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# Evaluation of Microwave Irradiation for Analysis of Carbonyl Sulfide, Carbon Disulfide, Cyanogen, Ethyl Formate, Methyl Bromide, Sulfuryl Fluoride, Propylene Oxide, and Phosphine in Hay

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Fumigant residues in hay were "extracted" by microwave irradiation. Hay, in gastight glass flasks, was placed in a domestic microwave oven, and fumigants were released into the headspace by microwave irradiation. Power settings for maximum release of fumigants were determined for carbonyl sulfide (COS), carbon disulfide ($CS_2$), cyanogen ($C_2N_2$), ethyl formate (EF), methyl bromide ($CH_3Br$), sulfuryl fluoride (SF), propylene oxide (PPO), and phosphine ($PH_3$). Recoveries of fortified samples were >91% for COS, $CS_2$, $CH_3Br$, SF, PPO, and $PH_3$ and >76% for $C_2N_2$ and EF. Completeness of extraction was assessed from the amount of fumigant retained by the microwaved hay. This amount was determined from further microwave irradiation and was always small (<5% of the amount obtained from the initial procedure). Limits of quantification were <0.1 mg/kg for COS, $CS_2$, $C_2N_2$, EF, and $PH_3$ and <0.5 mg/kg for $CH_3Br$, SF, and PPO. These low limits were essentially due to the absence of interference from solvents and no necessity to inject large-volume gas samples. The microwave method is rapid and solvent-free. However, care is required in selecting the appropriate power setting. The safety implications of heating sealed flasks in microwave ovens should be noted.

**KEYWORDS: Hay; carbonyl sulfide; carbon disulfide; cyanogen; ethyl formate; methyl bromide; sulfuryl fluoride; propylene oxide; phosphine; residue**

## INTRODUCTION

Hay is harvested, and dried plant material is used for animal feed. Grasses and lucerne (alfalfa) are common plants used for hay. Especially, lucerne hay is for high-quality or special purposes, e.g., for racing horses. Infestations of insects, such as Hessian fly (Diptera: Cecidomyiidae) and cereal leaf beetle (Coleoptera: Chrysomelidae), during storage and transport are frequently the cause of hygiene and quarantine concerns (*1*−*5*). Therefore, stored or imported or exported hays were treated with methyl bromide ($CH_3Br$) for rapid disinfestation and quarantine treatment. However, $CH_3Br$ is an ozone-depleting substance; since January 2005, it has been banned for use on stored commodities, and in January 2015, it will be phased out for quarantine treatment (*6*, *7*). Therefore, the alternative fumigants such as carbonyl sulfide (COS), carbon disulfide ($CS_2$), cyanogen ($C_2N_2$), ethyl formate (EF), sulfuryl fluoride (SF), and propylene oxide (PPO) were re-evaluated or developed to replace $CH_3Br$ for rapid fumigation and quarantine treatment (*8*−*12*).

After fumigation, residues are left in the treated commodities. After removal from the commodity matrix by either purge and trap techniques or by solvent extraction (*13*−*15*), they are usually analyzed by gas chromatography (GC). However, purge and trap methods are not suitable for less volatile fumigants such as $CS_2$, EF, and PPO that are unable to pass through the reflux condenser (*16*), while solvent extraction (*14*, *15*) has the problem of solvent interference and is time-consuming. None of the above methods are suitable for analysis of residues in hay, as hay floats and is difficult to immerse in water or a nonaqueous solvent. However, microwave irradiation is being increasingly used in the digestion of samples (*17*−*19*), and in recent work (*21*, *22*), excellent recoveries and precision have been obtained from microwave extractions of COS, $CH_3Br$, $PH_3$, and dimethyl sulfide (DMS) from wheat. It was considered that microwave treatment of fumigated hay in sealed containers would release sufficient fumigant from the hay into the headspace to enable residue determination. This hypothesis was therefore tested with samples of hay individually fumigated with COS, $CS_2$, $C_2N_2$, EF, $CH_3Br$, SF, PPO, and $PH_3$.

## MATERIALS AND METHODS

**Apparatus and Reagents.** COS (98.5% COS and 1.5% air and $CO_2$), $C_2N_2$ (98.0% $C_2N_2$ and 2.0% air and $CO_2$), and $CH_3Br$ (97.0% $CH_3Br$ and 3.0% air) were sourced from BOC gas Australia. SF (99.8% SF and 0.2% $CO_2$) was supplied by Dow AgroSciences LLC (Atascadero, CA). Tetrafluoroethane (>99.9%) was supplied by Actrol Ltd. (Australia). EF, PPO, and $CS_2$ were analytical grade and were purchased

