A MICROCOMBUSTION METHOD TO MEASURE FOREST FUEL EMISSIONS

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ABSTRACT

Thermogravimetry (TG) was applied to forest fuel as a microcombustion technique to study emissions by evolved gas analysis (EGA). Emission rates for carbon monoxide (CO), carbon dioxide (CO_2), and total hydrocarbons (THC) for both combustion and pyrolysis processes were determined. Thermoparticulates (TP) were emitted in copious amounts; TP emission factors were 85 g/kg and 249 g/kg for the combustion and pyrolytic conditions, respectively, while factors for volatile organic carbon (VOC) compounds collected in plugs of polyurethane foam (PUFS) were the same (~ 66 g/kg) for both conditions. A carbon mass balance on two series of the tests accounted for 95 percent of the fuel carbon content.

INTRODUCTION

MAN OFTEN BURNS FOREST FUELS FOR BENEFICIAL PURPOSES. Prescribed fires help to control wildfires and to solve land management problems. Many people use wood stoves and fireplaces. On a larger scale, wood-chip furnaces provide heat energy for some public buildings and the wood products industry.

Such fires release hundreds of combustion products into the atmosphere during flaming and smoldering combustion. Most of these products are carbonaceous substances that may adversely affect human

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health and welfare. There has been little success in describing relationships between the amounts of substances released during thermal degradation and factors that influence forest fuel burning.

Thermal analysis (TA) is a promising approach to this problem because it allows control over fuel temperature and atmospheric environment. It is especially suitable for the study of slow combustion such as the smoldering of forest fuels.

In TA, while a sample of material is heated or cooled according to a prescribed program, a property of the material is recorded as a function of temperature [1].

In a review of the literature on thermal analysis of wood, Beall and Eickner [2] cite 74 related studies from 1946 to 1968. Philpot [3] used TA and gas chromatography to examine the effects of plant ash on the pyrolysis of cottonwood and the resulting emissions. Duvvuri, et al. [4] derived kinetic parameters for predicting weight loss rates of wood and foliage samples undergoing pyrolysis. Shafizadeh, et al. [5] used thermogravimetry (TG) and evolved gas analysis (EGA) to investigate the role of extractives in the propagation of forest fires. Clements and McMahon [6] employed TG and EGA to study the emissions of nitrogen oxides from burning forest fuels. Susott [7] employed TG along with a "coulometric" detector for monitoring the total combustibles from a variety of forest fuels. Recently, Susott [8] used a differential scanning calorimeter to determine the heat requirements for pyrolysis of forest fuels, but he did not characterize the chemical species of emissions.

The purpose of our study was to evaluate the effectiveness of TA techniques to efficiently generate the products of combustion and pyrolysis, and to determine how these products were affected by oxygen level and heating rate.

METHODS

Experimental work for this study included fuel sampling, thermogravimetry, evolved gas analysis, and measurements of thermoparticulate and volatile organic compounds.

Sample Preparation

For the purpose of this experiment, any lignocellulosic material would have been suitable as the fuel. We used only one fuel—slash pine needles (*Pinus elliottii* Engelm. var *elliottii*)—in order to reduce variability in the experimental data. We selected slash pine needles because they are part of the understory fuel for many prescription fires in the Southeastern United States. A sample was taken from a supply of pine needles hand-collected from the upper litter layer under a plantation of slash pines at the Holt-Walton Experimental Forest near

Cordele, Georgia. The needles were air-dried at room temperature, ground in a Wiley mill to pass through a 60-mesh sieve, mixed thoroughly, and placed in a stoppered bottle until used.

Thermogravimetry

TG involves the continuous monitoring of the weight and temperature of a sample as its temperature is changed in a controlled manner. This technique was applied to the pine needle samples with a Perkin-Elmer[3] TGS-2 Thermogravimetric System (Figures 1 and 2). The system includes a high-precision electronic microbalance, a programmable controller, a temperature programmable furnace, and a two-pen strip-chart recorder. The microbalance is bottom loaded and electronically null. A platinum sample pan and counterbalance pan are suspended from opposite ends of the balance beam by inconel hang-down wires and platinum stirrups. The furnace is a hollow, thin-walled, cylindrical, alumina mandril wound with a platinum filament that

Figure 1. Photo of Perkin Elmer TGS-2 system.

[3]The use of trade names is for clarity of methods and does not constitute an endorsement by USDA.

