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ABSTRACT

This paper presents the design and evaluation results for a metal-coated multilayer Tedlar (MMT) bag that was developed for the collection of source emissions. The applicability of the MMT bag was evaluated for a number of important greenhouse relevant gases: CO, CO_2, CH_4, N_2O, and total hydrocarbons (THCs). The bag was tested for durability and stability for a range of concentrations of the tested compounds using both laboratory-prepared samples and real source samples. The results show that all tested compounds were more stable when stored in the MMT bag than when stored in a regular Tedlar bag. These compounds can be stored at room temperature for at least 3 months without significant changes in concentration. When properly packed, the MMT bag is durable and may be shipped by air. The MMT bag is lower in cost, lighter in weight, and easier to clean, and it requires less devices during the subsequent laboratory analysis compared with a stainless steel canister, which is often used to collect air and source samples.

IMPLICATIONS

The development of a reliable, economical, and easy-to-use storage and shipping procedure for samples of greenhouse gases and other air pollutants is important for accurately measuring these emissions from the wide variety of source types dispersed throughout the world. The MMT bag presented in this paper has proved to be a reliable sampling tool for the collection of gaseous pollutants, such as CO, CO_2, CH_4, and N_2O, from various emission sources. The bag can be used and reused for collection, transport, and storage of emission samples, which can be subsequently analyzed in a laboratory.

INTRODUCTION

There is an increasing interest in measuring greenhouse gases and other air pollutants emitted from various sources.[1-5] The major species of concern often include CO, CO_2, CH_4, N_2O, and total hydrocarbons (THCs). A commonly used method to measure emissions of these compounds from various sources involves the collection of source samples into a container. The samples collected are often subsequently analyzed in a laboratory using gas chromatography (GC) techniques.[6-9] The containers used for sample collection must be durable, chemically inert, and easy to handle in the field.

Two types of gas sample collectors (containers) are often used for source sampling: stainless steel canisters and Tedlar bags. However, the use of Tedlar bags can result in significant sample losses, mainly due to molecular diffusion and Tedlar film permeation, during sample transport and storage.[7,10,11] The use of stainless steel canisters can be difficult for many laboratories for a number of reasons. First, the canister is expensive (>$500 each). Second, the canister requires a special device for cleaning and sample preparation, which leads to a costly initial investment in the device and a costly long-term consumption of high purity gases. Third, the inner surface of the canister is designed mainly for low-concentration (trace gas) applications, not specifically for source samples that typically contain high concentrations of air pollutants. After collecting high-concentration source samples, the canisters are often difficult to clean completely and can be contaminated or "poisoned" permanently under extreme conditions. Finally, as the authors have experienced, shipping the canisters across international borders can often be problematic because of their "bomb-like" appearance.

To overcome the shortcomings of regular Tedlar bags and stainless steel canisters for emission source sampling, we developed a metal-coated multilayer Tedlar (MMT) sampling bag. The MMT bag is substantially lighter in weight and lower in cost (<$30 each) compared with a stainless steel canister. The applicability and performance of the MMT were evaluated using both laboratory-prepared gas samples and real source samples. In this paper, we present the bag design and the evaluation results.

EXPERIMENTAL METHODS

Design of the MMT Bag

The MMT bag is custom-made by Cole-Parmer Instrument Co. The bag has three layers. The inner and middle layers are made of 2-mil Tedlar film, which is believed to be chemically inert and have little tendency to sorb organic compounds.[7,8,10] The outer layer is made of Mylar film, which is more durable, less permeable, but chemically less inert than the Tedlar film. The outer surface of the bag is coated with a thin layer of aluminum to enhance the durability and further reduce the permeability of the bag. The bag features two fittings. One is a stainless steel hose/valve fitting for the attachment of Teflon tubing to fill the bag with the sample or to flush the bag prior to use. The other is a Teflon septum fitting, which provides a convenient means of taking the sample out of the bag with a syringe for GC analysis or other purposes. The capacity of the bag is 5 L, which provides sufficient sample volume for GC analysis and other possible analyses.

Evaluation Methods

The stability of CO, CH_4, CO_2, N_2O, and THCs stored in MMT bags was evaluated. For comparison purposes, the stability of those compounds stored in regular Tedlar bags was also tested. Gas samples containing CO, CH_4, CO_2, N_2O, and THCs were either prepared from gas standards in the laboratory or collected from real source samples in the field. All the samples were then analyzed at designated time intervals using a Hewlett Packard 6890 GC system equipped with a flame ionization detector (FID) and an electron capture detector (ECD). Compound loss/gain was determined by comparing the concentration measured at each subsequent time (day *X*) to the concentration measured initially (at day 0). The samples were all stored at room temperature during the course of the stability test.

Sample Preparation

All tested MMT and regular Tedlar bags were first leak-checked by filling the bags with air, examining for leaks in water, and visually examining for leaks after a 24-hr period. The leak-free bags were then cleaned 3 times with house air and 3 times with high-purity nitrogen (zero grade). After cleaning, the sampling bags were carefully rolled up to prevent puncturing by the stainless steel fittings. When shipped via air or transported to local fields, the bags were packed in plastic containers with polyurethane foam. The containers were highly rigid and well sealed to protect the bags. These types of containers are readily available at most hardware stores. For samples to be transported by air, the bags were only filled to 60% of their capacity, to account for possible expansion due to cabin pressure changes. After sample collection, the sampling bags were replaced into the containers for transport to the laboratory for analysis.

