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Carbon in fly ash analysis using photoacoustic spectroscopy

Jeffrey Raymond Dykstra
Iowa State University

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Dykstra, Jeffrey Raymond, Ph.D.

Iowa State University, 1993

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Carbon in fly ash analysis using photoacoustic spectroscopy

by

Jeffrey Raymond Dykstra

**A Dissertation Submitted to the
Graduate Faculty in Partial Fulfillment of the
Requirements for the Degree of
DOCTOR OF PHILOSOPHY**

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For the Graduate College

**Iowa State University
Ames, Iowa**

1993

DEDICATION

All things come from God, both the world we find ourselves in and our ability to understand a small part of God's creation. Therefore, it is only appropriate that I dedicate my dissertation to the Lord who created me as well as the world I'm trying to understand. My thankfulness to the Father of Jesus Christ is captured by the Psalmist David in Psalm 8:3 - 8:9, New International Version:

³When I consider your heavens, the work of your fingers,
the moon and the stars, which you have set in place,
⁴what is man that you are mindful of him,
the son of man that you care for him?
⁵You made him a little lower than the heavenly beings
and crowned him with glory and honor.
⁶You made him ruler over the works of your hands;
you put everything under his feet:
⁷all flocks and herds, and the beasts of the field,
⁸the birds of the air, and the fish of the sea,
all that swim the paths of the seas.
⁹O LORD, our Lord,
how majestic is your name in all the earth!

TABLE OF CONTENTS

NOMENCLATURE	viii
ACKNOWLEDGMENTS	xii
1. INTRODUCTION	1
2. BACKGROUND	3
2.1 Fly Ash Characteristics	3
2.2 Particle Interactions with Radiation	5
2.3 The Photoacoustic Signal	8
2.4 PAS Cell Design	11
2.5 Diesel Exhaust Measurements	12
2.6 Carbon Detection in Synthetic Fly Ash	14
2.7 On-Line Carbon Detection in Fluidized Bed Fly Ash	15
3. EXPERIMENTAL APPARATUS	21
3.1 Acoustical noise Reduction	21
3.2 Particle Entrainment Facility	27
3.3 Particulate Sampling	29
3.4 Data Acquisition System	30
3.5 Optical PAS System.....	31
3.6 Microwave PAS System	35
4. RESULTS AND DISCUSSION	40
4.1 Thermal Gravimetric Analysis of Fly Ash.....	40

4.2 Mono-Dispersed Fly Ash Tests with Optical PAS.....	49
4.3 Microwave PAS System	56
5. CONCLUSIONS	63
BIBLIOGRAPHY	65
APPENDIX A. ABSORPTION CROSS SECTION CALCULATION	70
APPENDIX B. DERIVATION OF THE PHOTOACOUSTIC EQUATION	83
APPENDIX C. PROPAGATION OF ERROR ANALYSIS	86
APPENDIX D. DATA ACQUISITION PROGRAM	88

LIST OF FIGURES

Figure 2.1	Mass specific absorption cross section for carbon spheroids at 632 nm	7
Figure 2.2	The influence of condensed tar on the PAS signal	16
Figure 2.3	Mass distribution of the laboratory scale fluidized bed fly ash	17
Figure 2.4	The PAS signal for fluidized bed fly ash	19
Figure 3.1	Reduction of pump noise at the microphone with a Helmholtz resonator ...	26
Figure 3.2	NO ₂ PAS signal verses frequency	27
Figure 3.3	The laboratory scale fluidized bed combustor	28
Figure 3.4	Optical PAS cell	33
Figure 3.5	Flow schematic for the optical PAS system	34
Figure 3.6	Flow schematic for the microwave PAS system	36
Figure 3.7	Electric and magnetic field lines for TE ₁₀ mode	37
Figure 3.8	Microwave PAS cell	39
Figure 4.1	Temperature verse time for the TGA stoker fly ash sample	42
Figure 4.2	TGA of the stoker fly ash sample	43
Figure 4.3	TGA of the pulverized coal fly ash sample	45
Figure 4.4	TGA of the atmospheric fluidized bed fly ash	46
Figure 4.5	TGA of the pressurized fluidized bed fly ash	47
Figure 4.6	Optical PAS signal for five specified size distributions	52
Figure 4.7	PAS linear regression slopes verse average particle size range diameter	54

Figure 4.8	Size distribution of Iowa State University stoker fly ash (top) and PAS contribution from each range (bottom)	55
Figure 4.9	Illustrative results of microwave PAS test # 4	60
Figure 4.10	Microwave PAS carbon in fly ash measurements	61

LIST OF TABLES

Table 3.1	Helmholtz design variables	23
Table 4.1	TGA fly ash samples	41
Table 4.2	Thermal gravimetric analysis of the fly ash samples.....	48
Table 4.3	Examination of TGA measurements	49
Table 4.4	Optical PAS signal for five specific size distributions	51
Table 4.5	Linear regression analysis of optical PAS results.....	51
Table 4.6	Size distribution of the Iowa State University stoker fly ash.....	53
Table 4.7	Results of the carbon in fly ash measurements using microwave PAS.....	62
Table 4.8	Linear regression results of the microwave PAS measurements.....	62
Table C.1	Parameters of propagation of error analysis	87

NOMENCLATURE

a	=	Radius of the sphere
a_n	=	Scattering coefficient
A_a	=	Mass specific absorption coefficient
A_D	=	Cross sectional area of the duct.
A_e	=	Mass specific extinction coefficient
b_a	=	Absorption coefficient
b_e	=	Extinction coefficient
b_n	=	Scattering coefficient
c_0	=	Speed of sound
C_a	=	Absorption cross section
C_e	=	Extinction cross section
C_p	=	Heat capacity per unit volume
C_s	=	Scattering cross section
D	=	Diameter
\overline{D}	=	Mean diameter
e	=	Electron charge
f_H	=	Resonant frequency of a Helmholtz resonator
$f(D)$	=	Diameter frequency distribution
g_{bj}	=	Damping constants of bound electrons
g_f	=	Damping constants of free electrons

$H_{(n+1/2)}^{(2)}(z)$	=	Half integral order Hankel function.
$H(r,t)$	=	Heat source
$I(z)$	=	light beam intensity
$J_{(n+1/2)}(z)$	=	Half integral order Bessel function.
k	=	Imaginary part of the refractive index
k	=	$\frac{2\pi}{\lambda}$
k_a	=	Thermal conductivity of air
L_e	=	Equivalent resonator neck length = $L + 8r/3\pi$
L	=	Resonator neck length
L	=	Optical path length
LOI	=	Loss-on-ignition
m	=	Refractive index in the form $n+ik$
m°	=	Effective electron mass $\approx m_e/18$
m_e	=	Electron mass in vacuum
$m(D)$	=	Mass distribution
M_c	=	Carbon mass loading
M_t	=	Total mass loading
n	=	Real part of the refractive index
n_f	=	Free electron number densities
n_j	=	Bound electron number densities
N	=	Total number of particles per unit volume
$N_{(n+1/2)}(z)$	=	Half integral order Neumann function
p	=	acoustic pressure
PAS	=	Photoacoustic Spectroscopy
ppm	=	Parts per million

R	=	Responsivity of the photoacoustic spectroscopy cell
r	=	Radius
r_{sample}	=	Radius of the sample lines
$r_{\text{PAS cell}}$	=	Radius of the PAS cell
\bar{r}	=	Mean radius
r_H	=	Radius of resonator neck
R_a	=	Mass specific absorption cross section
R_e	=	Mass specific extinction cross section
S	=	Photoacoustic spectroscopy signal
S_H	=	Cross sectional area of the resonator neck (πr^2)
SCFM	=	Standard cubic feet per minute
T	=	Pressure-amplitude transmission coefficient
TGA	=	Thermal gravimetric analysis
V	=	Volume of photoacoustic spectroscopy cell
V	=	Volume of the Helmholtz resonator
W	=	Optical power
Z_H	=	Impedance of a Helmholtz resonator

Greek Symbols

α	=	ka (size parameter)
β	=	$m\alpha$
$\chi_n(z)$	=	$-\left(\frac{\pi z}{2}\right)^{1/2} N_{(n+1/2)}(z)$ (Ricatti-Bessel function)
ε	=	Permissivity constant
γ	=	Ratio of specific heats

λ	=	Wavelength
λ_g	=	Waveguide wavelength
ρ_0	=	Mean density of fluid
ρ	=	Density
σ_m	=	Microphone sensitivity
τ	=	Thermal time constant
ω	=	Angular frequency (rad/s)
ω	=	Frequency of radiation
ω	=	Angular modulation frequency
ω_{bj}	=	Natural frequency of bound electrons
$\psi_n(z)$	=	$\left(\frac{\pi z}{2}\right)^{1/2} J_{(n+1/2)}(z)$ (Ricatti-Bessel function)
$\zeta_n(z)$	=	$\psi_n(z) + i\chi_n(z)$ (Ricatti-Bessel function)

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1. INTRODUCTION

The manual loss-on-ignition (LOI) is the current standard for the determination of the carbon content of fly ash. The value of LOI is used to determine the combustion efficiency of a combustor. This process is a tedious and sometimes inaccurate procedure [1]. Fly ash sampled from the exhaust of a combustor is dried and weighed before being subjected to an ashing furnace for several hours at 725°C. The sample is then reweighed and the change in weight is assumed to be due to the carbon present in the sample. The accuracy of this technique is limited by the assumption that only carbon in fly ash changes weight upon heating. However, fly ash may contain other constituents that change weight upon heating. For example, some calcium carbonate, commonly injected into combustors as a sulfur sorbent, can appear in fly ash. The conversion to calcium oxide upon heating is accompanied by a 44% reduction in the original weight of the sample.

An innovative and promising concept for on-line determination of the carbon loadings in coal combustion flue gas is photoacoustic spectroscopy (PAS). The photoacoustic effect is manifested as an acoustical wave that is produced when amplitude modulated electromagnetic radiation is absorbed by the gas or by particles suspended in a gas [2, 3]. The energy absorbed by the sample is transferred as heat to the surrounding gas where it results in a minute temperature rise. Subsequent expansion of the heated gas causes a periodic pressure fluctuation at the modulation frequency which propagates through the gas as an acoustical signal.

PAS work done with soot particles [4, 5] suggests that PAS would also be suited to the challenge of on-line determination of carbon loading because the PAS signal is based solely on the absorption of radiation and not on the energy lost from the incident beam due to absorption and scattering. Although elutriated carbon from coal combustion differs from diesel soot in both particles size and morphology, the technique has virtues that should allow the transition from diesel exhaust to fly ash.

Two radiation sources, a He-Ne laser ($\lambda = 632.8 \text{ nm}$) and microwaves ($\lambda = 12.24 \text{ cm}$), were investigated for producing PAS signals from carbon particles contained in a fly ash sample. At these wavelengths, combustion gases and mineral matter in the fly ash do not absorb appreciably compared with the strong absorption of radiation by the carbon particles [6, 7, 8, 9]. With these assumptions, the PAS signal produced is generated solely by the carbon loading and does not depend on the combustion gases or other particles present in the flue gas.

Brown and Dona [10] have shown, using synthetic fly ash, that the PAS signal is linearly dependent on the carbon concentration in the sample. Although this work with synthetic fly ash showed the promise of photoacoustics, later work by Dykstra and Brown [6] showed that the particle size distribution of carbon contained in the fly ash may affect the photoacoustic signal. An ideal instrument would be able to determine the carbon loadings without knowledge of the size distribution of the fly ash sample. It is therefore important to determine if a size dependence on the PAS signal exists and if it does, ascertain if changing the frequency of the radiation source rectifies the size dependence problem. The end goal of this research is the development an on-line carbon in fly ash monitor based on PAS. The instrument should require no information about the size distribution of the carbon in the fly ash sample.

2. BACKGROUND

The photoacoustic effect in both gaseous and non-gaseous matter was discovered by Alexander Graham Bell in 1880 [11]. Photoacoustic studies, however, remained dormant until the advent of the laser in the early seventies which provided a powerful excitation source. Photoacoustic applications were first limited to the study of gases and aerosols but have now been expanded to the study of solids [2, 3]. The use of photoacoustics for the determination of the carbon content in the fly ash requires a knowledge of both fly ash characteristics and photoacoustics.

2.1 Fly Ash Characteristics

Fly ash is particulate matter that is elutriated from the combustor. Fly ash is composed of ash and small coal particles in various stages of burnout[12]. The shape, size distribution and morphology of the fly ash are important parameters for the PAS technique because these properties affect the absorption coefficient of the fly ash particles.

The chemical characterization of fly ash is due to properties of the coal being burned. Mineral matter in the coal is composed of clays, shales, pyrite, quartz, calcite and lesser amounts of other materials [12] and can take two main forms in the coal: finely disseminated, generally submicroscopic particles and relatively coarse mineral inclusions. The first form includes the inorganic constituents of the original coal-forming plants and the second form comprises various minerals washed into the coal-forming material [13]. Only the first form is

important to the PAS signal. The second form of the mineral matter either stays in the bed or is elutriated as ash particles which have a very low absorption coefficient and produce a negligible PAS signal. In the first form, where the mineral matter is finely disseminated, it may stay attached to a carbon particle. Donsi, Massimilla and Maccio [14] using an optical microscope, describe some fly ash particles as having a heterogeneous structure that is composed of ash and carbon. When a particle is composed of both ash (small absorption coefficient) and carbon (large absorption coefficient) the effect on A_a (mass specific absorption coefficient) is uncertain.

Section 2.2 discusses particle interactions with radiation. This discussion assumes spherical particles. Unlike pulverized coal combustor fly ash, fluidized bed combustor fly ash particles are not spherical; with pulverized coal combustors the ash fusion temperature is reached and the ash forms droplets that later solidify into a spherical shape. With fluidized bed combustors the temperatures are usually below ash fusion temperatures resulting in irregularly shaped fly ash particles [15, 16]. This does not invalidate the use of the PAS technique, but makes the values of the absorption coefficient and the PAS signal more difficult to predict.

The particle size distribution influences the mass specific absorption coefficient, A_a . With fluidized bed combustors the mean size of the fly ash is almost solely dependent on the operating conditions (temperature, excess air, superficial gas velocity and fly ash recycle) and relatively independent of coal type [17, 18]. Two frequency distributions have been postulated for fluidized bed combustion, one distribution less than 100 μm and another distribution greater than 100 μm , where the largest size present in the fly ash is dependent on the gas velocity above the minimum for fluidization and the recycle of the fly ash [17, 18, 19, 20]. With high recycle of the fly ash, the mean particle diameter of the fly ash decreases significantly [15, 18]. Merrick and Highley [18] report that the size distribution of fines

produced is approximately constant for a particular bed material and is independent of the bed size distribution or operating conditions of the fluidized bed combustor. Particles in the second distribution (greater than 180 μm) contain mostly unburned carbon particles [21]. Pulverized coal combustors start with a much smaller feed (90% < 75 μm). Therefore, the top size of the pulverized coal fly ash is much smaller than the fluidized bed fly ash.

2.2 Particle Interactions with Radiation

The interactions between particles and radiation are dependent on the complex index of refraction of the particles, the diameter of the particles and the wavelength of the incident radiation. The interaction can be either scattering or absorption of the radiation; the sum of these two quantities is called extinction. Mie derived the classic formulation for the scattering and extinction of light which gives the scattering cross section, C_s , and extinction cross section, C_e , as a function of the above properties [22]. The absorption cross section, C_a , is given by the difference between C_e and C_s .

In the measurement of particulate mass concentrations, it is preferable to develop properties on a mass basis [23]. The absorption cross section on a mass basis for a single-sized particle system is

$$R_a = \frac{C_a}{\frac{1}{6}\pi\rho D^3} \quad (2.1)$$

where R_a is defined as the absorption cross section per unit mass of a particle of diameter D , and ρ is the particle density.

Kerker [24] developed an algorithm based on the complex index of refraction, $m = n - ik$, for calculation of C_a . Large values of $|m|$ are characteristic of particles that are highly reflective with a small value of C_a . Large values of C_a are produced when $|m - 1| \ll 1$

and $k \ll n$ [25]. The absorption cross section per unit mass, R_a , can then be calculated using Eq. (2.1). The algorithm, as well as a computer program implementing the algorithm, is listed in Appendix A. Using this program, R_a as a function of particle size was calculated for the absorption at the helium-neon laser wavelength (632 nm). This is shown in Figure 2.1. For Rayleigh particles ($D \ll \lambda$) the mass specific absorption cross section, R_a , is independent of particle size. In the limit of large particles ($D \gg \lambda$), R_a is inversely proportional to the particle diameter and can be estimated by [22]

$$R_a \approx \frac{3}{2} \left(\frac{1}{\rho D} \right) \quad (2.2)$$

This approximation assumes that the particles behave as blackbody radiation absorbers. Although soot and carbon approach the assumption of a black body, a correction is still needed. For a black body, the absorption efficiency, Q_a , for large particles is 1.0 [25]. Where Q_a for spheres is given by

$$Q_a = \frac{C_a}{\pi r^2} \quad (2.3)$$

For carbon particles interacting with helium-neon laser radiation ($\lambda = 632$ nm, $m = 1.8 - 0.4 i$), Q_a approaches 0.9 in the large particle limit ($D \gg \lambda$).

Since fly ash is not monodisperse in size, R_a must be adjusted to account for the effects of a size distribution. The absorption coefficient, b_a , takes into account a particle size distribution [23]

$$b_a = \int_0^{\infty} R_a(D) m(D) dD \quad (2.4)$$

where the total mass concentration of size D is given by

$$m(D) = \rho \left(\frac{\pi D^3}{6} \right) N f(D) \quad (2.5)$$

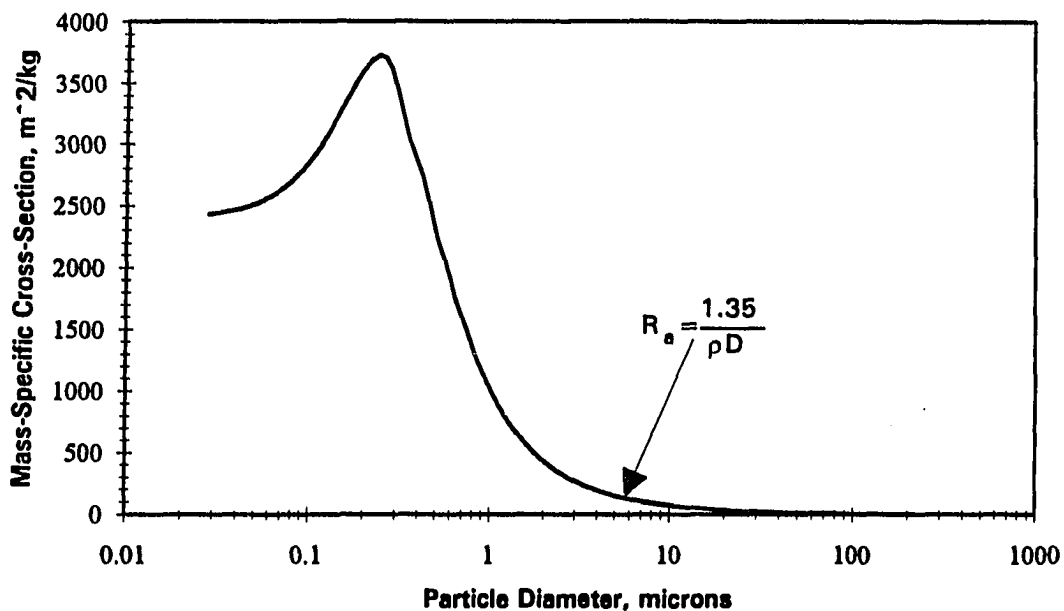


Figure 2.1: Mass specific absorption cross section for carbon spheroids at 632 nm

The total mass concentration satisfies

$$M_c = \int_0^{\infty} m(D) dD \quad (2.6)$$

where $f(D)$ is the diameter frequency distribution, N is the total number of absorbing particles and M_c is the mass concentration of absorbing particles. Combining Eq. (2.4) and Eq. (2.6) produces an expression for the mass specific absorption coefficient, A_a

$$A_a = \frac{b_a}{M_c} = \frac{\int_0^{\infty} R_a(D) m(D) dD}{\int_0^{\infty} m(D) dD} \quad (2.7)$$

where A_a was chosen to be based on the mass concentration of absorbing particles and not on the total mass concentration. The reason for this choice is explained in section 2.5.

The only restriction on the previous discussion is that multiple scattering does not occur [22]; that is the particles in the particle cloud interact independently with the incident radiation. This restriction is satisfied for

$$M_t < \frac{1}{A_e L} \quad (2.8)$$

where M_t is the total mass concentration, A_e is the mass specific extinction coefficient and L is the length of the PAS cell. For large mean diameters ($D \gg \lambda$) [23]

$$A_e \approx R_e \quad (2.9)$$

where R_e is given by

$$R_e \approx 3 \left(\frac{1}{\rho D} \right) \quad (2.10)$$

Using standard values for fluidized bed combustion fly ash: $\bar{D} = 20 \mu\text{m}$ (a conservative estimate) and $\rho = 2000 \text{ kg/m}^3$, and Eq. (2.8), Eq. (2.9), and Eq. (2.10), with $L = 0.1 \text{ m}$, the mass loading must be less than 130 g/m^3 . A mass loading of 130 g/m^3 is well in excess of mass loading expected from a bubbling fluidized bed combustor [26]. Dilution of the combustion exhaust is not necessary to meet this criteria.

2.3 The Photoacoustic Signal

The photoacoustic technique is based on periodic absorption of radiation from an amplitude modulated source. The energy absorbed by the sample is converted to heat which results in a minute temperature rise in the surrounding gas. Subsequent expansion of the heated gas causes a periodic pressure fluctuation at the light modulation frequency which

propagates through the gas as a sound wave. This wave can be detected by an appropriately sensitive microphone and a lock-in amplifier [2, 3].

To enhance the photoacoustic effect for absorption measurement, the sample is contained in a gas-filled chamber, called the photoacoustic cell. Roessler and Faxvog [27] derived an equation for the photoacoustic signal, S , from the wave equation for nonresonant operation with the assumption that the pressure rise is uniform throughout the cell (see Appendix B for the derivation):

$$S = R \left(\frac{b_a}{b_e} \right) [1 - e^{-b_e L}] W / L \quad (2.11)$$

where R is the cell responsivity (mV m/W), b_a is the absorption coefficient (m^{-1}), b_e is the extinction coefficient (m^{-1}), L is the optical path length of the sample (m), and W is the average optical power incident on the sample (W). For the case of weak attenuation, $b_e L \ll 1$, Eq. (2.11) simplifies to

$$S \approx R b_a W \quad (2.12)$$

on a mass basis

$$S \approx R A_a M_c W \quad (2.13)$$

where M_c is the mass concentration of absorbing particles in the sample (carbon) and A_a is the mass specific absorption coefficient based on the absorbing particles in the sample.

Both Eq. (2.12) and Eq. (2.13) show that the signal is proportional to the cell responsivity and excitation power. Responsivity, a measure of the instrument gain, is a function of cell geometry and material, modulation frequency, and microphone sensitivity. The theoretical cell responsivity, R , for nonresonant operation is [27]

$$R = \frac{4(\gamma - 1)\sigma_m L}{\pi \omega V \sqrt{2}} \quad (2.14)$$

where γ is the ratio of specific heats (C_p/C_v), σ_m is the microphone sensitivity (mV/Pa), L is the optical path length of the photoacoustic cell (m), V is the total volume of the cell, and ω is the angular modulation frequency (rad/s). In practice, the cell responsivity is determined by measuring the photoacoustic signal from a gas with well-defined absorption characteristics.

Propagation of Error analysis can be performed using Eq.(2.13) and Eq. (2.14) to determine the expected variance in the PAS signal. This was performed in Appendix C. The values of $(\Delta S/S)_{\text{microwave}} = 12.4\%$ and $(\Delta S/S)_{\text{optical}} = 11.6\%$ were calculated. The largest source of error is due to $\frac{\Delta M_c}{M_c}$, caused by the width of the screw auger.

Eq. (2.14) shows that low frequencies give the highest responsivity, however, the magnitude of the background noise is less at higher frequencies [28, 29]. The theoretical upper limit for the modulation frequency is given by the thermal time constant τ [30]:

$$\tau = \frac{r^2 C_p}{3k_a} \quad (2.15)$$

where r is the particle radius, C_p is the heat capacity per unit volume of the particle, and k_a is the thermal conductivity of the air or surrounding medium. This limit on the modulation frequency assumes that the entire particle is heated uniformly, which is satisfied for small particles ($\bar{r} \sim 6 \mu\text{m}$ for $f \sim 1000 \text{ Hz}$). Research done with carbon particles ($\bar{r} \sim 25 \mu\text{m}$) showed that frequencies higher than this limit can be used effectively [10].

The thermal time constant requires that the modulation frequency be less than 25 Hz for the size of particles involved in this study ($\bar{r}_{\text{max}} = 38 \mu\text{m}$). At a frequency of 25 Hz, the noise interference would be so great that the PAS signal could not be detected. Therefore, it is advantageous to exceed this limit to increase the signal-to-noise ratio to a workable level.

2.4 PAS Cell Design

The design of the PAS cell is the key to using PAS to measure carbon concentrations in fly ash. The cell responsivity, R (Eq. 2.14), contains all the PAS cell design parameters. The maximization of the cell responsivity is the first step in PAS cell design, but there are other important factors to consider.

The maximization of the cell responsivity involves many factors as can be seen from Eq. (2.14). The value of γ is fixed because the fly ash is entrained in air. A large value of σ can be obtained from a highly sensitive microphone. Both of these parameters do not require much consideration, but the other two parameters, ω , and the cross sectional area of the cell, require careful consideration.

As the modulation frequency is lowered, the PAS signal increases, but the noise on the signal also increases. Modulation frequencies of greater than 750 Hz are usually chosen to achieve workable signal to noise ratios.

The last term that influences the cell responsivity is the PAS cell cross-sectional area. As the cross-sectional area is decreased the responsivity increases as shown in Eq. (2.14). An important qualification on the PAS signal derivation is that the pressure rise is assumed uniform throughout the cell. This is a good assumption when the PAS cells are sealed, but when sample lines are added to the cell, this assumption breaks down slightly. The PAS pressure wave at the inlet and outlet ports will not be the same as the PAS pressure wave at the center of the PAS cell. Adams [29] has shown that the "inlet and outlet ports act as pneumatic short circuits for the induced pressure". PAS pressure waves are partially transmitted out the sampling lines. The fraction of the reverberant pressure wave that is lost is given by the pressure-amplitude transmission coefficient [31, 32].

$$T \approx \frac{2\pi r_{\text{sample}}^2}{\pi r_{\text{sample}}^2 + \pi r_{\text{PAS cell}}^2} \quad (2.16)$$

Therefore, the PAS cell diameter should be small, but the sample lines should also be small compared to the PAS cell diameter. This sets a practical limit for the PAS cell diameter since sample lines must be large enough to transport particulates.

2.5 Diesel Exhaust Measurements

Previous experimental work in measuring soot of diesel exhaust emissions with PAS serves as a starting point in designing a PAS system for measuring unburned carbon in fly ash. A PAS system for the measurement of soot in diesel vehicle exhaust consists of a constant flow dilution tube, horizontal PAS cell, microphone, signal processing equipment, and an amplitude modulated radiation source [33]. The combustion products enter a constant flow dilution tube where the dilution ratio is 3:1 to 10:1, depending on the engine operating conditions. This dilution assures isokinetic sampling without having to change the flow rate through the PAS cell. The microphone picks up the acoustic signal, and the signal is processed by a lock-in amplifier to extract the PAS signal.

The PAS method is suited for the detection of soot in diesel exhaust because the carbon in soot is composed of about 1% trace metals from fuel and lubrication oil additives and about 2% sulfates, neither of which contribute significantly to the photoacoustic signal. The remaining material, greater than 95% by mass, is carbonaceous in nature, a strong absorber of radiation [4].

If the mass specific absorption coefficient, A_m , remains constant, the signal will have a direct correlation to the mass concentration of the material in the sample. Roessler and Faxvog [34, 35] found that the mass specific absorption coefficient for diesel exhaust particles

varied with operating conditions by up to a factor of two. Although the mass specific absorption coefficient for diesel particles varied with operating conditions, Japar, Szkarlat and Pierson [36] found the mass specific absorption coefficient of the elemental carbon contained in the diesel exhaust particles did not vary significantly with vehicle operating conditions. Therefore, if A_a is based on the mass concentration of absorbing particles, M_c (carbon), and not on the total mass concentration, M_t (fly ash), the PAS signal can give quantitative information about the mass loading of elemental carbon in vehicle exhaust.

Two different radiation sources have been tried in conjunction with soot monitoring using PAS. CO_2 lasers ($\lambda = 10.6 \mu\text{m}$) have been used so that the soot particles satisfy Rayleigh scattering criteria ($D \ll \lambda$), but at this wavelength gaseous combustion products also absorb the infrared radiation and produce a significant photoacoustic signal. A two cell photoacoustic device was needed to subtract out the combustion gas contribution to the signal [4, 7, 37]. With the dual-cell setup care must be taken that the gaseous components going through each cell are equivalent; one flow with soot particulates and the other flow with soot particulates filtered out [4, 7, 37].