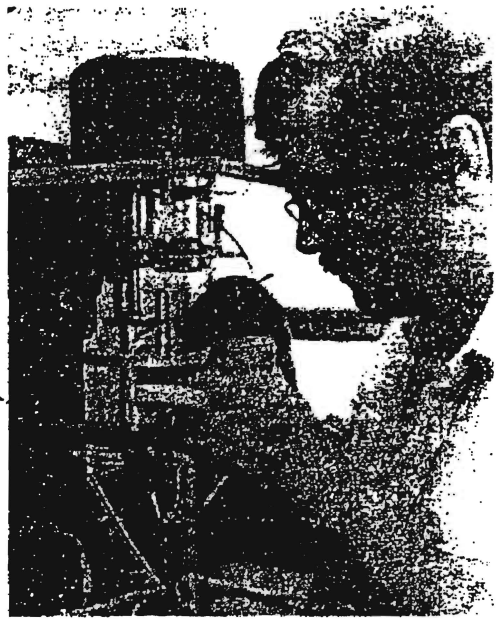

Figure 2. TGS-2 balance pan being loaded with fuel sample for an experimental run.

alternately serves as a resistance thermometer and a resistance heater. The system's two-pen recorder plots the sample's percent of initial weight and its temperature as a function of time on a strip-chart.

A glass combustion tube, surrounding the thermogravimetric furnace, allows both the furnace and the balance to be purged with a gas during normal operation. The purge gas serves three purposes: It prevents combustion products from entering the balance; it provides a desired environment in the furnace (inert or oxidative); and it carries away the products of the thermal treatment. Purge rates can range from about 10 cc/min (just enough to protect the balance's electronic parts) to above 200 cc/min (until weighing accuracy is affected). The purge gas and products leave the system through the combustion tube's exit port, where they can be sampled and analyzed.

Evolved Gas Analysis

Non-dispersive infrared monitors measured emissions of carbon monoxide (CO) and carbon dioxide (CO_2) continuously, while a flame ionization detector (FID) measured emissions of non-oxygenated total

hydrocarbons (THC). Measured emissions of CO, CO_2, and THC were plotted in parts per million against time on strip-chart recorders.

Thermoparticulate and Volatile Organic Compound Measurements

In TA, thermoparticulates are defined as the solid and liquid particles emitted when substances undergo thermal decomposition. They are equivalent to aerosols or particulate matter reported in air-quality studies. A thermoparticulate is not a pure compound but a mixture of substances whose chemical composition varies widely depending on the nature of the fuel and the burning process. Thermoparticulates were collected and weighed for sets of replicate TA treatment runs. During preliminary runs, thermoparticulate matter was found depositing on the inside walls of the glass combustion tube that encloses the TG furnace. To collect this thermoparticulate matter by collision or condensation, a heavy-gauge aluminum foil liner was fabricated to fit snugly against the inside walls of the combustion tube. Most of the remaining thermoparticulates, along with other products, were carried out the exit port of the combustion tube by the purge gas flow. All the exhausted products were passed through a trap consisting of a preweighed glass fiber filter. In addition, a train of several preweighed polyurethane foam (PUF) plugs was placed in line to trap volatile organic compounds (VOC). Yamasaki, et al. [9], Lewis and MacLeod [10], and Lewis and Jackson [11] reported that PUF is very efficient for trapping condensible organic chemicals in air while allowing high flow rates.

Gas Flow Scheme

The gas flow scheme (Figure 3) required some attention to assure continuous measurements of the gaseous products while the released thermoparticulates were being filtered. The purge gas flow splits into three paths. The first path feeds purge gas at about 90 cc/min directly into the combustion tube. A second path feeds about 10 cc/min purge gas through the balance chamber, then into the combustion tube. This second flow path protects the electronic balance parts from exposure to pyrolytic and combustion products. The third purge gas path fills a polyethylene gas bag as needed to serve as a plenum make-up supply for the analyzers. Exhaust gases from the combustion tube pass through the thermoparticulate trap and then are mixed and diluted with the make-up purge gases from the plenum bag. This mixture of gases is then pumped at 2.2 liters/min to a manifold that supplies equal amounts to each of the gas analyzers through three exit ports. At each exit port of the manifold, a portable pump receives its share of sample gas through a "T" dump that supplies 500 cc/min to a gas analyzer. The integrity of the flow system was tested by applying the