The laboratory-prepared samples were made using the following gas standards: CO_2 (10,000 ppm in nitrogen), CH_4 (100 ppm in nitrogen), CO (1000 ppm in nitrogen), and N_2O (50 ppm in nitrogen). All the gas standards were purchased from Scott Specialty Gases, Inc. Samples with different concentrations were obtained by diluting the gas standards with zero-grade nitrogen.

A number of real source samples were obtained locally and overseas. Local grab samples included those from a wood combustion source (a simulated campfire burning firewood) and those from room air while a kerosene heater was being used in the room. The radiant kerosene space heater (Kero-Sun Radiant 8) was used in a room with a volume of 19 m^3, and the fuel used was Sunnyside kerosene (1-K grade). A fan was running inside the room during each test to ensure the pollutants were well mixed. All grab sample bags were filled using Teflon sampling tubing and an SKC personal pump.

Two charcoal-making kiln emission samples were collected in MMT bags from each of three Brazilian round brick kilns and two Brazilian rectangular metal and brick kilns. The bags were filled by placing a copper sampling tube (1/2-in. diam) directly above the chimney of each kiln and pumping 3 L of emissions through a Whatman quartz fiber filter and then into the bag. All the samples used for the stability tests are summarized in Table 1.

Sample Analyses

All the samples were analyzed using a Hewlett Packard 6890 GC system equipped with an FID and an ECD. For THC analysis, GC/FID was used. The column was a 2 ft × 1/4 in. stainless steel column packed with glass beads (Alltech). The oven temperature was 35 °C, the injector temperature was 35 °C, and the FID temperature was 200 °C. The flow rates of air and hydrogen were 400 and 30 mL/min, respectively. Zero-grade nitrogen was used as carrier gas at a flow rate of 10 mL/min. The injections were made using a gas-tight syringe. The injection volume was 1 mL for N_2O and 50 μL for THCs, CH_4, CO, and CO_2. The concentration of THCs was calculated on a CH_4 basis, that is, mg/m^3 as CH_4.

Table 1. Information about the samples collected for the stability study on Tedlar and MMT sampling bags.

Source Type	Tedlar Bag		MMT Bag	
	Sample ID	Number of Samples	Sample ID	Number of Samples
Ambient air[a]			Air-M1 and Air-M2	2
			Air-M3 and Air-M7	5
Standard gases	Standard-T1 and Standard-T2	2	Standard-M1 to Standard-M4	4
Emissions from kerosene heater	Kerosene-T1 to Kerosene-T4	4	Kerosene-M1 to Kerosene-M5	5
Emissions from wood burning			Wood-M1 to Wood-M5	5
Emissions from charcoal-making process[b]			Charcoal-M1 to Charcoal-M10	10

[a]Samples Air-M1 to Air-M3 were collected from ambient air in New Jersey, samples Air-M4 and Air-M5 were collected inside a laboratory in New Jersey, and samples Air-M6 and Air-M7 were collected from ambient air in Brazil; [b]Samples Charcoal-M1 and Charcoal-M2 were collected at hour 64.5 of a 70-hr charcoal-making process, samples Charcoal-M3 and Charcoal-M4 were collected at hour 20 of a 96-hr charcoal-making process, samples Charcoal-M5 and Charcoal-M6 were collected at hour 44 of a 74-hr charcoal-making process, samples Charcoal-M7 and Charcoal-M8 were collected at hour 48 of a 157-hr charcoal-making process, and samples Charcoal-M9 and Charcoal-M10 were collected at hour 51 of a 118-hr charcoal-making process.

A nickel catalyst methanizer and FID were used in the analyses of CH_4, CO, and CO_2. Under this GC configuration, CO and CO_2 were converted to CH_4 by the nickel catalyst methanizer and then detected by the FID. A 10 ft × 1/8 in. stainless steel column packed with 80–100 mesh Carbosphere (Waters Associates, Inc.) was used for the separation of CO, CH_4, and CO_2. Zero-grade nitrogen was used as the carrier gas, and the flow rate was 30 mL/min. The oven temperature was held at 35 °C for 9 min, ramped to 200 °C at the rate of 25 °C /min, and held at the final temperature for 5 min. The injector temperature was 35 °C, the methanizer temperature was 375 °C, and the FID detector temperature was 200 °C. The flow rates of air and hydrogen were 400 and 30 mL/min, respectively.

Nitrous oxide (N_2O) was analyzed by GC/ECD. An 8 ft × 1/8 in. stainless steel column packed with 80–100 mesh Hayesep Q (Waters Associates, Inc.) was used for the separation of N_2O from other pollutants. Zero-grade nitrogen was used as the carrier gas, and the flow rate was 20 mL/min. The make-up gas flow rate was 60 mL/min. The oven temperature was held at 50 °C for 3.5 min, ramped to 200 °C at the rate of 20 °C/min, and held at the final temperature for 9 min. The injector temperature was 35 °C, and the ECD temperature was 350 °C.