PAS research with argon-ion lasers ($\lambda = 0.55 \mu\text{m}$) showed that light absorption by soot particulates could be approximated as Rayleigh scattering [4, 5]. The advantages of working at this wavelength are two fold. First, the mass specific optical absorption coefficient A_a is about an order of magnitude greater at this wavelength, resulting in a direct gain in the PAS sensitivity. Second, the dual-cell method is no longer needed because visible radiation, unlike infrared radiation, is not appreciably absorbed by combustion gases [7].

Roessler has worked with resonant [5] and nonresonant [38] PAS systems. Although the sensitivity can be increased by using a resonant system, the system must be continually tuned to keep the PAS cell in the resonant mode due to small variations in temperature and flow rate through the PAS cell. Although temperature affects the frequency at which the cell

operates in the resonant mode, there is no effect on the nonresonant PAS signal for temperatures above 28°C [39]. The nonresonant PAS system provides much greater calibration stability and allows for changes in the flow rate through the PAS cell without affecting the calibration [37].

2.6 Carbon Detection in Synthetic Fly Ash

The detection of carbon in fly ash differs from the detection of soot in diesel engines because the size of the fly ash particles is over an order of magnitude greater. This affects the absorption of the incident radiation (section 2.2) and more care must be taken to keep the particles entrained in the air flow.

Brown and Dona [10, 40] have used PAS to measure carbon in airborne synthetic fly ash. Their experimental set-up is similar to that used for diesel applications [33] with the following modifications: a vertical orientation of the photoacoustic cell was used, a helium-neon laser replaced an argon-ion laser, and no constant flow dilution tube was used with the particulate supply system. If a horizontal configuration was used, the air velocity would need to be very high to keep the particles entrained in the air stream. Osada et al. [37] have shown that photoacoustic response is independent of gas flow rate only for relatively low flow velocities. They suggest that flow velocities should be less than 0.40 m/s through the cell. The vertical orientation of the cell helps prevent particles from settling out of the air flow while maintaining a relatively low flow velocity. Brown and Dona used a fluidized bed to entrain the synthetic fly ash in air before passing it through the PAS cell. Synthetic fly ash is a combination of Illinois No. 5 coal screened to 270 by 325 mesh ($53 \times 45 \mu\text{m}$) and pulverized coal fly ash that has been previously heated in an oxidizing environment to remove all the carbon. Synthetic fly ash differs from actual fly ash in that the carbon did not pass through a

combustion zone and was monodisperse in size. With a total mass loading of 2 g/m^3 , Brown and Dona [10, 40] were able to measure carbon-in-fly ash in the range of 30% to 100%. Acoustical noise overwhelmed the PAS signal at carbon concentrations below 0.6 g/m^3 . This value is around the upper limit expected from industrial size fluidized bed combustors.

2.7 On-Line Carbon Detection in Fluidized Bed Fly Ash

Dykstra and Brown [6] performed on-line PAS measurements of carbon in fly ash using a helium-neon laser as an excitation source. The fly ash was sampled from a laboratory scale fluidized bed combustor with a sand bed to which 5% limestone had been added. Three different mass loadings through the PAS cell were obtained for each operating condition by dilution of the sampled flue gas (Figure 2.2). The results show that the PAS signal is linear with carbon loading for a given combustor operating condition (that is, for a given particle size distribution and morphology); however, a different line was obtained for each operating condition.

It is instructive to compare these results with the PAS signal expected for unburned carbon emitted from the laboratory scale fluidized bed combustor. If the carbon particles are large spheres ($D \gg \lambda$), then the mass specific cross section is give by Eq. (2.2). The integrated mass specific absorption coefficient, A_a , can be obtained by substituting Eq. (2.2), using ρ for char as approximately 1800 kg/m^3 and the mass distribution, $m(D)$, for unburned carbon into Eq. (2.7). In the absence of the mass distribution for char, substitution of the mass distribution for fly ash (a mixture of unburned carbon and mineral matter particles), which we obtained from Microtrac analysis, was used (see Figure 2.3).

This mass distribution, with an average particle diameter of $34 \text{ }\mu\text{m}$, yields an expected A_a of $0.078 \text{ m}^2/\text{g}$. The PAS signal predicted by Eq. (2.12) is plotted in Figure 2.2. The

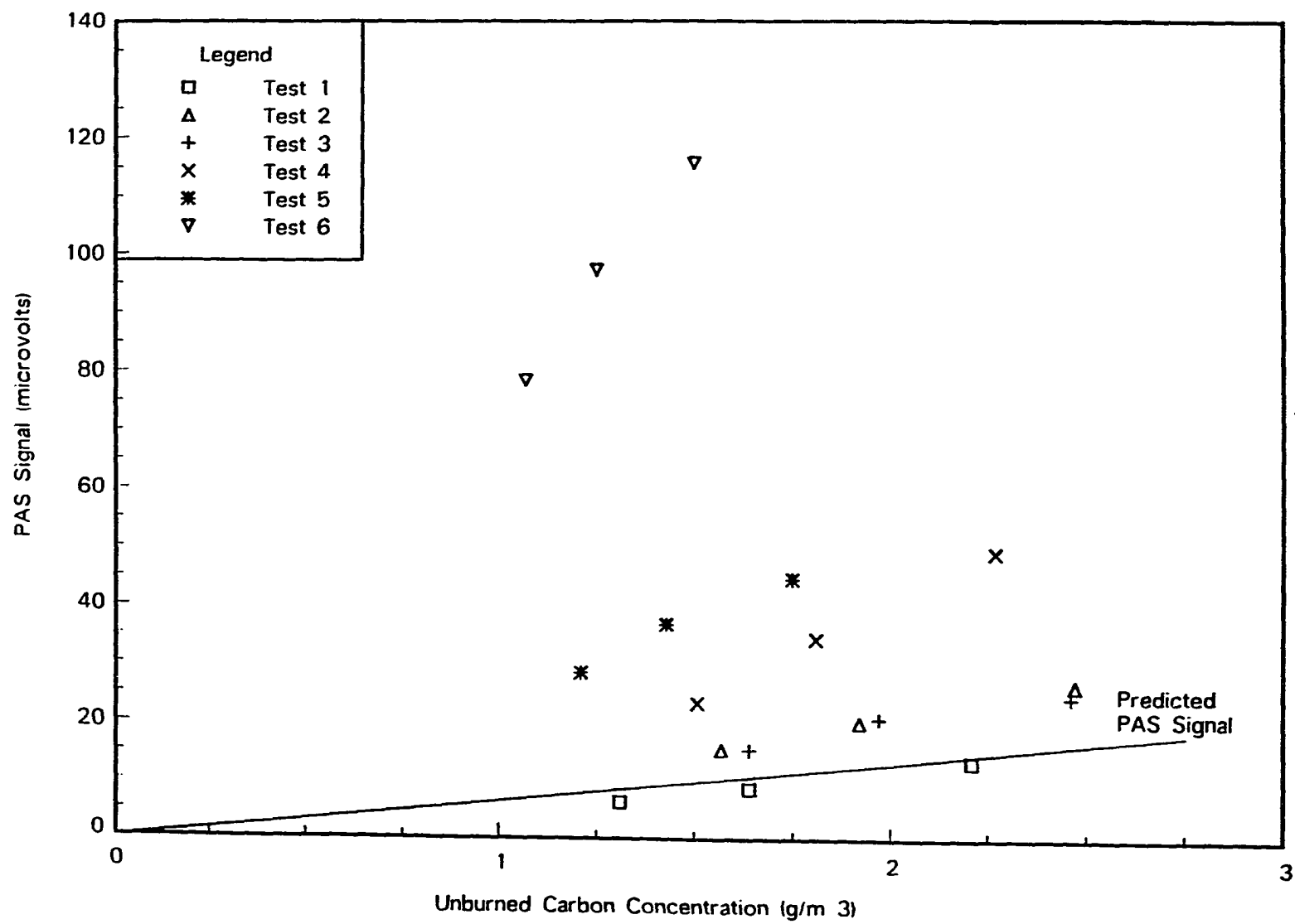


Figure 2.2 The influence of condensed tar on the PAS signal

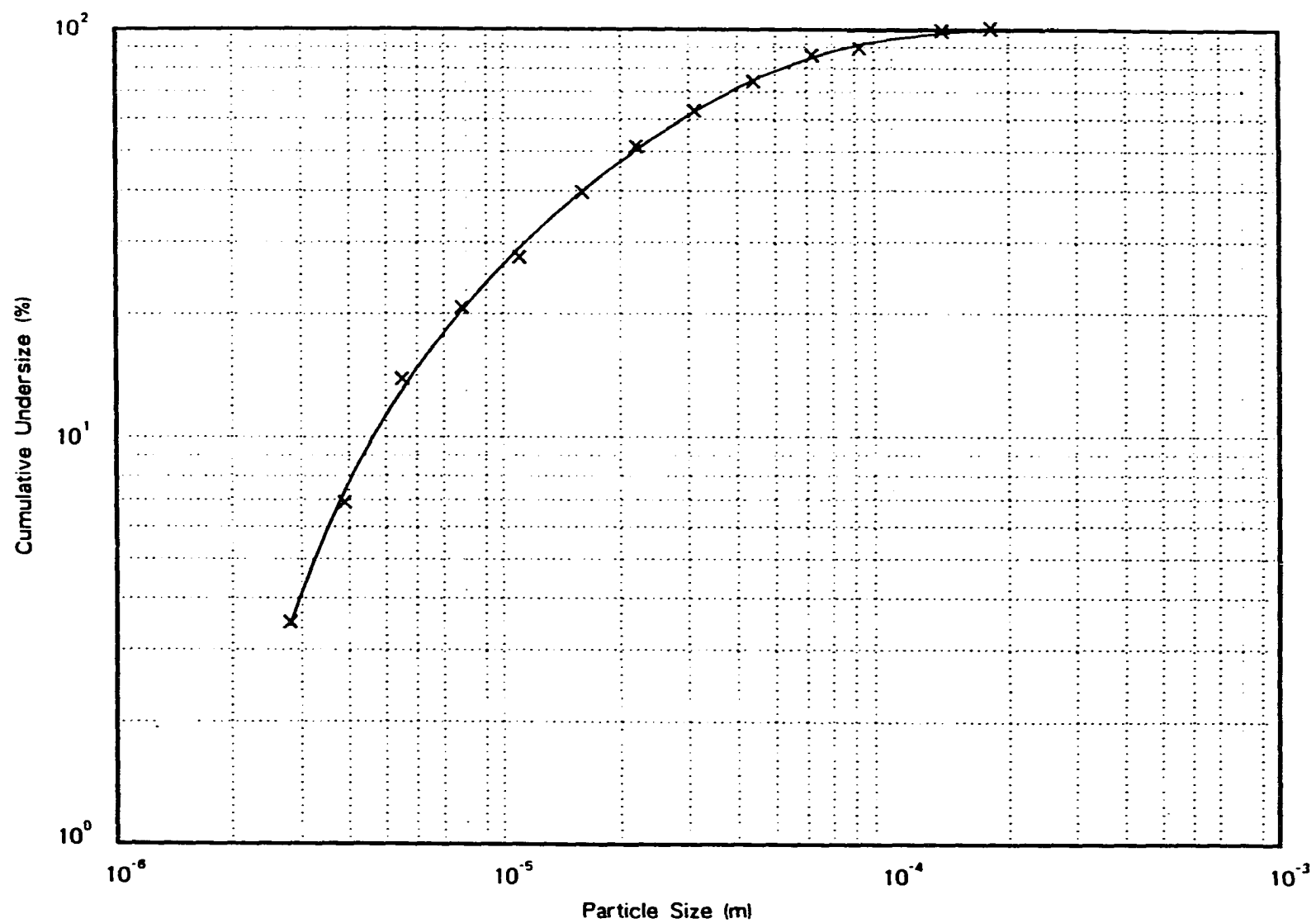


Figure 2.3 Mass distribution of the fluidized bed fly ash

predicted PAS signal is in close agreement with Test 1, which was the first test performed. The agreement between prediction and experiment becomes progressively worse for subsequent tests, especially for Tests 4 to 6. This was due to the formation of tar in the combustion zone which also produces a PAS signal. As the small amount of limestone attrited and elutriated from the bed, the amount of tar leaving the fluidized bed increased. The limestone serves as a catalysis for the dissociation of tars to low molecular weight hydrocarbons.

Although tar produced a PAS signal, tar is not a normal constituent of flue gas from industrial and utility scale boilers [16, 41, 42]. It appeared in these experiments as an artifact of the uninsulated freeboard of the laboratory-scale combustor. Accordingly, additional tests were performed to exclude both tar and uncalcined limestone from the flue gas. The second set of tests (Tests 9-14) employed a bed of 69% calcined and/or sulfated limestone and 31% sand.

Figure 2.4 shows that Tests 9-11, 13, and 14 are well correlated to a straight line running through the origin. The slope of this line yields an integrated mass specific absorption coefficient of $0.0494 \text{ m}^2/\text{g}$ which is within 37% of the value predicted from the mass distribution of the laboratory scale fluidized bed fly ash. This discrepancy is probably due to shadowing of the carbon in carbon and ash composite particles as well as an underestimation of \bar{D} . Test 12, which was performed at the lowest temperature and superficial velocity, produced a PAS signal much lower than expected when compared to the other tests. Scanning electron microscope (SEM) photographs were taken of the fly ash from test 9 and 12 to understand this abnormality. Examination of these photographs suggested that the low temperature of test 12 results in carbon particles roughly rectangular in shape, many with length-to-width ratios of 3 to 1, whereas higher temperatures (test 9) produce carbon particles that are more spherical, although still irregularly shaped. From examination of the

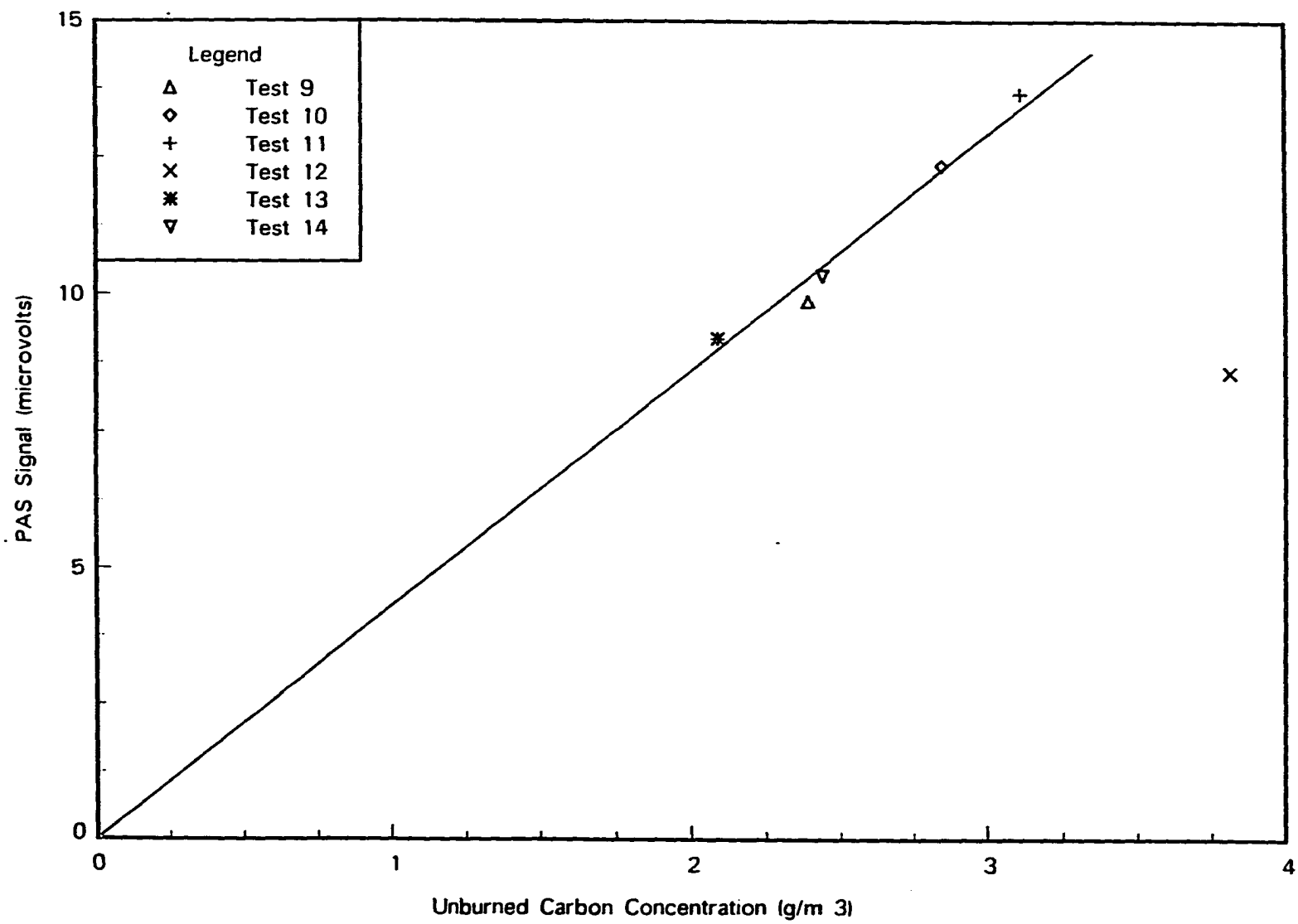


Figure 2.4 The PAS signal for fluidized bed fly ash

photographs, the average size of particles in Test 12 appears to be larger than for Test 9. The longer shapes and larger sizes of particles in test 12 will yield lower mass specific absorption coefficients than smaller, spherical particles and may explain the smaller PAS signal. Further tests are needed to determine if this contention is accurate. A useful carbon monitor should give values of the carbon loadings independent of the fly ash size distribution.

3. EXPERIMENTAL APPARATUS

Two different instruments for detecting unburned carbon were constructed and tested: one based on a helium-neon laser excitation source and the other based on microwaves as the excitation source. The particle entrainment facility and the data acquisition system are the same for both instruments.

PAS tests were performed using five different size ranges of stoker fly ash. The tests involved entrainment of the fly ash by 40 SCFM of air at room temperature through an empty fluidized bed. Airborne fly ash samples were extracted for PAS analysis. A variable speed auger was used to control the carbon loadings passing through the PAS cell.

Prior to the successful operation of the PAS cell, acoustical noise needed to be reduced. The effects of acoustical noise are two fold. First, the magnitude of these pressure waves can cause overloading of the microphone and the lock-in amplifier. Second, the PAS sensitivity is limited by noise interferences at the same frequency as the modulation frequency.

3.1 Acoustical Noise Reduction

Noise sources that can interfere with both microwave and optical PAS measurements include noise generated by: the absorption of radiation by cell windows and the cell wall, room noise, flow noise and pressure waves generated by the environment at the sampling extraction point that propagate through the sample line. A bandpass filter can significantly reduce noise, but additional methods are needed.

The magnitude of the window noise is small because absorption of radiant energy is small. The central chamber is long enough that the window heating effects dissipate before reaching the microphone.

The optical PAS cell was isolated from room noise by construction of one acoustical isolation box inside another (described in section 3.5). When running PAS tests there was no noticeable difference between operation with the doors to the acoustical boxes open or closed. Therefore, at the modulation frequency of the PAS tests (> 800 Hz), room noise does not play a significant role and an isolation box was not used with the microwave instrument.

Flow noise can be eliminated by having stagnation conditions in the PAS cell [37]. For tests with particulates, this is impractical because the particulates would settle out of the gas. Therefore, a vertical orientation is used and flow rates are kept as low as is practical and the flow passages for the flue gas entering the cells are angled upward to reduce eddy currents which generate acoustical noise in the cell.

External acoustical noise entering the PAS cell comes from two main sources: the sampling extraction point and the vacuum pump, with the sampling extraction point being the more serious problem. Faxvog and Roessler [5] isolated their PAS apparatus from the pressure fluctuation generated from the exhaust of a diesel engine by using a constant flow dilution tube with dilution ratios of 3:1 to 10:1. The high dilution ratio damps out any pressure fluctuations that were generated by the operation of the diesel engine. Although this method works well for their situation, the PAS signal generated by a diluted sample of flue gas would not be large enough to measure in the present apparatuses.

The flow of air through a fluidized bed to entrain the fly ash particles causes pressure fluctuations that propagate through the sample line. If these pressure fluctuations are not dampened, they will cause overloading of the microphone and lock-in amplifier. One approach to reducing these pressure fluctuations is the use of a Helmholtz resonator.

Helmholtz resonators have been used in the noise control industry to reduce noise of selected frequency ranges at a measuring point [32, 43, 44]. The classic model of a Helmholtz resonator consists of a rigid-walled volume connected to an external environment by a small opening. Looking at the specific impedance of a Helmholtz resonator provides insight into how a Helmholtz resonator reduces the sound pressure at a measurement location [44].

$$Z_H = \frac{1}{2\pi} \rho_0 \frac{\omega^2}{c_0} + i \rho_0 \left(\frac{c_0^2 S_H}{\omega V_H} - \omega L_e \right) \quad (3.1)$$

This equation is valid for $\omega r_H / c_0 \ll 1$, which is the case for this application. The variables are listed in Table 3.1.

Table 3.1 Helmholtz design variables

Variable Name	Variable interpretation
ρ_0	mean density
c_0	speed of sound
ω	angular frequency (rad/s)
S_H	cross sectional area of the resonator neck (πr_H^2)
V_H	volume of the Helmholtz resonator
L_e	equivalent resonator neck length = $L_H + 8r_H/3\pi$
L_H	resonator neck length
r_H	radius of resonator neck

The resonant frequency is calculated by setting the imaginary part of Z_H to zero.

$$f_H = \frac{c_0}{2\pi} \sqrt{\frac{S_H}{L_H V_H}} \quad (3.2)$$

A Helmholtz resonator incorporated as a side branch in the duct system greatly reduces the sound transmitted near the resonant frequency. The pressure wave traveling down the duct is reflected back towards the source of the pressure wave. Therefore, a

Helmholtz resonator must be placed between the source of noise and the PAS cell. This will insure minimum transmission of unwanted pressure waves into the PAS cell. The fraction of the pressure wave that travels past the Helmholtz resonator towards the PAS cell is given by the pressure-amplitude transmission coefficient, T . The pressure-amplitude transmission coefficient is a function of frequency and geometry, and is given by [32]

$$T = 1 - \frac{\rho_0 c_0 / A_D}{2Z_H + \rho_0 c_0 / A_D} \quad (3.3)$$

where A_D is the cross sectional area of the duct.

Near the resonant frequency the transmission drops off to a small fraction of the incident pressure wave because the value of Z_H becomes much less than $\rho_0 c_0 / A_D$. If an absorbing material is added inside the Helmholtz resonator the pressure-amplitude transmission coefficient, T , no longer approaches zero, but the value of T remains small over a larger frequency range than predicted by Eq. (3.3) [43]. Fiberglass was added inside the Helmholtz resonators because the frequency from the fluidized bed contains a range of low frequency noise and the design equations only estimate the resonant frequency. The actual resonant frequency may be slightly higher or lower.

The Helmholtz resonator used for the reduction of low frequency noise of the fluidized bed had a resonant frequency of 20 Hz (Eq. 3.2, $V=254.5 \text{ in.}^3$, $L_e=0.553 \text{ in.}$, $S_H=0.012 \text{ in.}$). The Helmholtz resonator is connected to the sample line via a tee in the sample line between the fluidized bed and the PAS cell. This resonator is located approximately 4 feet from the PAS cell. The exact positioning of the Helmholtz resonator is not important provided the Helmholtz resonator is between the fluidized bed and the PAS cell because noise pick-up through the sample line walls was minimal. The fly ash sample passes uninterrupted past the Helmholtz resonator, but the low frequency noise is reflected back to the fluidized bed.

The pump was a very steady noise source at 33 Hz with a sound pressure level of 120 dB. This needed to be reduced before any PAS measurements could be attempted. The pump, through vacuum action, creates an unwanted pressure wave that travels upstream towards the PAS cell. Therefore, the pressure wave does not pass by the Helmholtz resonator used to control the noise generated by the fluidized bed.

The first approach to damping out the pump noise was to use a venturi instead of the vacuum pump to draw the sample through the PAS cell. Although the use of the venturi decreased the total magnitude of the noise by a factor of 2, the higher frequency terms (>800 Hz) actually increased as compared to a vacuum pump. This caused severe interference with the PAS pressure wave (825 Hz). Therefore, a vacuum pump was used to draw the sample through the PAS cell and methods were explored to reduce the noise that the vacuum pump generated. Eventually, the vacuum pump noise was reduced by the addition of a second Helmholtz resonator placed between the vacuum pump and the PAS cell. This resonator was placed 5 feet downstream of the PAS cell. Again, the exact position of the Helmholtz resonator is not important provided the resonator is placed between the PAS cell and the pump. This Helmholtz resonator is made from a 3 inch diameter PVC pipe 18 inches long filled with fiberglass and had a resonant frequency of 28 Hz (Eq. 3.2, $V=127.25$ in³, $L_e=0.553$ in, $S_H=0.012$ in²). The Helmholtz resonator provided significant reduction in pump noise (Figure 3.1).

A high-pass filter was designed to further eliminate low frequency noise [45]. The filter had a cutoff frequency of 500 Hz and eliminated the low frequency noise that the microphone detected.

After the noise was reduced to an acceptable level, the band pass filter on the lock-in amplifier in conjunction with a well-chosen modulation frequency further increased the PAS sensitivity. The modulation frequency is chosen by filling the PAS cell with NO₂ and then

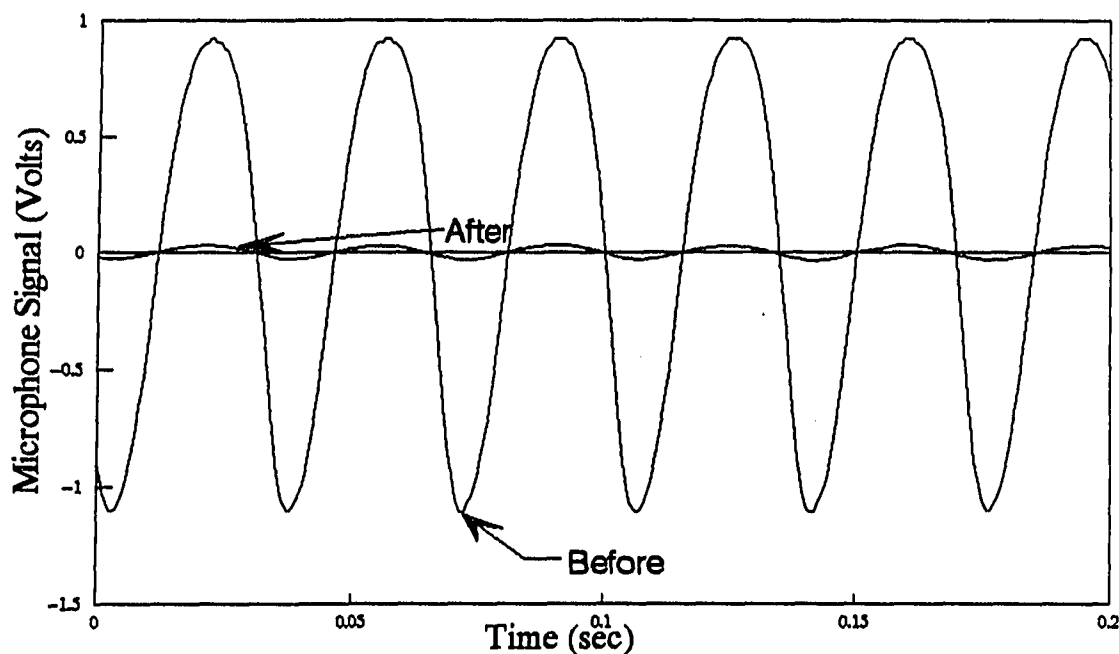


Figure 3.1 Reduction of pump noise at the microphone with a Helmholtz resonator

tabulating the PAS signal as a function of frequency and comparing these result with the PAS signal from an empty PAS cell at the same frequency. A large ratio of the NO_2 PAS signal to the empty PAS cell signal is desired. Roessler [38] has observed that noise interferences from the exhaust of a diesel engine are greatest at low frequencies, but the PAS signal is inversely proportional to the modulation frequency. Roessler [38] found that the noise interference decreased faster than the PAS signal as the modulation frequency was increased. He concluded that 2-3 kHz was a reasonable range for operation. The same functional relationship was also observed in the PAS apparatus under investigation, but with the conclusion that frequencies above 800 Hz were acceptable. The PAS signal of NO_2 is displayed in Figure 3.2 as a function of frequency and shows the $1/f$ dependence of the PAS signal.