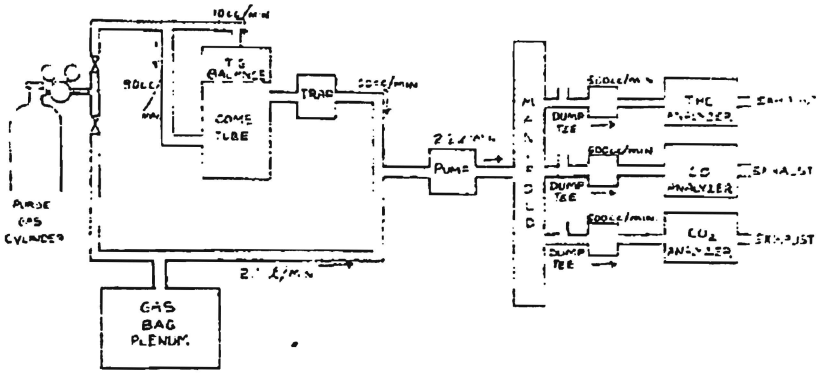

Figure 3. Schematic flow diagram of gases including combustion products from thermogravimetric furnace to gas analyzers.

analyzers to the exhaust gases during a blank run with primary standards of CO and CO_2 injected into the exit port of the combustion tube.

Test Run Procedure

The same general procedure was employed for each of 32 experimental runs. Typically, a 10-mg pine needle sample was heated to 100°C, held at that temperature until dry (2 min), heated to 900°C, and then cooled back to 100°C. Three heating rates were applied: 200°C/min[4] was used in 20 of the runs; 100°C/min for 6 runs; and 50°C/min for 6 runs. Half of the experimental runs used oxygen as a purge gas and half used nitrogen. Prior to the runs, the time lag for each analyzer to respond to a gas sample injected at the exit port of the combustion tube was measured. Corrections for these time lags were applied to the data from the gas analyzers in an attempt to correlate emission rates with sample weight losses and temperature readings from the TG system. The applied time lag corrections do not account for possible errors caused by diffusion of the gaseous products within the combustion tube before they arrive at the exit port. This diffusion may be the main cause for a lack of close correlation of emission rates with temperature and weight loss data.

RESULTS AND DISCUSSION

Emissions of the three gaseous products in parts per million are plotted against time for a typical run with nitrogen as the purge gas in

[4]Maximum for the TGS-2 System.

Figure 4a. The maximums for each gas are in the same order of magnitude (50 to 150 ppm). The CO_2 was released first, followed by CO, and then THC. Emission factors for the gases based on integrated values under their curves will be discussed later.

Figure 5a shows the weight-ratio curve and temperature curve for the same experimental run. The weight-ratio curve drops rapidly at about 300 °C with nearly a constant slope until about 380 °C. During that time, about 65 percent of the fuel's weight is removed by decomposition. After about 550 °C, the weight-ratio curve again has nearly a constant but much smaller slope (1/10 of the first slope). This two-step decomposition appears to be much the same as that suggested by Akita [12] for the wood cellulose and lignin components. About 23 percent of the initial fuel weight remained as charred residue after the temperature reached 900 °C and dropped back to 100 °C at the end of the run.

In Figure 4b, the emissions for CO_2, CO, and THC are plotted against time for a typical TG run with oxygen as the purge gas. In this run, the CO is released first, with both the CO_2 and THC following closely behind. The peak values for CO_2 and CO are about an order of magnitude greater than for the run with nitrogen as purge, while the THC peak values are about the same in both cases.

