concentration under these conditions was not observed, *vide infra*. Small chamber initial and final CO concentrations were 2,168 and 2,538 ppm (1.9 to 2.9 g/m³) at the 50 g/m³ load. At the 80 g/m³ load initial and final CO concentrations were 6,548 and 6,360 ppm respectively. In the 56 m³ chamber long sampling lines and a large dead space (in-line filter assembly required to remove particles from the sample) in the CO analysis system lead to large sample lag time (\approx 30 min.). Consequently, peak CO concentrations were not seen until the 30 min. sample point. Only moderate concentration decay was observed, which may have been sampling artifact. Peak CO concentrations in the 56 m³ chamber were, for the 50 and 80 g/m³ loads respectively, 2,992 and 4,810 ppm. O₂ concentration in the 56 m³ varied from 19.1 to 19.9% and ranged from 20.1 to 20.6% in the 0.7 m³ chamber.

DISCUSSION

Systems Mass Balance and Comparative Efficiencies

Mass balance calculations, based on peak concentrations, (Figures 7a & 7b) indicate that in the 0.7m^3 chamber test system **10.7** and **23.4 %** of the **mass** of SFE ignited, for the **50** and **80 g/m³** loads, remained unaccounted for by the aerosol, CO, and CO₂ yields. Unaccounted for mass in the **56 m³** chamber system corresponded to **24.8** and **25 %** of the SFE ignited for the **50** and **80 g/m³** loads respectively. Thus, irrespective of target load the **0.7m³** system used bulk SFE more efficiently than its **56 m³** counterpart. However, with respect to aerosol generation **12.3** and **12.4%** of the SFE mass was aerosolized at the **50** and **80 g/m³** loads in the **0.7m³** chamber; whereas aerosolization was **19** and **17.4%** of the SFE **mass** at the **50** and **80 g/m³** loads in the **56 m³** chamber. The average ratio (both target loads) of aerosol formation efficiency the chambers was **56:0.7 m³ = 1.5:1**. Hence the large system was **50 %** more efficient at aerosol dispersal if not formation. A slightly greater overall efficiency for pyrolysis of SFE for the **0.7m³** chamber system suggested that transport loss of aerosol and not aerosol production was the cause for the observed difference in aerosol dispersement efficiency. CO₂ concentration in the **0.7m³** system was greater at the lower target load than that of the higher target load. At the **80 g/m³** level two varieties of SFE (both marked formulation A - from the supplier) may have been used. It was observed that the atmosphere produced by the material yielding **0.5** of the expected CO₂ also deposited very **sooty** residue (similar to that seen with ignition of SFE formulation C). Consequently the mass percentages of CO₂ yield were not **similar**, at **38.2** and **21.8%** for the **50** and **80 g/m³** loads respectively. Yield of CO₂ (using peak concentrations) in the **56 m³** system were similar at **49.6** and **50.6%** for the **50** and **80 g/m³** loads. The relative CO₂ production efficiencies for the systems were **56:0.7 m³ chambers = 1.3** at **50 g/m³** load and **2.3** at the **80 g/m³** load. Thus at the lower target load the **56 m³** system produces **30 %** to **130%** more CO₂, per **mass** of SFE ignited than does the **0.7m³** system depending on target load. CO₂ production accounts for **5.9** and **9.5%** of the SFE mass used in the **56 m³** system at **50** and **80 g/m³** loads. CO₂ yield in the **0.7m³** system was **6.5** and **6.9%** for the **50** and **80 g/m³** loads respectively. Therefore CO₂ production efficiency ratio for the systems was **56:0.7 = 0.8** at **50 g/m³** and **1.5** at **80 g/m³** targets. Differences of Carbon combustion efficiency between the generation systems was responsible for the discrepancy in relative CO₂ and CO₂ concentration between the test systems.

Aerosol Growth

For aerosols with $\cong 3.0\ \mu\text{m}$ MMAD's and σ_g 's of **1.7** the **high** initial mass concentrations observed in the study of **14, 10.2, 10.0** and **6.3 g/m³** [(assuming a particle density of **2.0 g/cm³** for SFE - (4)] the corresponding particle number concentrations were **1.78x 10⁶**, **1.30x 10⁶**, **1.27x 10⁶** and **0.8 x 10⁶ particles/cm³**. Aerosols with a number concentration of **1.0 x 10⁶ particles/cm³** have a number concentration T_n of $\cong 33$ **min.** due to coagulation and agglomeration of particles, without loss of total **mass** in the particle distribution (5). Therefore, part of the particle growth and **shift** of the particle distribution toward larger MMAD could be attributed to this phenomena. Electron micrography and X-ray dispersive analysis showed that the SFE aerosol particles were cuboidal crystals composed ($\cong 97.0\%$) of KCl. Given this

composition the particles were expected to be hygroscopic and highly soluble. The apparent acceleration of particle growth in the 56 m³ chamber is most likely a result of increased adsorption of water due to RH % differences, 30 & 100% for the 0.7 and 56 m³ chambers respectively. Particle growth due to agglomeration was greater for the higher load. However, regardless of particle concentration hygroscopic growth exerted the greatest influence on shifts of aerosol distribution MMAD. Interestingly, the shift of the particle size distribution toward larger MMAD (Figures 5a & 5b) continued despite early gravitational settling of the larger particles as shown by the disappearance of the minor mode.

System Atmosphere Differences and Particle Growth: Toxicity Implications

An average (both load levels) relative aerosol dispersal efficiency ratio of $\cong 1.5$ for the 56 m³ chamber conditions vs. the 0.7 m³ conditions would favor a comparable 1.5 ratio for total aerosol mass deposition in lungs at a given minute ventilation (Ve). For example at the 80 g/m³ load level an individual with a resting Ve of 7 LPM (assuming 0.9 deposition fraction, *vide infra*) exposed to the 56 m³ chamber atmosphere, at peak aerosol concentration of 14.0 g/m³, would have 9.8×10^{-2} grams/min. total lung deposition rate. The same individual exposed to the 0.7 m³ chamber atmosphere, at peak aerosol concentration of 10.0 g/m³, would have 7.0×10^{-2} g/min. total lung deposition rate. At the 50 g/m³ load the corresponding deposition rates would be 7.1×10^{-2} g/min and 4.4×10^{-2} g/min.

Based on the current ICRP (International Commission on Radiation Protection) human particle deposition curves (6) the particle size increases observed in all experimental conditions would increase total lung deposition (TL) - (Figure 8). One μm increases in MMAD in the 0.7 m³ chamber seen for the 50 and 80 g/m³ loads correspond to increases in total lung deposition of 5 and 3 %. In the 56 m³ chamber 2 μm increase of MMAD would result in a 7 % increase in TL while an increase of MMAD of 3.9 μm at the 80 g/m³ load would result in a 20 % increase in TL. Particle growth induced deposition changes would be greater for deposition in various regions of the lung. In the naso-oro-pharyngeal (NP) region particle deposition would increase by as little as 8 % (0.7 m³ - 80 g/m³) to as much as 45 % (56 m³ - 80 g/m³) - (Figure 9). Tracheobronchial (TB) region deposition (7) would increase by 6 % at either target load in the 0.7 m³ chamber and by 14 and 33 % in 56 m³ chamber at 50 and 80 g/m³ respectively (Figure 10). With initial MMAD's and magnitude of particle growth observed in the present study pulmonary (P) regional deposition would decrease. In the 0.7 m³ chamber P deposition would decrease 17 and 16% at the low and high loads. Decreases P region fractional deposition would be greater in the 56 m³ chamber at 26 and 44 %. These large decreases in the P region would have greater significance for less soluble aerosols because of the relatively slower clearance from this compartment. However, since SFE particles are highly soluble shifts in regional deposition pattern and thus clearance and retention pattern assume secondary relevance to TL deposition changes, primarily because of circumvention of normal clearance mechanisms.

Breathing 1% (10000 ppm) atmospheres of CO, has been shown to increase Ve from 7 to 9 LPM in humans in a few minutes and 2 % atmospheres increase Ve to 11 LPM (8). These increases in Ve would result in proportional increases in particle deposition. CO, yield ratios of 1.3 for and 2.3 for the 56 m³ vs 0.7 m³ chamber at the 50 and 80 g/m³ loads would result in

proportional increases in particle deposition caused by CO₂ stimulated hyperventilation. At an 80 g/m³ load the 2.8 x 10⁻² g/min. TL deposition difference rate between the 56 and 0.7m³ chamber conditions would increase to a 6.2x 10⁻² g/min. TL deposition difference between the two atmospheres.

Change in the percent baseline carboxyhemoglobin (HbCO) level in man caused by breathing CO can be predicted by the following empirical equation (9). This algorithm was found applicable for humans breathing CO concentrations from 90 to 21000 ppm for periods of 20 to 300 min. but with limited reliability at HbCO concentrations above 80 Yo.

$$A [\text{Hb CO}] \% = (\text{CO} \times V_e \times t) / 2.5 \times 10^4,$$

where V_e is LPM, t is min. and CO is ppm. As a point of reference baseline HbCO in non-smokers is 1.3 % and in smokers may vary from 5 to 10 %.

Thus at V_e = 7 LPM and exposure to the 0.7m³ chamber 50 and 80 g/m³ atmospheres for 20 min. the predicted HbCO levels would be ≅ 14.4 and 37.4%. In the 56 m³ chamber corresponding HbCO levels would be 20.6 and 27.5%, if concentration decay did not occur. Although this simple mathematical description is adequate for rudimentary estimates of HbCO formation and homeostasis, further investigation which includes measurement of pertinent physiological responses to CO exposure are required. The potential differences of physiological response from exposure to steady state concentrations of CO and CO₂ and exposure to atmospheres with decaying CO and CO₂ concentrations require further examination.