The instrument was calibrated periodically using a set of standards diluted from cylinders of gas standards prepared by Scott Specialty Gases. For each batch of analysis, at least one standard (usually at the middle of the calibration curve) was analyzed to correct for response factor changes. Two injections were made for each sample. If the change in response from the 2 injections was >10% for CO, CO_2, CH_4, and THCs and 20% for N_2O, additional injections were made until satisfactory reproducibility was obtained. The concentrations were calculated using the average response values from replicate injections.

RESULTS AND DISCUSSION

Analytical System Performance

All calibrations for CO, CO_2, CH_4, and THCs had R^2 values of >0.997. The difference in the response factors obtained from all calibration sets over the test course was <10%. The relative standard deviation (%RSD) from 14 repeat analyses of the middle-level standard was 6% for THCs and ~10% for CO, CO_2, and CH_4.

The N_2O calibration had an average R^2 value of 0.978. The accuracy from 7 analyses was 8% and the precision, determined from 14 repeat analyses of the middle level standard over the test course, was 22%. The %RSD of 7 repeat injections of the N_2O standard in the same day was 20%. The linearity and reproducibility of the instrument for N_2O analysis were higher in our previous study.[1] We suspect that the larger variation of the instrument for N_2O analysis in this stability study was probably due to an insufficient equilibration time for the ECD. All types of analyses (THCs, CO_2-CO-CH_4, and N_2O) were done by one GC/FID/ECD system in our laboratory. We had to switch GC configurations one after another for each different type of analysis. The ECD, used for the N_2O analysis, requires a longer equilibration time than does the FID. Unfortunately, the stability test requires the sample analyses to be performed at predesignated time intervals. This resulted in occasional N_2O analysis without a fully equilibrated ECD.

The analytical detection limit (ADL) was determined as 3 times the SD derived from seven "blank" air samples. These "blank" samples were spiked with trace amounts of some test compounds (CO and CH_4) in order to get near-baseline lowest detectable responses. The ADL values were ~0.7 ppm for CO, CH_4, CO_2, and THCs, and 250 ppb for N_2O.

Stability for Laboratory-Prepared Samples

Two samples were prepared in Tedlar bags using gas standards (Standard-T1 and Standard-T2). For sample 1, the initial concentration was 46 ppm for CO, 9 ppm for CH_4, and 3045 ppm for CO_2. As shown in Figure 1, significant losses (>75%) of CO, CH_4, and CO_2 were observed after storage in the Tedlar bag for 32 days. For sample 2, more than 80% of CO was lost after storage for 46 days, but only 18% of CO_2 was lost, and CH_4 was almost stable after storage of 46 days (see Figure 1). This was probably because the concentrations of CO_2 and CH_4 in sample 2 were much lower than those in sample 1. The CO initial concentration in sample 2 was 211 ppm, which was much higher than ambient level; therefore, a large degradation of CO was observed. The CH_4 and CO_2 initial concentrations were 1.8 and 482 ppm, respectively, which were close to the ambient levels of approximately 1.4–1.6 ppm of CH_4 and 470 ppm of CO_2. There was probably an equilibrium between the CH_4 and CO_2 in sample bag 2 and that present in the ambient air, and thus the net loss of CH_4 and CO_2 during storage in sample bag 2 was small (<20%).

Four samples were prepared in MMT bags using gas standards (Standard-M1 to Standard-M4). The initial concentrations of the four MMT bag samples were 5.6–55 ppm for CO, 5.0–33 ppm for CH_4, and 59.3–5922 ppm for CO_2. Results for the percent recovery of each compound over the 75-day storage time are presented in Table 2. As shown in Table 2 and Figure 2, all three compounds were relatively stable with >80% remaining after storage for 75 days. We found that CO_2 in sample 4 was stable for the first 25 days and started increasing after that. This sample had an initial CO_2 concentration of 59.3 ppm, much less than the ambient CO_2 level of ~470 ppm. We suspect that multiple needle punctures of the septum of the sampling bag caused increased infusion rates of ambient air during each injection, thereby increasing the concentration of CO_2.

Stability of Real Source Samples

Kerosene emission samples were collected in four Tedlar bags (samples Kerosene-T1 to Kerosene-T4) and five MMT bags (samples Kerosene-M1 to Kerosene-M5). The initial concentrations in the four Tedlar bag samples were 1.0–3.2 ppm for CO, 2.0–2.1 ppm for CH_4, and 2314.3–3776.9 ppm for CO_2, and the initial concentrations in the five MMT bag samples were 3.9–8.0 ppm for CO, 2.4–4.3 ppm for CH_4, and 2357.1–2581.5 ppm for CO_2. The Tedlar sample bags were tested for 82 days, and the MMT sample bags were tested for 97 days.