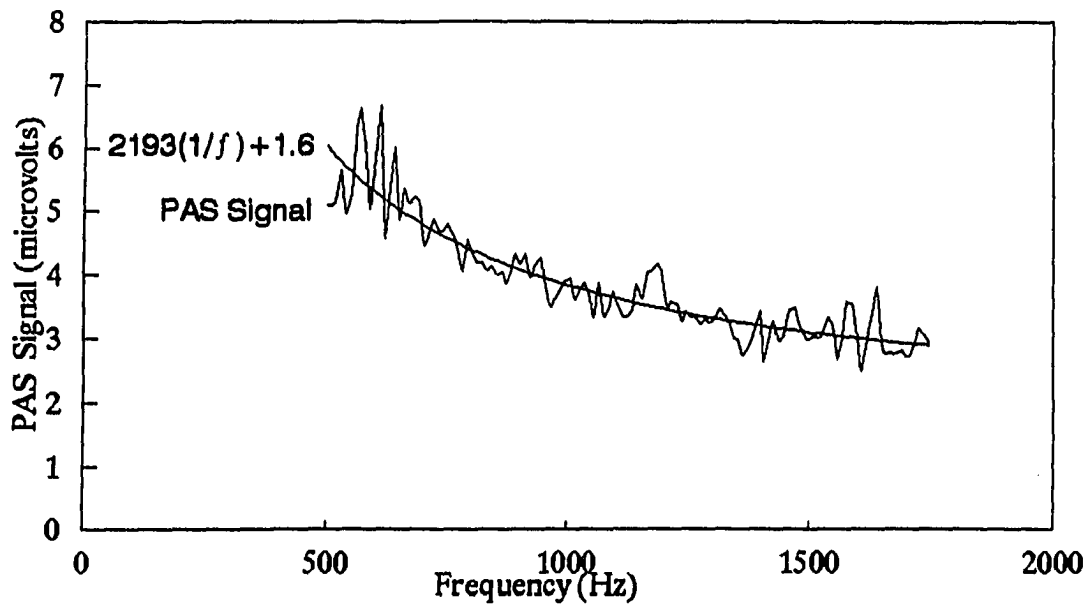


Figure 3.2 NO₂ PAS signal verses frequency

3.2 Particle Entrainment Facility

An eight inch diameter fluidized bed combustor has been utilized to entrain fly ash particles. The fluidized bed combustor is shown in Figure 3.3. The fluidizing medium was removed from the bed, but no other modifications were needed.

The distributor plate consists of 250 3/32 inch diameter holes evenly distributed in 1/2 inch stainless steel. Spot welded to the distributor plate is a 100-mesh stainless steel screen. The inner wall of the combustion bed is made of stainless steel lined with a 1 inch thick layer of Kaocast RFT castable refractory. An insulated, mild steel freeboard extends 4 feet above the distributor plate. The freeboard length allows time for the fly ash to evenly distribute in the air before the sample is extracted for PAS analysis. The majority of the flue gas exits from

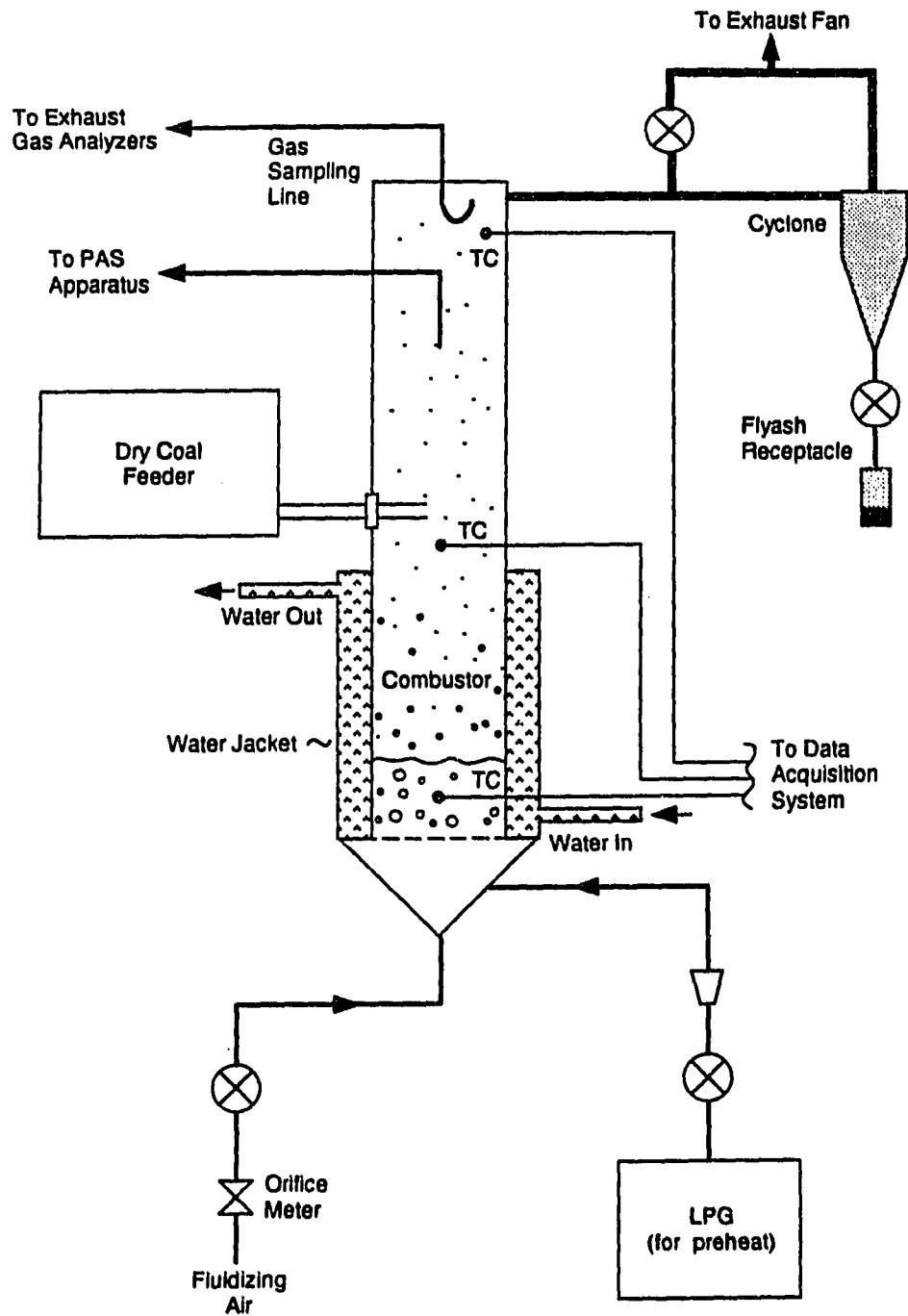


Figure 3.3 The laboratory scale fluidized bed combustor

the top of the combustor through a high efficiency cyclone that captures over 97% (on a mass basis) of the fly ash particles.

Airflow through the bed was calculated from pressure drops across orifice flow meters. A linear variable displacement transformer (LVDT) pressure transducer was used to measure the pressure drop across the orifice meter. The analog output of the transducer was converted by a DAS-8 A/D converter. The digital signal was used by the microcomputer to convert the pressure drop to a corresponding flow rate using calibration test data.

The fly ash was metered into the combustor by an AccuRate dry chemical feeder (model 602), equipped with a 3/4 inch diameter, variable speed helix and a 5 gallon vinyl hopper. The feeder provides feed rates of fly ash from 1 to 50 pounds per hour. The hopper was sealed during operation to prevent back flow of fly ash.

3.3 Particulate Sampling

Particulate sampling is the same in both the optical PAS and the microwave PAS systems. A probe descends 12 inches down the center of the freeboard and provides isokinetic sampling of the fly ash for PAS analysis. Isokinetic sampling involves removing a sample without disturbing the streamlines of the flow system. This is important because the carbon loadings passing through the PAS cell are determined from LOI analysis [1] of the cyclone catch. Fly ash samples passing through the PAS cell are not large enough to perform LOI on. This method of determining the carbon loadings passing through the PAS cell is valid only if isokinetic sampling is employed.

3.4 Data Acquisition System

Measurement of the photoacoustic signal requires the use of several electronic instruments: a microphone system, electronic filters, a lock-in amplifier, and a computer. These components are the same for both the optical and microwave PAS systems.

The pressure measurements were done with an ACO Pacific model MK224 microphone recessed into the midpoint of the PAS cell. The microphone is used in conjunction with a preamplifier (ACO Pacific model 4012) and powered by an ACO Pacific model PS 9200 power supply. The 1/2 inch electret condenser microphone has a sensitivity of -26 dB referenced to 1 V/Pa (50 mV/Pa). The microphone is protected from any fly ash impingement by a 10 μm screen.

The microphone signal is carried via coax cable to a high pass filter before it enters the signal port of the lock-in amplifier. The high pass filter was designed to have a cut-off frequency of 500 Hz [45]. The filter greatly attenuates any low frequency noise.

A Stanford Research Systems model SR530 lock-in amplifier extracts the PAS signal from the microphone signal. The lock-in amplifier has two inputs: the conditioned microphone signal and a reference signal. The reference signal is at the same frequency as the frequency of modulation. For the optical PAS system the reference signal is provided from a Stanford Research Systems model SR540 optical chopper. The microwave system has a hard wired modulator that modulates the microwave energy and also provides a reference signal to the lock-in amplifier. The lock-in amplifier displays both the signal magnitude and the phase shift from the reference signal.

The microphone power supply, high-pass filter, and the lock-in amplifier are all contained inside an aluminum box to shield these electronic components from electromagnetic radiation.

A Zenith 286 computer controls the experimental apparatus. The computer and the lock-in communicate to each other through the serial port on the computer. The computer remotely controls the lock-in amplifier. A data acquisition program (see Appendix D) is used to control all functions on the lock-in amplifier and optical chopper, as well as run routines that allow for PAS signals to be measured as the computer steps through a series of different chopping frequencies. The computer displays the lock-in settings, the modulation frequency and the PAS signal. The PAS signal is also graphed in real time as a function of time. This data can be recorded on the hard disk and later imported into a spread sheet for analysis.

3.5 Optical PAS System

The optical PAS system is designed to measure the carbon content in fly ash. In diesel exhaust measurement a horizontal PAS cell was used, but for fly ash measurements, settling of the fly ash would create a problem. A horizontal configuration with high gas velocities could keep the particles entrained in the gas stream, but Osada et al. [37] have shown that the PAS signal is independent of gas flow rates only for low flow velocities. They suggest that flow velocities should be less than 0.40 m/s through the cell. Therefore, a vertical orientation for the PAS cell was used. Figure 3.4 is a detailed drawing of the optical PAS cell.

The optical PAS cell has windows on the top and bottom of the cell to allow the helium-neon laser beam to enter and exit the PAS cell. The top window, a high quality circular slide cover, is mounted flat and is held in place with a Teflon fitting. The bottom window is similar, but is mounted at the Brewster angle to minimize beam scattering at the bottom window. This reduces noise that would be produced at the PAS modulation frequency. A small amount of purge air is introduced at the bottom window to prevent the fly ash from settling on the bottom window. The laser is aligned through the cell with the use of

a photovoltaic cell located under the bottom window. If the laser is misaligned the voltage produced by the photovoltaic cell is much less than when the laser is aligned correctly.

The entrained fly ash sample enters at the top of the PAS cell. The vertical inlet insures minimal flow disruptions as the fly ash sample enters the PAS cell. Any flow disruptions will generate acoustical noise in the PAS cell. The helium-neon laser radiation interacts with the fly ash in the main section of the PAS cell and the entrained fly ash sample is removed out the bottom of the cell. A complete schematic of the optical PAS system is shown in Figure 3.5.

The PAS cell is housed in an acoustical box inside a second acoustical box to isolate the cell from background room noise (not shown on the optical PAS schematic). The outer box is constructed of 3/4 inch plywood lined with aluminum foil and 1.5 inches of foam. The inner box is constructed of 1/2 inch plywood lined with 2 inches of fiberglass. All openings in the boxes for sample lines are packed with foam.

The optical PAS cell was calibrated with 500 ppm NO_2 in N_2 ($b_a = 0.030 \text{ m}^{-1}$ [46]) at a chopping frequency of 825 Hz. Average modulated power into the optical cell was approximately 13 mW based on power meter measurements at the laser aperture and estimates of reflection and absorption losses at mirrors and windows. The calibration gas was expanded to atmospheric pressure and was pumped through the optical PAS cell. A value of 4 μV was recorded for the PAS signal. Using this information, the optical cell responsivity was calculated to be 10.26 mV-m/W. Eq. (2.14) estimates the responsivity to be 27.42 mV-m/W.



Figure 3.4 Optical PAS cell

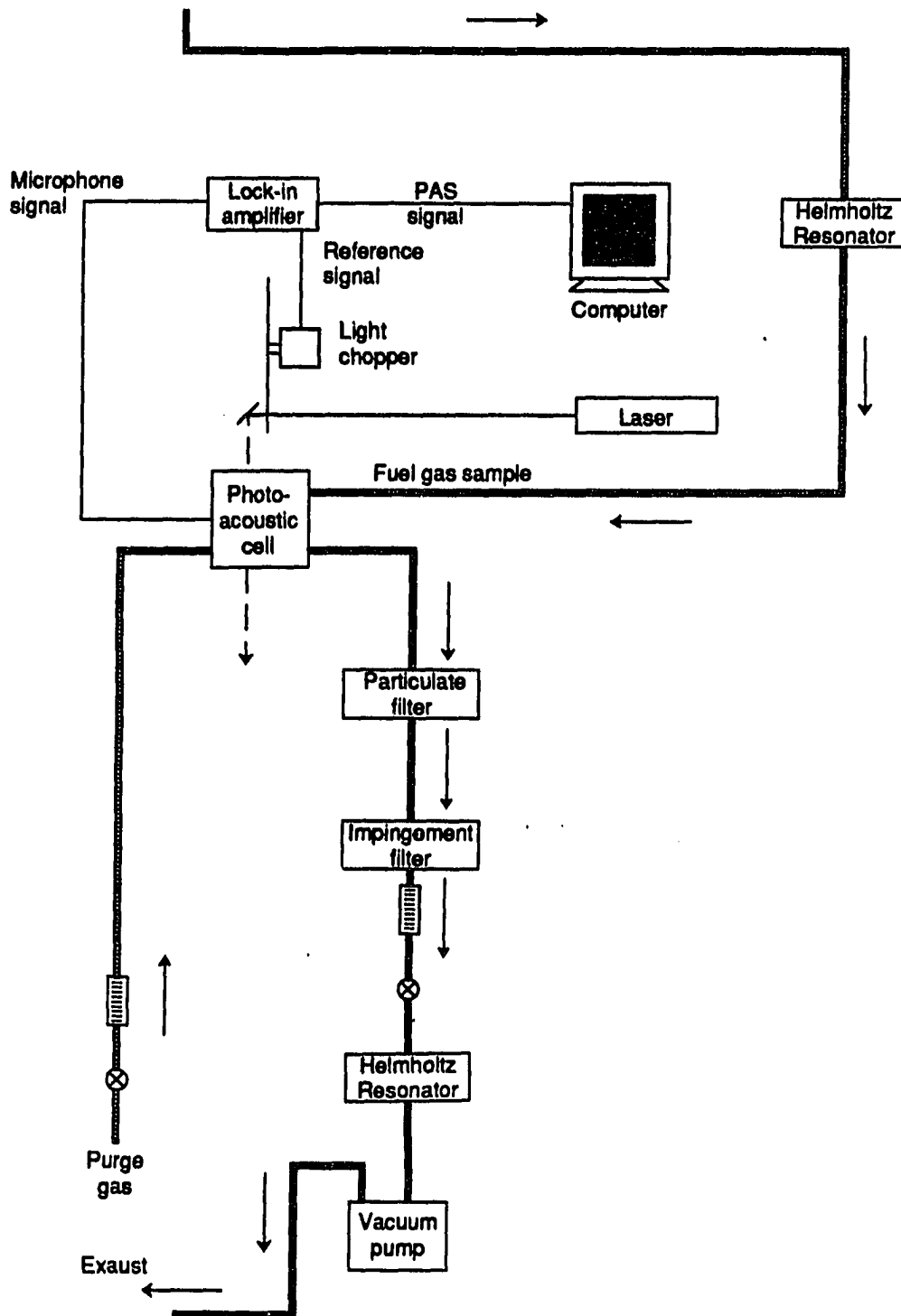


Figure 3.5 Flow schematic for the optical PAS system

3.6 Microwave PAS System

The microwave source is a modified microwave oven [47] ($f = 2450$ MHz) that can be modulated from 980 to 1030 Hz. The modulated radiation is produced by using a vacuum tube switch to turn the magnetron on and off. The maximum forward power that is produced is 600 watts (rms). The power can be adjusted by varying the percent of time that the microwave oven is energized. Although the power is changed, the frequency of modulation remains the same. The power can also be adjusted by varying the amount of power that is produced by the magnetron in the microwave unit, but this is not the desired method of changing the power because it can cause overheating of the microwave unit.

The microwave energy is transported to the PAS cell via a brass waveguide (see Figure 3.6). The microwave energy is transmitted through a 0.003 inch thick piece of Teflon. At the bottom of the waveguide is a 60 degree termination where the microwaves are reflected back up through the PAS cell and the waveguide. The reflected microwaves exit the top of the PAS cell and are directed to a water load by a microwave circulator. The termination of the waveguide establishes the end node for the standing wave inside the waveguide. This is important because a node was also desired at the microphone interface to reduce electromagnetic leakage to the microphone.

The magnetron produces electromagnetic radiation at 2450 MHz. This modulated energy (1000 Hz) is transported to the PAS cell via a waveguide. The waveguide allows transmission of the TE_{10} mode only (see Figure 3.7), thus allowing easy calculations for the cutoff frequency and the waveguide wavelength of the standing wave. This is important because electromagnetic interference with the microphone was a problem. Therefore, having a microwave standing wave node at the microphone interface is important.

The standing wave inside the rectangular waveguide has a different wavelength than

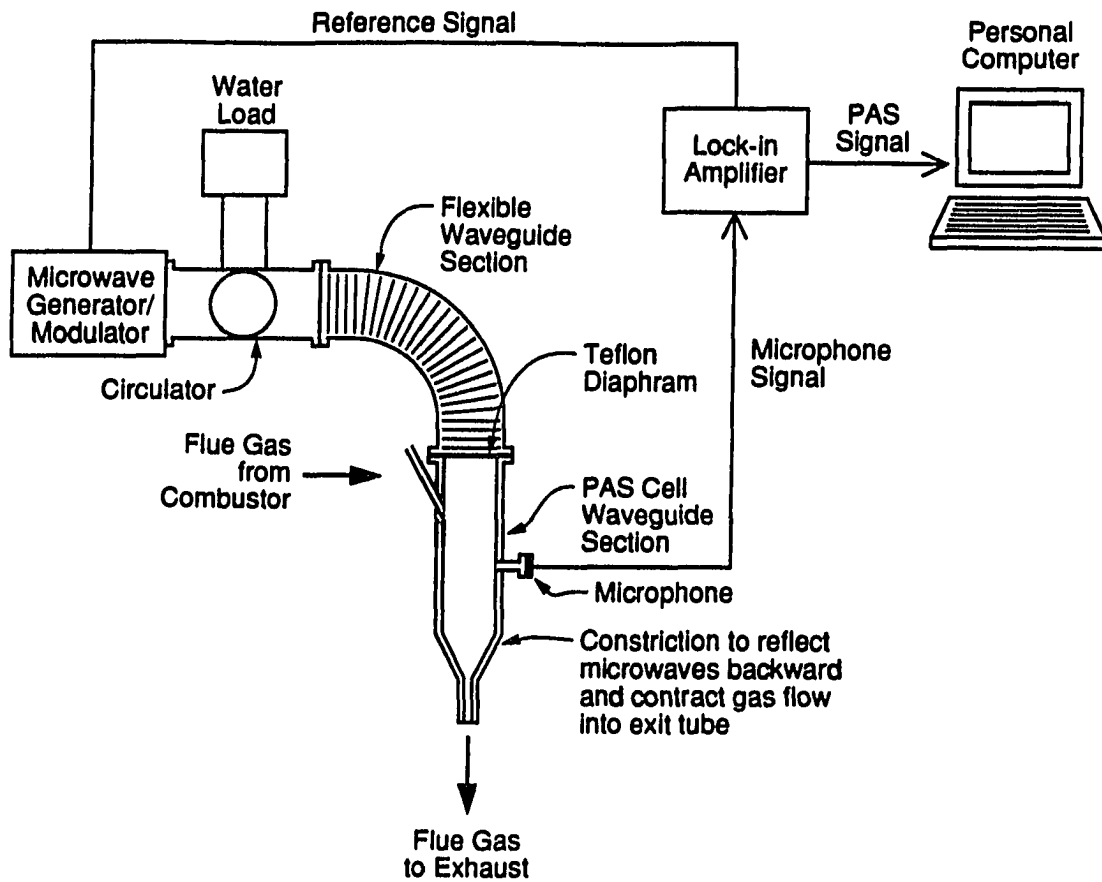


Figure 3.6 Flow schematic for the microwave PAS system

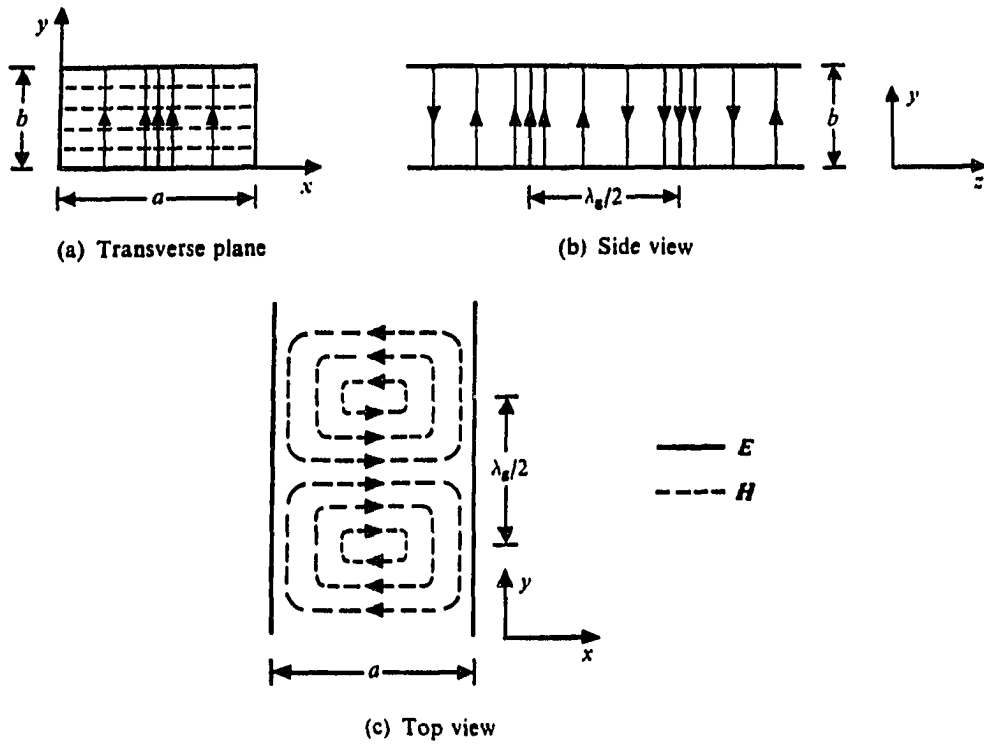


Figure 3.7 Electric and magnetic field lines for TE_{10} mode

the same frequency wave in open space [48]. The waveguide wavelength is given by

$$\lambda_g = \frac{\lambda_0}{\sqrt{1 - \left(\frac{\lambda_0}{\lambda_c}\right)^2}} \quad (3.4)$$

where λ_0 and λ_c are defined as follows:

$$\lambda_0 = \frac{c}{f} \quad (3.5)$$

$$\lambda_c = \frac{1}{\sqrt{\left(\frac{m}{2a}\right)^2 + \left(\frac{n}{2b}\right)^2}} \quad (3.6)$$

The values for the variables are as follows: a (width of waveguide cross section) is 7.144 cm, b (height of waveguide cross section) is 2.064 cm, c (the speed of light) is 3×10^8 m/s, and f is 2450 MHz. The transverse electric modes are represented by the short hand TE_{mn} . The only mode that can propagate in the waveguide is the TE_{10} mode ($m=1$, $n=0$). Accordingly, the following values were obtained: λ_0 (wavelength in free space) of 12.245 cm, λ_c (cut-off wavelength) of 14.2875 cm, and λ_g (waveguide wavelength) of 23.765 cm. This value of λ_g was used to assure that the node of the standing wave was present at the microphone interface to the PAS cell.

Figure 3.8 shows a schematic drawing of the microwave PAS cell. As with the optical PAS cell, the flow is parallel to the vertical axis. The PAS cell is constructed from 3/32 inch brass plate, soldered together. The particulates enter the top of the PAS cell and are allowed to interact with the microwave radiation and the particulates are removed at the bottom of the cell. There is no need for purge air in this design because there is no bottom window.

The neck at the top of the cell, above the Teflon window serves as a smooth transition for the microwave energy into the PAS cell. The smooth transition prevents reflections that would occur from an abrupt transition. The transition also decreases the waveguide cross section, thereby increasing the intensity of the microwave field.

The microphone is recessed away from the PAS cell by a half inch and the interface between the microphone and the cell is 5/64 of an inch. This small hole attenuated the electromagnetic field which can interfere with PAS measurements.

i

4. RESULTS AND DISCUSSION

4.1 Thermal Gravimetric Analysis of Fly Ash

The manual loss-on-ignition (LOI) test is the standard method for determination of the carbon content in coal combustion fly ash. LOI involves weighing a dried fly ash sample, then placing the sample in an oven at 725°C until there is no more weight loss. The change in the sample weight is assumed to be due to the carbon content of the fly ash sample. Thermal gravimetric analysis (TGA) was used to determine the validity of this assumption.

Seven fly ash samples were analyzed using TGA. All seven samples were dried (150°C for 5 hours) before analysis. Table 4.1 describes each of the seven samples. The fly ash samples were produced by four different combustors, each combustor uses a different method of combustion. The two pressurized fluidized bed fly ash samples were produced from different coals burned in the same combustor.

TGA was performed with 50 mg of each sample. The samples were contained in a small stainless steel cup that hung on a sensitive balance. Changes in the weight of the sample were record as a function of both time and temperature. The cup was contained in a positive pressure chamber with a throughput of 100 ml of gas per minute. The samples were placed in a nitrogen atmosphere at room temperature for 30 minutes to stabilize the samples' weight. This process was followed by heating of the chamber in a nitrogen environment at the rate of 20°C per minute until a temperature of 725°C was reached. At this point, the environment was switched to air by an automatic gas handling system. The sample remained in the air

Table 4.1 TGA fly ash samples

	Combustor Type	Coal Type	LOI (dry basis)
Sample #1	Stoker	Iowa	40.30%
Sample #2	Pulverized Coal	Iowa	1.69%
Sample #3	Atmospheric Fluidized Bed	Iowa	3.13%
Sample #4	Atmospheric Fluidized Bed	Iowa	2.01%
Sample #5	Atmospheric Fluidized Bed	Iowa	3.96%
Sample #6	Pressurized Fluidized Bed	Rawhide (Wyoming)	1.59%
Sample #7	Pressurized Fluidized Bed	Illinois #6	3.18%

environment for 30 minutes. Figure 4.1 shows the time verses temperature curve for the stoker fly ash sample; other samples were subject to a similar temperature profile.

All the samples showed a slightly greater weight loss than obtained from LOI measurements. This result is probably due to absorption of water from the air as the samples were being prepared for TGA.

The nitrogen environment prevents the particulate carbon from oxidizing and leaving the sample as CO_2 . Therefore, changes at temperatures less than 725°C are not due to oxidation of the particulate carbon in the sample. Changes in the sample weight at 725°C when the air is added to the sample chamber are due to the particulate carbon oxidizing and leaving the sample as CO_2 .