With exception of potential CO concentration effects on HbCO the 0.7 m³ at the higher target load, characteristics of the SFE atmospheres generated in the larger test chamber appear to pose a higher relative potential inhalation toxicity risk.

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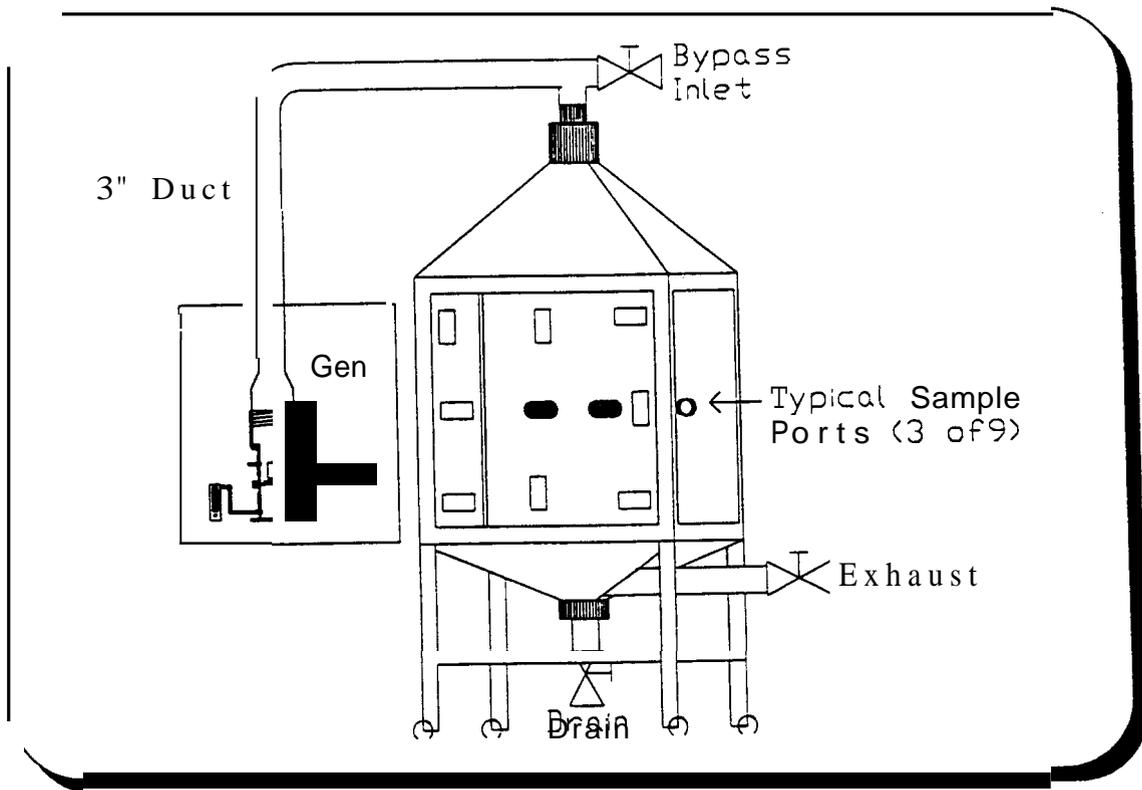
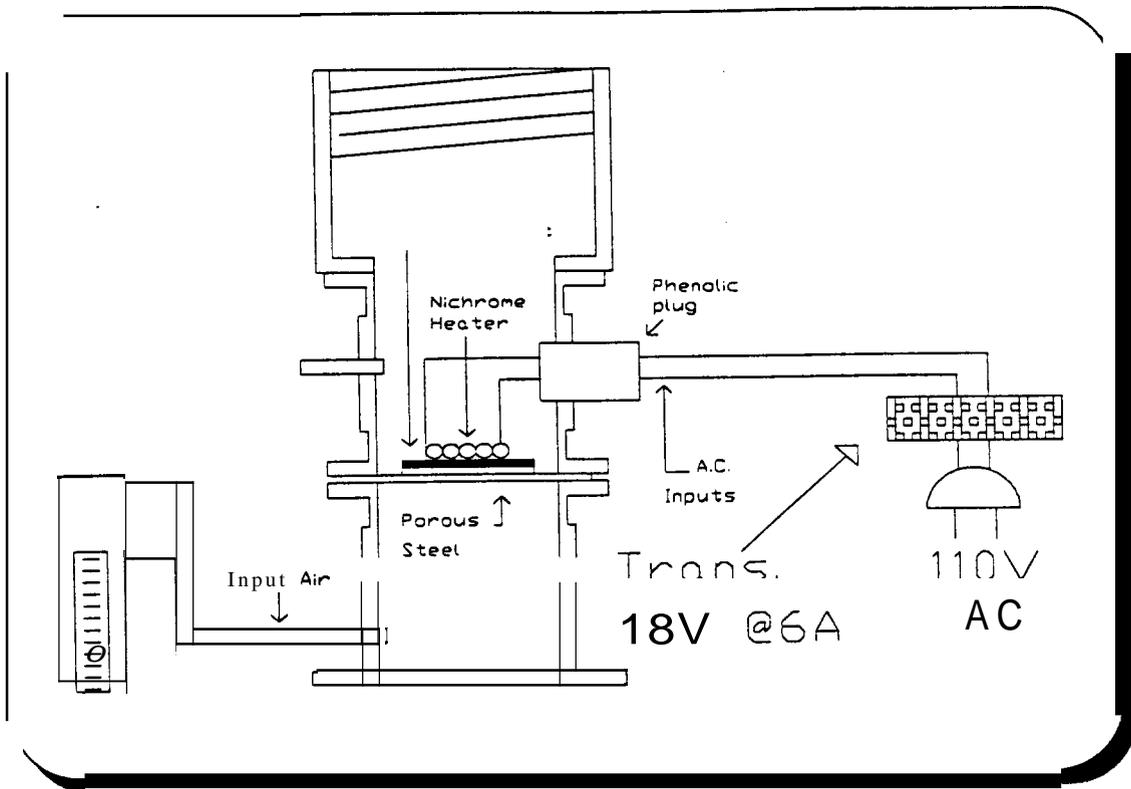


Figure 1. Aerosol Generator & Exposure Chamber - 0.7m³

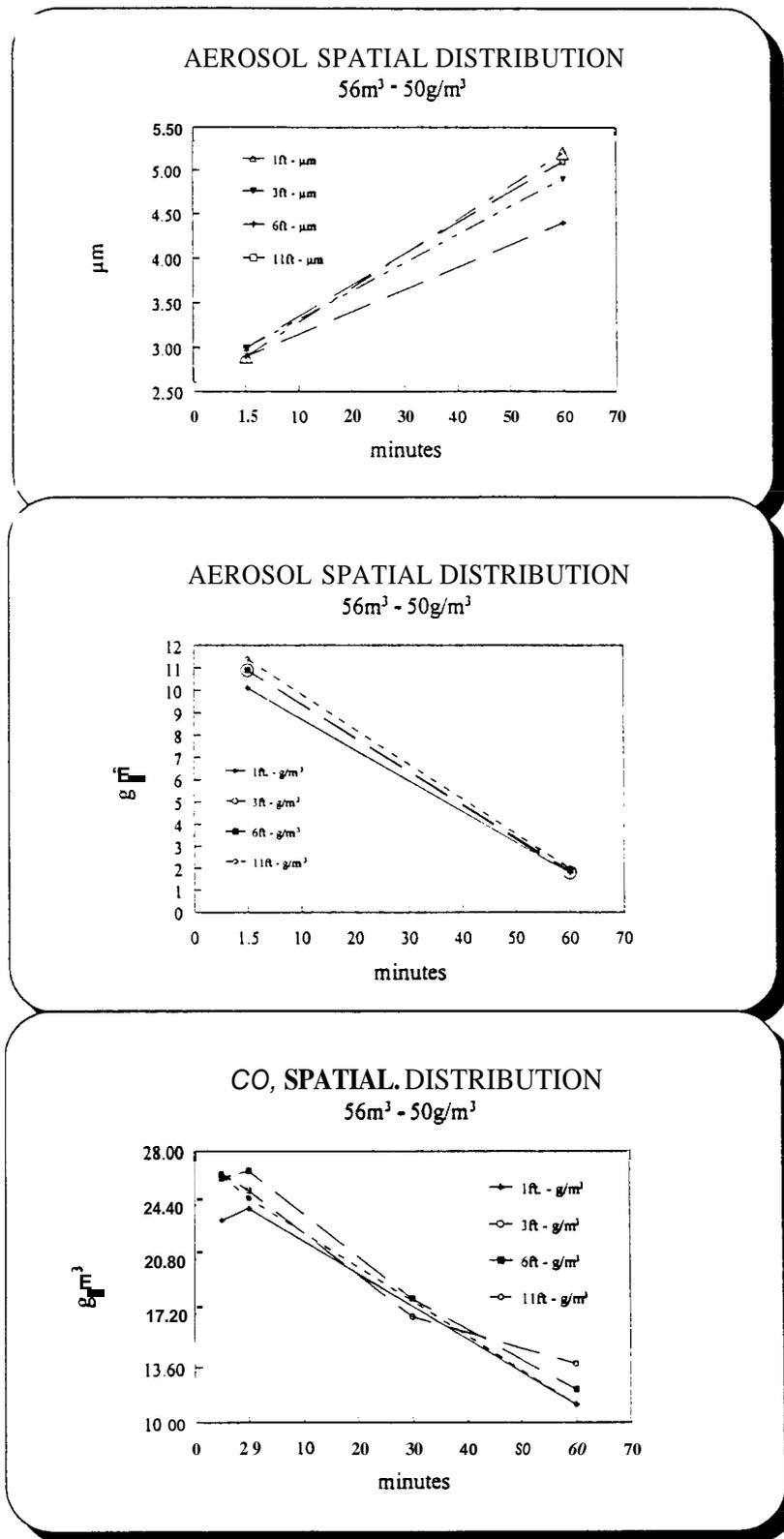
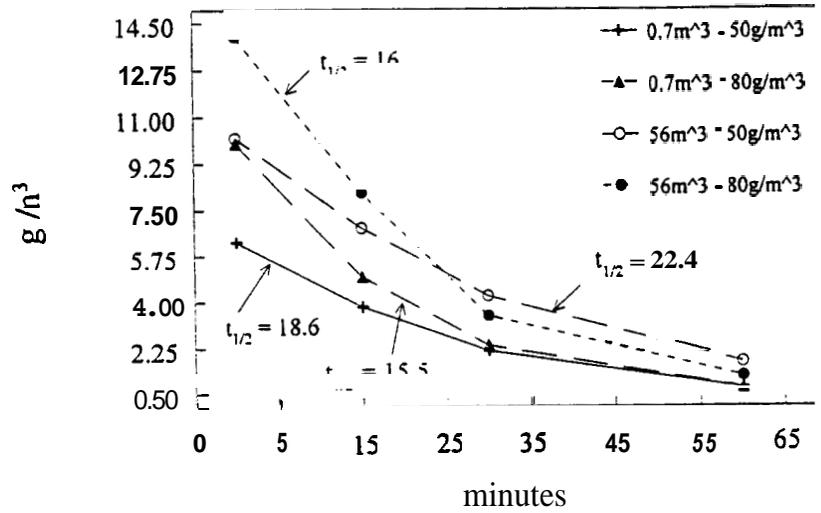