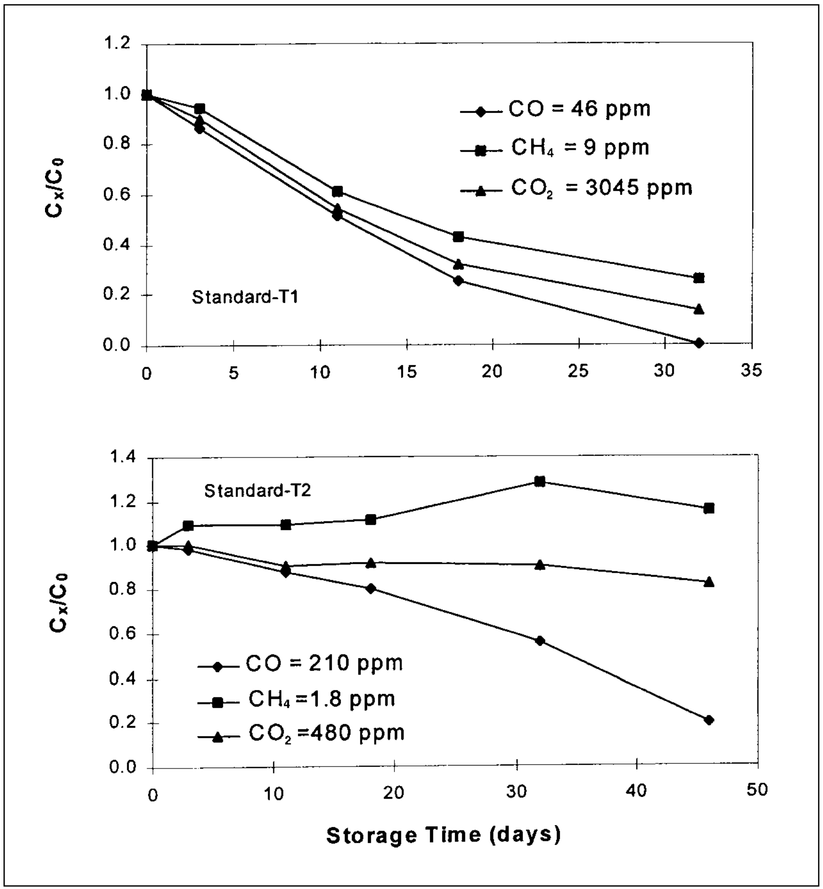

Figure 1. Stability of CO, CH_4, and CO_2 from standard gas samples stored in Tedlar bags. The *y* axis represents the ratio of the concentration at day *X* (C_x) over the concentration at day 0 (C_0). The concentrations shown in the figure are C_0 values.

A large decay of CO and CO_2 (but not CH_4) was also observed when samples were stored in the regular Tedlar bags (Figure 3). More than 50% of the CO and CO_2 was lost after storage for 82 days. However, as explained earlier, the CH_4 concentration in the four Tedlar sampling bags was similar to the ambient CH_4 level; therefore, the loss of the sample CH_4 was negligible. When stored in the MMT bags, all the compounds were stable for 96 days for the kerosene emission samples (see Figure 3), and the average recovery was 85% for CO, 89% for CH_4, and 76% for CO_2. Besides the significant sample losses found during storage in Tedlar bags, a large variation of the performance among the Tedlar sampling bags was also observed. The %RSD for the four Tedlar bags was 102% for CO, 12% for CH_4, and 58% for CO_2, but the %RSD for the performance of the five MMT sampling bags was only ~16% for CO, 6% for CH_4, and 18% for CO_2 (see Table 2), much smaller than those for Tedlar bags.

Two ambient air samples (Air-M1 to Air-M2) and 10 charcoal-making emission samples (Charcoal-M1 to Charcoal-M10) were collected in Brazil for this stability study, but the sample bag for charcoal

Table 2. Recovery of CO, CH_4, and CO_2 after storage in Tedlar and MMT sampling bags.[a]

		Tedlar Bags					MMT Bags			
Source Type	Sample ID	Storage	Recovery (%)			Sample ID	Storage	Recovery (%)		
		Time (days)	CO	CH_4	CO_2		Time (days)	CO	CH_4	CO_2
Standard gases	Standard-T1	32	0	26	13	Standard-M1	75	88	92	107
	Standard-T2	46	56	128	91	Standard-M2	75	96	96	105
						Standard-M3	75	64	71	69
						Standard-M4	75	90	89	152
	Average		28	77	52	**Average**		85	87	108
	%RSD		141	94	105	**%RSD**		17	13	32
Emissions from kerosene heater	Kerosene-T1	82	0	90	10	Kerosene-M1	96	100	94	87
	Kerosene-T2	82	88	101	44	Kerosene-M2	96	98	85	86
	Kerosene-T3	82	12	117	24	Kerosene-M3	96	80	83	77
	Kerosene-T4	82	100	93	50	Kerosene-M4	96	82	90	78
						Kerosene-M5	96	68	93	53
	Average		50	100	32	**Average**		86	89	76
	%RSD		102	12	58	**%RSD**		16	6	18

[a] Recovery was calculated as the concentration remaining in the sampling bag after the storage time over the initial concentration measured at day 0.