The stoker fly ash sample is shown in Figure 4.2. The majority of the weight loss occurs at 725°C after air has been introduced. Therefore, the majority of the weight loss is due to particulate carbon in the sample and the LOI assumption that the majority of the weight

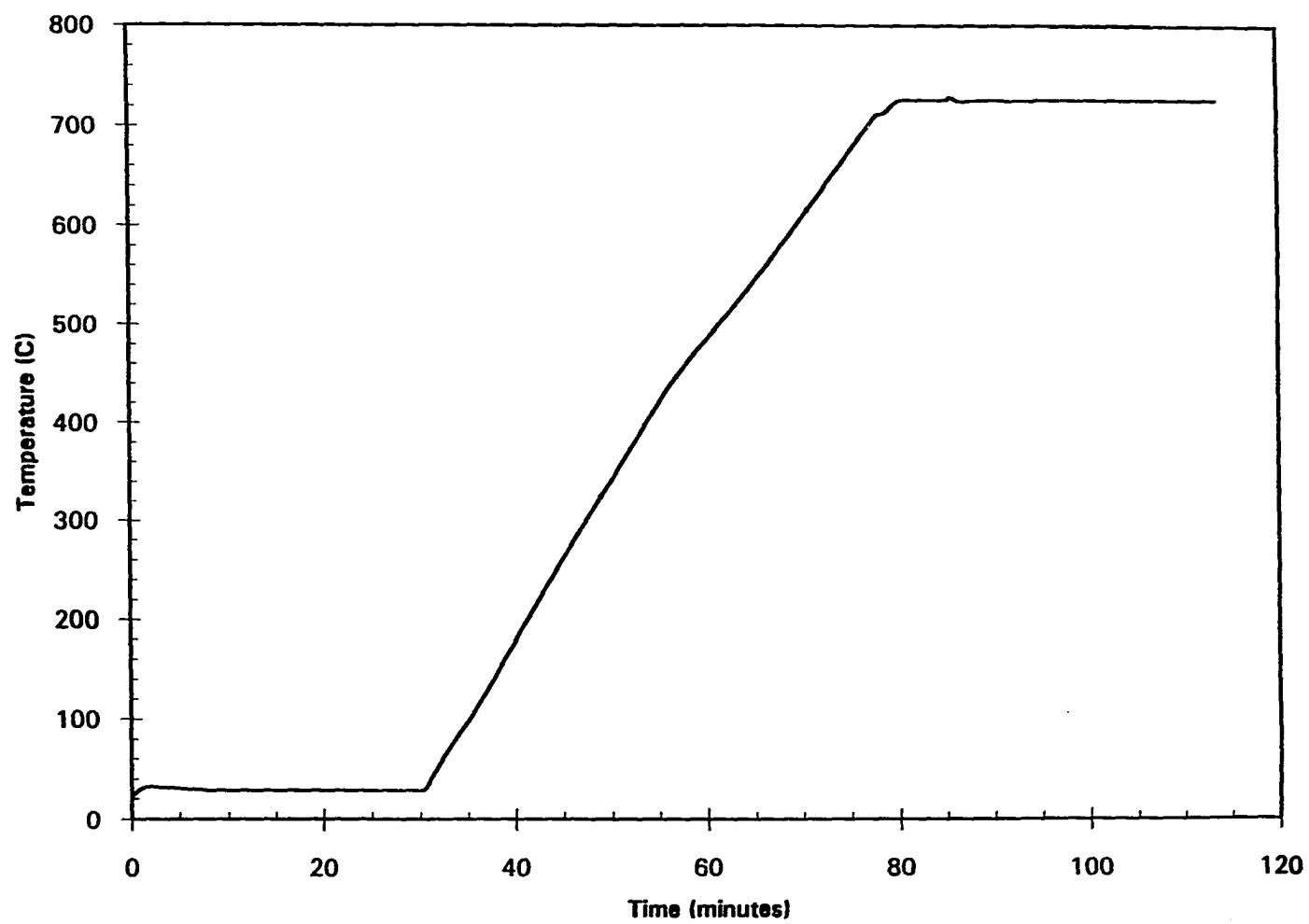


Figure 4.1 Temperature verse time for the TGA stoker fly ash sample

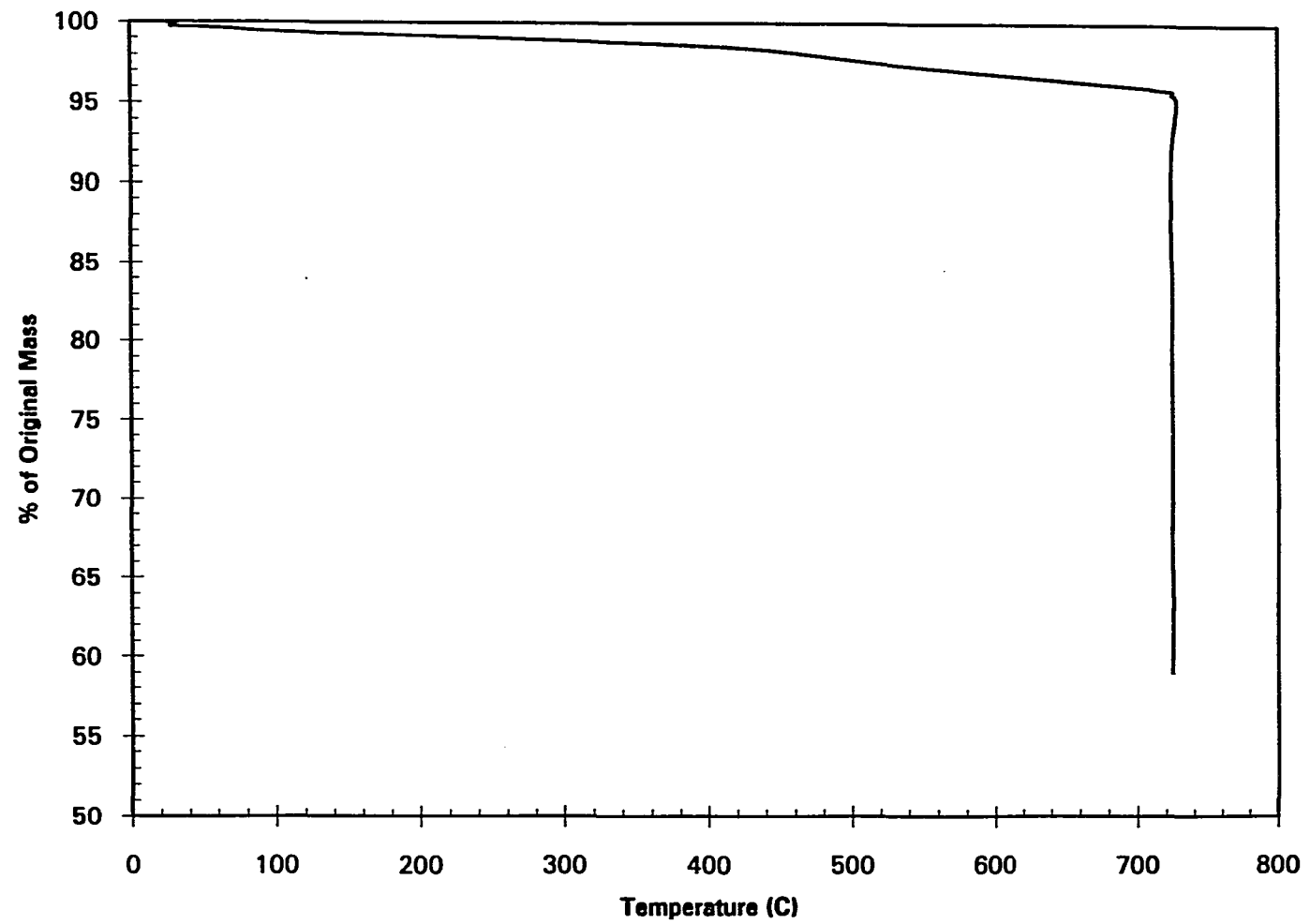


Figure 4.2 TGA of the stoker fly ash sample

loss is due to carbon loss appears to be valid for this stoker fly ash sample.

The pulverized coal fly ash sample is shown in Figure 4.3. The pulverized coal sample shows two very distinct weight loss regions. The first weight loss occurs at about 400°C. This is assumed to be due to the devolatilization of tars (high molecular weight hydrocarbons) [49] from the sample. The second weight loss occurs at 725°C after the air has been added. Again, this is assumed to be due to the particulate carbon oxidizing and leaving the sample as CO₂. Although the weight loss is not due solely to carbon in the sample, the approximation of all carbon is still fairly accurate. Therefore the use of LOI for the calculation of combustion efficiency is still valid for pulverized coal fly ash.

Figure 4.4 shows the TGA of the atmospheric fluidized bed fly ash. In these three samples there are three distinct weight changes. As with the pulverized fly ash sample, there is a weight loss at around 400°C. This is again assumed to be due to tars leaving the sample. The second weight loss occurs in the range from 550°C to 650°C. This weight loss is assumed to be due to a calcination [50] reaction such as $\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$. This does not represent a combustion loss. Therefore, using LOI to calculate combustion efficiency will underestimate the actual combustion efficiency. The source of CaCO₃ in the fluidized bed fly ash samples is from limestone added to the fluidized bed combustor to capture SO₂ produced from the burning of high sulfur Iowa coal. The third weight loss at 725°C is due to the oxidation of the particulate carbon.

The pressurized fluidized bed TGA is shown in Figure 4.5. Sample #6 shows two distinct changes in the sample weight and sample #7 shows three changes in the sample weight. Both samples show a small weight loss at 725°C due to a small amount of particulate carbon in the sample. Apparently, the efficiency of pressurized fluidized bed combustors is so high that very little particulate carbon is left in the sample. Sample #7 also shows a gradual weight loss from room temperature to approximately 550°C. The reason for this change is

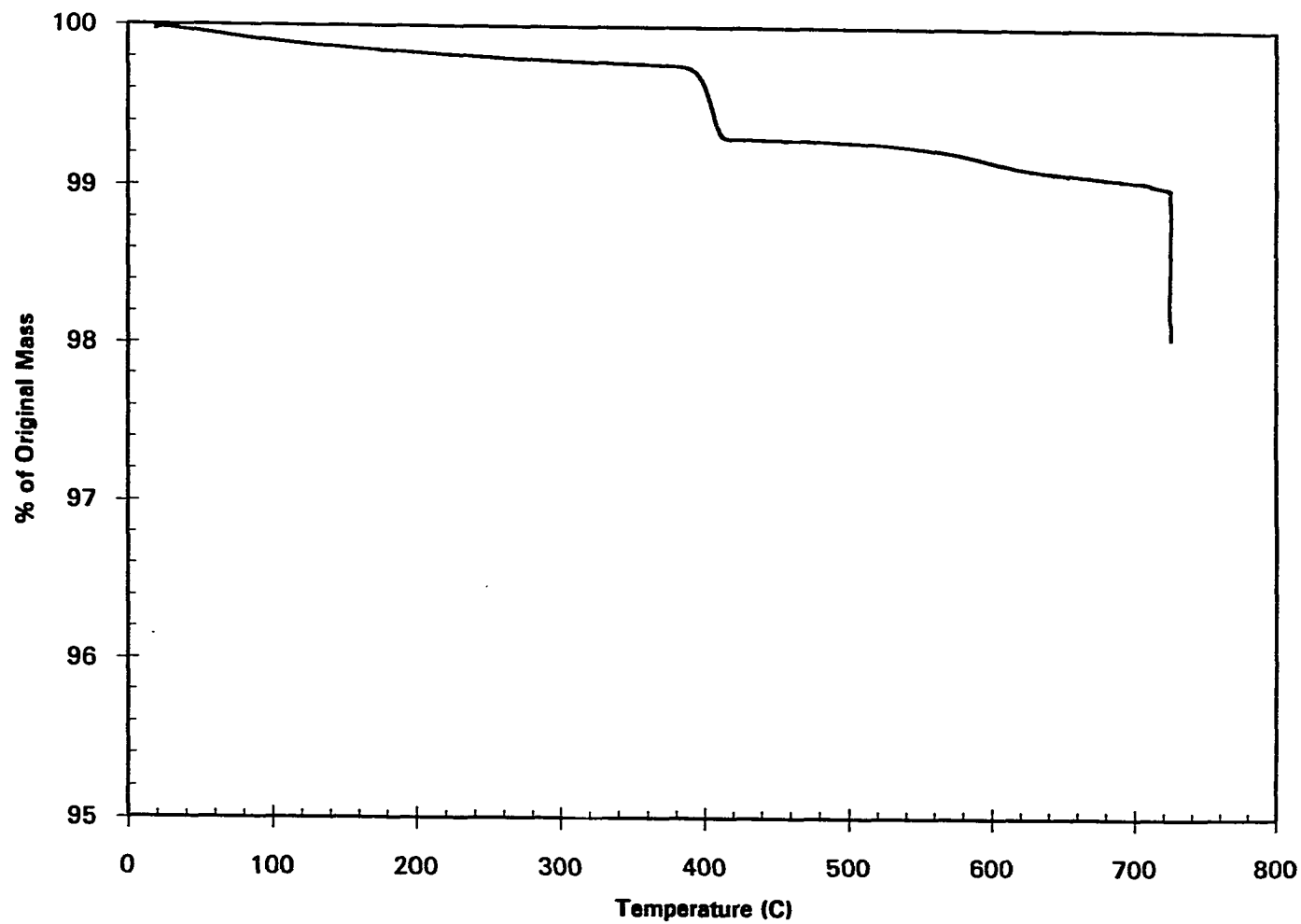


Figure 4.3 TGA of the pulverized coal fly ash sample

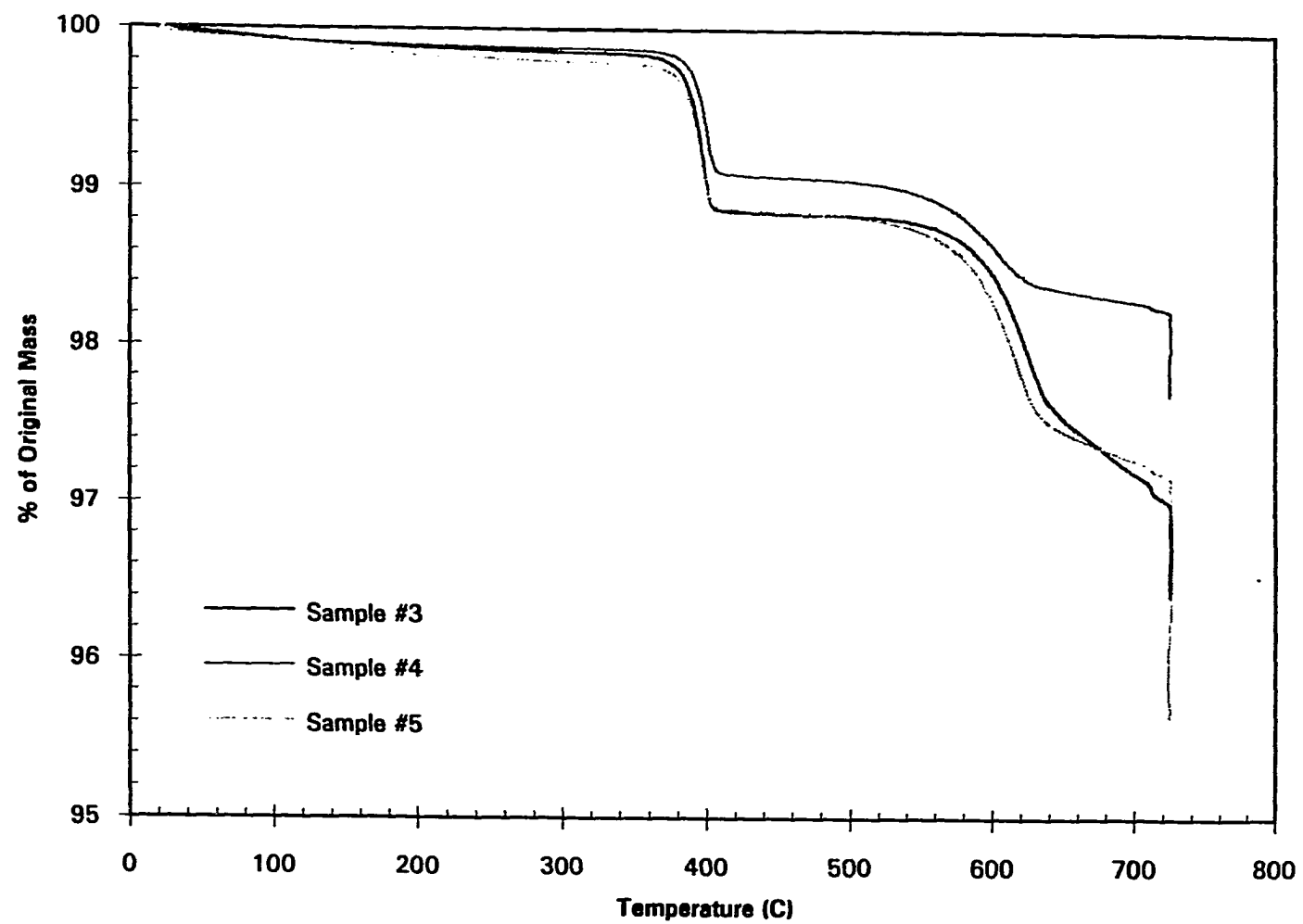


Figure 4.4 TGA of the atmospheric fluidized bed fly ash

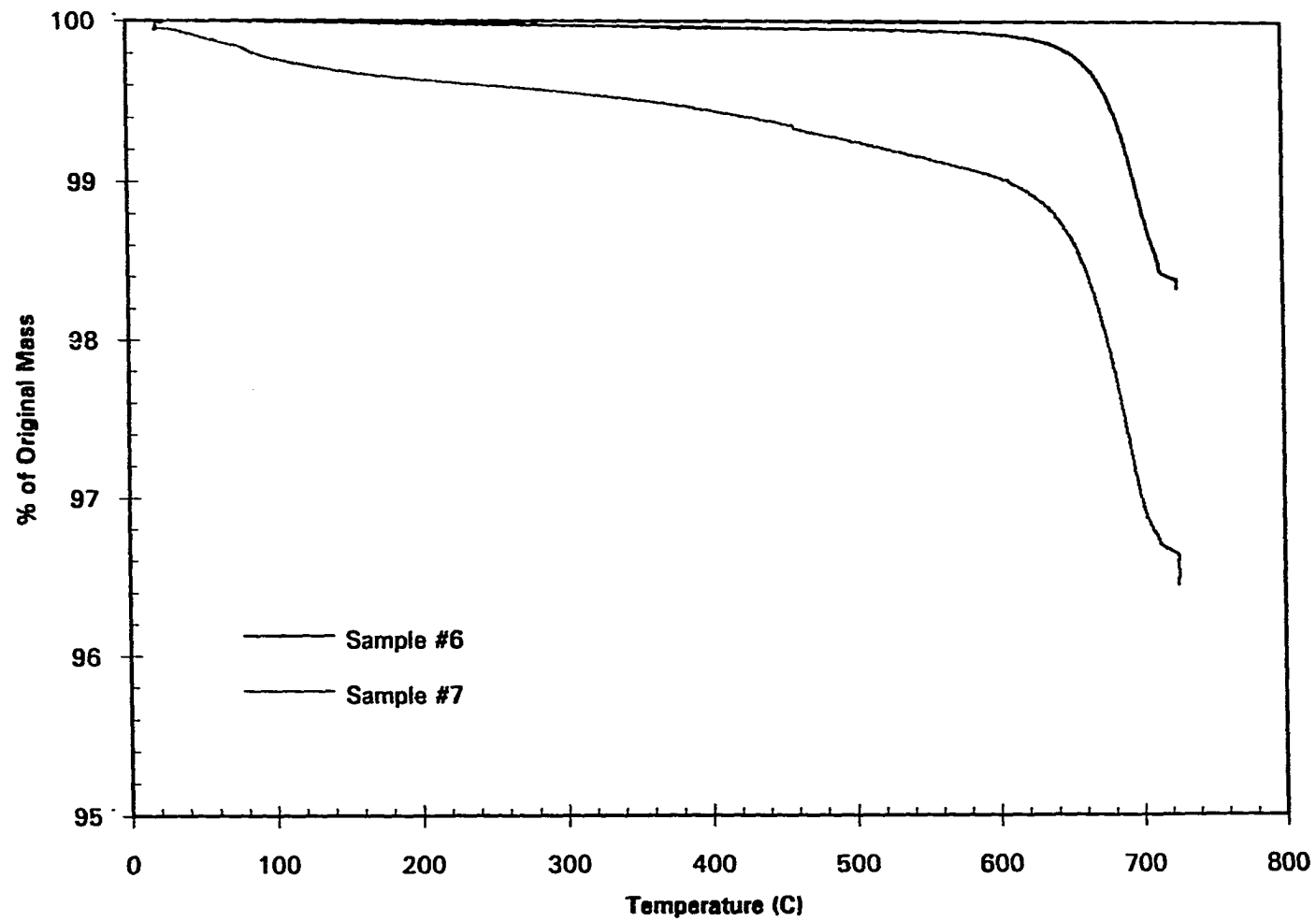


Figure 4.5 TGA of the pressurized fluidized bed fly ash

unclear, but may be due to devolatilization of light hydrocarbons from the sample. Both samples show a loss in weight at 620°C to 700°C due to calcination of the sample. Sample #6 has a smaller change in weight than sample #7 because a western coal has less sulfur than an Illinois coal, hence a lower feed rate of limestone per pound of feed coal would be needed to capture the SO₂.

Table 4.2 summarizes the fluidized bed fly ash samples. The error in assuming that the weight loss is equivalent to the loss in combustibles is also calculated on a dry basis. The error in assuming that the weight loss is equivalent to the loss in combustibles indicates that a new method is needed to determine the amount of combustibles in the fly ash sample.

All the fly ash samples used in tests with the carbon monitors were from stoker boilers with high values of LOI (Figure 4.2). Therefore, the assumption that LOI is equivalent to particulate carbon in the sample is fairly accurate.

Table 4.2 Thermal gravimetric analysis of the fly ash samples

Sample #	Combustor Type	Coal Type	H ₂ O	Tars	Carbonate	Particulate Carbon
			(%)	(%)	(%)	(%)
3	Atmospheric Fluidized Bed	Iowa	0.10	1.07	1.85	0.57
4	Atmospheric Fluidized Bed	Iowa	0.09	0.86	0.82	0.53
5	Atmospheric Fluidized Bed	Iowa	0.14	1.03	1.67	1.52
6	Pressurized Fluidized Bed	Rawhide	0.00	0.06	1.61	0.06
7	Pressurized Fluidized Bed	Illinois #6	0.23	0.85	2.26	0.21

Table 4.3 Examination of TGA measurements

Sample #	Combustor Type	Coal Type	Total Weight Loss (dry)	Combustibles (Tar + Carbon)	Error in LOI Assumption
			(%)	(%)	(%)
3	Atmospheric Fluidized Bed	Iowa	3.49	1.64	112.8
4	Atmospheric Fluidized Bed	Iowa	2.21	1.39	58.9
5	Atmospheric Fluidized Bed	Iowa	4.23	2.55	65.8
6	Pressurized Fluidized Bed	Rawhide	1.73	0.12	1341.7
7	Pressurized Fluidized Bed	Illinois #6	3.33	1.06	214.2

4.2 Monodispersed Fly Ash Tests with Optical PAS

Previous PAS experiments with synthetic fly ash demonstrated the feasibility of detecting PAS signals from unburned char using a helium-neon laser as the excitation source [10]. However, the size distribution of particles was fixed in these tests. More recent on-line tests from a laboratory scale fluidized bed combustor yielded a calibration curve that was independent of combustion conditions except for one outlier [6]. The reason for the outlier was postulated to be due to a larger carbon particle size distribution as compared to the other tests. Experiments are required that control the particle size distribution to determine if the PAS signal is independent of the carbon particle size distribution. The present experiments control the particle size distributions by screening the fly ash into distinct size distributions.

Photoacoustic signals were measured from five different size ranges. Using standard sieves, fly ash from an Iowa State University stoker boiler was sized into samples of

75 x 63 μm , 63 x 53 μm , 53 x 45 μm , 45 x 38 μm , and minus 38 μm .

An empty fluidized bed was used to entrain the fly ash for the PAS measurements. The fly ash was augered into the side of the bed and mixed with 40 SCFM of air. A quarter inch sampling probe at the top of the freeboard removes an airborne sample from the exhaust. The airborne fly ash leaving the bed is collected in a cyclone for conventional analysis. The cyclone results are then compared with the PAS results.

Several tests were performed on each size distribution. Each test was five minutes in duration and the end of one test was the start of the next test. At the five minute mark the cyclone valve was closed and a mark was placed in the PAS data file. The sample collected was weighed and the collection jar was emptied and replaced at the bottom of the cyclone. The cyclone valve was then reopened. At the end of 5 minutes the procedure was repeated. The carbon loadings were determined by performing an LOI test from a single sample taken from each size range and multiplying this value by the total fly ash loading as determined from the cyclone catch.

The results of the PAS measurements are presented in Table 4.4 and in Figure 4.6. The PAS signal values were calculated by integrating the PAS data (trapezoidal rule) and then dividing by the total time interval (~ 5 minutes).

Linear regression was performed on each size range to determine the mass specific absorption coefficient for each size range. The Y-intercept was set to the average background signal for each set of tests. The slope calculated by linear regression is proportional to the mass specific absorption coefficient (Eq. 2.13). Table 4.5 presents the linear regression slopes as well as the calculated mass specific absorption coefficients ($W = 13 \text{ mW}$, $R = 10.26 \text{ mV-m/W}$). The values compare well with the mass specific absorption coefficients predicted by Eq. (2.2) and Eq. (2.3) ($\rho = 1800 \text{ kg/m}^3$, $Q = 0.9$).

Table 4.4 Optical PAS signal for five specific size distributions

Size Range	Carbon Loading (g/m ³)	PAS Signal (μV)	Size Range	Carbon Loading (g/m ³)	PAS Signal (μV)
75 x 63 μm	7.28	21.23	45 x 38 μm	6.76	19.69
	8.08	19.49		7.00	21.81
	8.38	22.29		7.25	22.61
63 x 53 μm				7.85	20.23
	7.64	23.19		7.77	22.19
	7.74	24.96		6.39	17.61
	7.66	23.69	Minus 38 μm	5.36	34.63
53 x 45 μm	7.61	23.78		5.89	38.41
				6.18	41.63
	6.53	19.86		6.08	39.91
	7.11	19.99		6.33	44.75
	7.43	20.40		6.41	44.65
	7.64	20.88		4.97	33.55
	7.88	19.98		4.37	33.43
	8.06	22.91		4.31	30.74
	8.32	25.40		4.26	30.14
	8.34	25.25		4.24	30.03
	7.58	21.63		1.46	13.19
	4.02	10.75			

Table 4.5 Linear regression analysis of optical PAS results

Size Range (μm)	Average Diameter (μm)	Linear Regression Slope (μV-m ³ /g)	Standard Error of Slope Estimate	Calculated Mass Specific Absorption Coefficient (m ² /g)	Estimated Mass Specific Absorption Coefficient (m ² /g)
75 x 63	69.0	2.01	0.1240	0.0151	0.0109
63 x 53	58.0	2.57	0.0416	0.0193	0.0129
53 x 45	49.0	2.09	0.0750	0.0157	0.0153
45 x 38	41.5	2.23	0.0841	0.0167	0.0181
Minus 38	19.0	5.28	0.0810	0.0396	0.0395

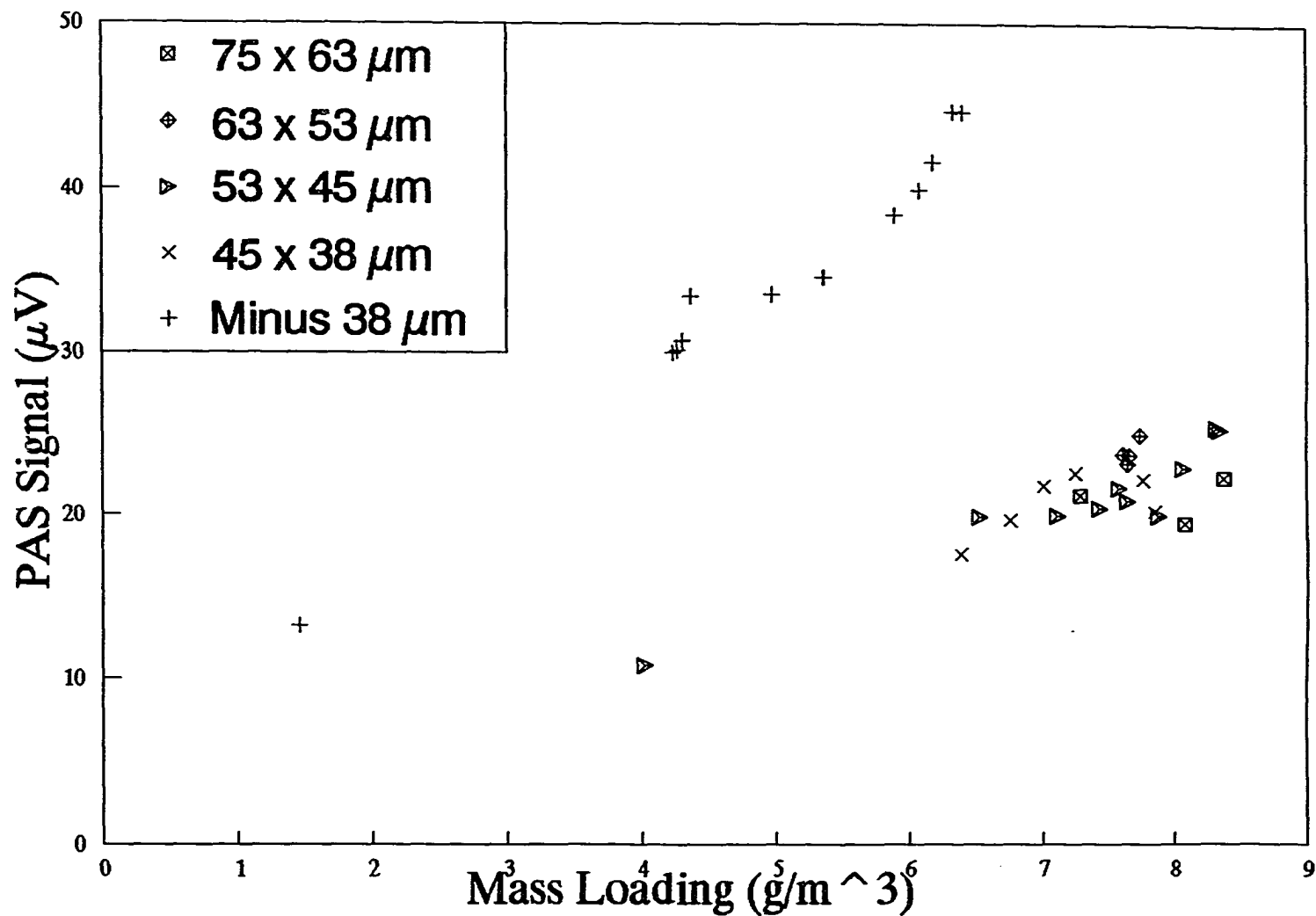


Figure 4.6 Optical PAS signal for five specified size distributions

A $1/D$ relationship was found to fit the five values of the calculated mass specific absorption coefficient (Figure 4.7). Error bars for the data points were calculated using a 95% confidence interval on both the mass specific absorption coefficient and \bar{D} . \bar{D} for the minus 38 μm size range was calculated by Microtrac analysis. The error in \bar{D} for this range was very small. The minus 38 μm size range was run twice to assure that the large change in slope was not due to experimental error; only the first set of tests run are presented.