Figure 2. Aerosol/CO₂ Distribution in the 56m³ chamber

AEROSOL CONCENTRATION DECAY



AEROSOL CONCENTRATION DECAY

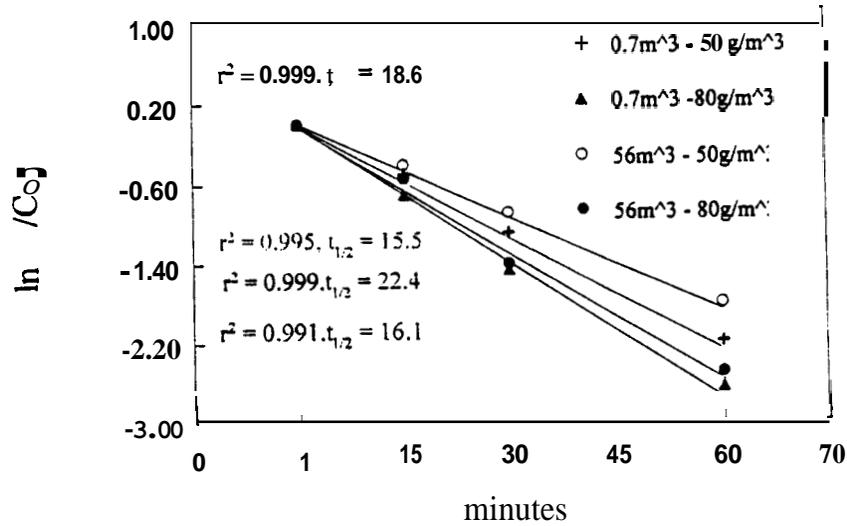


Figure. 3. Aerosol Concentration Decay

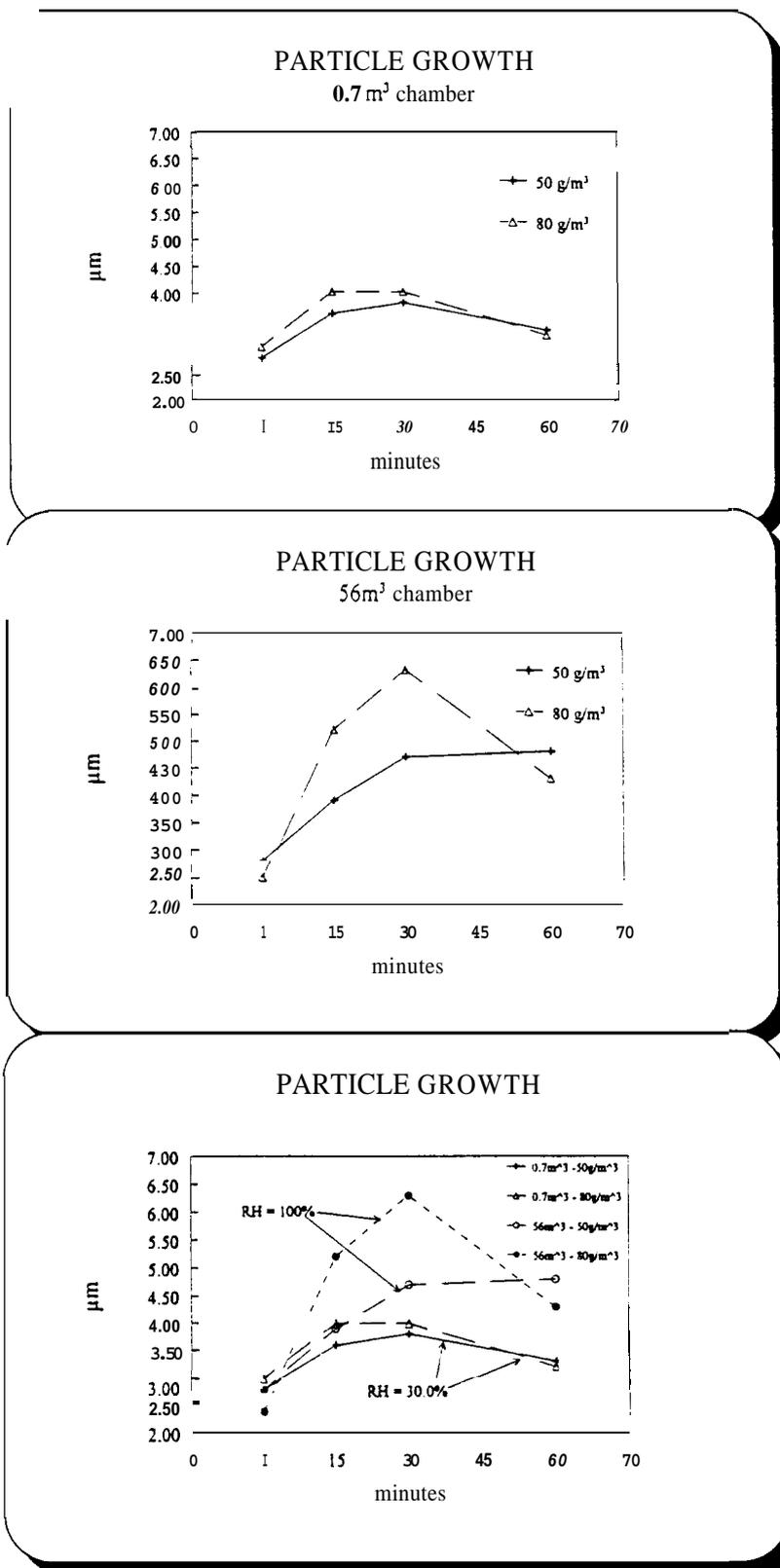