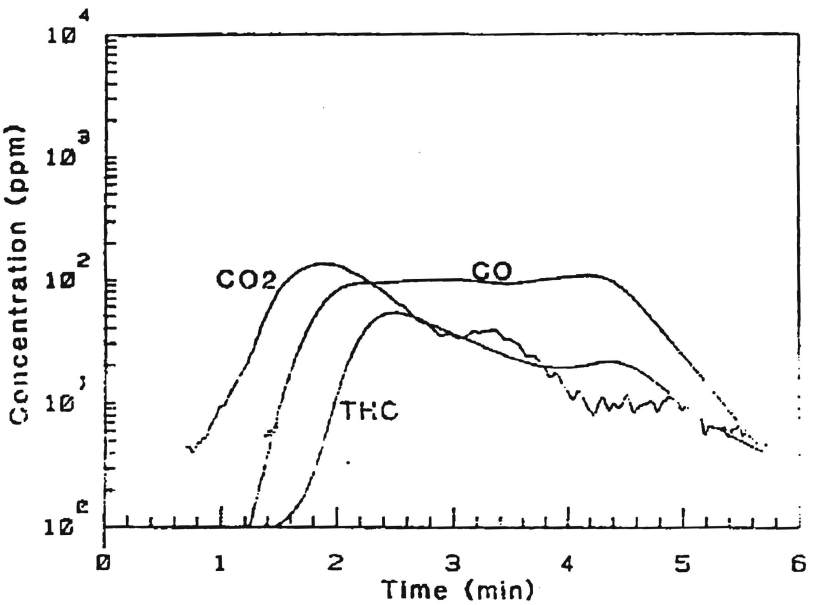

Figure 4a. Evolved gas analysis curves for CO_2, CO, and THC produced by pyrolyzing slash pine needles in nitrogen.

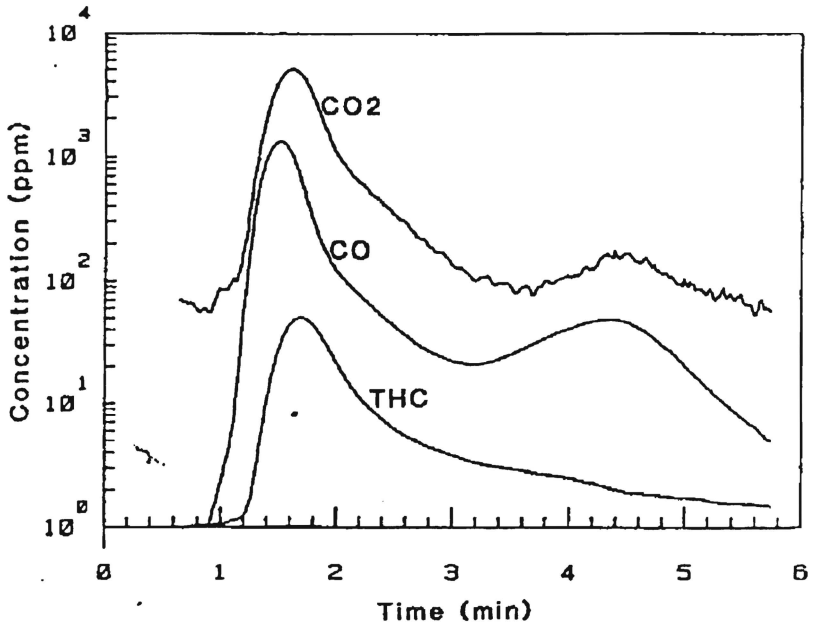

Figure 4b. Evolved gas analysis curves for CO_2, CO, and THC produced by burning slash pine needles in oxygen.

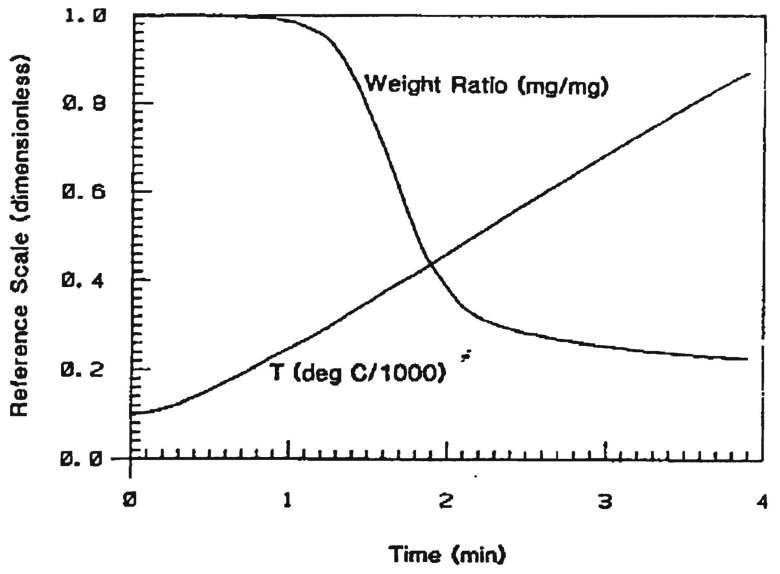

Figure 5a. Thermogravimetric plot for slash pine needles pyrolyzed in nitrogen.