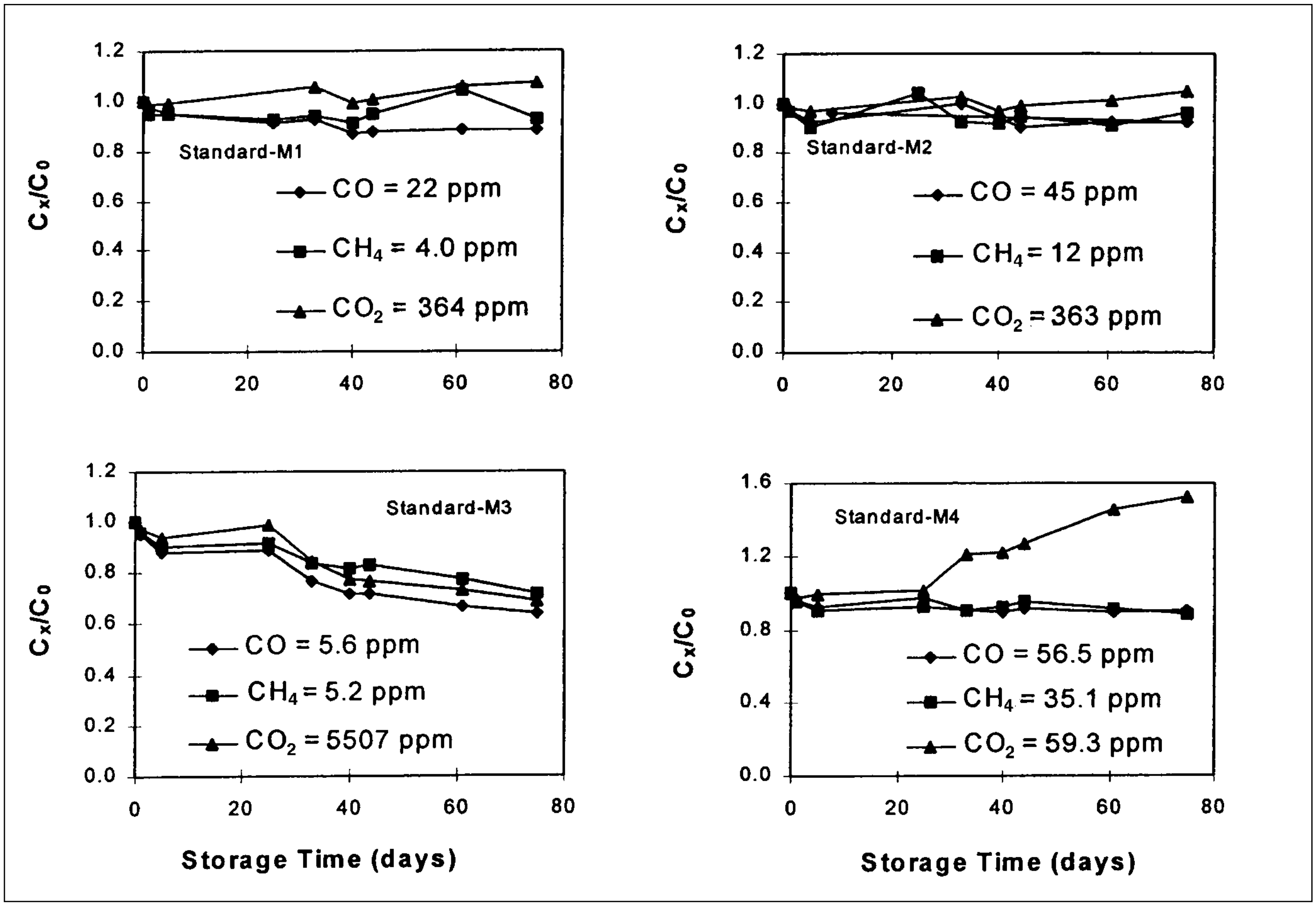

Figure 2. Stability of CO, CH_4, and CO_2 from standard gas samples stored in MMT bags. The *y* axis represents the ratio of the concentration at day *X* (C_x) over the concentration at day 0 (C_0). The concentrations shown in the figure are C_0 values.

sample 1 was spoiled during the sampling process. The initial concentrations of CO_2 and N_2O in the two ambient air samples were ~800 and ~400 ppb, respectively. CO, CH_4, and THCs were not detected in the ambient air samples. The initial concentrations of the nine charcoal-making kiln emission samples were 10,455–66,221 ppm for CO, 7882–26,701 ppm for CH_4, 775–150,542 ppm for CO_2, 1.22–5.04 ppm for N_2O, and 4775–36,361 ppm for THCs. The septum of the sample bag for charcoal sample 5 was dislodged, so only 10 samples (2 ambient air samples and 8 charcoal-making kiln emission samples) were tested for the stability study. The samples were tested up to 4 months. The recoveries of CO, CH_4, CO_2, THCs, and N_2O in the 10 samples after storage for 4 months are presented in Table 3. The average recovery was more than 80% for CO, CH_4, CO_2, and THCs, and the %RSD for the 10 MMT sampling bags was <16%. An example of the behavior of all the compounds stored in the MMT bag is shown in Figure 4. The average recovery of N_2O was 71%, and the %RSD for the 10 samples was <27%. As discussed in the section on Analytical System Performance, the large variations of the N_2O results were probably due to the variation of the instrument.