Figure 4. Aerosol Particle Growth

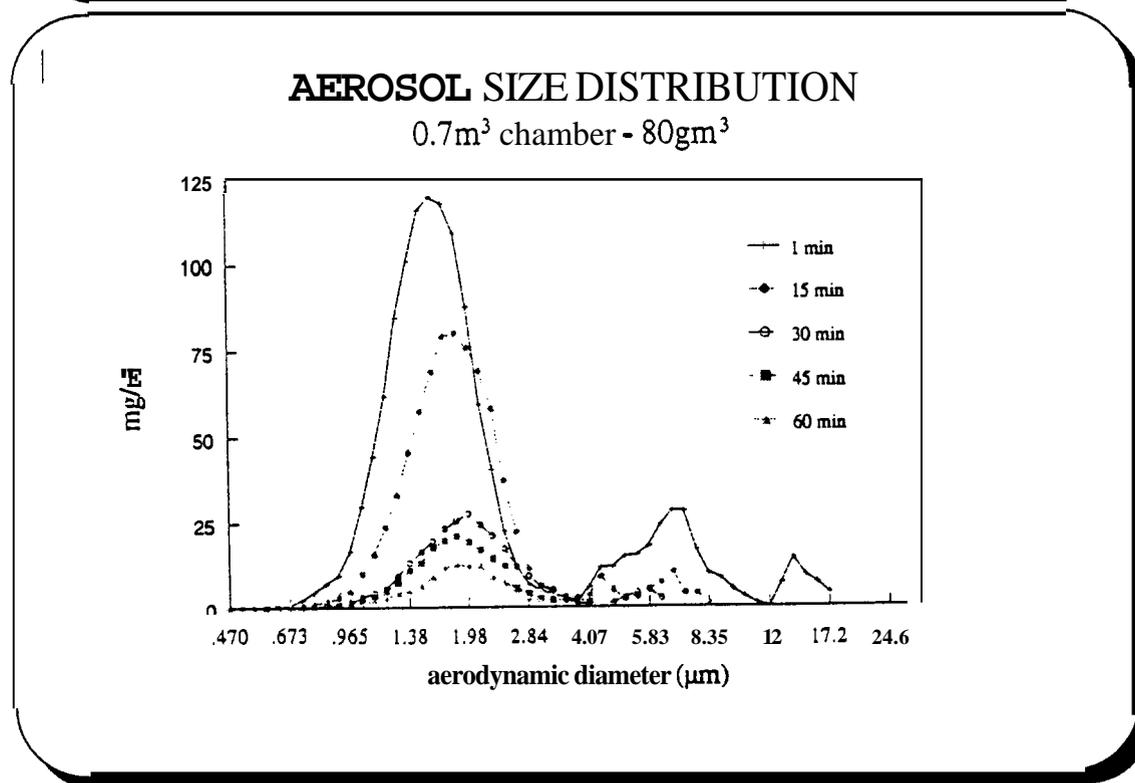
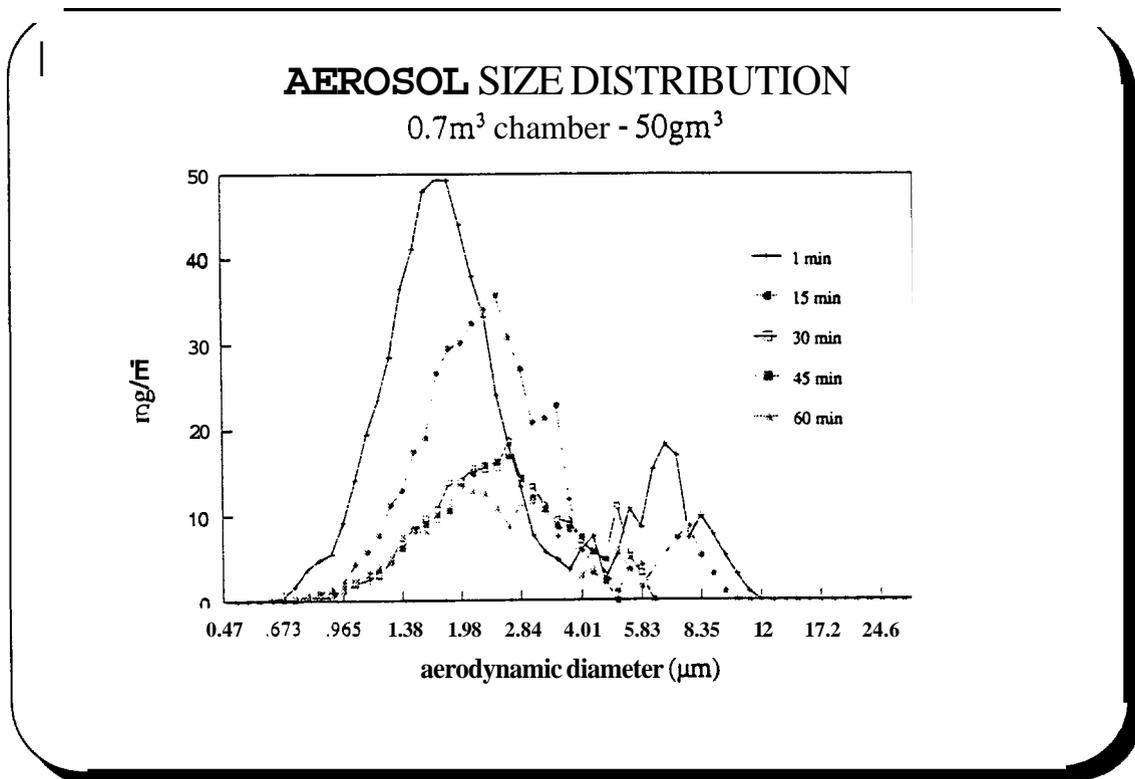


Figure 5a. Aerosol Size Distribution - 0.7m³ chamber

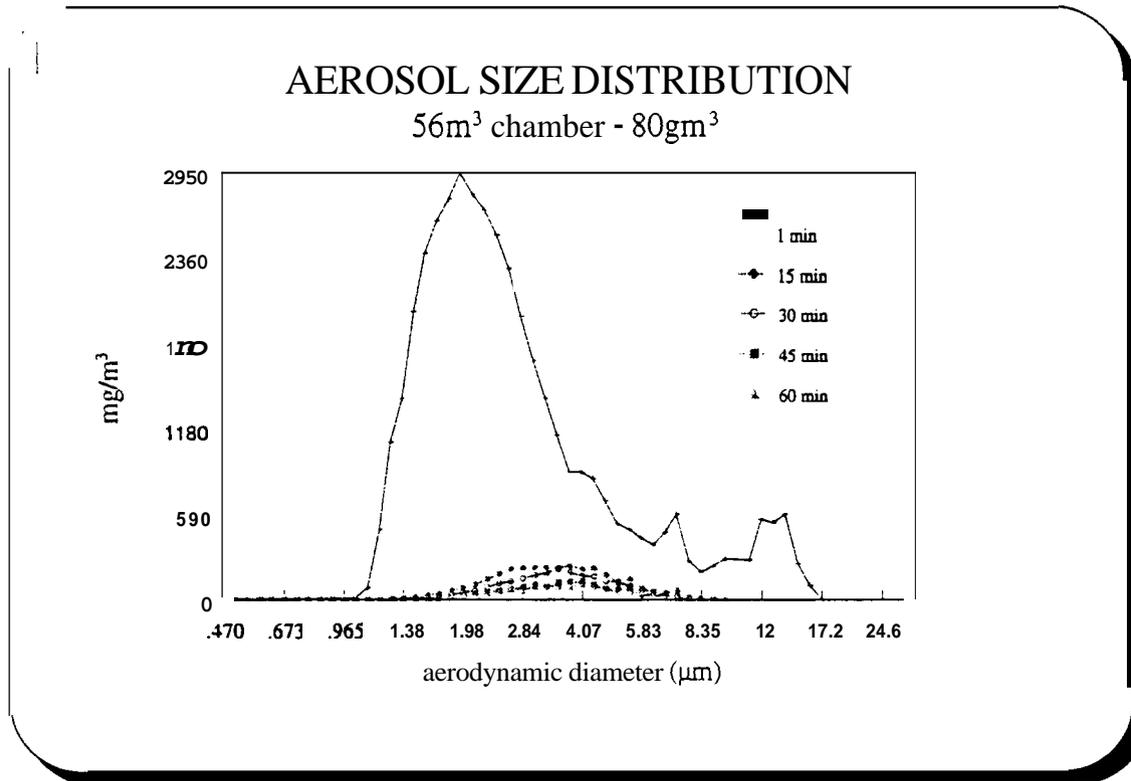
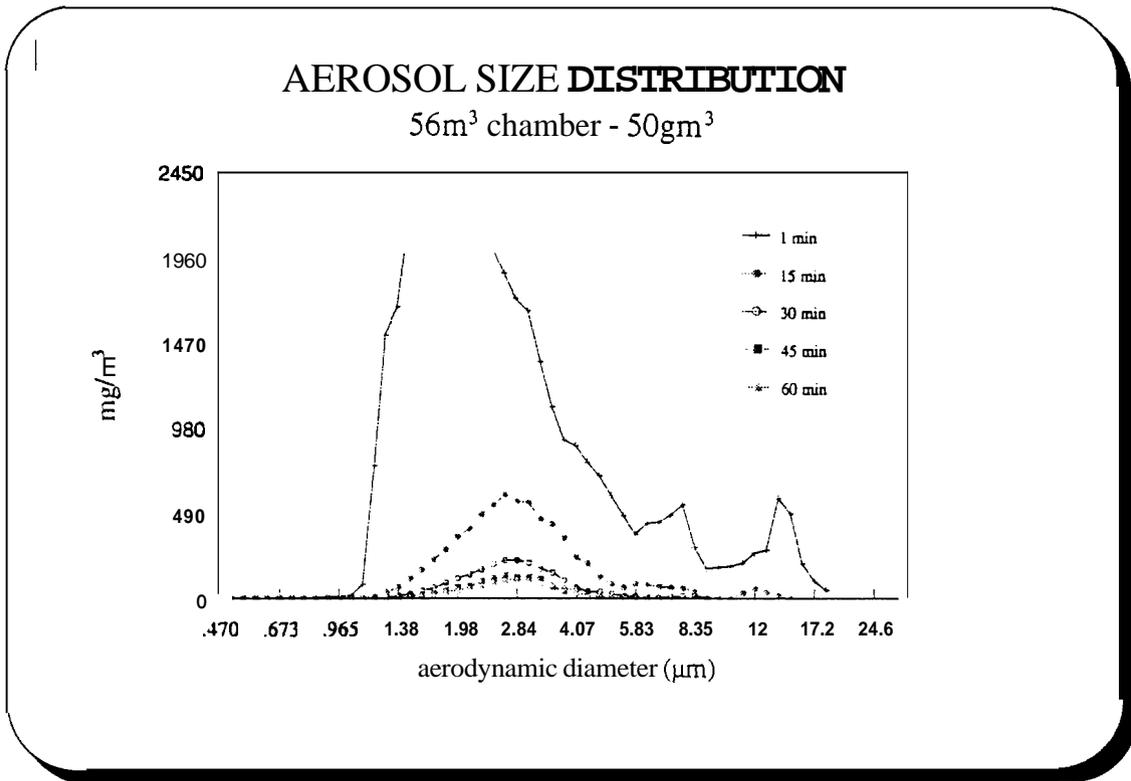