For accurate comparisons of the CO and CO_2 curves, it was convenient to normalize the data for the amounts of fuel consumed: Data for each curve were multiplied by 10 mg and divided by the weight of fuel consumed. At about the time the CO curve peaks and just before the CO_2 curve peaks, there is a corresponding surge in the temperature curve when CO_2 is the purge gas (Figure 5b). This temperature surge indicates that a very rapid and strong exothermic reaction (combustion) is occurring in the furnace which prevents the furnace's cooling fan from controlling the temperature rise rate. The weight-ratio curve in Figure 5b drops very rapidly in one step, reaching a final weight at about the peak of the temperature surge. In this case, the final weight (ash) was only about 2 percent of the initial fuel weight, as compared to about 23 percent residue in the run with nitrogen.

The emission factor[3] for each gas product was determined from the mole weight of the gas, the integrated value under the parts per million curve, the total flow rate, and weight of fuel burned. The weights and emission factors for CO, CO_2, and THC are given in Table 1 for each run, identified according to heating rate and purge gas used. Test

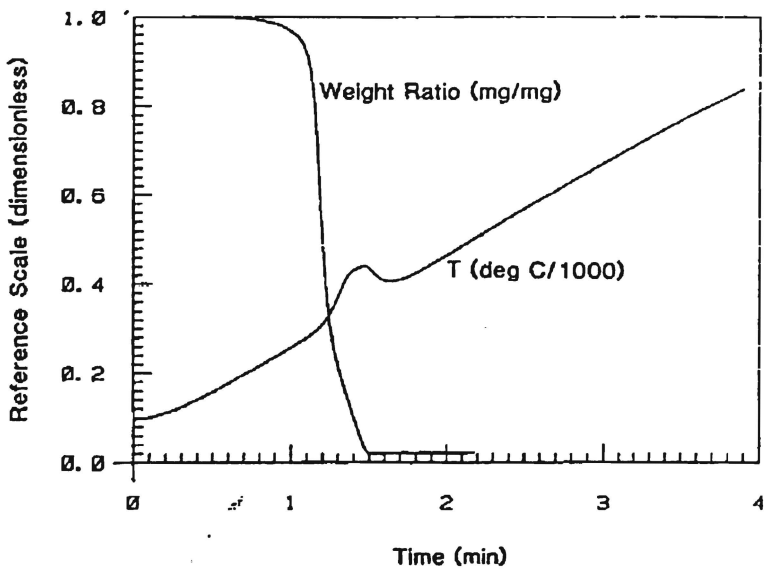

Figure 5b. Thermogravimetric plot for slash pine needles burned in oxygen.

[3]An emission factor for a given effluent is the mass of the effluent produced per mass of fuel consumed (dry weight basis), usually expressed as g/kg.

Table 1. Evolved gases from thermogravimetric analyses on slash pine needles.

Test Number	Fuel Burned mg	Cumulative weight THC µg	CO µg	CO_2 µg	Emission factors[1] THC g/kg	CO g/kg	CO_2 g/kg	Purge gas	Ramp °C/min
10	4.85	23.41	792.8	5512.	4.83	163.5	1136	O_2	200
11	5.06	22.09	750.3	5683.	4.37	148.3	1123.	O_2	200
12	4.71	18.97	716.3	5548.	4.03	152.1	1176.	O_2	200
13	4.93	23.42	775.0	5570.	4.75	157.2	1130.	O_2	200
14	4.56	19.53	743.7	4996.	4.28	163.1	1129.	O_2	200
15	4.55	26.52	789.0	5138.	5.83	173.4	1129.	O_2	200
16	4.74	5.03	225.2	8194.	1.06	47.5	1729.	O_2	200
17	4.71	20.59	745.2	5186.	4.37	158.2	1102.	O_2	200
18	4.44	29.75	–	5172.	6.70	–	1165.	O_2	200
19	4.63	24.71	755.9	5067.	5.34	163.3	1094.	O_2	200
40	9.30	134.5	715.3	670.3	14.46	76.91	72.08	N_2	200
41	9.24	135.5	717.1	633.1	14.66	77.61	68.52	N_2	200
42	8.74	126.0	669.4	671.9	14.42	76.59	76.88	N_2	200
43	8.02	114.6	628.9	565.7	14.27	78.32	70.32	N_2	200
44	9.66	143.1	780.3	669.7	14.81	80.78	69.33	N_2	200
50	9.22	121.6	789.5	1055.	13.19	85.63	114.4	N_2	200
51	9.17	135.6	770.7	657.9	14.79	84.05	71.74	N_2	200
52	9.55	144.2	783.4	1189.	15.10	82.03	124.5	N_2	200
53	9.62	149.9	792.1	767.6	15.58	82.34	79.79	N_2	200
54	9.06	129.3	731.8	690.9	14.27	80.77	76.26	N_2	200
60	10.85	148.4	1049.	1163.	13.68	96.68	107.2	N_2	100
61	9.07	133.3	863.7	795.9	14.70	95.23	87.75	N_2	100
62	9.48	139.1	917.6	824.7	14.67	96.79	86.99	N_2	100
63	9.84	142.4	877.7	854.2	14.47	89.20	86.81	N_2	100
64	9.86	136.7	828.4	659.5	13.86	84.02	66.89	N_2	100
65	9.30	122.3	939.0	605.2	13.15	101.0	65.08	N_2	100
70	4.80	18.65	845.3	3977.	3.89	176.1	828.5	O_2	50
71	4.44	19.52	712.3	5241.	4.40	160.4	1180.	O_2	50
72	4.64	4.97	221.9	7842.	1.07	47.82	1690.	O_2	50
73	4.28	16.36	773.0	4781.	3.82	180.6	1117.	O_2	50
74	4.60	28.00	890.5	5330.	6.09	193.6	1159.	O_2	50
75	4.76	5.50	278.4	8719.	1.11	58.49	1832	O_2	50