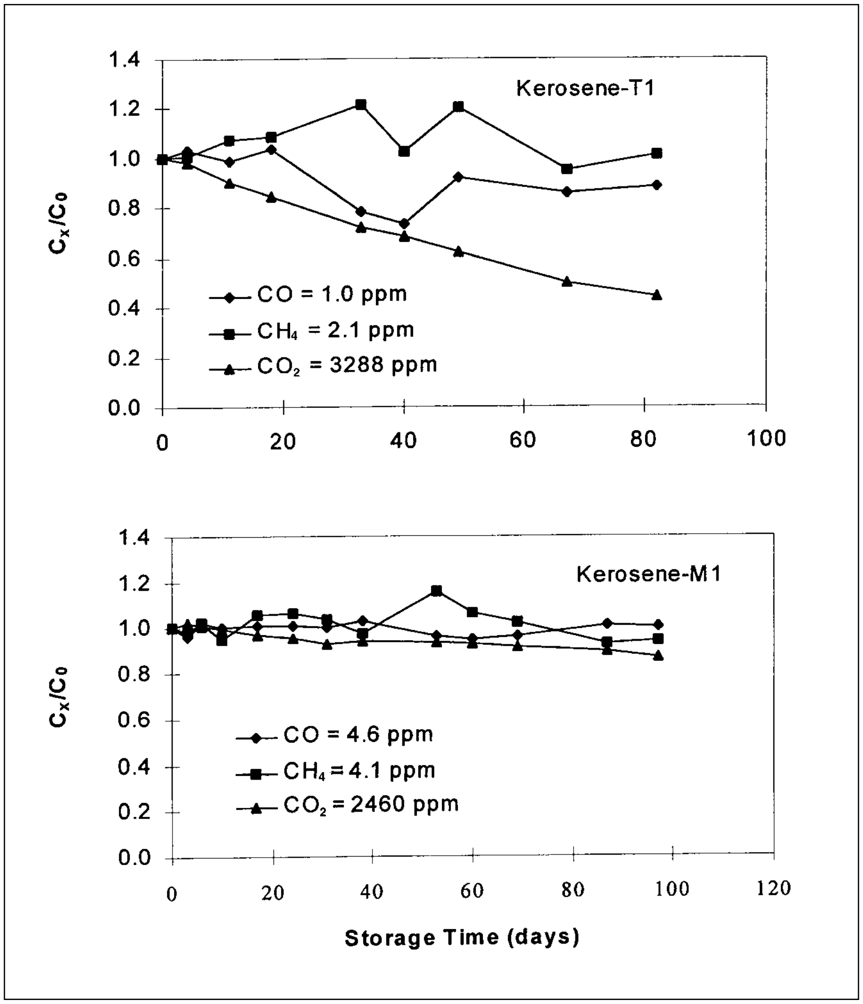

Figure 3. Stability of CO, CH_4, and CO_2 from kerosene heater emission samples stored in the Tedlar bag (top) and the MMT bag (bottom). The *y* axis represents the ratio of the concentration at day *X* (C_x) over the concentration at day 0 (C_0). The concentrations shown in the figure are C_0 values.

Table 3. Recovery of CO, CH_4, CO_2, THCs, and N_2O after storage in MMT bags for 125 days.[a]

Source Type	Sample ID	Storage Time (days)	Recovery (%)				
			THCs	CO	CH_4	CO_2	N_2O
Ambient air	Air-M1	125	ND[b]	ND[b]	ND[b]	94	103
Ambient air	Air-M2	125	ND[b]	ND[b]	ND[b]	105	111
Charcoal-making emissions	Charcoal-M2	125	92	113	107	93	73
Charcoal-making emissions	Charcoal-M3	125	80	96	88	76	72
Charcoal-making emissions	Charcoal-M4	125	84	123	107	93	73
Charcoal-making emissions	Charcoal-M6	125	70	92	107	93	53
Charcoal-making emissions	Charcoal-M7	125	69	89	81	69	86
Charcoal-making emissions	Charcoal-M8	125	91	119	107	93	60
Charcoal-making emissions	Charcoal-M9	125	63	92	79	59	107
Charcoal-making emissions	Charcoal-M10	125	86	119	107	93	45
	Average		80	105	98	83	71
	RSD		13	14	13	16	27

[a] Recovery was calculated as the concentration remaining in the sampling bag after storage of 125 days over the initial concentration measured at day 0; [b] Not detected. The detection limit was ~0.7 ppm for THCs, CO, and CH_4.