Figure 4.7 confirms the postulate that there is a carbon particle size dependence at the helium-neon wavelength. To determine the effect of this carbon size distribution dependence on quantitative PAS carbon determinations, the relative contributions to the PAS signal of each size range of the stoker fly ash (Table 4.6) were calculated using the $1/D$ relationship shown in Figure 4.7. Figure 4.8 shows the size distribution of the stoker fly ash as well as the relative contributions to the PAS signal. These results show that although only 15.1% of the carbon is contained in the -38 μm size range, it contributes approximately 40% of the total PAS signal.

Table 4.6 Size distribution of the Iowa State University stoker fly ash

Size Range of Fly Ash Sample	Percent Carbon	Percent Water	Percent Ash	Mass Percent Total	Mass Percent Carbon
Plus 30 mesh or plus 600 μm	40.0	0.8	59.2	3.5	3.9
30 x 40 mesh or 600 x 425 μm	40.8	1.6	57.6	7.7	8.7
40 x 50 mesh or 425 x 300 μm	40.0	1.3	58.7	7.7	8.5
50 x 100 mesh or 300 x 150 μm	39.3	1.2	59.5	20.5	22.4
100 x 200 mesh or 150 x 75 μm	38.6	1.5	59.9	16.6	17.8
200 x 400 mesh or 75 x 38 μm	35.9	1.5	62.6	23.7	23.6
Minus 400 mesh or minus 38 μm	26.8	1.2	72.0	20.3	15.1

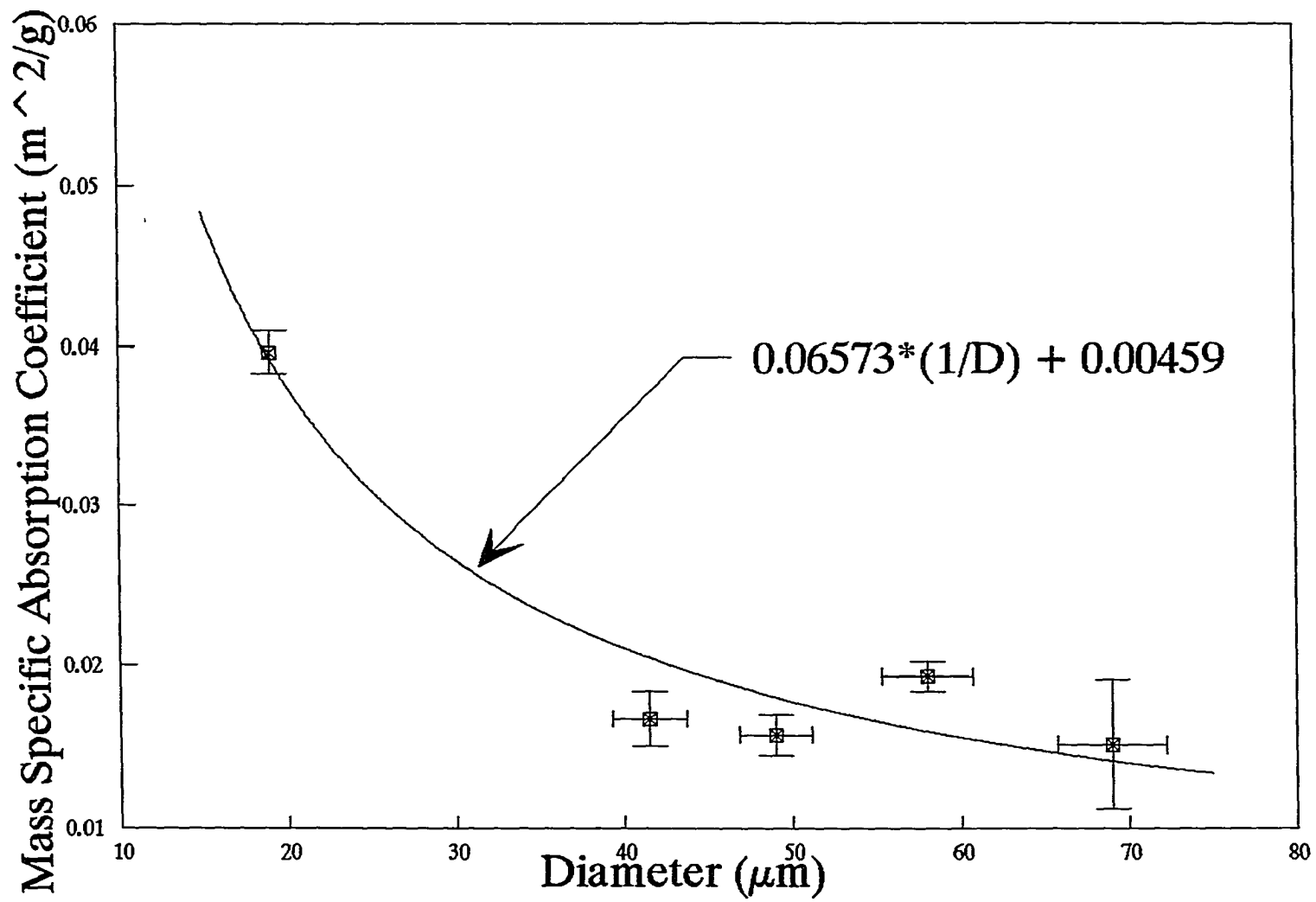


Figure 4.7 PAS linear regression slopes verse average particle size range diameter

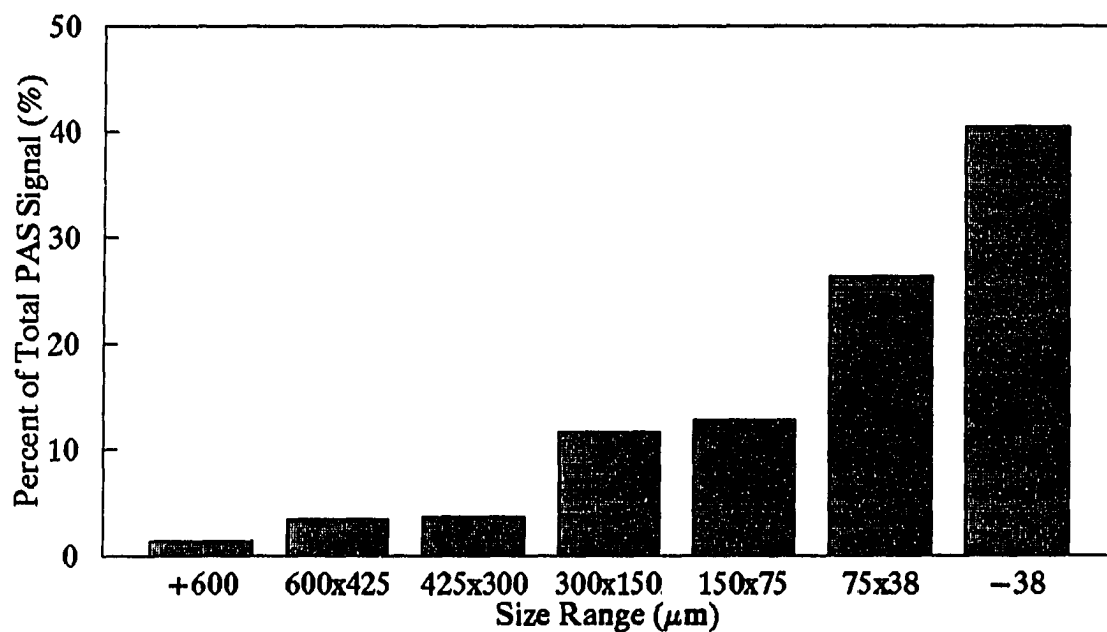
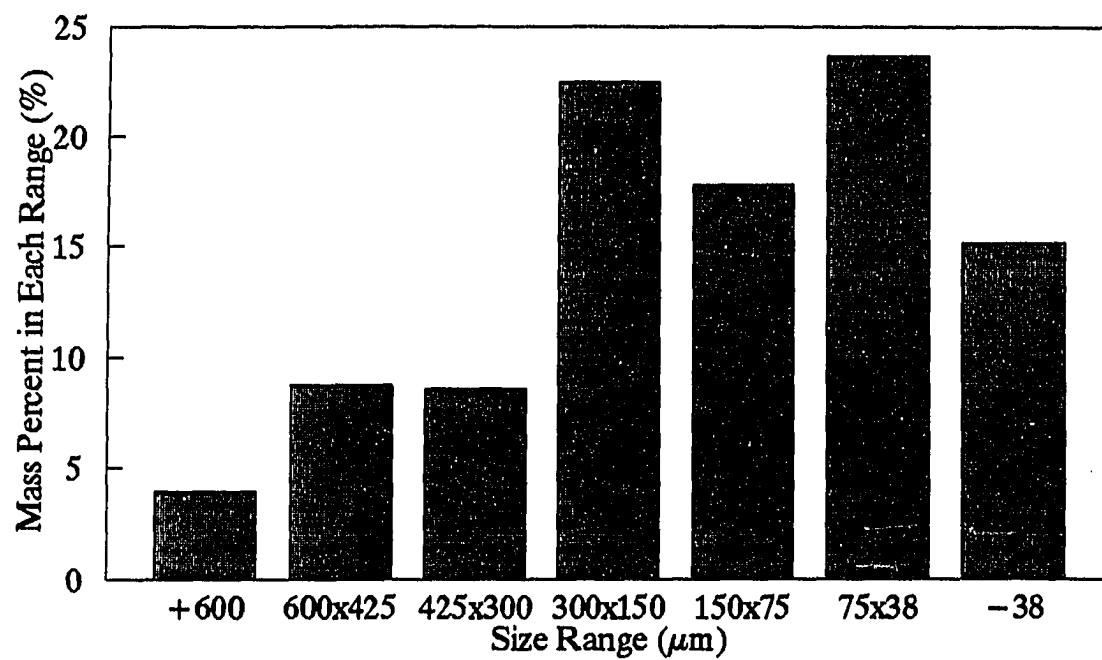


Figure 4.8 Size distribution of Iowa State University stoker fly ash (top) and PAS contribution from each range (bottom)

A longer wave radiation would not have this short fall because at long wavelengths ($\lambda \gg D$) radiation absorption is no longer a surface area phenomenon, but is a volumetric phenomenon [24]. Therefore, the mass specific absorption coefficient would be independent of the carbon particle size distribution. This is the motivation for the study of microwave ($\lambda = 12.24$ cm) PAS.

4.3 Microwave PAS System

Previous photoacoustic studies of solids using microwaves as the excitation source are very limited [51, 52]. No on-line sampling PAS measurements have been performed with microwaves.

The reason for using microwaves as a radiation source is that absorption of the radiation by the carbon particles would be characterized by the small particle limit ($D \ll \lambda$). The wavelength of the microwave radiation is 12.24 cm. Therefore, the small particle limit is appropriate. In the limit of small particles, the mass specific absorption coefficient is independent of the particle size distribution [24]. This is not the case for the optical PAS system. An ideal carbon monitor would be able to give qualitative results without knowledge of the size distribution.

Although use of microwaves provides a mass specific absorption coefficient that is independent of particle size distribution, the value of the mass specific absorption coefficient is much smaller than at the helium-neon wavelength. The exact value for the mass specific absorption coefficient is elusive because there are no published values for either the mass specific absorption coefficient or the refractive index. Using approximations for the refractive index and the program in Appendix A that calculates mass specific absorption cross sections, a

reduction by a factor of 1000 in the mass specific absorption coefficient as compared to the optical mass specific absorption coefficient was expected.

The first approach involved using a slotted waveguide with a water load at the end of the waveguide. A Plexiglas PAS cell was inserted into the waveguide so that the microwave energy would pass through the Plexiglas cell and produce a PAS signal. Although this approach did not provide useful PAS measurements, many important problems were identified and solved. First, electromagnetic leakage was picked up by some of the electronics. The unshielded electronic equipment acted as antennas for the electromagnetic radiation. This caused problems because the electromagnetic leakage was also at the PAS modulation frequency and lock-in readings of over 100 μV were obtained with the microphone disconnected. Second, the microwave intensity transmitting through the Plexiglas was not enough to produce a measurable PAS signal.

The second approach involved sectioning off a portion of the waveguide for use as a PAS cell, thereby utilizing all the microwave power in the waveguide. The waveguide was tapered down to increase the microwave intensity. A Teflon window separated the waveguide from the PAS cell. The fly ash was introduced just below the Teflon window and exited out the bottom of the waveguide. The waveguide was terminated at the bottom and the microwave energy was reflected back through the waveguide where a circulator channeled the microwaves to a water load. The microwave standing wave had a node at the termination point and also at the microphone interface at the center of the PAS cell. This was done to reduce the electromagnetic leakage reaching the microphone. As an added precaution in this regard, the microphone interface hole was only 5/64 of an inch and the microphone was recessed one half inch. This approach produced a PAS signal, but the back ground signal continued to increase as the measurements proceeded due to a PAS signal being generated by fly ash settling at the bottom of the PAS cell. There were also problems of repeatability

because alignment of the PAS cell components is critical. If an edge presented itself to the microwave radiation, there was localized heating that felt warm to the touch. Also, when a solder joint partially failed, sparks arced from one surface to the other.

The final approach involved a new end plate for the PAS cell. The angle of the end plate was changed from 5 degrees to 60 degrees to prevent settling of the fly ash at the bottom of the cell. Also, all PAS cell components were aligned and pinned into place to prevent misalignment.

The microwave PAS cell modifications reduced the background signal, although a significant background signal persisted. Each microwave PAS measurement involved establishing a background, after which fly ash was added to the microwave PAS system. After establishing a steady state with the fly ash, the signal was measured for 5 minutes. The fly ash auger was then turned off and another background signal was measured (see Figure 4.9). The background signal needed to be subtracted from the fly ash PAS signal. An average was taken from the pre-test and post-test background signals. The average background was then subtracted from the total PAS signal to determine the contribution due to the carbon in the fly ash. The background signal varied from day to day depending on the humidity in the air. Therefore, dry samples of fly ash were used in the testing of the microwave PAS system because moisture in the fly ash sample would produce a PAS signal and the effect of the carbon in the fly ash could not be determined .

Three different size ranges of an Iowa State stoker fly ash were tested as well as a bulk sample of the fly ash. The size distribution for the bulk sample is shown in Figure 4.8, although particles larger than 425 μm did not report to the cyclone catch. Figure 4.10 and Table 4.7 presents the results.

Linear regression was performed on each set of data. These results are shown in Table 4.8. The slope of these lines is proportional to the mass specific absorption coefficient

(Eq. 2.14). The slopes calculated for each set show that there is no size dependence for the microwave PAS carbon in fly ash measurements. The y-intercept values diverge from the origin as the particle size ranges get smaller. This may be due to the subtraction of the background signal from the total fly ash signal. The 40 SCFM of air passing by the fly ash auger pulls a small amount of fly ash through the screw auger. The smaller particles are pulled through the auger at a faster rate causing the background to be based on a non-empty cell condition. The bulk sample is very large in comparison to the other samples. Therefore, the fly ash is not pulled through the screw auger and a y-intercept is close to the expected value of zero.

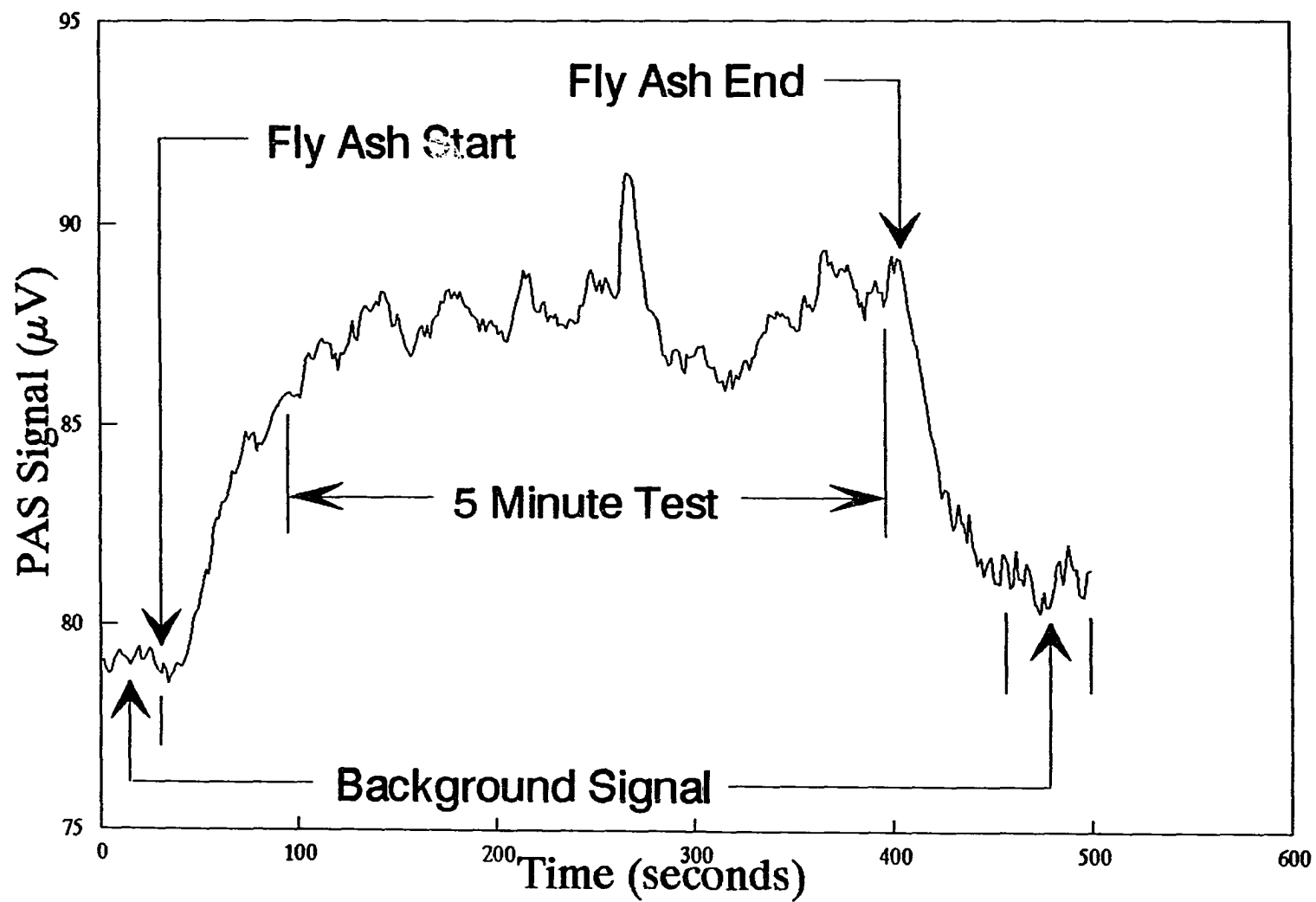


Figure 4.9 Illustrative results of microwave PAS Test # 4

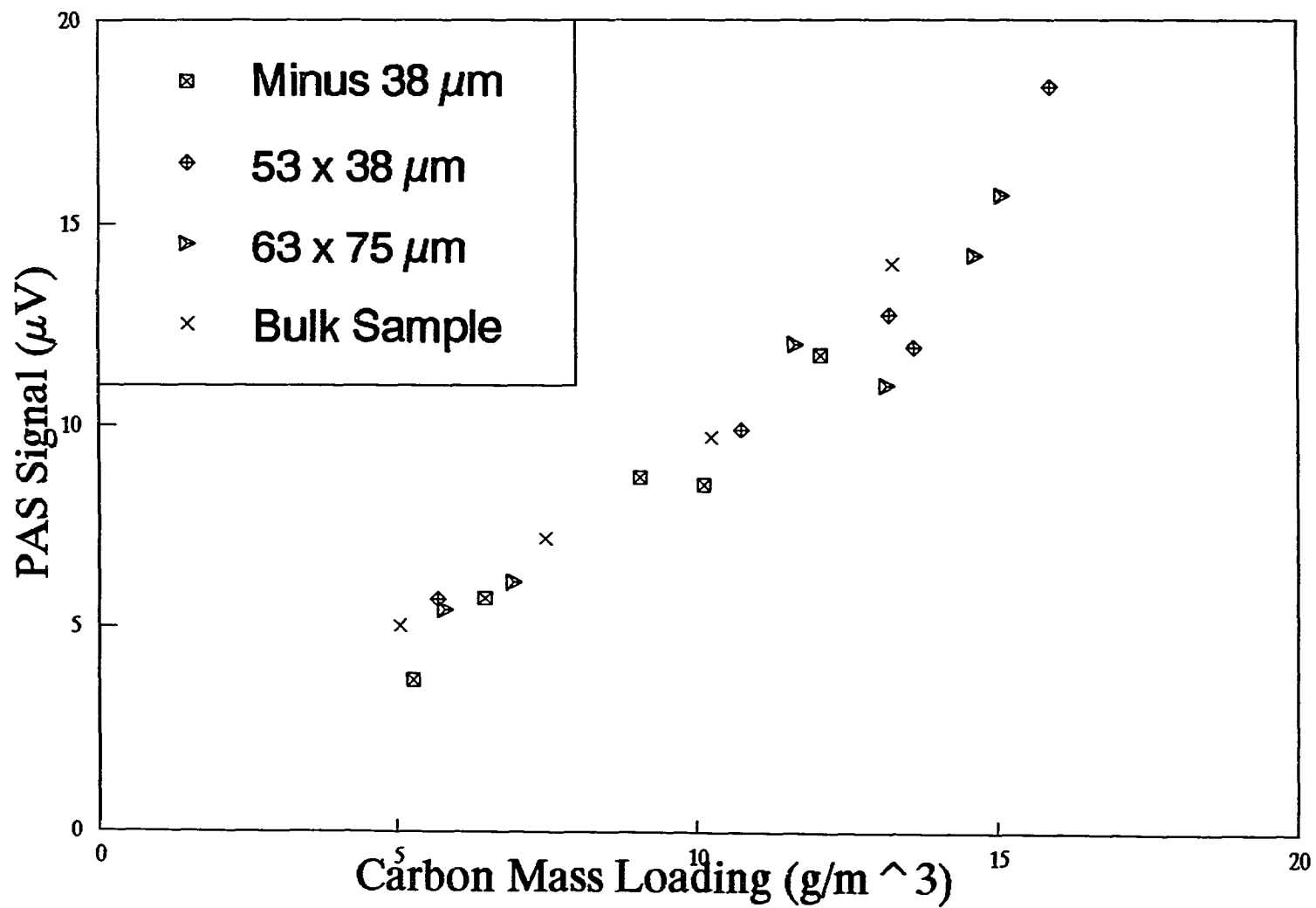


Figure 4.10 Microwave PAS carbon in fly ash measurements

Table 4.7 Results of the carbon in fly ash measurements using microwave PAS

Test Number	Sample	Carbon Loading (g/m ³)	PAS Signal (μV)
1	Minus 38 μm	12.07	11.36
2	Minus 38 μm	5.27	3.54
3	Minus 38 μm	10.13	8.76
4	Minus 38 μm	9.05	8.78
5	Minus 38 μm	6.47	5.56
6	53 x 38 μm	15.92	18.01
7	53 x 38 μm	13.60	12.42
8	53 x 38 μm	13.21	12.63
9	53 x 38 μm	5.70	5.36
10	53 x 38 μm	10.78	10.15
11	75 x 63 μm	15.10	15.77
12	75 x 63 μm	14.64	14.61
13	75 x 63 μm	5.82	5.44
14	75 x 63 μm	11.66	11.56
15	75 x 63 μm	13.17	11.38
16	75 x 63 μm	6.96	5.87
17	Bulk	13.25	14.06
18	Bulk	5.05	4.92
19	Bulk	7.49	7.57
20	Bulk	10.25	9.52

Table 4.8 Linear regression results of the microwave PAS measurements

Sample	Slope (μV m ³ /g)	Standard Error of slope estimate	Y-intercept	Standard Error of slope estimate
Minus 38 μm	1.10	0.105	-1.84	0.575
53 x 38 μm	1.14	0.172	-1.78	1.338
75 x 63 μm	1.07	0.103	-1.27	0.911
Bulk	1.08	0.114	-0.69	0.698

5. CONCLUSIONS

PAS shows genuine promise for measuring the carbon content in a fly ash sample. Many of the problems of implementing PAS for carbon in fly ash detection have been overcome. Acoustical backgrounds can be attenuated to a workable level using Helmholtz resonators to isolate the PAS from pressure waves propagating through the sample lines. Solutions to electromagnetic radiation picked up by the electronic equipment were also found. The future of PAS for carbon in fly ash detection is promising, but, there are areas that need scrutiny.

PAS investigation with two radiation sources: helium-neon laser and microwaves, shows that PAS can detect carbon in fly ash and that PAS has potential for on-line carbon in fly ash measurement, although an optimum radiation source has not been found. Both the helium-neon laser source and the microwave source have attributes that are desirable for carbon in fly ash measurements, but they also have shortfalls.

The helium-neon source produces a large mass specific absorption coefficient for the carbon in the fly ash. Although the optical PAS system uses less than one forty thousandths of the power as the microwave system, it still has twice the sensitivity ($D < 75 \mu\text{m}$). The helium-neon laser source is easy to work with and there is no pick-up of the electromagnetic field by the electronic equipment, but there is also a size dependence of the PAS signal to the carbon size distribution. This is a serious drawback because a carbon monitor should be able to perform without knowledge of the carbon particle size distribution.

The microwave source produces a signal that is independent of the carbon particle size distribution, but the low mass specific absorption coefficient at 12.24 cm would require a microwave source producing 12000 watts to measure the carbon content of pulverized coal combustors. This is impractical. There are also problems with electronic equipment picking up the electromagnetic field generated by the magnetron leaking from the waveguide.

At long wavelengths, the concerns about the form of carbon in fly ash are no longer warranted. The PAS signal produced by either heterogeneous particles of carbon and mineral matter, or homogeneous carbon particles would be proportional to the amount of the carbon present in the fly ash sample, independent of its form in the fly ash.

The key to future developments in PAS for carbon in fly ash detection lies in the selection of an optimum radiation source. An optimum radiation source may be found in the spectrum between the helium-neon laser source ($\lambda = 632 \text{ nm}$) and the microwave source ($\lambda = 12.24 \text{ cm}$). It may be possible to find a source that has all the positive characteristics with manageable drawbacks.