Figure 5b. Aerosol Size Distribution - 56m³ chamber

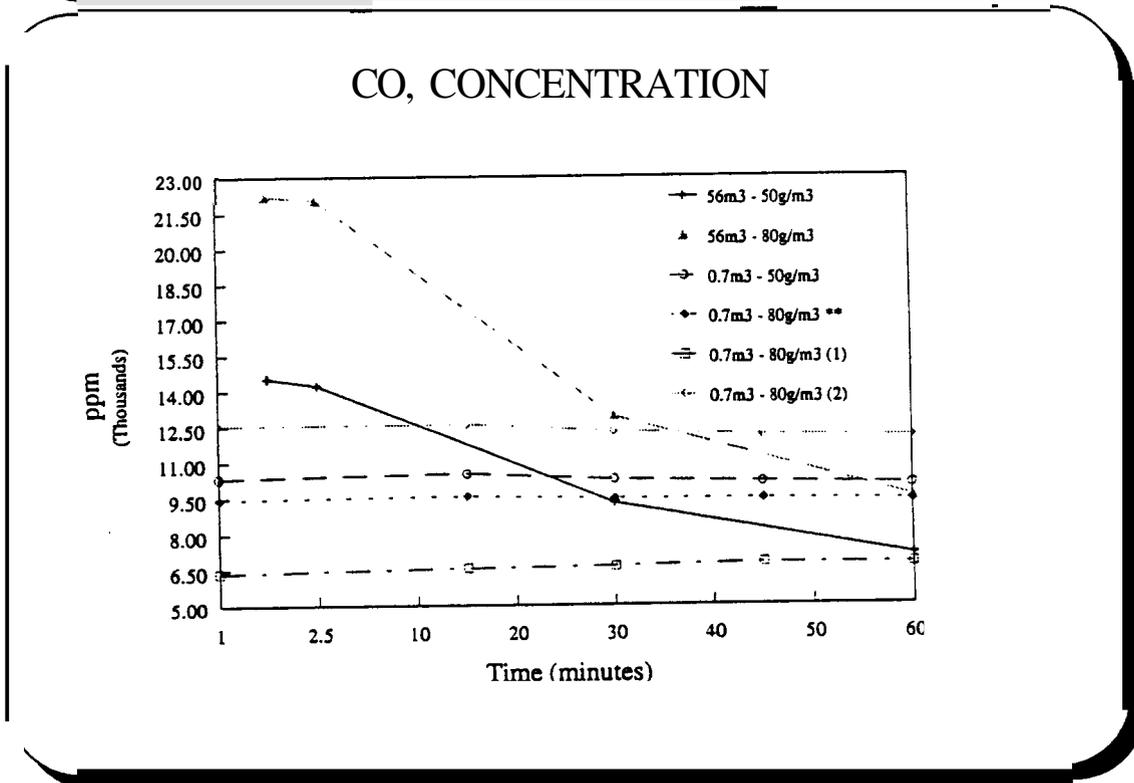
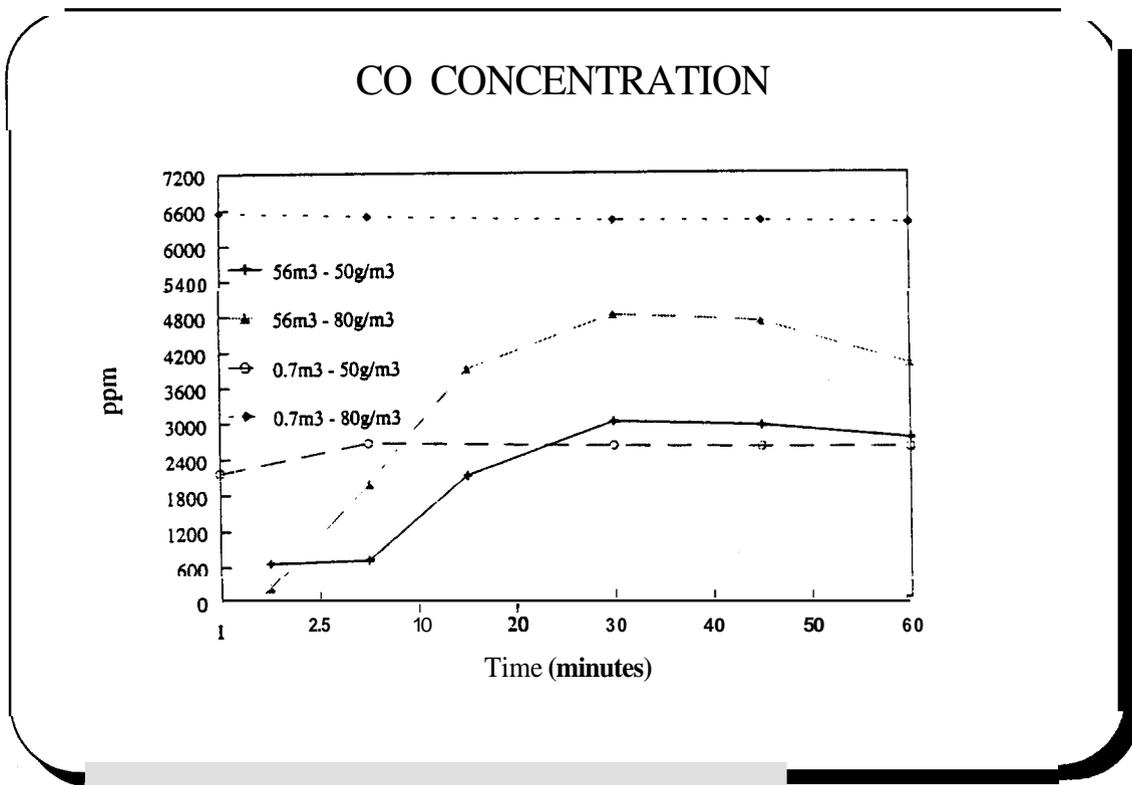


Figure 6. CO and CO₂ Concentrations

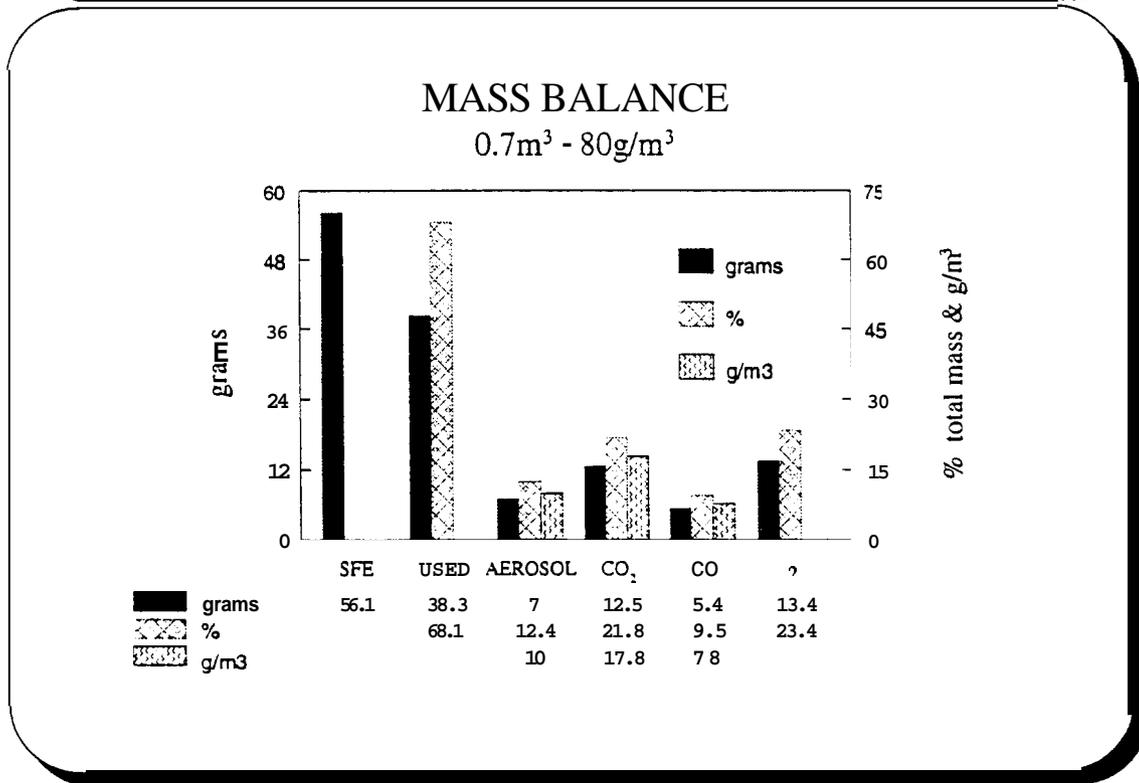
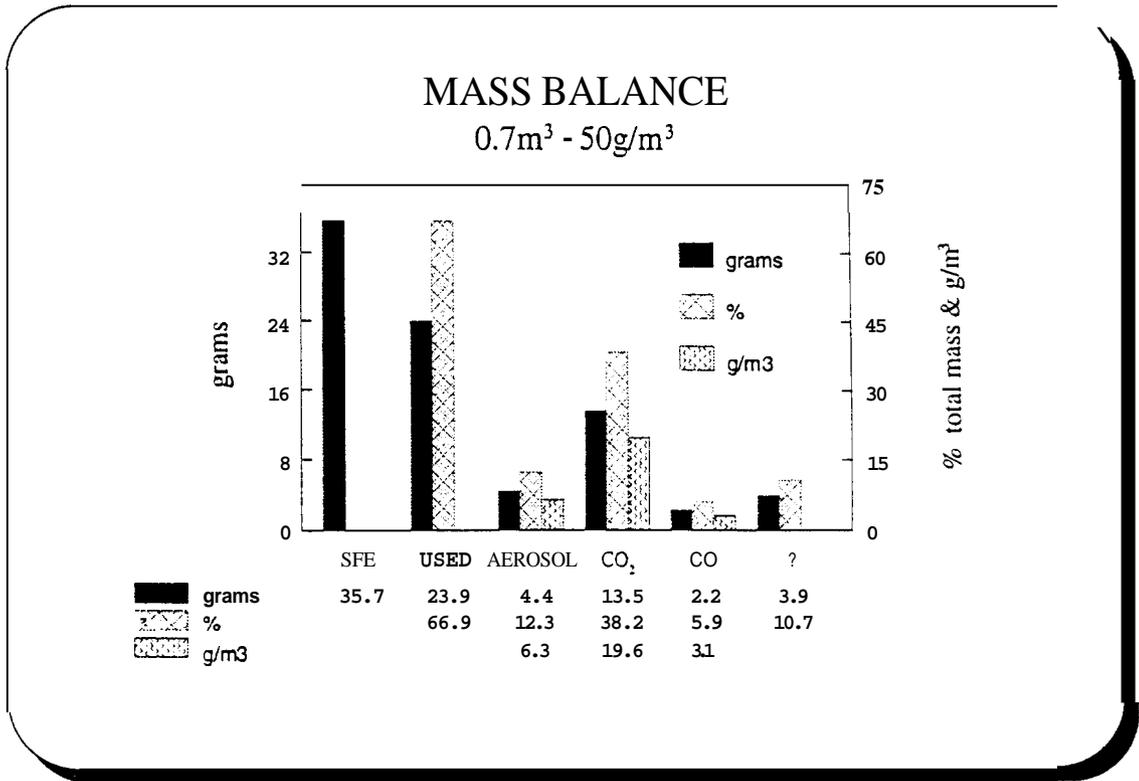