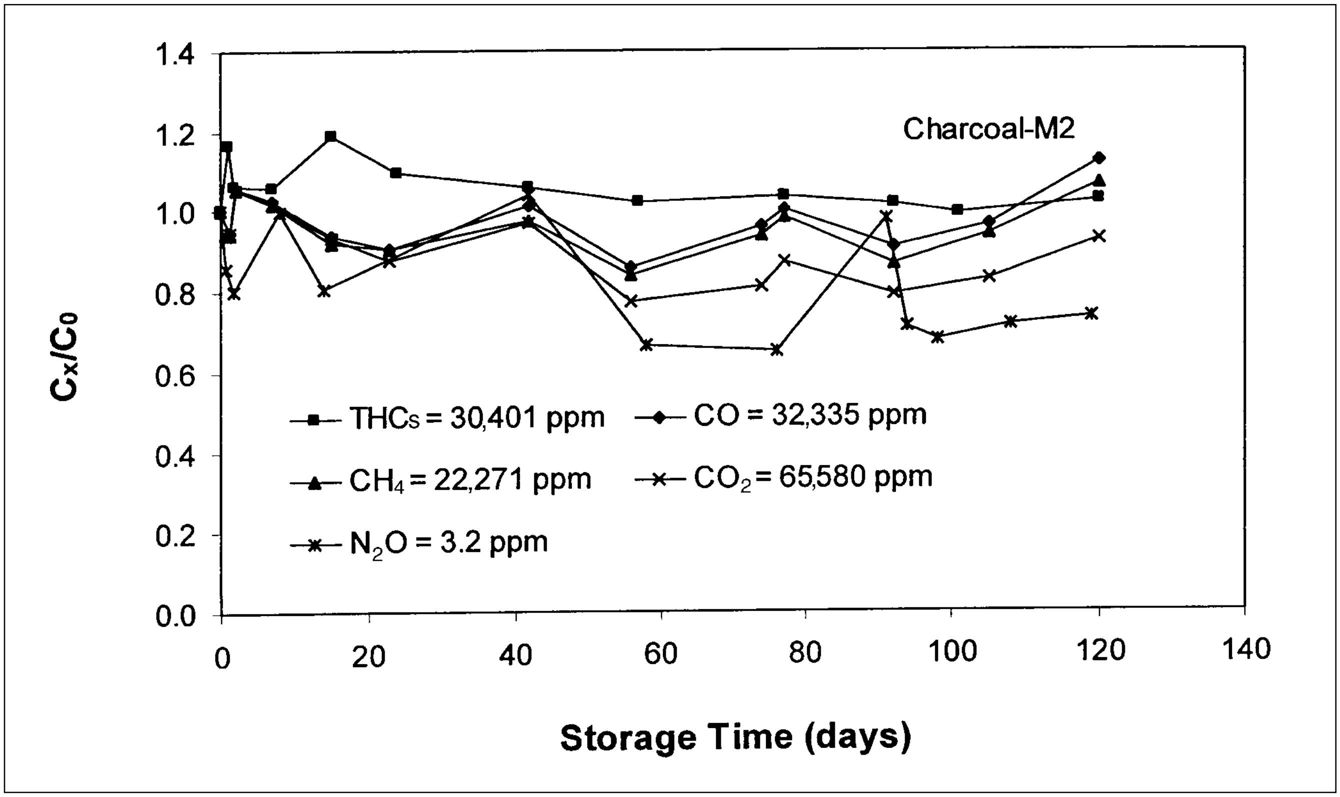

Figure 4. Stability of CO, CH_4, CO_2, N_2O, and THCs from a charcoal-making emission sample (Charcoal-M2) stored in a MMT bag for 125 days. The *y* axis represents the ratio of the concentration at day *X* (C_x) over the concentration at day 0 (C_0). The concentrations shown in the figure are C_0 values.

Potential N_2O Artifact

It has been found that N_2O can be produced in stainless steel canisters during sample storage.[12] Significant N_2O formation (artifact) in the canister was observed within a couple hours after combustion gas samples were collected.[13] Concerning the potential artifact formation of N_2O during the sample storage in MMT bags, we collected three samples from ambient outdoor air (Air-M3 to Air-M5), two samples from indoor room air (Air-M6 and Air-M7), and five samples from wood combustion emissions (Wood-M1 to Wood-M5) with MMT bags. The wood combustion source was the simulated campfire. The ambient indoor and outdoor samples were taken on the Busch Campus of Rutgers University in Piscataway, NJ. These samples were analyzed for N_2O concentrations as a function of storage time from 0.5 to 9 hr. The results, shown in Figure 5, indicate that no significant changes in N_2O concentrations occurred during the first few hours (a critical time period for the artifact) when both ambient air (indoor and outdoor) samples and wood-burning emission samples were stored in the MMT bags.

Durability of MMT Sampling Bags

The durability of the MMT bags was tested in transportation and handling processes. In the pilot test stage, three MMT bags were filled with a mixture of diluted standard gases and packed at Rutgers University as previously described. These bags were sent to the University of California at Berkeley via FedEx. It was found that the bags were maintained in their original conditions upon return from California 2 weeks later, and that the concentrations of the species contained in the bags were not decreased after the bags were returned.

After the pilot tests, the MMT bags were deployed from our New Jersey laboratory to collect samples of emissions from various charcoal-making kilns in Brazil and Kenya. All the cleaned empty bags and sample-filled bags were transported by air, and all analyses were performed in New Jersey. Samples collected in New Jersey were transported by automobile. Information on the bag breakage and other failures is summarized in Table 4. The results from our tests demonstrate that the MMT bag can be reused several times if no physical damage occurs to the bag. The successful use of the MMT bag in the field studies further proved that the MMT bag is a reliable means for the collection of emission source samples.