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APPENDIX A. ABSORPTION CROSS SECTION CALCULATION

Using the following particle parameters a , m , k , α , and β the absorption cross section can be calculated.

where

a = radius of the sphere

m = the refractive index in the form $n+ik$

$$k = \frac{2\pi}{\lambda} \quad \text{where } \lambda \text{ is the wavelength of the incident radiation.} \quad (\text{A.1})$$

$$\alpha = ka \quad (\text{A.2})$$

$$\beta = m\alpha \quad (\text{A.3})$$

The following procedure for calculating the absorption cross section was developed by Kerker [24].

$$C_s = \left(\frac{\lambda^2}{2\pi} \right) \sum_{n=1}^{\infty} (2n+1) \{ |a_n|^2 + |b_n|^2 \} \quad (\text{A.4})$$

$$C_e = \left(\frac{\lambda^2}{2\pi} \right) \sum_{n=1}^{\infty} (2n+1) \{ \text{Re}(a_n + b_n) \} \quad (\text{A.5})$$

$$C_a = C_e - C_s \quad (\text{A.6})$$

where a_n and b_n are scattering coefficients for a sphere:

$$a_n = \frac{\psi'_n(\beta)\psi_n(\alpha) - m\psi_n(\beta)\psi'_n(\alpha)}{\psi'_n(\beta)\zeta_n(\alpha) - m\psi_n(\beta)\zeta'_n(\alpha)} \quad (\text{A.7})$$

$$b_n = \frac{m\psi'_n(\beta)\psi_n(\alpha) - \psi_n(\beta)\psi'_n(\alpha)}{m\psi'_n(\beta)\zeta_n(\alpha) - \psi_n(\beta)\zeta'_n(\alpha)} \quad (\text{A.8})$$

where:

$$\psi_n(z) = \left(\frac{\pi z}{2}\right)^{1/2} J_{(n+1/2)}(z) \quad (\text{Ricatti-Bessel function}) \quad (\text{A.9})$$

$J_{(n+1/2)}(z)$ = half integral order Bessel function.

$$\zeta_n(z) = \left(\frac{\pi z}{2}\right)^{1/2} H_{(n+1/2)}^{(2)}(z) = \psi_n(z) + i\chi_n(z) \quad (\text{A.10})$$

$H_{(n+1/2)}^{(2)}(z)$ = half integral order Hankel function.

$$\chi_n(z) = -\left(\frac{\pi z}{2}\right)^{1/2} N_{(n+1/2)}(z) \quad (\text{Ricatti-Bessel function}) \quad (\text{A.11})$$

$N_{(n+1/2)}(z)$ = half integral order Neumann function

The scattering coefficients can also be written in terms of the logarithmic derivatives of the Ricatti-Bessel functions.

$$a_n = \frac{\psi_n(\alpha)}{\zeta_n(\alpha)} \left[\frac{\eta_n^{(1)}(\beta) - m\eta_n^{(1)}(\alpha)}{\eta_n^{(1)}(\beta) - m\eta_n^{(3)}(\alpha)} \right] \quad (\text{A.12})$$

$$b_n = \frac{\psi_n(\alpha)}{\zeta_n(\alpha)} \left[\frac{\eta_n^{(1)}(\alpha) - m\eta_n^{(1)}(\beta)}{\eta_n^{(3)}(\alpha) - m\eta_n^{(1)}(\beta)} \right] \quad (\text{A.13})$$

The Ricatti-Bessel functions can be evaluated using recurrence relations:

$$f_{n+1}(z) = [(2n+1)/z]f_n(z) - f_{n-1}(z) \quad (\text{A.14})$$

$$\frac{df_n(z)}{dz} = \frac{n f_{n-1}(z) - (n+1) f_{n+1}(z)}{(2n+1)} \quad (\text{A.15})$$

where

$$\psi_0(z) = \sin(z) \quad \text{and} \quad \psi_1(z) = \sin(z)/z - \cos(z) \quad (\text{A.16})$$

$$\chi_0(z) = \cos(z) \quad \text{and} \quad \chi_1(z) = \cos(z)/z + \sin(z) \quad (\text{A.17})$$

$$\zeta_n(z) = \psi_n(z) + i\chi_n(z) \quad (\text{A.10})$$

The logarithmic derivation functions, $\eta_n^{(1)}(z)$ and $\eta_n^{(3)}(z)$ can be evaluated using these simple recursion formulas.

$$\eta_n^{(3)}(z) = \zeta_{n-1}(z)/\zeta_n(z) - n/z \quad (\text{A.18})$$

$$\eta_n^{(1)}(z) = \frac{z^2 + n z \eta_{n-1}^{(1)}(z) - n^2}{n z - z^2 \eta_{n-1}^{(1)}(z)} \quad (\text{A.19})$$

where

$$\eta_0^{(1)} = \cot(z) = \cot(a - ib) = \frac{\sin(2a) + i \sinh(2b)}{\cosh(2b) + \cos(2a)} \quad (\text{A.20})$$

The algorithm described above assumes knowledge of the refractive index. If the refractive index is not known the computer program assigns a value for the refractive index based on an algorithm developed by Lee and Tien [53] for soot particles. The algorithm is based on a dispersion model of two bound and one free electrons. The parameters for the equations are obtained from experimentation. Two equations are presented from which the values of n and k are obtained.

$$n^2 - k^2 = 1 + \frac{e^2}{m_e \epsilon} \sum_{j=1}^2 \frac{n_j (\omega_{bj}^2 - \omega^2)}{(\omega_{bj}^2 - \omega^2) + \omega^2 g_{bj}^2} - \frac{e^2}{m^o \epsilon} \frac{n_f}{(\omega^2 + g_f^2)} \quad (\text{A.21})$$

$$2nk = \frac{e^2}{m_e \epsilon} \sum_{j=1}^2 \frac{n_j \omega g_{bj}}{(\omega_{bj}^2 - \omega^2) + \omega^2 g_{bj}^2} + \frac{e^2}{m^{\circ} \epsilon} \frac{n_f g_f}{\omega (\omega^2 + g_f^2)} \quad (\text{A.22})$$

where

e = electron charge

m_e = electron mass in vacuum

ϵ = permissivity constant

n_j = bound electron number densities

n_f = free electron number densities

ω_{bj} = natural frequency of bound electrons

ω = frequency of radiation

g_{bj} = damping constants of bound electrons

g_f = damping constants of free electrons

m° = effective electron mass $\approx m_e/18$

The value for these parameters are given in subroutine REFRACTIVEINDEX. The following is a listing of the computer code incorporating the equations above.

Program name: ABSORPT1.BAS

```
REM      This program calculates the extinction and scattering
REM cross-sections, from which the absorption cross-section is
REM determined. The absorption efficiency and the mass specific
REM absorption cross-section are also determined. Inputs to the
REM program are input by modification of the subroutine INPUTS.
```

```
REM      If N(max.) reaches 150 there will be rounding error and the
REM information will not be valid at that point. If you know the
REM value for the refractive index then override the subroutine
REM REFRACTIVEINDEX by typing the values as m= and mi= at the
REM end of the subroutine.
```

REM The program follows the algorithm presented by Milton
 REM Kerker in The Scattering of Light and Other Electromagnetic
 REM Radiation, pp. 27-69.

REM The variable name designation follows that of Kerker with
 REM the following exceptions:

REM
 REM VARIABLENAME = REAL part of the variable
 REM VARIABLENAMEi = IMAGINARY part of the variable
 REM mVARIABLENAME = Magnitude of the vector
 REM aVARIABLENAME = Angle of the vector from the horizontal

DECLARE SUB REFRACTIVEINDEX (lambda#, m#, mi#)
 DECLARE SUB INPUTS (m#, mi#, mm#, am#, lambda#, rho#)
 DECLARE SUB BESSEL (n%, alpha#, gn1#, gn#, gn.1#)
 DECLARE SUB ZETAFUNCTION (psin#, chin#, zetareal#, zetaim#, zetamag#, zetaangle#)
 DECLARE SUB ETABETA1 (n%, beta#, betai#, mbeta#, abeta#, eta.1#, etai.1#, meta.1#,
 aeta.1#, eta#, etai#, meta#, aeta#)
 DECLARE SUB A (m#, mi#, mm#, am#, etabeta#, etabetai#, metabeta#, aetabeta#,
 etalalph#, etalalphi#, metalalph#, aetalalph#, eta3alph#, eta3alphi#, meta3alph#, aeta3alph#,
 psin#, zetan#, zetani#, mzetan#, azetan#, ann#, anni#, mann#, aann#)
 DECLARE SUB B (m#, mi#, mm#, am#, etabeta#, etabetai#, metabeta#, aetabeta#,
 etalalph#, etalalphi#, metalalph#, aetalalph#, eta3alph#, eta3alphi#, meta3alph#, aeta3alph#,
 psin#, zetan#, zetani#, mzetan#, azetan#, bnn#, bnai#, mbnn#, abnn#)
 DECLARE SUB ETA1ALPHAN (n%, alpha#, gn#, gn.1#, eta#, etai#, meta#, aeta#)
 DECLARE SUB ETA3ALPHAN (n%, alpha#, gn#, gni#, mgn#, agn#, gn.1#, gni.1#, mgn.1#,
 agn.1#, eta#, etai#, meta#, aeta#)
 DECLARE SUB SINH (argu#, result#)
 DECLARE SUB COSH (argu#, result#)
 DECLARE SUB INITIALETA1 (A#, B#, eta1#, etai1#, meta1#, aeta1#)
 DECLARE SUB arctan (x#, y#, ang#)
 DECLARE SUB INITIALVALUE (alpha#, psi0#, psi1#, chi0#, chi1#, zeta0#, zeta0i#,
 mzeta0#, mzeta0#)
 DEFDBL A-H, L-M, O-Z
 DEFINT I-K, N

 DIM psi(0 TO 160)
 DIM chi(0 TO 160)
 DIM zeta(0 TO 160), zetai(0 TO 160), mzeta(0 TO 160), azeta(0 TO 160)
 DIM etalalpha(0 TO 160), etalalphi(0 TO 160)
 DIM metalalpha(0 TO 160), aetalalpha(0 TO 160)
 DIM eta3alpha(0 TO 160), eta3alphi(0 TO 160)

```

DIM meta3alpha(0 TO 160), aeta3alpha(0 TO 160)
DIM eta1beta(0 TO 160), eta1betai(0 TO 160)
DIM meta1beta(0 TO 160), aeta1beta(0 TO 160)
DIM an(0 TO 160), ani(0 TO 160), man(0 TO 160), aan(0 TO 160)
DIM bn(0 TO 160), bni(0 TO 160), mbn(0 TO 160), abn(0 TO 160)

```

```

COMMON SHARED pi, n, r, alpha, m, mi, mm, am, lambda, beta, betai, mbeta, abeta
CLS
INPUT "what is the name of the output file"; output$
OPEN output$ FOR OUTPUT AS #1

```

```

CALL INPUTS(m, mi, mm, am, lambda, rho)
FOR j = 1 TO 4
FOR i = 1 TO 99
n = 0
sumscat = 0#
sumext = 0#

```

```

counter = i / 10#
multiplier = .00000001# * (10# ^ j)
r = multiplier + counter * multiplier

```

```

alpha = 2# * pi * r / lambda
beta = m * alpha
betai = mi * alpha
mbeta = (beta ^ 2# + betai ^ 2#) ^ .5#
CALL arctan(beta, betai, abeta)
CALL INITIALVALUE(alpha, psi(0), psi(1), chi(0), chi(1), zeta(0), zetai(0), mzeta(0),
azeta(0))

```

```

CALL INITIALETA1(beta, betai, eta1beta(0), eta1betai(0), meta1beta(0), aeta1beta(0))

```

```

DO
n = n + 1
CALL BESSEL(n, alpha, psi(n + 1), psi(n), psi(n - 1))
CALL BESSEL(n, alpha, chi(n + 1), chi(n), chi(n - 1))

```

```

CALL ZETAFUNCTION(psi(n), chi(n), zeta(n), zetai(n), mzeta(n), azeta(n))
CALL ETA1ALPHAN(n, alpha, psi(n), psi(n - 1), eta1alpha(n), eta1alphai(n), meta1alpha(n),
aeta1alpha(n))
CALL ETA3ALPHAN(n, alpha, zeta(n), zetai(n), mzeta(n), azeta(n), zeta(n - 1), zetai(n - 1),
mzeta(n - 1), azeta(n - 1), eta3alpha(n), eta3alphai(n), meta3alpha(n), aeta3alpha(n))

```

```
CALL ETABETA1(n, beta, betai, mbeta, abeta, etalbeta(n - 1), etalbetai(n - 1), metalbeta(n - 1), aetalbeta(n - 1), etalbeta(n), etalbetai(n), metalbeta(n), aetalbeta(n))
```

```
CALL A(m, mi, mm, am, etalbeta(n), etalbetai(n), metalbeta(n), aetalbeta(n), etalalpha(n), etalalphai(n), metalalpha(n), aetalalpha(n), eta3alpha(n), eta3alphai(n), meta3alpha(n), aeta3alpha(n), psi(n), zeta(n), zetai(n), mzeta(n), azeta(n), an(n), _  
ani(n), man(n), aan(n))
```

```
CALL B(m, mi, mm, am, etalbeta(n), etalbetai(n), metalbeta(n), aetalbeta(n), etalalpha(n), etalalphai(n), metalalpha(n), aetalalpha(n), eta3alpha(n), eta3alphai(n), meta3alpha(n), aeta3alpha(n), psi(n), zeta(n), zetai(n), mzeta(n), azeta(n), bn(n), _  
bni(n), mbn(n), abn(n))
```

```
checkext = sumext
```

```
checkscat = sumscat
```

```
sumscat = sumscat + (2# * n + 1#) * (man(n) ^ 2# + mbn(n) ^ 2#)
```

```
sumext = sumext + (2# * n + 1#) * (an(n) + bn(n))
```

```
LOOP UNTIL ((sumext - checkext) = 0# AND (sumscat - checkscat) = 0#) OR n >= 150
```

```
cscat = lambda ^ 2# / 2# / pi * sumscat
```

```
cext = lambda ^ 2# / 2# / pi * sumext
```

```
cabs = cext - cscat
```

```
qabs = cabs / pi / r ^ 2#
```

```
rb = cabs / rho / (4# / 3# * pi * r ^ 3#)
```

```
CLS
```

```
LOCATE 10, 10
```

```
PRINT "PROGRAM IS RUNNING DO NOT 'BREAK'"
```

```
LOCATE 15, 10
```

```
PRINT "r =", r * 1000000#, "micrometers"
```

```
LOCATE 19, 10
```

```
PRINT "N(max) =", n
```

```
PRINT #1, r; alpha; rb; cabs; qabs, 2# * r, n
```

```
NEXT i
```

```
NEXT j
```

```
CLOSE #1
```

```
END
```

```
SUB A (m, mi, mm, am, etabeta, etabetai, metabeta, aetabeta, etalalph, etalalphi, metalalph,  
aetalalph, eta3alph, eta3alphi, meta3alph, aeta3alph, psin, zetan, zetani, mzetan, azetan, ann,  
anni, mann, aann)
```

```

mterm1 = mm * meta1alph
aterm1 = am + aeta1alph
term1 = mterm1 * COS(aterm1)
term1i = mterm1 * SIN(aterm1)
mterm2 = mm * meta3alph
aterm2 = am + aeta3alph
term2 = mterm2 * COS(aterm2)
term2i = mterm2 * SIN(aterm2)
term3 = etabeta - term1
term3i = etabetai - term1i
mterm3 = (term3 ^ 2# + term3i ^ 2#) ^ .5#
CALL arctan(term3, term3i, aterm3)
term4 = etabeta - term2
term4i = etabetai - term2i
mterm4 = (term4 ^ 2# + term4i ^ 2#) ^ .5#
CALL arctan(term4, term4i, aterm4)
mterm5 = mterm3 / mterm4
aterm5 = aterm3 - aterm4
mpsin = ((psin) ^ 2#) ^ .5#
mann = mpsin / mzetan * mterm5
CALL arctan(psin, 0, apsin)
aann = apsin - azetan + aterm5
ann = mann * COS(aann)
anni = mann * SIN(aann)

```

END SUB

DEFINT L

SUB arctan (x, y, ang)

ang = ATN(y / x)

IF (x < 0# AND y <= 0#) THEN ang = ATN(y / x) + pi

IF (x < 0# AND y > 0#) THEN ang = ATN(y / x) + pi

IF (x > 0# AND y < 0#) THEN ang = ATN(y / x)

IF (x > 0# AND y > 0#) THEN ang = ATN(y / x)

END SUB

DEFDBL L

SUB B (m, mi, mm, am, etabeta, etabetai, metabeta, aetabeta, eta1alph, eta1alphi, meta1alph, aeta1alph, eta3alph, eta3alphi, meta3alph, aeta3alph, psin, zetan, zetani, mzetan, azetan, bnn, bnni, mbnn, abnn)

```

mterm1 = mm * metabeta
aterm1 = am + aetabeta
term1 = mterm1 * COS(aterm1)
term1i = mterm1 * SIN(aterm1)
mterm2 = mm * metabeta
aterm2 = am + aetabeta
term2 = mterm2 * COS(aterm2)
term2i = mterm2 * SIN(aterm2)
term3 = eta1alph - term1
term3i = eta1alphi - term1i
mterm3 = (term3 ^ 2# + term3i ^ 2#) ^ .5#
CALL arctan(term3, term3i, aterm3)
term4 = eta3alph - term2
term4i = eta3alphi - term2i
mterm4 = (term4 ^ 2# + term4i ^ 2#) ^ .5#
CALL arctan(term4, term4i, aterm4)
mterm5 = mterm3 / mterm4
aterm5 = aterm3 - aterm4
mpsin = ((psin) ^ 2#) ^ .5#
mbnn = mpsin / mzetan * mterm5
CALL arctan(psin, 0, apsin)
abnn = apsin - azetan + aterm5
bnn = mbnn * COS(abnn)
bnni = mbnn * SIN(abnn)

```

END SUB

DEFINT L

SUB BESSEL (n, alpha, gn1, gn, gn.1)

```
gn1 = ((2# * n + 1#) / alpha) * gn - gn.1
```

END SUB

SUB COSH (argu, result)

```
IF argu > 706# THEN argu = 706#
```

```
result = (EXP(argu) + EXP(-argu)) / 2#
```

END SUB

SUB ETA1ALPHAN (n, alpha, gn, gn.1, eta, etai, meta, aeta)

```

eta = (gn.1 / gn) - (n / alpha)
etai = 0#
meta = ((eta) ^ 2#) ^ .5#
CALL arctan(eta, 0#, aeta)

```

END SUB

DEFDBL L

```

SUB ETA3ALPHAN (n, alpha, gn, gni, mgn, agn, gn.1, gni.1, mgn.1, agn.1, eta, etai, meta,
aeta)
mterm1 = mgn.1 / mgn
aterm1 = agn.1 - agn
eta = mterm1 * COS(aterm1) - n / alpha
etai = mterm1 * SIN(aterm1)
meta = (eta ^ 2# + etai ^ 2#) ^ .5#
CALL arctan(eta, etai, aeta)

```

END SUB

DEFINT L

```

SUB ETABETA1 (n, beta, betai, mbeta, abeta, eta.1, etai.1, meta.1, aeta.1, eta, etai, meta,
aeta)
mterm1 = mbeta ^ 2#
aterm1 = abeta * 2#
mterm2 = n * mbeta * meta.1
aterm2 = abeta + aeta.1
mterm3 = n ^ 2#
aterm3 = 0#
mterm4 = n * mbeta
aterm4 = abeta
mterm5 = mbeta ^ 2# * meta.1
aterm5 = abeta * 2# + aeta.1
term6 = mterm1 * COS(aterm1) + mterm2 * COS(aterm2) - mterm3 * COS(aterm3)
term6i = mterm1 * SIN(aterm1) + mterm2 * SIN(aterm2) - mterm3 * SIN(aterm3)
mterm6 = (term6 ^ 2# + term6i ^ 2#) ^ .5#
CALL arctan(term6, term6i, aterm6)
term7 = mterm4 * COS(aterm4) - mterm5 * COS(aterm5)
term7i = mterm4 * SIN(aterm4) - mterm5 * SIN(aterm5)

```



```

mterm7 = (term7 ^ 2# + term7i ^ 2#) ^ .5#
CALL arctan(term7, term7i, aterm7)
meta = mterm6 / mterm7
aeta = aterm6 - aterm7
eta = meta * COS(aeta)
etai = meta * SIN(aeta)

```

END SUB

```

SUB INITIALETA1 (aa, bb, etal, etali, metal, aetal)
term1 = SIN(2# * aa)
CALL SINH(-2# * bb, term2)
CALL COSH(-2# * bb, term3)
term4 = COS(2# * aa)
etal = term1 / (term3 - term4)
etali = term2 / (term3 - term4)
metal = (etal ^ 2# + etali ^ 2#) ^ .5#
CALL arctan(etal, etali, aetal)

```

END SUB

```

SUB INITIALVALUE (alpha, psi0, psi1, chi0, chi1, zeta0, zeta0i, mzeta0, azeta0)
psi0 = SIN(alpha)
psi1 = ((SIN(alpha)) / alpha) - COS(alpha)
chi0 = COS(alpha)
chi1 = ((COS(alpha)) / alpha) + SIN(alpha)
zeta0 = psi0
zeta0i = chi0
mzeta0 = (psi0 ^ 2# + chi0 ^ 2#) ^ .5#
CALL arctan(zeta0, zeta0i, azeta0)
END SUB

```

DEFDBL L

SUB INPUTS (m, mi, mm, am, lambda, rho)

CLS

```

pi = ATN(1#) * 4#
lambda = .000000632#      'm
rho = 2000#                'kg/m^3
CALL REFRACTIVEINDEX(lambda, m, mi)
mm = (m ^ 2# + mi ^ 2#) ^ .5#
CALL arctan(m, mi, am)

```

```

PRINT #1, "complex refraction index ="; m; "- i"; -mi
PRINT #1, "rho ="; rho; "kg/m^3"
PRINT #1, "lambda ="; lambda; "m"
PRINT #1, "      r      alpha      Rb      Cabs      Qabs
Diam      N(max)"
PRINT #1, "      (m)      (m^2/kg)      (m^2)
(m)      "
END SUB

```

```
DEFDBL K, N
```

```
SUB REFRACTIVEINDEX (lambda, m, mi)
```

```

' This algorithm is based on a paper by S. C. Lee and C. L. Tien
' "Optical Constants of Soot in Hydrocarbon Flames.", Eighteenth Symposium
' (International) on Combustion, The Combustion Institute, 1981.

```

```

e = 1.6022D-19      'electron charge      C
emass = 9.109500000000001D-31 'electron mass in vacuum      kg
effmass = emass / 18#      'effective electron mass      kg
epsilon = 8.85411D-12      'permittivity constant      C^2/N/m^2
bn1 = 4.07D+27      'bound electron number density      m^-3
bn2 = 4.47D+28      'bound electron number density      m^-3
free = 4D+25      'free electron number density      m^-3
wb1 = 12500000000000000#      'natural frequency of bound electrons s^-1
wb2 = 72500000000000000#      'natural frequency of bound electrons s^-1
w = 299792500# / lambda      'frequency of radiation      s^-1
'properties at 1450 K
'gb1 = 59000000000000000#      'damping constant of bound electron s^-1
'gb2 = 56000000000000000#      'damping constant of bound electron s^-1
'gf = 12000000000000000#      'damping constant of free electron s^-1
'properties at 300 K
gb1 = 25000000000000000#      'damping constant of bound electron s^-1
gb2 = 25300000000000000#      'damping constant of bound electron s^-1
gf = 54000000000000000#      'damping constant of free electron s^-1

```

```

'FOR j = 0 TO 7
'FOR i = 1 TO 99
'counter = i / 10#
'multiplier = .0000001# * (10# ^ j)
'lambda = multiplier + counter * multiplier

```

```

w = 2# * pi * 299792500# / lambda
term1 = bn1 * (wb1 ^ 2# - w ^ 2#) / ((wb1 ^ 2# - w ^ 2#) ^ 2# + w ^ 2# * gb1 ^ 2#)
term2 = bn2 * (wb2 ^ 2# - w ^ 2#) / ((wb2 ^ 2# - w ^ 2#) ^ 2# + w ^ 2# * gb2 ^ 2#)
term3 = term1 + term2
term4 = term3 * e ^ 2# / emass / epsilon
term5 = e ^ 2# / effmass / epsilon * free / (w ^ 2# + gf ^ 2#)
c2 = 1# + term4 - term5

```

```

term1 = bn1 * w * gb1 / ((wb1 ^ 2# - w ^ 2#) ^ 2# + w ^ 2# * gb1 ^ 2#)
term2 = bn2 * w * gb2 / ((wb2 ^ 2# - w ^ 2#) ^ 2# + w ^ 2# * gb2 ^ 2#)
term3 = term1 + term2
term4 = e ^ 2# / emass / epsilon * term3
term5 = e ^ 2# / effmass / epsilon * free * gf / w / (w ^ 2# + gf ^ 2#)
c1 = term4 + term5

```

```

n = ((c2 + (c2 ^ 2# + c1 ^ 2#) ^ .5#) / 2#) ^ .5#
k = c1 / 2# / n
m = n
mi = -k

```

```

PRINT #1, lambda, n, k

```

```

'NEXT i
LOCATE 10, 10
PRINT "lambda = "; lambda
'NEXT j

```

```

END SUB

```

```

DEFINT K-L, N
SUB SINH (argu, result)
IF argu > 706# THEN argu = 706#
result = (EXP(argu) - EXP(-argu)) / 2#
END SUB

```

```

SUB ZETAFUNCTION (psin, chin, zetareal, zetaim, zetamag, zetaangle)
zetareal = psin
zetaim = chin
zetamag = (zetareal ^ 2# + zetaim ^ 2#) ^ .5#
CALL arctan(zetareal, zetaim, zetaangle)

```

```

END SUB

```

APPENDIX B. DERIVATION OF THE PHOTOACOUSTIC EQUATION

The following is a derivation of the PAS signal equation, Eq. (2.11), as developed by Roessler and Faxvog [27]. Neglecting the effects of acoustic losses produced by heat conduction and viscosity, the wave equation can be written as

$$\nabla^2 p - \frac{1}{c^2} \frac{\partial^2 p}{\partial t^2} = \frac{(\gamma - 1)}{c^2} \frac{\partial H}{\partial t} \quad (\text{B.1})$$

The absorbed light is assume to be equivalent to a heat source $H(r,t)$ producing a change in acoustic pressure p . The symbol c is the velocity of sound in the sample chamber medium and γ is the ratio of the specific heats (c_p/c_v).

At modulation frequencies well away from acoustic resonances of the chamber and assuming that the pressure rise is uniform throughout the cell volume V , Eq. (B.1) becomes

$$\left(\frac{\partial p}{\partial t} \right) = (\gamma - 1)H \quad (\text{B.2})$$

The heat generated in a volume element $A dz$ is given by

$$dH = b_a I(z) A dz \quad (\text{B.3})$$

where $I(z) = I_0 \exp(-b_E z)$ is the light beam intensity at a distance z into the sample, b_a and b_e are the absorption and extinction coefficients, respectively, and A is the cross sectional area of

the radiation source. The average heat generated per unit volume throughout the cell is $\int dH / V$ or

$$\left(\frac{AI}{V}\right)\left(\frac{b_a}{b_e}\right)[1 - \exp(-b_e L)] \quad (B.4)$$

where L is the cell length. Heat generated by cell wall absorption of any light scattered from the main beam is assumed to be negligible.

The modulated radiation is square-wave time dependent and can be expressed as its Fourier sum

$$I(t) = I_0 + \frac{4}{\pi} I_0 \sum_{n=1}^{\infty} \frac{\sin[(2n-1)\omega t]}{(2n-1)} \quad (B.5)$$

Here, I_0 is the half peak intensity (i. e., the average intensity) and ω is the angular chopping frequency, $2\pi f$.

Substituting for $I(t)$ in Eq. (B.4) gives

$$H = \frac{W}{V} \frac{b_a}{b_e} (1 - e^{-b_e L}) \left\{ 1 + \frac{4}{\pi} \sum_{n=1}^{\infty} \frac{\sin[(2n-1)\omega t]}{(2n-1)} \right\} \quad (B.6)$$

where the average incident power $W = AI_0$.