Figure 7a. Mass Balance - 0.7m³ chamber

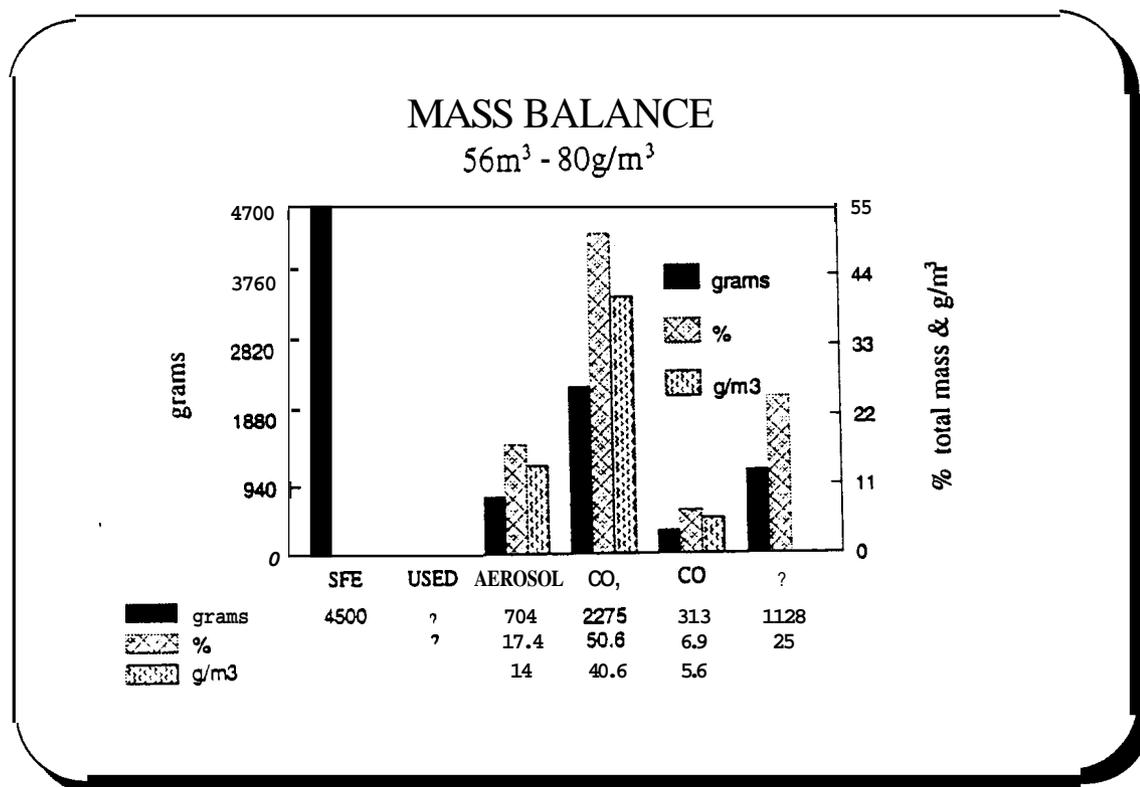
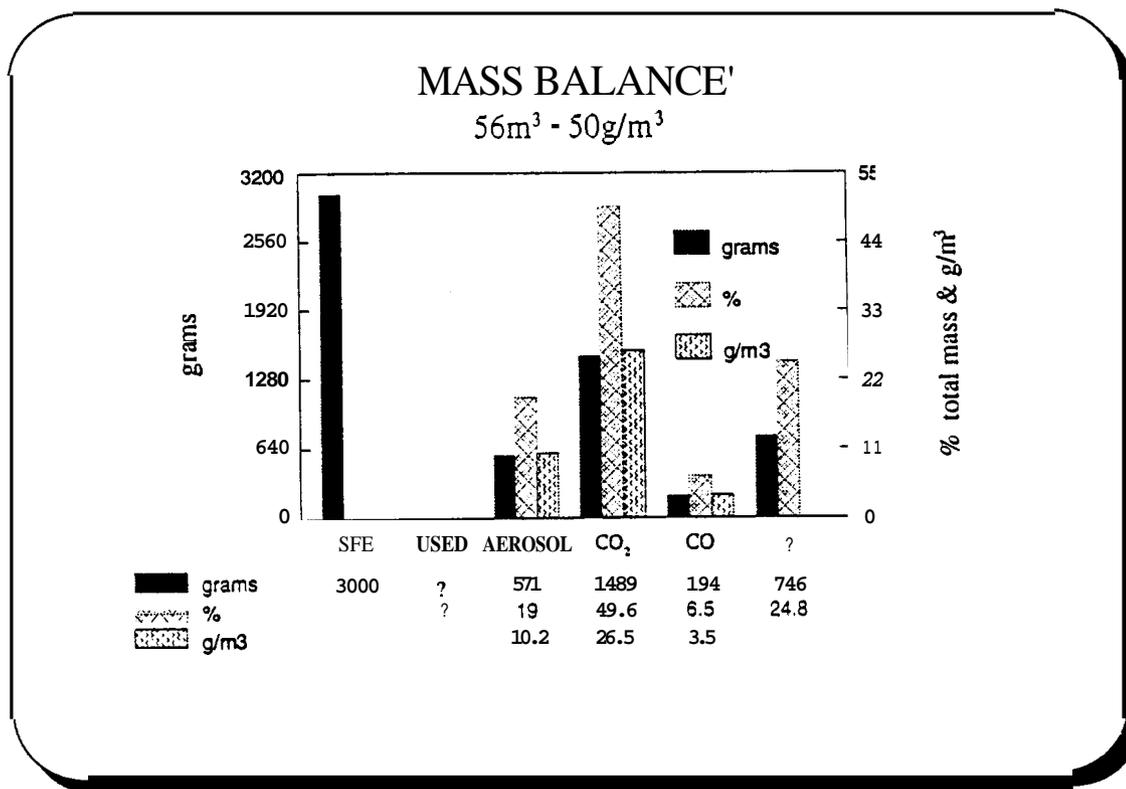