[1]All calculations based on initial dry weight of fuel.

numbers 16, 72, and 75 apparently burned quite efficiently, as indicated by the high CO_2 emission factors. Missing data for CO in test number 18 was caused by failure of the sampling pump for the CO analyzer.

Duncan's multiple-range test was applied to the emission factor data

at the 5 percent level. As expected, significant effects on the outputs of CO, CO_2, and THC are explained by the nature of purge gas, whereas no effects were found related to heating rates. However, the heating rates used in this experiment represent a very narrow range of possible rates available in open burning situations.

Thermoparticulate (TP) and Volatile Organic Compounds (VOC)

Amounts of TP and VOC were measured by gravimetric analysis. Because 5- to 10-mg samples of fuel produce very small amounts of TP and VOC, the collection was accumulated for 10 runs in each of two sets, all heated at 200 °C/min. Oxygen was the purge gas for one set of runs, nitrogen for the other set. Table 2 gives the emission factors of evolved gases, thermoparticulates (EF_{TP}), and volatile organic carbon (EF_{VOC}) for the two sets of runs. As expected, the set with nitrogen as purge gas had emission factors for THC and TP about three times those of the set with oxygen as purge gas.

The relatively slow thermal degradation which occurs in the TG apparatus approximates the slow smoldering combustion of forest fuels. The EF_{TP} found in the oxygen series is roughly equivalent to the total suspended particulate emission factors reported for smoldering combustion of forest fuels during open burning and laboratory experiments [13,14]. The very high EF_{TP} for the nitrogen series illustrates the importance of maintaining a good oxygen supply during the open burning of forest fuels in order to minimize smoke production. While EF_{TP} increased three-fold in the nitrogen series, the EF_{VOC} as determined with the PUF sampler did not change significantly. The term VOC is used by air quality personnel to describe the numerous volatile carbonaceous substances that may have an impact on air quality and are not accounted for in the national ambient air quality

Table 2. Average emission factors for evolved gases, volatile organic compounds and thermoparticulates from thermogravimetric runs on slash pine needles.

Purge Gas	Emission Factors				
	THC	CO	CO_2 g/kg	VOC[1]	TP[2]
Oxygen (16 runs)	4.1 ± 1.7	143 ± 49	1233 ± 270	63.3[3]	84.9[3]
Nitrogen (16 runs)	14.4 ± 0.6	86 ± 8	83 ± 18	67.9[4]	248.5[4]

[1]As determined with PUF sampler.
[2]As determined by combining weights of aluminum foil and filter collectors
[3]Series 10 (ten runs).
[4]Series 40 and 50 (ten runs)

standards for particulate matter and hydrocarbon. While quality assurance and quality control procedures exist for measuring particulate matter and hydrocarbons, few specific guidelines are available for measurements of VOC [15].