Integrating Eq. (B.2) and substituting for H from Eq. (B.6) gives

$$p(t) = \frac{(\gamma - 1)W}{V} \frac{b_a}{b_e} (1 - e^{-b_e L}) \left(t - \frac{4}{\omega\pi} \sum_{n=1}^{\infty} \frac{\cos(2n-1)\omega t}{(2n-1)^2} \right) \quad (B.7)$$

The linear term in the time dependence simply represents the dc heating of the cell and is ultimately balanced by the cell heat losses which have been neglected in Eq. (B.1). The sinusoidal voltage produced by the microphone will be $\sigma_m p$, where σ_m is the microphone sensitivity (mV/Pa) at the modulation frequency f . This signal, fed to a lock-in amplifier set at the fundamental frequency, produces a dc output voltage which is the rms value of the input, i.e., $\sigma_m p / \sqrt{2}$. This final output voltage S is as follows, from Eq. (B.7)

$$S = \frac{4(\gamma - 1)\sigma_m}{\sqrt{2}\pi\omega V} \frac{b_a}{b_e} (1 - e^{-b_e L}) W \quad (\text{B.8})$$

It is convenient to express this in the form

$$S = R \left(\frac{b_a}{b_e} \right) [1 - e^{-b_e L}] W/L \quad (\text{B.9})$$

where R is the responsivity, given as

$$R = \frac{4(\gamma - 1)\sigma_m L}{\pi\omega V\sqrt{2}} \quad (\text{B.10})$$

APPENDIX C. PROPAGATION OF ERROR ANALYSIS

Propagation of error on the PAS signal can be performed using Eq. (2.13) and Eq. (2.14). Propagation of error analysis states that:

$$\left(\frac{\Delta S}{S}\right)^2 = \left(\frac{\partial S}{\partial R} \frac{\Delta R}{S}\right)^2 + \left(\frac{\partial S}{\partial A_a} \frac{\Delta A_a}{S}\right)^2 + \left(\frac{\partial S}{\partial M_c} \frac{\Delta M_c}{S}\right)^2 + \left(\frac{\partial S}{\partial W} \frac{\Delta W}{S}\right)^2 \quad (C.1)$$

Using Eq. (2.13) and performing the differentiation, Eq. (C.1) becomes:

$$\frac{\Delta S}{S} = \sqrt{\left(\frac{\Delta R}{R}\right)^2 + \left(\frac{\Delta A_a}{A_a}\right)^2 + \left(\frac{\Delta M_c}{M_c}\right)^2 + \left(\frac{\Delta W}{W}\right)^2} \quad (C.2)$$

Where $\frac{\Delta R}{R}$ is obtained from Eq. (2.14):

$$\frac{\Delta R}{R} = \sqrt{\left(\frac{\delta R}{\delta \gamma} \frac{\Delta \gamma}{R}\right)^2 + \left(\frac{\delta R}{\delta \sigma_m} \frac{\Delta \sigma_m}{R}\right)^2 + \left(\frac{\delta R}{\delta L} \frac{\Delta L}{R}\right)^2 + \left(\frac{\delta R}{\delta \omega} \frac{\Delta \omega}{R}\right)^2 + \left(\frac{\delta R}{\delta V} \frac{\Delta V}{R}\right)^2} \quad (C.3)$$

After performing the differentiation with L and V equal to constants, Eq. (C.3) becomes:

$$\frac{\Delta R}{R} = \sqrt{\left(\frac{\Delta \gamma}{\gamma - 1}\right)^2 + \left(\frac{\Delta \sigma_m}{\sigma_m}\right)^2 + 0 + \left(\frac{-\Delta \omega}{\omega}\right)^2 + 0} \quad (C.4)$$

Table C.1 lists the parameters used in Eq. (C.2) and Eq. (C.4)

Table C.1 Parameters of propagation of error analysis

Parameter	Value	Comments
$\frac{\Delta M_c}{M_c}$	~ 0.10	Auger calibration data
$\frac{\Delta R}{R}$	~ 0.02	Eq. (C.4), assumes closed volume
$\frac{\Delta A_n}{A_n}$	~ 0.05	Error for each size range
$\left(\frac{\Delta W}{W}\right)_{\text{microwave}}$	~ 0.05	Power measurements
$\left(\frac{\Delta W}{W}\right)_{\text{optical}}$	~ 0.025	Power measurements
$\frac{\Delta \omega}{\omega}$	~ 0.02	Drifting of the modulation frequency
$\frac{\Delta \sigma_m}{\sigma_m}$	~ 0.000	Microphone calibration data
$\frac{\Delta \gamma}{\gamma - 1}$	~ 0.000	Temperature variations were small

Using the above values, $\left(\frac{\Delta S}{S}\right)_{\text{optical}} = 11.6\%$ and $\left(\frac{\Delta S}{S}\right)_{\text{microwave}} = 12.4\%$. The largest factor

in the error is due to the calculation of the carbon mass loadings. In chapter 4, Figure 4.9, the variations in the PAS signal can be seen. The oscillations in the PAS signal are probably due to changes in the mass loading caused by the width of the auger, producing an oscillatory pattern in the PAS signal as the auger turns.

APPENDIX D. DATA ACQUISITION PROGRAM

The following is a listing of the data acquisition program for the PAS project. It takes data from the lock-in amplifier as well as controls remotely all lock-in settings. Specific subroutines control the optical chopper which enables data collection of PAS signal verses chopping frequency. The subroutines are names after the path that must be taken to get into the subroutine. For example: if the user presses F1 followed by F3 the subroutine that would be executed is F1F3. A full list of the lock-in commands is contained in the Stanford lock-in manual.

Program name: LOCK-IN2.BAS

```

DECLARE SUB WriteData ( )
DECLARE SUB GraphData ( )
DECLARE SUB GraphBox ( )
DECLARE SUB WriteSettings ( )
DECLARE SUB Sensitivity ( )
DECLARE SUB Arrow (i%, j%, l%, m%, n%)
DECLARE SUB LockinRead ( )
DECLARE SUB F1F1 ( )
DECLARE SUB F1F2 ( )
DECLARE SUB F1F3 ( )
DECLARE SUB F1F4 ( )
DECLARE SUB F1F5 ( )
DECLARE SUB F1F6 ( )
DECLARE SUB F1F7 ( )
DECLARE SUB F1F8 ( )
DECLARE SUB UpdateScreen ( )
DECLARE SUB F1 ( )
DECLARE SUB F2 ( )
DECLARE SUB F3 ( )
DECLARE SUB F4 ( )

```

```

DECLARE SUB F5 ( )
DECLARE SUB F6 ( )
DECLARE SUB F7 ( )
DECLARE SUB DisplayWindow ( )
DEFINT I-K
DIM PlotData(1 TO 256)

```

```

COMMON SHARED RCOS$, RSIN$, RCOS, RSIN, Freq, sensitiv$, isensitiv, sensitiv
COMMON SHARED DynRes$, linex1$, linex2$, bandpass$, pretime$, posttime$
COMMON SHARED ENBW$, datafile$, Time, time0, delt, RefPhase, PlotData( )

```

```

SCREEN 9           ' Allows graphics to be used.
COLOR 7           ' Assigns the correct color for the screen.
KEY 1, CHR$(1)    ' Assigns the function
KEY 2, CHR$(2)    ' keys to different
KEY 3, CHR$(3)    ' character strings
KEY 4, CHR$(4)
KEY 5, CHR$(5)
KEY 6, CHR$(6)
KEY 7, CHR$(7)
KEY 8, CHR$(8)
KEY 9, CHR$(19)

CLS 2             ' Clears the computer screen.
OPEN "COM1:19200,N,8,2,CS,DS,CD" FOR RANDOM AS #1
                  ' Sets up com1 port to 19,200 baud, no parity, 8 data bits,
                  ' ignore CTS (clear to send), DSR (data set ready),
                  ' and CD (carrier detect).
PRINT #1, "      " ' Clears port by sending spaces

```

' *** SETS LOCK-IN TO INITIAL VALUES *****'**

```

PRINT #1, "Z"      ' Resets the SR530 lock-in Amplifier.
FOR j = 1 TO 1600: NEXT j ' Waits for RESET to finish.
PRINT #1, "B1"     ' Turns the BANDPASS FILTER on.
PRINT #1, "C0"     ' Displays the REFERENCE FREQUENCY.
PRINT #1, "G12"    ' Sets the SENSITIVITY to 50  $\mu$ V.
PRINT #1, "D1"     ' Sets the DYNAMIC RESERVE to HIGH.
PRINT #1, "L1,0"   ' Turns the LINE FILTER OFF.
PRINT #1, "L2,0"   ' Turns the 2x LINE FILTER OFF.
PRINT #1, "N1"     ' Sets the ENBW to 10 Hz.
PRINT #1, "P0.0"   ' Sets the REFERENCE PHASE SHIFT to ZERO.
PRINT #1, "S2"     ' Selects an output of R, $\theta$ .

```

```

PRINT #1, "T1,7"      ' Sets PRE TIME CONSTANT at 1 SECOND.
PRINT #1, "T2,0"      ' Sets POST TIME CONSTANT OFF.
PRINT #1, "R0"        ' Sets the TRIGGER to SQUARE WAVE.
delt = 1!              ' Default of 1 second between writing to data file.
datafile$ = ""         ' Sets data filename to null.
                        ' #1 communications between the lock-in and the computer
                        ' #2 communications between the data file and the computer

```

```

CALL DisplayWindow
CALL GraphBox
DO

```

```

key$ = INKEY$
IF key$ = CHR$(1) THEN CALL F1
IF key$ = CHR$(2) THEN CALL F2
IF key$ = CHR$(3) THEN CALL F3
IF key$ = CHR$(4) THEN CALL F4
IF key$ = CHR$(5) THEN CALL F5
IF key$ = CHR$(6) THEN CALL F6
IF key$ = CHR$(7) THEN CALL F7
IF key$ = CHR$(8) THEN CLS 1: FOR i = 5 TO 255: PlotData(i) = 0: NEXT: CALL
GraphBox
IF key$ = "d" OR key$ = "D" THEN CALL WriteData
CALL LockinRead
CALL UpdateScreen
CALL GraphData

```

```

LOOP UNTIL key$ = CHR$(19)
chopvolt = 0#          ' Sets the chopping frequency to zero.
PRINT #1, USING "X6,##.#####"; chopvolt  ' Sets the voltage of X6 (D/A output).
CLOSE #1
CLOSE #2
END

```

```

DEFINT L-N
SUB Arrow (i, j, l, m, n)

```

```

    'i,j = upper left corner
    'l = bottom width - 1
    'm = length - 2
    'n = top width - 2
FOR k1 = 1 TO m
LOCATE (i + k1), (j)

```

```

PRINT "|"
NEXT k1
LOCATE (i + k1), j
PRINT "L"
LOCATE i, j
PRINT "r"

```

```

FOR k2 = 1 TO l
LOCATE (i + k1), (j + k2)
PRINT "-"
NEXT k2

```

```

FOR k2 = 1 TO n
LOCATE (i), (j + k2)
PRINT "-"
NEXT k2
LOCATE i, j + k2
PRINT ">"

```

```

END SUB

```

```

DEFSNG L-N
SUB DisplayWindow

```

```

CLS 2
LOCATE 1, 36
PRINT "MAIN MENU"
LOCATE 3, 45
PRINT "F1 LOCK-IN SETTINGS"
LOCATE 4, 45
PRINT "F2 DEFINE OUTPUT FILE"
LOCATE 5, 45
PRINT "F3 OPEN OR CLOSE A DATA FILE"
LOCATE 6, 45
PRINT "F4 WRITE DATA TO THE DATA FILE"
LOCATE 7, 45
PRINT "F5 WRITE SETTINGS TO THE DATA FILE"
LOCATE 8, 45
PRINT "F6 WRITE NOTES TO THE DATE FILE"
LOCATE 9, 45
PRINT "F7 LOCK-IN SIGNAL VS. FREQUENCY"
LOCATE 10, 45

```

```

PRINT "F8 CLEAR GRAPH DISPLAY"
LOCATE 11, 45
PRINT "F9 END PROGRAM"
LOCATE 14, 14
PRINT "LOCK-IN SETTINGS"
LOCATE 16, 8
PRINT "Sensitivity"
CALL Sensitivity
LOCATE 16, 32
PRINT sensitiv$
LOCATE 17, 8
PRINT "Dynamic Reserves"
PRINT #1, "D"
INPUT #1, in
IF in = 0 THEN DynRes$ = "low  "
IF in = 1 THEN DynRes$ = "normal"
IF in = 2 THEN DynRes$ = "high  "
LOCATE 17, 32
PRINT DynRes$
LOCATE 18, 8
PRINT "Pre Time Constant"
PRINT #1, "T1"
INPUT #1, in
IF in = 1 THEN pretime$ = "1 mS  "
IF in = 2 THEN pretime$ = "3 mS  "
IF in = 3 THEN pretime$ = "10 mS "
IF in = 4 THEN pretime$ = "30 mS "
IF in = 5 THEN pretime$ = "100 mS"
IF in = 6 THEN pretime$ = "300 mS"
IF in = 7 THEN pretime$ = "1 S   "
IF in = 8 THEN pretime$ = "3 S   "
IF in = 9 THEN pretime$ = "10 S  "
IF in = 10 THEN pretime$ = "30 S  "
IF in = 11 THEN pretime$ = "100 S "
LOCATE 18, 32
PRINT pretime$
LOCATE 19, 8
PRINT "Post Time Constant"
PRINT #1, "T2"
INPUT #1, in
IF in = 0 THEN posttime$ = "none "
IF in = 1 THEN posttime$ = "0.1 S"
IF in = 2 THEN posttime$ = "1 S  "

```

```

LOCATE 19, 32
PRINT posttime$
LOCATE 20, 8
PRINT "Band Pass Filter"
PRINT #1, "B"
INPUT #1, in
IF in = 0 THEN bandpass$ = "out"
IF in = 1 THEN bandpass$ = "in "
LOCATE 20, 32
PRINT bandpass$
LOCATE 21, 8
PRINT "Line Filter"
PRINT #1, "L1"
INPUT #1, in
IF in = 0 THEN linex1$ = "out"
IF in = 1 THEN linex1$ = "in "
LOCATE 21, 32
PRINT linex1$
LOCATE 22, 8
PRINT "Line x2 Filter"
PRINT #1, "L2"
INPUT #1, in
IF in = 0 THEN linex2$ = "out"
IF in = 1 THEN linex2$ = "in "
LOCATE 22, 32
PRINT linex2$
LOCATE 14, 54
PRINT "LOCK-IN SIGNAL"
PRINT #1, "S"
INPUT #1, in
IF in = 0 THEN RCOS$ = " X Volts": RSIN$ = " Y Volts"
IF in = 1 THEN RCOS$ = " X Offset": RSIN$ = " Y Offset"
IF in = 2 THEN RCOS$ = " R Volts": RSIN$ = "  $\theta$  Deg. "
IF in = 3 THEN RCOS$ = " R Offset": RSIN$ = "  $\theta$  Offset"
IF in = 4 THEN RCOS$ = " X Noise": RSIN$ = " Y Noise"
IF in = 5 THEN RCOS$ = " X6 (D/A)": RSIN$ = " X6 (D/A)"
PRINT #1, "N"
INPUT #1, in
IF in = 1 THEN ENBW$ = "10 Hz"
IF in = 0 THEN ENBW$ = "1 Hz"
PRINT #1, "P"
INPUT #1, RefPhase
LOCATE 16, 46

```

```

PRINT RCOS$
LOCATE 17, 46
PRINT "-----"
LOCATE 16, 63
PRINT RSIN$
LOCATE 17, 63
PRINT "-----"
LOCATE 20, 56
PRINT "Frequency"
LOCATE 21, 56
PRINT "-----"

```

'draw box

```

i = 13
j = 5
LOCATE i, j
PRINT "r"

FOR k2 = 1 TO 72
LOCATE (i + k1), (j + k2)
PRINT "—"
NEXT k2

```

```

LOCATE (i + k1), (j + k2)
PRINT "┐ "

```

```

FOR k1 = 1 TO 9
LOCATE (i + k1), (j)
PRINT "| "
LOCATE (i + k1), (j + k2)
PRINT "| "
LOCATE (i + k1), 42
PRINT "| "
NEXT k1

```

```

LOCATE (i + k1), j
PRINT "L"

```

```

FOR k2 = 1 TO 72
LOCATE (i + k1), (j + k2)
PRINT "—"
NEXT k2

```

```

LOCATE (i + k1), (j + k2)

```

```

PRINT "J "
LOCATE i, 42
PRINT "T"
LOCATE (i + k1), (42)
PRINT "L"
CALL GraphBox
END SUB

```

SUB F1

```

CLS 2
DO
i = 3
j = 29
LOCATE i, 32
PRINT "LOCK-IN SETTINGS"
LOCATE i + 1, 32
PRINT "_____ "
LOCATE i + 4, j
PRINT "F1 Bandpass and Notch Filters"
LOCATE i + 5, j
PRINT "F2 Sensitivity"
LOCATE i + 6, j
PRINT "F3 Dynamic Reserves"
LOCATE i + 7, j
PRINT "F4 Channel #1 and #2 Output"
LOCATE i + 8, j
PRINT "F5 Reference Phase Shift (Deg.)"
LOCATE i + 9, j
PRINT "F6 Time Constants"
LOCATE i + 10, j
PRINT "F7 Chopping Frequency"
LOCATE i + 11, j
PRINT "F8 Off Set Selected Channels"
LOCATE i + 12, j
PRINT "F9 Return to Main Menu"

keyF1$ = INKEY$
IF keyF1$ = CHR$(1) THEN CALL F1F1: keyF1$ = CHR$(19)
IF keyF1$ = CHR$(2) THEN CALL F1F2: keyF1$ = CHR$(19)
IF keyF1$ = CHR$(3) THEN CALL F1F3: keyF1$ = CHR$(19)
IF keyF1$ = CHR$(4) THEN CALL F1F4: keyF1$ = CHR$(19)

```



```

IF keyF1$ = CHR$(5) THEN CALL F1F5: keyF1$ = CHR$(19)
IF keyF1$ = CHR$(6) THEN CALL F1F6: keyF1$ = CHR$(19)
IF keyF1$ = CHR$(7) THEN CALL F1F7: keyF1$ = CHR$(19)
IF keyF1$ = CHR$(8) THEN CALL F1F8: keyF1$ = CHR$(19)
LOOP UNTIL keyF1$ = CHR$(19)
CLS 2
CALL DisplayWindow
END SUB

```

SUB F1F1

```

CLS 2
DO
i = 5
j = 29

LOCATE i, 32
PRINT "SIGNAL FILTERS"
LOCATE i + 1, 32
PRINT "_____ "
LOCATE i + 3, j
PRINT "F1 BandPass Filter"
LOCATE i + 3, j + 21
PRINT bandpass$
LOCATE i + 5, j
PRINT "F2 Line Filter"
LOCATE i + 5, j + 21
PRINT linex1$
LOCATE i + 7, j
PRINT "F3 Line x2 Filter"
LOCATE i + 7, j + 21
PRINT linex2$
LOCATE i + 9, j
PRINT "F4 Return to Main Menu"
PRINT #1, "B"
INPUT #1, in
IF in = 0 THEN bandpass$ = "out"
IF in = 1 THEN bandpass$ = "in "
PRINT #1, "L1"
INPUT #1, in
IF in = 0 THEN linex1$ = "out"
IF in = 1 THEN linex1$ = "in "

```

```

PRINT #1, "L2"
INPUT #1, in
IF in = 0 THEN linex2$ = "out"
IF in = 1 THEN linex2$ = "in "
KEYF1F1$ = INKEY$
IF KEYF1F1$ = CHR$(1) THEN PRINT #1, "K32" 'Toggles the bandpass filter.
IF KEYF1F1$ = CHR$(2) THEN PRINT #1, "K31" 'Toggles the linex1 filter.
IF KEYF1F1$ = CHR$(3) THEN PRINT #1, "K30" 'Toggles the linex2 filter.
LOOP UNTIL KEYF1F1$ = CHR$(4)
END SUB

```

SUB F1F2

```

CLS 2
DO
keyF1F2$ = INKEY$
LOCATE 11, 29
PRINT "SENSITIVITY ="
LOCATE 11, 44
PRINT sensitiv$
LOCATE 14, 14
PRINT "Press the '+' key to increase the sensitivity value"
LOCATE 15, 14
PRINT "Press the '-' key to decrease the sensitivity value"
LOCATE 20, 22
PRINT "PRESS F1 to return to the MAIN MENU"
IF keyF1F2$ = "+" THEN PRINT #1, "K27"
IF keyF1F2$ = "-" THEN PRINT #1, "K28"
PRINT #1, "G"
INPUT #1, isensitiv
CALL Sensitivity
LOOP UNTIL keyF1F2$ = CHR$(1)
END SUB

```

SUB F1F3

```

CLS 2
DO
LOCATE 5, 28
PRINT "DYNAMIC RESERVES"
LOCATE 5, 46

```

```

PRINT DynRes$
LOCATE 8, 26
PRINT "F1 Dynamic Reserves 'high'"
LOCATE 9, 26
PRINT "F2 Dynamic Reserves 'normal'"
LOCATE 10, 26
PRINT "F3 Dynamic Reserves 'low'"
LOCATE 11, 26
PRINT "F4 Return to the MAIN MENU"
PRINT #1, "D"
INPUT #1, in
IF in = 0 THEN DynRes$ = "low  "
IF in = 1 THEN DynRes$ = "normal"
IF in = 2 THEN DynRes$ = "high  "
keyF1F3$ = INKEY$
IF keyF1F3$ = CHR$(1) THEN PRINT #1, "D2"
IF keyF1F3$ = CHR$(2) THEN PRINT #1, "D1"
IF keyF1F3$ = CHR$(3) THEN PRINT #1, "D0"
LOOP UNTIL keyF1F3$ = CHR$(4)
END SUB

```

SUB F1F4

```

CLS 2
DO
LOCATE 5, 17
PRINT "CHANNEL #1 AND #2 OUTPUT"; RCOS$, " &"; RSIN$
PRINT #1, "S"
INPUT #1, in
IF in = 0 THEN RCOS$ = " X Volts"; RSIN$ = " Y Volts"
IF in = 1 THEN RCOS$ = " X Offset"; RSIN$ = " Y Offset"
IF in = 2 THEN RCOS$ = " R Volts"; RSIN$ = "  $\theta$  Deg. "
IF in = 3 THEN RCOS$ = " R Offset"; RSIN$ = "  $\theta$  Offset"
IF in = 4 THEN RCOS$ = " X Noise"; RSIN$ = " Y Noise"
IF in = 5 THEN RCOS$ = " X5 (D/A)"; RSIN$ = " X5 (D/A)"
LOCATE 8, 26
PRINT "F1  X Volts & Y Volts"
LOCATE 9, 26
PRINT "F2  X Offset & Y Offset"
LOCATE 10, 26
PRINT "F3  R Volts &  $\theta$  Deg."
LOCATE 11, 26

```

```

PRINT "F4  R Offset &  $\theta$  Deg."
LOCATE 12, 26
PRINT "F5  X Noise & Y Noise"
LOCATE 13, 26
PRINT "F6  X5 (D/A) & X6 (D/A)"
LOCATE 14, 26
PRINT "F7  Return to the MAIN MENU"
keyF1F4$ = INKEY$
IF keyF1F4$ = CHR$(1) THEN PRINT #1, "S0"
IF keyF1F4$ = CHR$(2) THEN PRINT #1, "S1"
IF keyF1F4$ = CHR$(3) THEN PRINT #1, "S2"
IF keyF1F4$ = CHR$(4) THEN PRINT #1, "S3"
IF keyF1F4$ = CHR$(5) THEN PRINT #1, "S4"
IF keyF1F4$ = CHR$(6) THEN PRINT #1, "S5"
LOOP UNTIL keyF1F4$ = CHR$(7)
END SUB

```

SUB F1F5

```

CLS 2
DO
LOCATE 5, 17
PRINT USING "The current Reference Phase Shift is ###.# Deg."; RefPhase
LOCATE 10, 23
PRINT "F1  Change Reference Phase Shift"
LOCATE 11, 23
PRINT "F2  Return to the MAIN MENU"
keyF1F5$ = INKEY$
IF keyF1F5$ = CHR$(1) THEN
    LOCATE 18, 17
    INPUT "Enter the Reference Phase Shift in degrees."; RefPhase
    PRINT #1, "P"; RefPhase
    PRINT #1, "P"
    INPUT #1, RefPhase
    CLS 2
END IF
LOOP UNTIL keyF1F5$ = CHR$(2)
END SUB

```

SUB F1F6

```

CLS 2
DO
LOCATE 5, 33
PRINT "TIME CONSTANTS"
LOCATE 6, 33
PRINT "_____ "
LOCATE 10, 28
PRINT "F1  Pre Time Constant"
LOCATE 11, 28
PRINT "F2  Post Time Constant"
LOCATE 12, 28
PRINT "F3  Set ENBW"
LOCATE 13, 28
PRINT "F4  Return to the MAIN MENU"
keyF1F6$ = INKEY$
IF keyF1F6$ = CHR$(1) THEN
  CLS 2
  DO
    keyF1F61$ = INKEY$
    LOCATE 9, 22
    PRINT "The current Pre Time Constant is "; pretime$
    LOCATE 14, 15
    PRINT "Press the '+' key to increase the Pre Time Constant"
    LOCATE 15, 15
    PRINT "Press the '-' key to decrease the Pre Time Constant"
    LOCATE 20, 22
    PRINT "PRESS F1 to return to the MAIN MENU"
    IF keyF1F61$ = "+" THEN PRINT #1, "K3"
    IF keyF1F61$ = "-" THEN PRINT #1, "K4"
    PRINT #1, "T1"
    INPUT #1, in
    IF in = 1 THEN pretime$ = "1 mS "
    IF in = 2 THEN pretime$ = "3 mS "
    IF in = 3 THEN pretime$ = "10 mS "
    IF in = 4 THEN pretime$ = "30 mS "
    IF in = 5 THEN pretime$ = "100 mS"
    IF in = 6 THEN pretime$ = "300 mS"
    IF in = 7 THEN pretime$ = "1 S  "
    IF in = 8 THEN pretime$ = "3 S  "
    IF in = 9 THEN pretime$ = "10 S  "
    IF in = 10 THEN pretime$ = "30 S  "
    IF in = 11 THEN pretime$ = "100 S "
    LOOP UNTIL keyF1F61$ = CHR$(1)
  
```

```

    keyF1F6$ = CHR$(4)
END IF
IF keyF1F6$ = CHR$(2) THEN
    CLS 2
    DO
        keyF1F62$ = INKEY$
        LOCATE 9, 21
        PRINT "The current Post Time Constant is "; posttime$
        LOCATE 14, 21
        PRINT "F1  Change Post Time Constant to none"
        LOCATE 15, 21
        PRINT "F2  Change Post Time Constant to 0.1 S"
        LOCATE 16, 21
        PRINT "F3  Change Post Time Constant to 1 S"
        LOCATE 17, 21
        PRINT "F4  Return to the MAIN MENU"
        IF keyF1F62$ = CHR$(1) THEN PRINT #1, "T2,0"
        IF keyF1F62$ = CHR$(2) THEN PRINT #1, "T2,1"
        IF keyF1F62$ = CHR$(3) THEN PRINT #1, "T2,2"
        PRINT #1, "T2"
        INPUT #1, in
        IF in = 0 THEN posttime$ = "none "
        IF in = 1 THEN posttime$ = "0.1 S"
        IF in = 2 THEN posttime$ = "1 S. "
        LOOP UNTIL keyF1F62$ = CHR$(4)
        keyF1F6$ = CHR$(4)
    END IF
IF keyF1F6$ = CHR$(3) THEN
    CLS 2
    DO
        keyF1F63$ = INKEY$
        LOCATE 9, 27
        PRINT "The current ENBW is "; ENBW$
        LOCATE 14, 27
        PRINT "F1  Change ENBW to 1 Hz"
        LOCATE 15, 27
        PRINT "F2  Change ENBW to 10 Hz"
        LOCATE 16, 27
        PRINT "F3  Return to the MAIN MENU"
        IF keyF1F63$ = CHR$(1) THEN PRINT #1, "N0"
        IF keyF1F63$ = CHR$(2) THEN PRINT #1, "N1"
        PRINT #1, "N"
        INPUT #1, in

```

```

IF in = 0 THEN ENBW$ = "1 Hz "
IF in = 1 THEN ENBW$ = "10 Hz"
LOOP UNTIL keyF1F6$ = CHR$(3)
keyF1F6$ = CHR$(4)
END IF
LOOP UNTIL keyF1F6$ = CHR$(4)
END SUB

```

SUB F1F7

```

CLS 2
PRINT #1, "X6"
INPUT #1, chopvolt
keyf1f7$ = ""
DO
keyf1f7$ = INKEY$
LOCATE 4, 24
PRINT "OPTICAL CHOPPER FREQUENCY ADJUST"
LOCATE 5, 24
PRINT "_____ "
LOCATE 9, 25
PRINT "F1 Set the Chopping Frequency"
LOCATE 10, 25
PRINT "F2 RETURN TO THE MAIN MENU"
LOCATE 13, 25
PRINT "Use the '+' and '-' keys for small adjustments"
LOCATE 14, 25
PRINT "in the chopping frequency"
IF keyf1f7$ = CHR$(1) THEN
  CLS 2
  LOCATE 9, 25
  INPUT "Input the desired chopping frequency"; chopfreq
  chopvolt = chopfreq * .0025
  PRINT #1, USING "X6,##.#####"; chopvolt ' sets the voltage of X6 (D/A output)
  keyf1f7$ = CHR$(2)
END IF
IF keyf1f7$ = "+" THEN
  chopvolt = chopvolt + .0025
  PRINT #1, USING "X6,##.#####"; chopvolt ' sets the voltage of X6 (D/A output)
  keyf1f7$ = ""
END IF
IF keyf1f7$ = "-" THEN

```

```

    chopvolt = chopvolt - .0025
    PRINT #1, USING "X6,##.#####"; chopvolt ' sets the voltage of X6 (D/A output)
    keyf1f7$ = ""
END IF
LOOP UNTIL keyf1f7$ = CHR$(2)
END SUB

```

SUB F1F8

```

CLS 2
DO
LOCATE 5, 17
PRINT "This function allows you to zero the selected displays"
LOCATE 12, 26
PRINT "F1  Apply the REL Function to Channel #1"
LOCATE 13, 26
PRINT "F2  Apply the REL Function to Channel #2"
LOCATE 14, 26
PRINT "F3  Return to the MAIN MENU"
keyF1F8$ = INKEY$
IF keyF1F8$ = CHR$(1) THEN PRINT #1, "K 21"
IF keyF1F8$ = CHR$(2) THEN PRINT #1, "k 13"
LOOP UNTIL keyF1F8$ = CHR$(3)
END SUB

```

SUB F2

```

CLS 2
LOCATE 3, 30
PRINT "DEFINE OUTPUT FILE"
LOCATE 4, 30
PRINT "_____ "
LOCATE 7, 13
PRINT "The fastest reading time is 0.25 seconds"
LOCATE 8, 13
PRINT "If t < 0.45 sec then only channel #1 will be read"
LOCATE 9, 13
PRINT "If 0.45 ≤ t < 0.60 sec then only channels #1 & #2 will be read"
LOCATE 10, 13
PRINT "If 0.6 ≤ t < 0.88 sec then the graph will not be updated"
LOCATE 11, 13