Figure 7b . Mass Balance - 56m^3 chamber

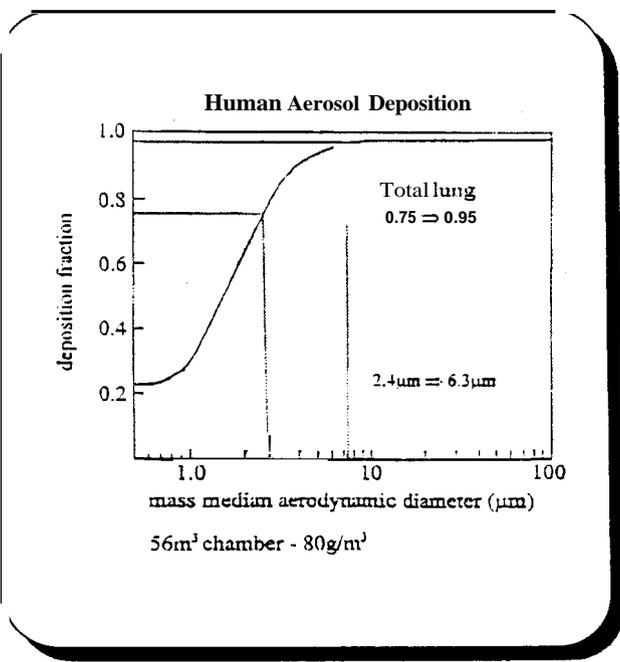
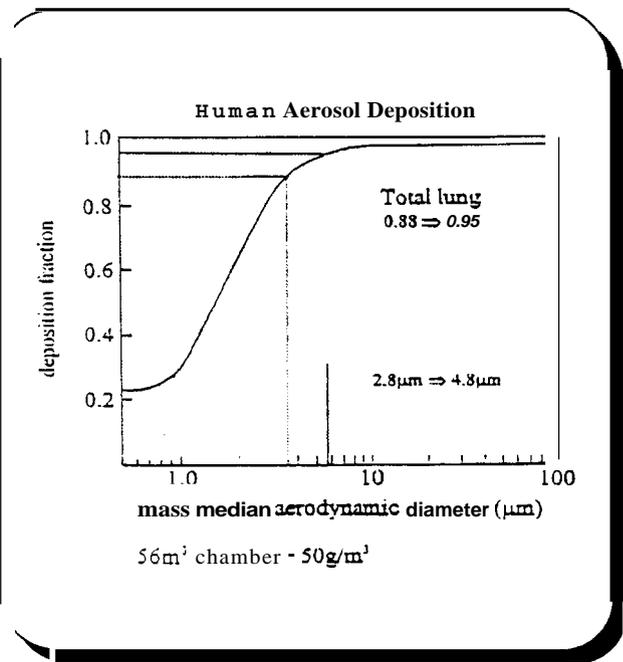
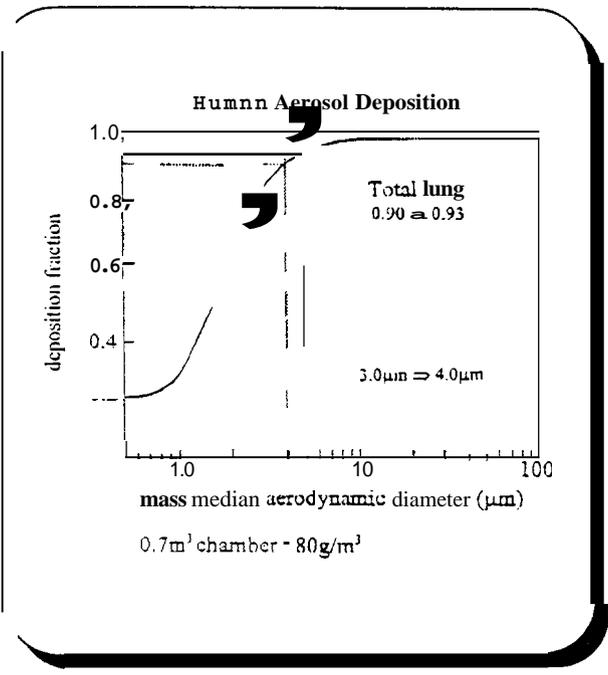
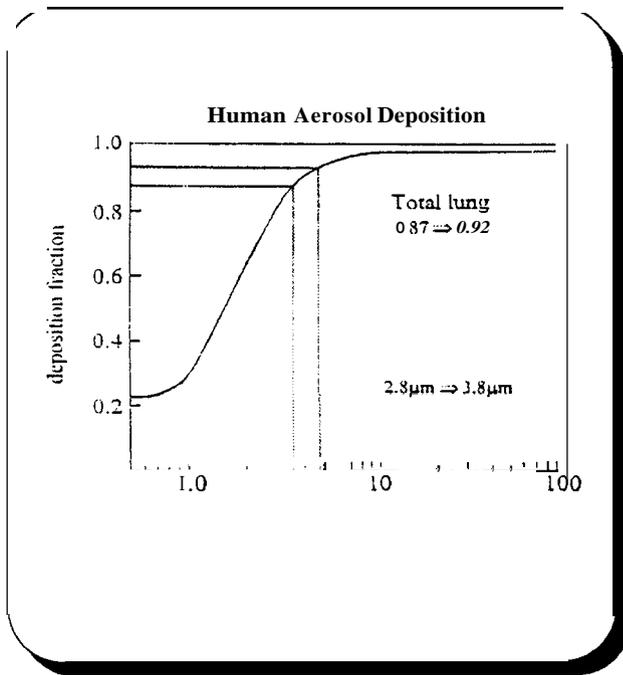


Figure 8. Total Lung Deposition

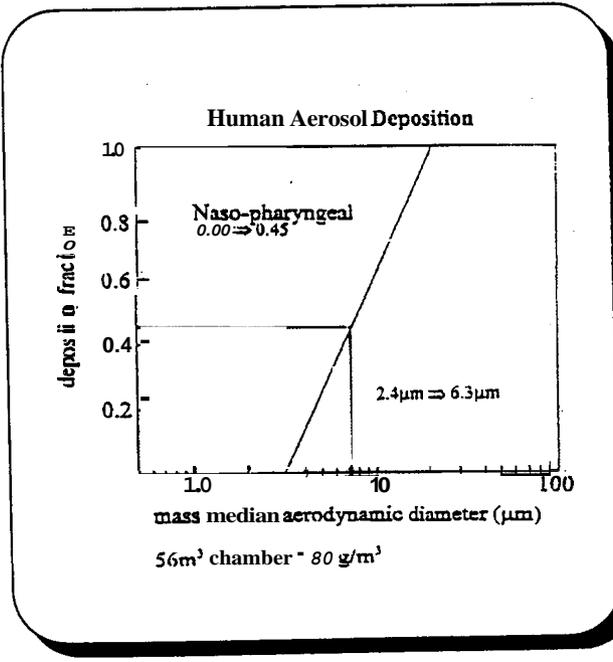
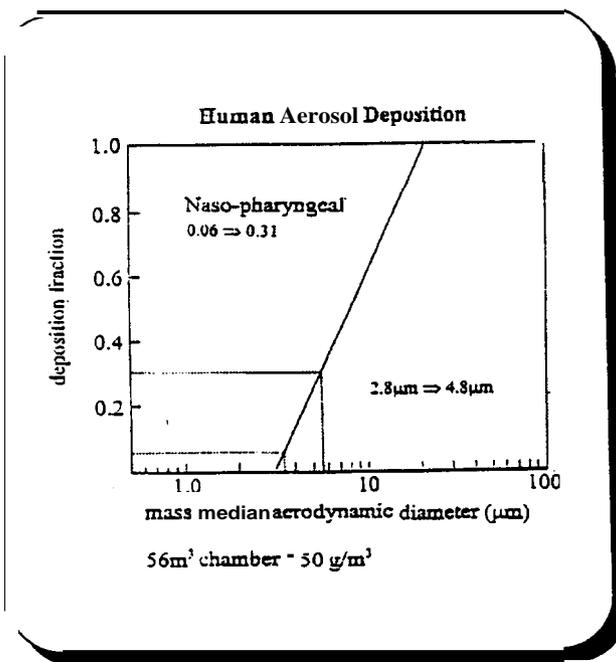
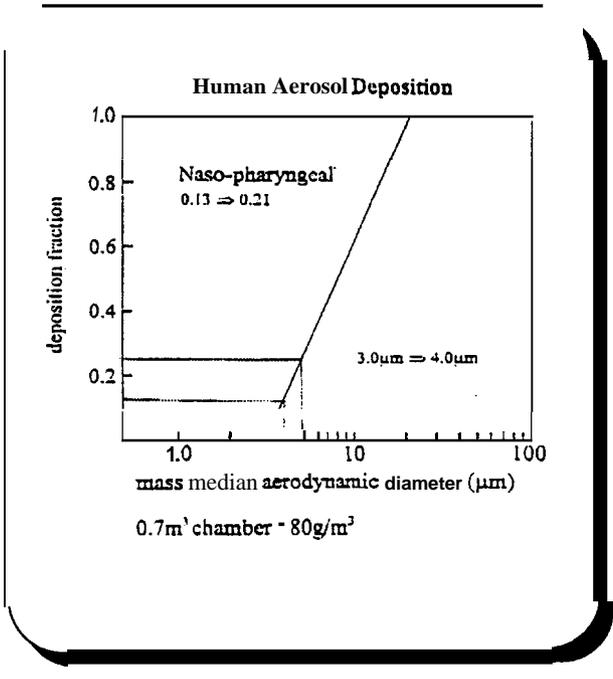
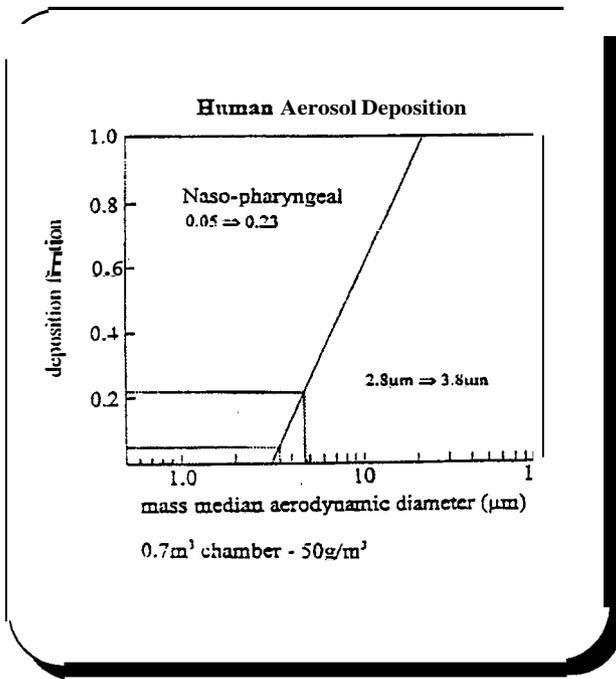