The VOC and TP samples obtained in this study were only analyzed gravimetrically. Chromatographic and spectrophotometric techniques could be employed to further characterize the emissions as a function of the experimental conditions. Applications of the methods used in this study combined with such techniques include, for example, determining conditions that minimize or maximize production of hazardous pollutants such as the polynuclear aromatic hydrocarbons. Another application could involve rigorously quantifying fuel nutrient losses and distribution under controlled heating rates. The method could also be applied to measure the release of toxic compounds released from controlled heating of wood treated with pesticides or wood preservatives.

Carbon Balance

Ward, et al. [16] and Nelson [17] used a carbon mass balance method in relating amounts of fuel burned to the measured emissions of carbon-containing combustion products. In this method, the fuel's carbon content is balanced with that of its combustion products. The mass of carbon for substance i can be expressed as

$$m_i = F_i \cdot M_i \tag{1}$$

where

F_i = the fraction of carbon to total mass of substance,
M_i = total mass of substance.

The carbon mass balance can be written as

$$m_{fuel} = \sum_{i=1}^{n} m_i \tag{2}$$

or

$$F_{fuel} \cdot M_{fuel} = \sum_{i=1}^{n} F_i \cdot M_i \tag{3}$$

The emission factor for substance i may be written as

$$EF_i = 1000\left(\frac{M_i}{M_{fuel}}\right) \tag{4}$$

Rearranging Equation (3) and combining with Equation (4) gives an expression for percent deviations from theoretical values:

$$\%\ Dev = 100\left[\left(\frac{1}{F_{fuel}} \cdot \sum_{i=1}^{n} F_i \cdot \frac{EF_i}{1000}\right) - 1\right] \tag{5}$$

For our experimental runs the values of carbon fractions of the gaseous products are

$$F_{CO} = \frac{C}{CO} = \frac{12}{28} = 0.429 \tag{6}$$

$$F_{CO_2} = \frac{C}{CO_2} = \frac{12}{44} = 0.273 \tag{7}$$

$$F_{THC} = \frac{C}{CH_4} = \frac{12}{16} = 0.75\ (THC\ as\ Methane) \tag{8}$$

$$F_R = 0\ (mineral\ ash\ with\ oxygen\ purge) \tag{9}$$

$$F_R = 0.91\ (residue\ with\ nitrogen\ purge—estimated) \tag{10}$$

The residue from the oxygen runs was considered as mineral ash. Residue from nitrogen runs was considered to consist of mineral ash plus char (carbon). The combined dry weight of the fuel from runs 40–44 and 50–54 was 91.23 mg and produced 1.82 mg mineral ash (2 percent) and 20.95 mg total residue (22.9 percent).

For the pine needle fuel, the empirical formula $C_6H_9O_4$ used by Byram [18] in combustion calculations was taken to yield

$$F_{fuel} = \frac{6C}{C_6H_9O_4} = \frac{72}{145} = 0.497 \tag{11}$$

This value agrees well with the 49.9 ± .8 percent carbon found for samples of slash pine needles [19]. For the thermoparticulates and volatile organic carbon compounds, a mean value of $F_{Part} = 0.595$ was used. A separate tube furnace experimental set-up provided a convenient way to collect *TP* on glass filters for carbon analysis. The procedure involved filtering purge gases and exhausted products from heating a ½-gram sample of pine needles in the furnace heated slowly (20 °C/min) to 500 °C. Four samples—two with nitrogen and two with

air as purge gases—gave a mean of 59.5 percent carbon with a standard deviation of 1.7 percent.

Substituting the appropriate carbon fraction values along with the corresponding emission factors into the left side of Equation 5 yields the following percent deviations from the theoretical value:

Purge Gas	*Percent Deviation*
oxygen	−1.6
nitrogen	−6.0

Thus, the carbon content of the products measured account for 98.4 and 94.0 percent of the fuel carbon content in the two series.

CONCLUSIONS

TA is applicable to the study of combustion and pyrolysis products from the thermal decomposition of forest fuels. This microcombustion technique accounted for an average of 95 percent of the carbon products released as thermoparticulates, volatile organic carbon, total hydrocarbons (as detected by FID), carbon monoxide, and carbon dioxide. TA seems to best simulate slow oxidative smoldering combustion and oxygen-starved pyrolytic conditions of fuel decomposition.

Thermoparticulate emission factors increased three-fold, 85 g/kg to 249 g/kg, while volatile organic carbon products remained the same, ~ 66 g/kg, when purge gas was switched from oxygen to nitrogen. The limited range of heating rates used in the study did not have an effect on emission factors of the evolved gases.

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