CONCLUSIONS

An MMT bag was developed for the collection of gaseous pollutants from emission sources. The MMT bag is more durable and less permeable than a regular Tedlar sampling bag and lighter than a stainless steel canister.

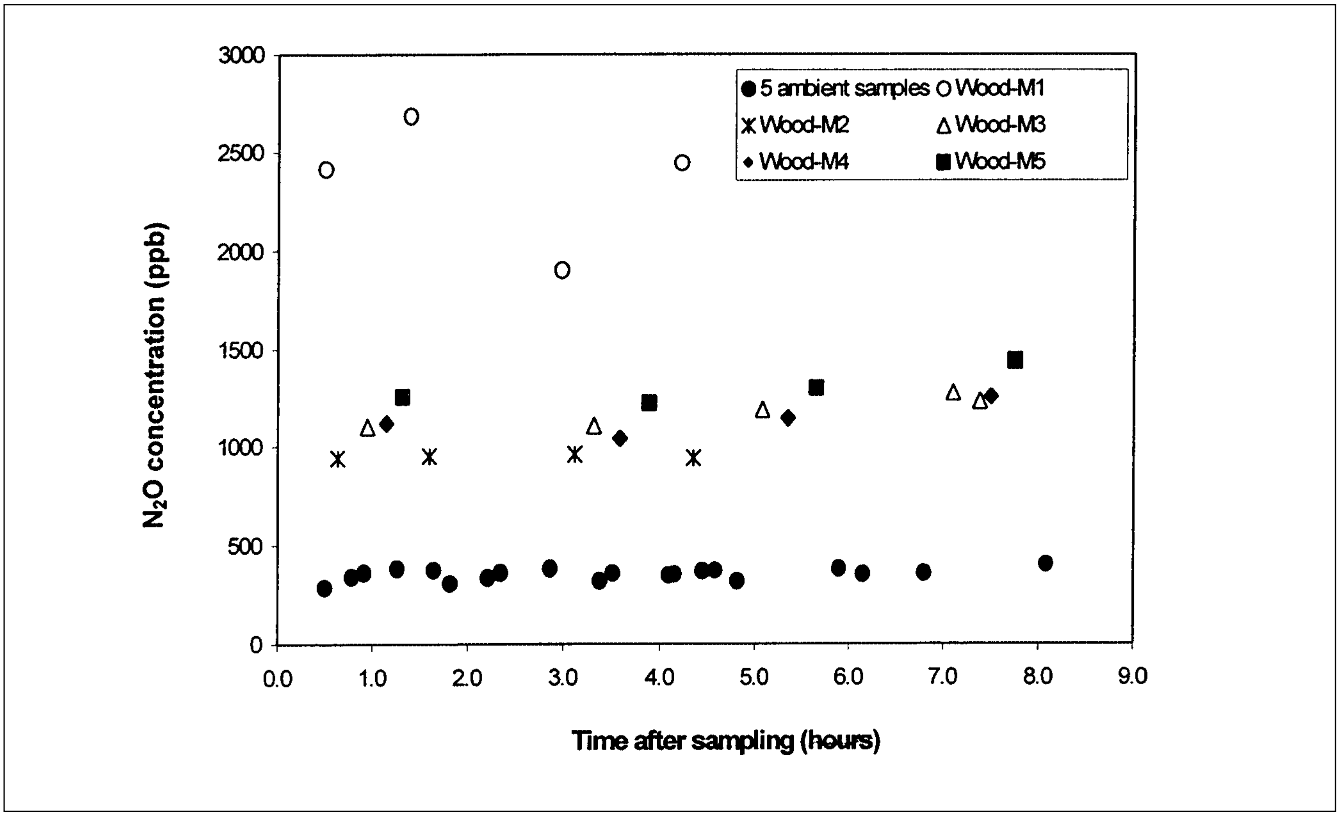

Figure 5. Test for artifact formation of N_2O stored in MMT sampling bags. Samples were collected from wood-burning emissions and ambient air.

Compared with the use of stainless steel canisters, the use of the MMT bag reduces the measurement cost substantially due to the lower material expense, lower shipping cost, lower cleaning cost, and less accessory requirements during the subsequent GC analysis. When properly packed, the MMT bag is durable even when shipped by air. As a collection and storage container, the MMT bag is suitable for several gaseous pollutants of typical concern (e.g., CO_2, CO, CH_4, THCs, and N_2O). These compounds can be stored in an MMT bag at room temperature for at least 3 months without significant changes in concentration.

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Table 4. Summary of MMT bag durability test: valid samples collected with MMT bags.

Source Type	Location	Number of Samples Collected	Number of Valid Samples	Number of Invalid Samples[a]
Ambient air	New Jersey	5	5	0
	Brazil	2	2	0
Emissions from kerosene heater	Residential house in New Jersey	5	5	0
Emissions from wood burning	Residential house in New Jersey	5	5	0
Emissions from charcoal-making process	Brazil	51	49	2
Emissions from charcoal-making process	Kenya	86	85	1

[a] Invalid samples: sample bags leaked or spoiled.

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