```



```

PRINT "If t > 0.88 sec then all attributes will be updated"
10 LOCATE 14, 13
INPUT "Enter a value for the time between readings (seconds)"; delt
IF delt < 0 THEN
    LOCATE 17, 13
    PRINT "The time between reading must be greater then 0"
    LOCATE 14, 66
    PRINT "          "
    CALL Arrow(14, 3, 8, 2, 7)
    GOTO 10
END IF
LOCATE 17, 13
PRINT "The time between readings will be"; delt; "seconds          "
LOCATE 20, 23
PRINT "HIT ANY KEY TO RETURN TO MAIN MENU"
DO
LOOP UNTIL INKEY$ <> ""
CLS 2
CALL DisplayWindow
END SUB

```

SUB F3

```

CLS 2
DO
LOCATE 3, 25
PRINT "OPEN OR CLOSE A DATA FILE"
LOCATE 4, 25
PRINT "_____ "
LOCATE 7, 25
PRINT "F1  Open a data file"
LOCATE 8, 25
PRINT "F2  Close a data file"
LOCATE 9, 25
PRINT "F3  Return to the main Menu"
keyF3$ = INKEY$
IF keyF3$ = CHR$(1) THEN
    LOCATE 13, 20
    IF datafile$ <> "" THEN
        PRINT "A data file is all ready open."
        LOCATE 14, 20
        PRINT "You must first close "; datafile$
    
```

```

    LOCATE 16, 20
    PRINT "PRESS ANY KEY TO CONTINUE"
    DO
    LOOP UNTIL INKEY$ <> ""
ELSE
    INPUT "Enter the name of the data file "; datafile$
    OPEN datafile$ FOR OUTPUT AS #2
    time0 = -999
END IF
CLS 2
END IF
IF keyF3$ = CHR$(2) THEN
    IF datafile$ = "" THEN
        LOCATE 13, 25
        PRINT "No data file has been opened."
        LOCATE 16, 25
        PRINT "PRESS ANY KEY TO CONTINUE"
        DO
        LOOP UNTIL INKEY$ <> ""
    ELSE
        LOCATE 13, 25
        PRINT "Do you wish to close "; datafile$; " (y/[n])";
        INPUT YES$
        IF YES$ = "y" OR YES$ = "Y" THEN
            CLOSE #2
            LOCATE 14, 25
            PRINT datafile$; " has been closed"
            datafile$ = ""
            LOCATE 16, 25
            PRINT "PRESS ANY KEY TO CONTINUE"
            DO
            LOOP UNTIL INKEY$ <> ""
        END IF
    END IF
END IF
CLS 2
END IF
LOOP UNTIL keyF3$ = CHR$(3)
CLS 2
CALL DisplayWindow
END SUB

```

SUB F4

```

mark$ = "mark @ time ="
IF time0 < 0 THEN
    time0 = TIMER
    WRITE #2, "time", RCOS$, RSIN$, "freq", "TIMER"
END IF
IF datafile$ = "" THEN
    CLS 2
    LOCATE 7, 26
    PRINT "NO DATA FILE HAS BEEN OPENED"
    LOCATE 14, 23
    PRINT "HIT ANY KEY TO RETURN TO MAIN MENU"
    DO
        LOOP UNTIL INKEY$ <> ""
ELSE
    COLOR 15
    LOCATE 3, 40
    PRINT "                                "
    LOCATE 4, 40
    PRINT "                                "
    LOCATE 5, 40
    PRINT "                                "
    LOCATE 6, 40
    PRINT "                                "
    LOCATE 7, 40
    PRINT "                                "
    LOCATE 7, 40
    PRINT "Data file -- "; datafile$
    LOCATE 8, 40
    PRINT "                                "
    LOCATE 9, 40
    PRINT "Press 'm' to mark an event        "
    LOCATE 10, 40
    PRINT "                                "
    LOCATE 11, 40
    PRINT "Hit 's' to stop writing data        "
    IF delt < .6 THEN
        LOCATE 3, 40
        PRINT "The time between reading is"; delt; "seconds"
        LOCATE 4, 40
        PRINT "There is not enough time to read"
        LOCATE 5, 40
        PRINT "all the lock-in values."
    
```

```

END IF
COLOR 7
CALL LockinRead
CALL UpdateScreen
PRINT #2, Time - time0, RCOS, RSIN, Freq, Time
DO
keyF4$ = INKEY$
IF keyF4$ = "m" OR keyF4$ = "M" THEN
    WRITE #2, mark$, TIMER - time0
END IF
IF (TIMER - Time) > delt THEN
    IF delt < .45 THEN
        PRINT #1, "q1"
        INPUT #1, RCOS
        Time = TIMER
        PRINT #2, Time - time0, RCOS, Blank123, Blank123, Time
        LOCATE 18, 47
        PRINT USING "+#.###^ ^ ^ ^"; RCOS
    END IF
    IF .45 <= delt AND delt < .6 THEN
        PRINT #1, "q1"
        INPUT #1, RCOS
        PRINT #1, "q2"
        INPUT #1, RSIN
        Time = TIMER
        PRINT #2, Time - time0, RCOS, RSIN, Blank123, Time
        LOCATE 18, 47
        PRINT USING "+#.###^ ^ ^ ^"; RCOS
        LOCATE 18, 64
        PRINT USING "+#.###^ ^ ^ ^"; RSIN
    END IF
    IF .6 <= delt AND delt < .88 THEN
        CALL LockinRead
        CALL UpdateScreen
        PRINT #2, Time - time0, RCOS, RSIN, Freq, Time
    END IF
    IF delt >= .88 THEN
        CALL LockinRead
        CALL UpdateScreen
        CALL GraphData
        PRINT #2, Time - time0, RCOS, RSIN, Freq, Time
    END IF
END IF
END IF

```

```

    LOOP UNTIL keyF4$ = "s" OR keyF4$ = "S"
END IF
CALL DisplayWindow
END SUB

```

SUB F5

```

CLS 2
IF datafile$ = "" THEN
    CLS 2
    LOCATE 7, 26
    PRINT "NO DATA FILE HAS BEEN OPENED"
    LOCATE 14, 23
    PRINT "HIT ANY KEY TO RETURN TO MAIN MENU"
    DO
        LOOP UNTIL INKEY$ <> ""
        CALL DisplayWindow
    ELSE
        LOCATE 3, 25
        PRINT "WRITE SETTINGS TO THE DATA FILE"
        LOCATE 4, 25
        PRINT "_____ "
        LOCATE 10, 14
        LOCATE 17, 23
        PRINT "HIT ANY KEY TO RETURN TO MAIN MENU"
        CALL WriteSettings
    END IF
    DO
        LOOP UNTIL INKEY$ <> ""
        CALL DisplayWindow
    END SUB

```

SUB F6

```

CLS 2
IF datafile$ = "" THEN
    CLS 2
    LOCATE 7, 26
    PRINT "NO DATA FILE HAS BEEN OPENED"
    LOCATE 14, 23
    PRINT "HIT ANY KEY TO RETURN TO MAIN MENU"

```

```

DO
  LOOP UNTIL INKEY$ <> ""
ELSE
  LOCATE 3, 26
  PRINT "WRITE NOTES TO THE DATA FILE"
  LOCATE 6, 18
  PRINT "Print the note that you want in the data file"
  LOCATE 8, 18
  INPUT note$
  WRITE #2, note$
  LOCATE 10, 18
  PRINT "The note "; note$, ""
  LOCATE 11, 18
  PRINT "is now in the data file."
  LOCATE 17, 23
  PRINT "HIT ANY KEY TO RETURN TO MAIN MENU"
DO
  LOOP UNTIL INKEY$ <> ""
END IF
CALL DisplayWindow
END SUB

```

```

DEFINT O
SUB F7

```

```

CLS 2
IF datafile$ = "" THEN
  CLS 2
  LOCATE 7, 26
  PRINT "NO DATA FILE HAS BEEN OPENED"
  LOCATE 14, 23
  PRINT "HIT ANY KEY TO RETURN TO MAIN MENU"
  DO
    LOOP UNTIL INKEY$ <> ""
ELSE
  PRINT #1, "X6"
  INPUT #1, chopvolt
  DO
    keyf7$ = INKEY$
    LOCATE 3, 30
    PRINT "SIGNAL VS FREQUENCY      "
    LOCATE 4, 30

```

```

PRINT "_____ "
LOCATE 8, 21
PRINT "F1  Referance signal generated by the lock-in"
LOCATE 9, 21
PRINT "F2  Referance signal generated by the chopper"
LOCATE 10, 21
PRINT "F3  Return to the MAIN MENU "
IF keyf7$ = CHR$(3) THEN exitsub$ = "yes"
IF keyf7$ = CHR$(1) THEN
  CLS 2
  PRINT #1, "R1" ' Sets the trigger to a SINE WAVE
  DO
    keyf7$ = INKEY$
    LOCATE 3, 30
    PRINT "SIGNAL VS FREQUENCY "
    LOCATE 4, 30
    PRINT "_____ "
    LOCATE 8, 21
    PRINT "F1  Frequency from 0 to 1000 Hz "
    LOCATE 9, 21
    PRINT "F2  Frequency from 1,000 to 10,000 Hz "
    LOCATE 10, 21
    PRINT "F3  Return to the MAIN MENU "
    IF keyf7$ = CHR$(3) THEN exitsub$ = "yes"
    IF keyf7$ = CHR$(1) THEN
      CLS 2
      LOCATE 3, 31
      PRINT "F1  0 to 1000 Hz"
      LOCATE 4, 31
      PRINT "_____ "
      LOCATE 8, 25
      INPUT "Input the lower limit in Hz"; low
      LOCATE 9, 25
      INPUT "Input the upper limit in Hz"; high
      LOCATE 10, 25
      INPUT "Input the frequency step in Hz"; delf
      IF low > 1000! THEN
        LOCATE 20, 19: PRINT "The lower limit must be lower than 1,000"
        CALL Arrow(8, 5, 12, 11, 13)
      END IF
      IF high > 1000! THEN
        LOCATE 21, 19: PRINT "The upper limit must be lower than 1,000"
        CALL Arrow(8, 5, 12, 12, 13)
      END IF
    END IF
  LOOP
END IF

```

```

END IF
IF low >= high THEN
    LOCATE 22, 14
    PRINT "The upper limit must be larger than the lower limit"
    CALL Arrow(8, 5, 7, 13, 13)
END IF
IF low < high AND high <= 1000! THEN keyf7$ = CHR$(3)
range$ = "100 Hz"
range = 100!
END IF
IF keyf7$ = CHR$(2) THEN
    CLS 2
    LOCATE 3, 31
    PRINT "F2  1,000 to 10,000 Hz"
    LOCATE 4, 31
    PRINT "_____ "
    LOCATE 8, 25
    INPUT "Input the lower limit in Hz"; low
    LOCATE 9, 25
    INPUT "Input the upper limit in Hz"; high
    LOCATE 10, 25
    INPUT "Input the frequency step in Hz"; delf
    IF low < 1000! THEN
        LOCATE 20, 19: PRINT "The lower limit must be greater than 1,000"
        CALL Arrow(9, 5, 12, 10, 13)
    END IF
    IF low > 10000! THEN
        LOCATE 21, 19: PRINT "The lower limit must be less than 10,000"
        CALL Arrow(9, 5, 12, 11, 13)
    END IF
    IF high > 10000! THEN
        LOCATE 22, 19: PRINT "The upper limit must be lower than 10,000"
        CALL Arrow(9, 5, 12, 12, 13)
    END IF
    IF low >= high THEN
        LOCATE 23, 14
        PRINT "The upper limit must be larger than the lower limit"
        CALL Arrow(9, 5, 7, 13, 13)
    END IF
    IF low < high AND high <= 10000! AND low >= 1000 THEN keyf7$ = CHR$(3)
range$ = "10 kHz"
range = 10000!
END IF

```



```

LOOP UNTIL keyf7$ = CHR$(3)
CLS 2
LOCATE 5, 20
PRINT "THE FOLLOWING CONNECTIONS NEED TO BE MADE"
LOCATE 6, 20
PRINT "_____ "
LOCATE 9, 4
PRINT "1. Connect the X5 (D/A output, rear panel) to the VCO INPUT (rear panel)"
LOCATE 10, 4
PRINT "2. Connect the REF OUTPUT (rear panel) to the REF INPUT (front panel)"
LOCATE 11, 4
PRINT "3. Set the VCO RANGE (rear panel) to "; range$
LOCATE 16, 20
PRINT "F1 Start the Signal vs Frequency test"
LOCATE 17, 20
PRINT "F2 Return to the MAIN MENU"
keyf7$ = ""
DO
keyf7$ = INKEY$
IF keyf7$ = CHR$(1) THEN
  CLS 2
  CALL WriteSettings
  WRITE #2, "Signal vs Frequency"
  WRITE #2, RCOSS$, RSIN$, "freq", "Sensitivity", "X5 voltage"
  IF ENBW$ = "10 Hz" THEN hold = 15      ' Sets the time for the
  IF ENBW$ = "1 Hz" THEN hold = 30      ' noise measurement to stabilize.
  PRINT #1, "G4"                        ' Sets the sensitivity to 100 nV.
  CALL Sensitivity
  FOR x = high TO low STEP -delf
  print2$ = "no"                        ' Flag to state that data has
                                         ' not been written to the data file.
  DO
  IF range$ = "100 Hz" THEN X5 = x * .00984 + .0295
  IF range$ = "10 kHz" THEN X5 = x * .000106 + .06295
  PRINT #1, USING "X5,##.#####"; X5    ' Sets the voltage of X5 (D/A output).
  PRINT #1, "X5"
  INPUT #1, X5
  CALL LockinRead
  CALL Sensitivity
  LOCATE 3, 30
  PRINT "SIGNAL VS FREQUENCY"
  LOCATE 4, 30
  PRINT "_____ "

```

```

LOCATE 7, 26
PRINT USING "Current Frequency##### Hz"; Freq
LOCATE 8, 26
PRINT USING "Current voltage output ##.#### volts"; X5
LOCATE 11, 30
PRINT USING "Upper Limit##### Hz"; high
LOCATE 12, 30
PRINT USING "Lower Limit##### Hz"; low
LOCATE 13, 30
PRINT USING "Step Size###.# Hz"; delf
LOCATE 16, 10
PRINT "The program proceeds from upper limit to the lower limit"
LOCATE 18, 10
PRINT "Press 'i' to interrupt the subroutine and return to the MAIN MENU"
SLEEP (hold)
CALL LockinRead
CALL Sensitivity
IF INKEY$ = "i" OR INKEY$ = "I" THEN
    exitsub$ = "yes"
    x = low - 1
    print2$ = "yes"
    OVERLOAD = 0
    RCOS = 0#
END IF
IF (RCOS < .8 * sensitiv AND RCOS > .25 * sensitiv) THEN
    DO
        CALL LockinRead
        CALL Sensitivity
        PRINT #1, "Y4" ' Checks overload of the lock-in.
        INPUT #1, OVERLOAD
        IF OVERLOAD = 0 THEN
            PRINT #2, RCOS, RSIN, Freq, sensitiv, X5
            print2$ = "yes"
        ELSE
            PRINT #1, "K27"
        END IF
        SLEEP (hold)
    IF INKEY$ = "i" OR INKEY$ = "I" THEN
        exitsub$ = "yes"
        x = low - 1
        print2$ = "yes"
        OVERLOAD = 0
        RCOS = 0#

```

```

END IF
  LOOP UNTIL OVERLOAD = 0
ELSE
  IF RCOS < .25 * sensitiv THEN PRINT #1, "K28"
  IF RCOS < .25 * sensitiv AND sensitiv$ = "100 nV " THEN
    PRINT #2, RCOS, RSIN, Freq, sensitiv, X5
    print2$ = "yes"
  END IF
  IF RCOS > .8 * sensitiv THEN PRINT #1, "K27"
  IF RCOS > .8 * sensitiv AND sensitiv$ = "500 mV" THEN
    PRINT #2, RCOS, RSIN, Freq, sensitiv, X5
    print2$ = "yes"
  END IF
END IF
LOOP UNTIL print2$ = "yes"
NEXT x
END IF
IF keyf7$ = CHR$(2) THEN keyf7$ = CHR$(1)
LOOP UNTIL keyf7$ = CHR$(1) OR exitsub$ = "yes"
END IF
IF keyf7$ = CHR$(2) THEN
  CLS 2
  DO
    keyf7$ = INKEY$
    LOCATE 3, 14
    PRINT "          SIGNAL VS FREQUENCY          "
    LOCATE 4, 14
    PRINT "          _____          "
    LOCATE 8, 25
    PRINT "F1  Give instructions to the chopper      "
    LOCATE 9, 25
    PRINT "                      "
    LOCATE 10, 25
    PRINT "F3  Return to the MAIN MENU              "
    IF keyf7$ = CHR$(3) THEN exitsub$ = "yes"
    IF keyf7$ = CHR$(1) THEN
      CLS 2
      LOCATE 3, 14
      PRINT "Limits on the chopping frequency are 500 to 3000 Hz."
      LOCATE 4, 14
      PRINT "_____ "
      LOCATE 8, 25
      INPUT "Input the lower limit in Hz"; low
    
```

```

LOCATE 9, 25
INPUT "Input the upper limit in Hz"; high
LOCATE 10, 25
INPUT "Input the frequency step in Hz"; delf
IF low < 499 THEN
    LOCATE 19, 19: PRINT "The lower limit must be greater than 500"
    CALL Arrow(8, 5, 12, 10, 13)
END IF
IF low > 3000! THEN
    LOCATE 20, 19: PRINT "The lower limit must be lower than 3,000"
    CALL Arrow(8, 5, 12, 11, 13)
END IF
IF high > 3000! THEN
    LOCATE 21, 19: PRINT "The upper limit must be lower than 3,000"
    CALL Arrow(8, 5, 12, 12, 13)
END IF
IF low >= high THEN
    LOCATE 22, 14
    PRINT "The upper limit must be larger than the lower limit"
    CALL Arrow(8, 5, 7, 13, 13)
END IF
IF low < high AND high <= 3000! THEN keyf7$ = CHR$(3)
END IF
LOOP UNTIL keyf7$ = CHR$(3)
CLS 2
LOCATE 5, 20
PRINT "THE FOLLOWING CONNECTIONS NEED TO BE MADE"
LOCATE 6, 20
PRINT "_____ "
LOCATE 9, 4
PRINT "1. Connect the X6 (D/A output, rear panel lock-in) to the CONTROL"
LOCATE 10, 4
PRINT "    VOLTAGE on the front panel of the optical chopper."
LOCATE 11, 4
PRINT "2. Connect the REF MODE (front panel of chopper) to"
LOCATE 12, 4
PRINT "    the REF INPUT (front panel lock-in)."
LOCATE 16, 20
PRINT "F1 Start the Signal vs Frequency test"
LOCATE 17, 20
PRINT "F2 Return to the MAIN MENU"
keyf7$ = ""
DO

```

```

keyf7$ = INKEY$
IF keyf7$ = CHR$(1) THEN
  CLS 2
  CALL WriteSettings
  WRITE #2, "Signal vs Frequency"
  WRITE #2, RCOS$, RSIN$, "freq", "Sensitivity", "X6 voltage"
  IF ENBW$ = "10 Hz" THEN hold = 15      ' Sets the time for the
  IF ENBW$ = "1 Hz" THEN hold = 30      ' noise measurement to stabilize.
  PRINT #1, "G4"                        ' Sets the sensitivity to 100 nV.
  CALL Sensitivity
  FOR x = high TO low STEP -delf
  print2$ = "no"                        ' Flag to state that data has
                                         ' not been written to the data file.

  DO
  X6 = x * .0025
  PRINT #1, USING "X6,##.#####"; X6    ' sets the voltage of X6 (D/A output).
  PRINT #1, "X6"
  INPUT #1, X6
  CALL LockinRead
  CALL Sensitivity
  LOCATE 3, 30
  PRINT "SIGNAL VS FREQUENCY"
  LOCATE 4, 30
  PRINT "_____ "
  LOCATE 7, 26
  PRINT USING "Current Frequency##### Hz"; Freq
  LOCATE 8, 26
  PRINT USING "Current voltage output ##.#### volts"; X6
  LOCATE 11, 30
  PRINT USING "Upper Limit##### Hz"; high
  LOCATE 12, 30
  PRINT USING "Lower Limit##### Hz"; low
  LOCATE 13, 30
  PRINT USING "Step Size###.# Hz"; delf
  LOCATE 16, 10
  PRINT "The program proceeds from upper limit to the lower limit"
  LOCATE 18, 10
  PRINT "Press 'i' to interrupt the subroutine and return to the MAIN MENU"
  SLEEP (hold)
  CALL LockinRead
  CALL Sensitivity
  IF INKEY$ = "i" OR INKEY$ = "I" THEN
    exitsub$ = "yes"

```

```

x = low - 1
print2$ = "yes"
OVERLOAD = 0
RCOS = 0#
END IF
IF (RCOS < .8 * sensitiv AND RCOS > .25 * sensitiv) THEN
DO
CALL LockinRead
CALL Sensitivity
PRINT #1, "Y4" ' Checks overload of the lock-in.
INPUT #1, OVERLOAD
IF OVERLOAD = 0 THEN
PRINT #2, RCOS, RSIN, Freq, sensitiv, X6
print2$ = "yes"
ELSE
PRINT #1, "K27"
END IF
SLEEP (hold)
IF INKEY$ = "i" OR INKEY$ = "I" THEN
exitsub$ = "yes"
x = low - 1
print2$ = "yes"
OVERLOAD = 0
RCOS = 0#
END IF
LOOP UNTIL OVERLOAD = 0
ELSE
IF RCOS < .25 * sensitiv THEN PRINT #1, "K28"
IF RCOS < .25 * sensitiv AND sensitiv$ = "100 nV " THEN
PRINT #2, RCOS, RSIN, Freq, sensitiv, X6
print2$ = "yes"
END IF
IF RCOS > .8 * sensitiv THEN PRINT #1, "K27"
IF RCOS > .8 * sensitiv AND sensitiv$ = "500 mV" THEN
PRINT #2, RCOS, RSIN, Freq, sensitiv, X6
print2$ = "yes"
END IF
END IF
LOOP UNTIL print2$ = "yes"
NEXT x
END IF
IF keyf7$ = CHR$(2) THEN keyf7$ = CHR$(1)
LOOP UNTIL keyf7$ = CHR$(1) OR exitsub$ = "yes"

```

```

END IF
LOOP UNTIL keyf7$ = CHR$(1) OR exitsub$ = "yes"
END IF
PRINT #1, "R0" ' Sets the trigger to a SQUARE WAVE.
chopvolt = 0# ' Sets the chopping frequency to zero.
PRINT #1, USING "X6,##.#####"; chopvolt ' Sets the voltage of X6 (D/A output).
CALL DisplayWindow
END SUB

```

DEFSNG O **SUB GraphBox**

```

VIEW (27, 30)-(282, 150)
WINDOW (0, 0)-(255, 130)
i = 5
j = 2
LOCATE i, j
PRINT "V"
LOCATE i + 1, j
PRINT "O"
LOCATE i + 2, j
PRINT "L"
LOCATE i + 3, j
PRINT "T"
LOCATE i + 4, j
PRINT "S"
LOCATE 12, 13
PRINT "250 Data Points"
LOCATE 2, 2
PRINT sensitiv$
LINE (5, 5)-(255, 5)
LINE (255, 5)-(255, 130)
LINE (255, 130)-(5, 130)
LINE (5, 130)-(5, 5)
LINE (5, 5)-(5, 0)
LINE (55, 5)-(55, 0)
LINE (105, 5)-(105, 0)
LINE (155, 5)-(155, 0)
LINE (205, 5)-(205, 0)
LINE (255, 5)-(255, 0)
LINE (5, 5)-(0, 5)
LINE (5, 30)-(0, 30)

```

```

LINE (5, 55)-(0, 55)
LINE (5, 80)-(0, 80)
LINE (5, 105)-(0, 105)
LINE (5, 130)-(0, 130)
END SUB

```

SUB GraphData

```

'VIEW (27, 30)-(282, 150)
'WINDOW (0, 0)-(255, 130)
FOR i = 7 TO 240
  PlotData(i - 1) = PlotData(i)
NEXT i
IF INT(PlotData(239) * 125 / sensitiv + 5) > 5 THEN
  IF INT(PlotData(239) * 125 / sensitiv + 5) < 130 THEN
    PRESET (240, PlotData(239) * 125 / sensitiv + 5)
  END IF
END IF
PlotData(240) = RCOS
PSET (240, PlotData(240) * 125 / sensitiv + 5)
FOR i = 239 TO 7 STEP -1
  IF INT(PlotData(i - 1) * 125 / sensitiv + 5) > 5 THEN
    IF INT(PlotData(i - 1) * 125 / sensitiv + 5) < 130 THEN
      PRESET (i, PlotData(i - 1) * 125 / sensitiv + 5)
    END IF
  END IF
  PSET (i, PlotData(i) * 125 / sensitiv + 5)
NEXT i
END SUB

```

SUB LockinRead

```

PRINT #1, "q1"
INPUT #1, RCOS
PRINT #1, "q2"
INPUT #1, RSIN
Time = TIMER
PRINT #1, "f"
INPUT #1, Freq
END SUB

```


SUB Sensitivity

```

PRINT #1, "G"
INPUT #1, isensitiv
IF isensitiv = 1 THEN sensitiv$ = "10 nV ": sensitiv = .00000001#
IF isensitiv = 2 THEN sensitiv$ = "20 nV ": sensitiv = .00000002#
IF isensitiv = 3 THEN sensitiv$ = "50 nV ": sensitiv = .00000005#
IF isensitiv = 4 THEN sensitiv$ = "100 nV": sensitiv = .0000001#
IF isensitiv = 5 THEN sensitiv$ = "200 nV": sensitiv = .0000002#
IF isensitiv = 6 THEN sensitiv$ = "500 nV": sensitiv = .0000005#
IF isensitiv = 7 THEN sensitiv$ = "1 μV ": sensitiv = .000001#
IF isensitiv = 8 THEN sensitiv$ = "2 μV ": sensitiv = .000002#
IF isensitiv = 9 THEN sensitiv$ = "5 μV ": sensitiv = .000005#
IF isensitiv = 10 THEN sensitiv$ = "10 μV ": sensitiv = .00001#
IF isensitiv = 11 THEN sensitiv$ = "20 μV ": sensitiv = .00002#
IF isensitiv = 12 THEN sensitiv$ = "50 μV ": sensitiv = .00005#
IF isensitiv = 13 THEN sensitiv$ = "100 μV": sensitiv = .0001#
IF isensitiv = 14 THEN sensitiv$ = "200 μV": sensitiv = .0002#
IF isensitiv = 15 THEN sensitiv$ = "500 μV": sensitiv = .0005#
IF isensitiv = 16 THEN sensitiv$ = "1 mV ": sensitiv = .001#
IF isensitiv = 17 THEN sensitiv$ = "2 mV ": sensitiv = .002#
IF isensitiv = 18 THEN sensitiv$ = "5 mV ": sensitiv = .005#
IF isensitiv = 19 THEN sensitiv$ = "10 mV ": sensitiv = .01#
IF isensitiv = 20 THEN sensitiv$ = "20 mV ": sensitiv = .02#
IF isensitiv = 21 THEN sensitiv$ = "50 mV ": sensitiv = .05#
IF isensitiv = 22 THEN sensitiv$ = "100 mV": sensitiv = .1#
IF isensitiv = 23 THEN sensitiv$ = "200 mV": sensitiv = .2#
IF isensitiv = 24 THEN sensitiv$ = "500 mV": sensitiv = .5#
END SUB

```

SUB UpdateScreen

```

COLOR 15
LOCATE 18, 47                                ' Prints the value of channel #1
PRINT USING "+#.###^~^~"; RCOS              ' to the screen.
LOCATE 18, 64                                ' Prints the value of channel #2
PRINT USING "+#.###^~^~"; RSIN              ' to the screen.
COLOR 7
LOCATE 22, 58                                ' Prints the value of the frequency
PRINT USING "#### Hz "; Freq ' to the screen.

```

END SUB

SUB WriteData

```
IF datafile$ <> "" THEN
  PRINT #2, Time - time0, RCOS, RSIN, Freq, Time
END IF
END SUB
```

SUB WriteSettings

```
WRITE #2, "LOCK-IN SETTINGS"
WRITE #2, ""
WRITE #2, "Sensitivity", sensitiv$
WRITE #2, "Dynamic Reserves", DynRes$
WRITE #2, "Pre Time Constant", pretime$
WRITE #2, "Post Time Constant", posttime$
WRITE #2, "Band Pass Filter", bandpass$
WRITE #2, "Line Filter", linex1$
WRITE #2, "Line x2 Filter", linex2$
WRITE #2, "ENBW", ENBW$
WRITE #2, "Ref Phase", RefPhase
WRITE #2, ""
END SUB
```