Figure 9. Naso-oro-pharyngeal Deposition

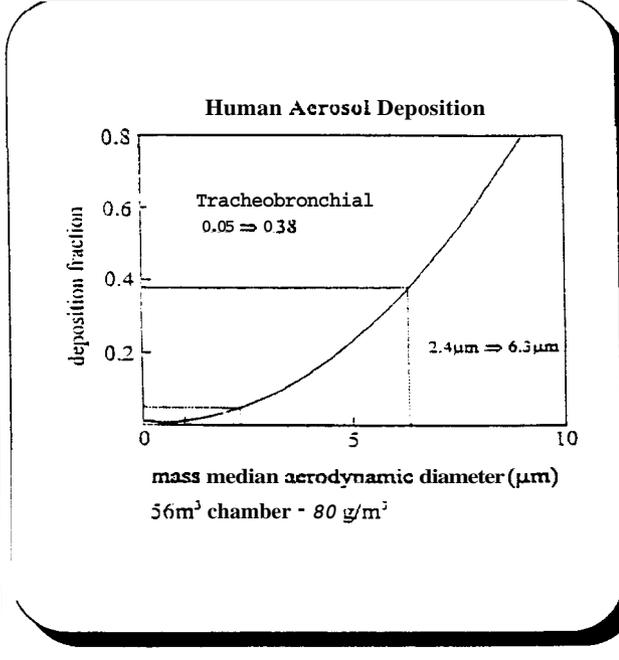
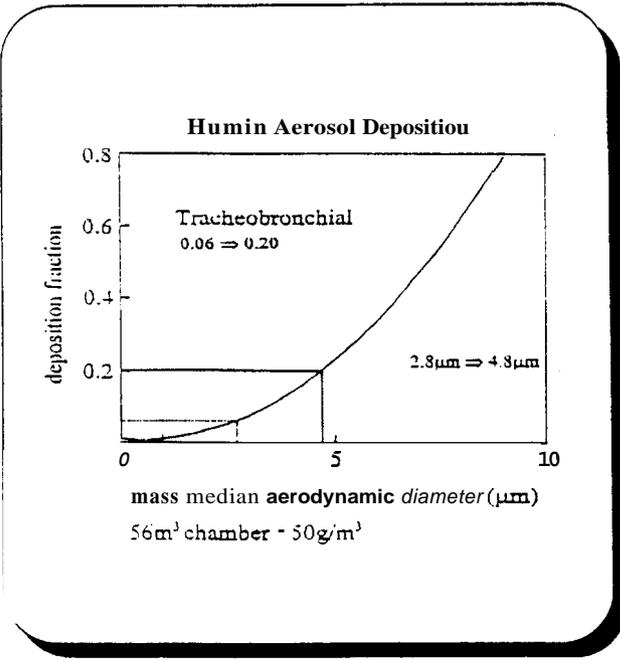
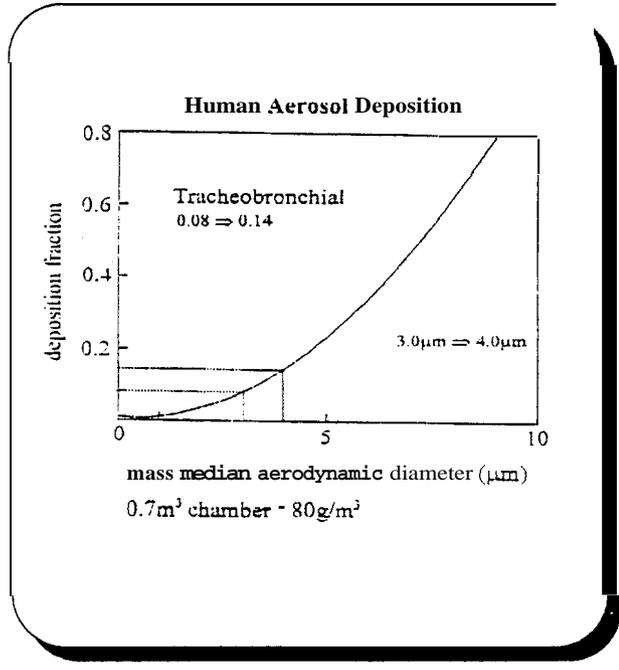
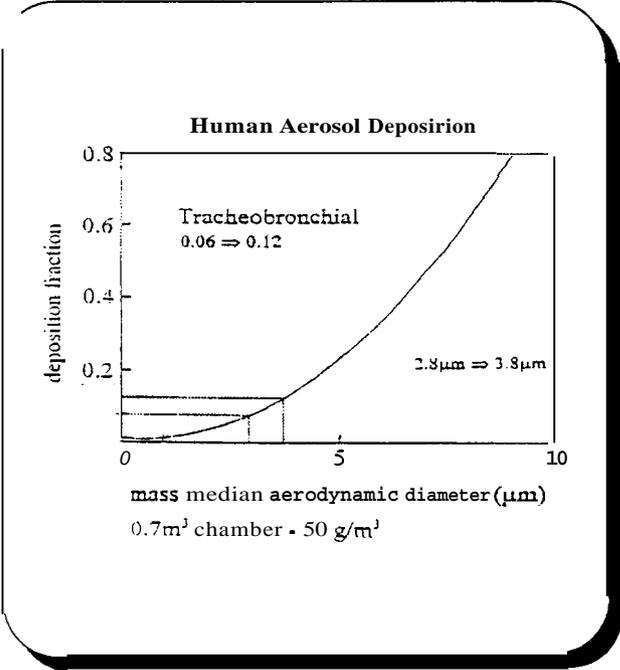


Figure 10. Tracheobronchial Deposition

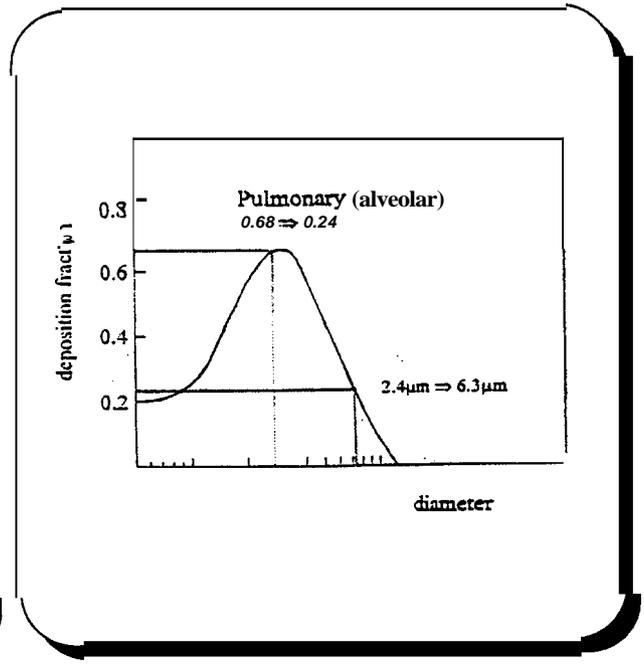
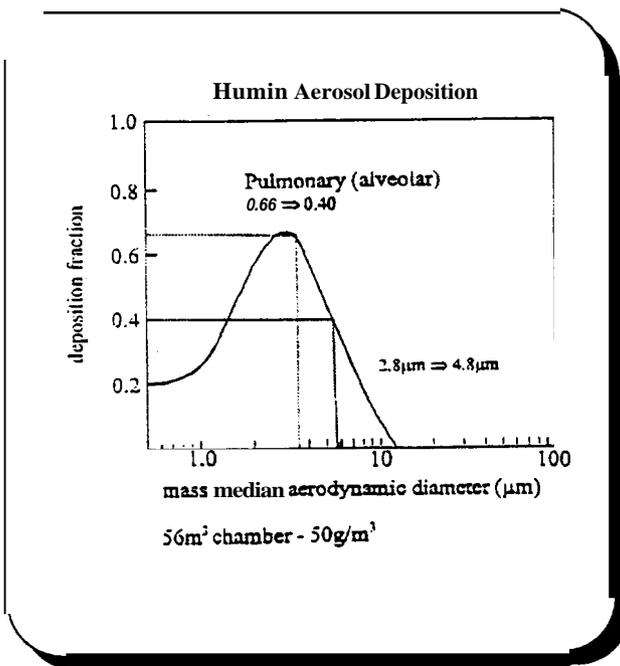
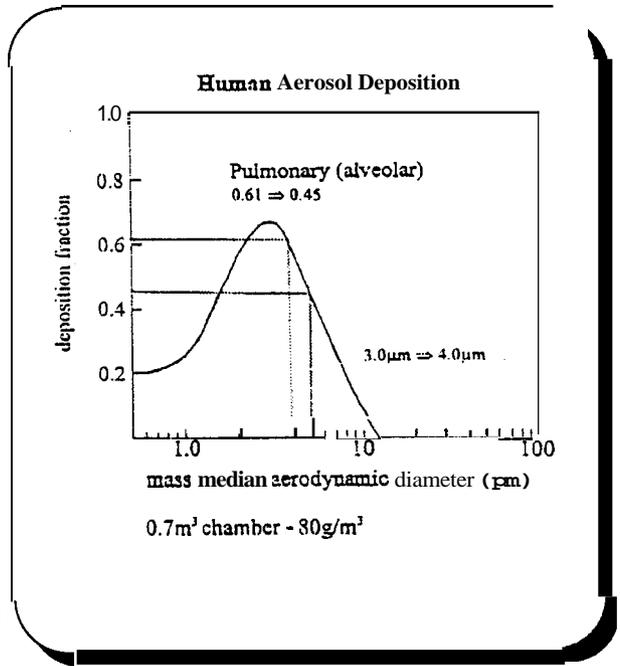
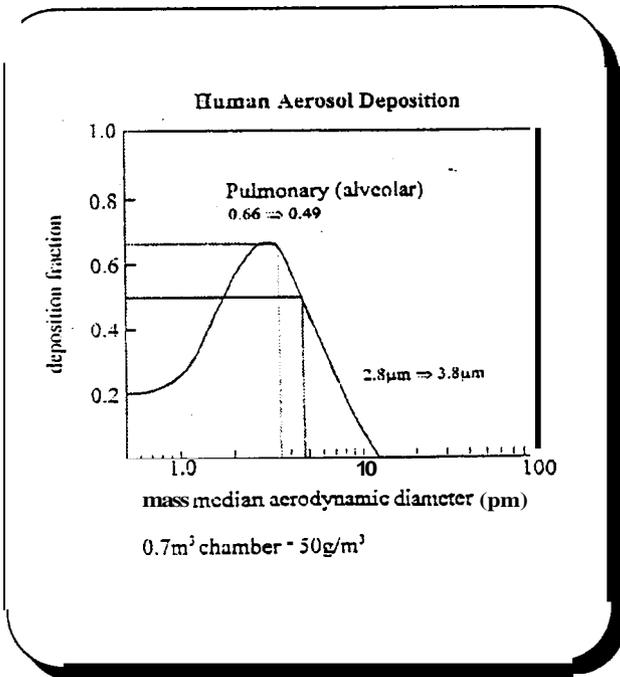


Figure 11. Pulmonary (Alveolar) Deposition