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**Table 1.** Optimal Power Settings (W) for Release of Fumigant from Hay and Power Settings Used to Evaluate the Procedure

| fumigant | power[a] (W) | time for first stage (s) | time for second stages (s) | recommended no. of stages[b] |
|---|---|---|---|---|
| COS | 495 | 50 | 30 | 3 |
| | 630 | 50 | 30 | 2 |
| $CS_2$ | 495 | 60 | 40 | 3–4 |
| | 630 | 60 | 40 | 2–3 |
| $C_2N_2$ | 495 | 50 | 30 | 3 |
| | 630 | 50 | 30 | 2 |
| EF | 495 | 50 | 30 | 3 |
| | 630 | 50 | 30 | 2 |
| $CH_3Br$ | 495 | 50 | 30 | 4 |
| | 630 | 50 | 30 | 3 |
| SF | 495 | 60 | 40 | 3–4 |
| | 630 | 60 | 40 | 2–3 |
| PPO | 495 | 60 | 40 | 3–4 |
| | 630 | 60 | 40 | 2–3 |
| $PH_3$ | 495 | 60 | 40 | 3–4 |
| | 630 | 60 | 40 | 2–3 |

[a] Power outputs of 630 and 495 W are those rated by the manufacturer for the settings medium high and medium. [b] The times of third and fourth stages (s) treatment are the same as the second stage (s) and were left for 2 min before further irradiation.

from Ajax Chemicals (Sydney, Australia). $PH_3$ (85.0% $PH_3$ and 15.0% air and $CO_2$) was laboratory prepared by the FAO method (*23*).

One liter Erlenmeyer flasks (Bibby Sterilin, Staffordshire, catalog no. FE 1 L/3) were used for preparation of standards; 2.5 L desiccators (Bibby Sterilin, catalog no. FE 2L/4) were used for the fumigation of the hay samples; and 250 mL bottles (Alltech catalog no. 9535) were used for the microwave "extraction". Each bottle was fitted with a Mininert valve equipped with septa (Alltech catalog no. 95326). The measured volume of each Erlenmeyer flask and inlet system was calculated from the weight of water required to fill the container and was used for calculations.

A 5 $\mu$L syringe (SGE, Melbourne, Australia; catalog no. 5R-GT) was used for the transfer of liquid fumigants. A 2.5 mL gastight syringe (SGE; catalog no. 008510) was used for the transfer of gases, and a 100 $\mu$L airtight syringe with valve (SGE; catalog no. 005279) was used for the injection of microwaved gas samples into GC.

COS, SF, and $CS_2$ (with sulfur filter) and $PH_3$ (with phosphorus filter) were determined on a Varian CP-3800 (Varian Instruments, Sunnyvale, CA), equipped with a pulsed flame photometric detector. Separation was achieved on a 30 m $\times$ 0.53 mm i.d., AT-Q column (Alltech Associates, catalog no. 0810025, part no. 13939) at 140 °C with a carrier flow ($N_2$) of 8.2 mL/min at 5.0 psi. Injection volumes of gases were 40 $\mu$L. A minimum interval of 5 min was kept between injections, in order to elute interfering chemicals.

EF, PPO, and $CH_3Br$ were determined on a Varian 3400 GC (Varian Instruments), equipped with a flame ionization detector (FID), after separation on a 50 m $\times$ 0.53 mm i.d., GS-Q column at 140 °C with a carrier flow ($N_2$) of 6 mL/min at 10 psi. Injection volumes of gases were 60 $\mu$L.

The purity of fumigant (e.g., from lecture bottles, cylinders, or laboratory generated) of COS, SF, $C_2N_2$, $CH_3Br$, and $PH_3$ was determined on a GOW-MAC mass density balance (GOW-MAC Instrument Co., Madison, NJ), after separation on a 1 m $\times$ 5 mm i.d. Porapak Q 100/120 mesh (Alltech Associates, catalog no. 2702) at 105 °C with a carrier flow ($N_2$) of 150 mL/min. The reference gas was tetrafluoroethane (>99.9%).

The microwave oven was a domestic model (Panasonic, Matsushita Electrical Industrial Co., Ltd., Osaka, Japan, model NN-5454) purchased at a local retail outlet. The rated maximum power output was 900 W, at an operating frequency of 2450 MHz.

**Hay Sample and Fumigation of Hay.** Hay used in this study was Australian Lucerne (*Medicago sativa*) hay, 9.4% moisture content (m.c.), w/w, wet basis. The moisture content of the hay was measured by oven drying at 105 °C for 2 h.

**Table 2.** Recoveries of Fortified Samples ($n = 4$)

| fumigant | amount added (ng/L) | amount added (mg/kg) | mean recovery[a] $\pm$ SD[b] (%) |
|---|---|---|---|
| COS | 1.0 | 0.05 | 91.2 $\pm$ 8.6 |
| $CS_2$ | 2.0 | 0.1 | 98.4 $\pm$ 9.2 |
| $C_2N_2$ | 1.0 | 0.05 | 76.5 $\pm$ 8.6 |
| EF | 2.0 | 0.1 | 78.4 $\pm$ 11.4 |
| $CH_3Br$ | 1.0 | 0.05 | 94.7 $\pm$ 7.3 |
| SF | 1.0 | 0.05 | 101.7 $\pm$ 8.8 |
| PPO | 2.0 | 0.1 | 97.3 $\pm$ 10.1 |
| $PH_3$ | 0.5 | 0.025 | 102.3 $\pm$ 9.5 |

[a] Calculated base on maximum release of fumigant. [b] Standard deviation.

Hay samples (100 g) were fumigated in sealed desiccators (2.5 L) equipped with a lid-fitted septum injection system for 48 h at concentrations of 50 (COS, $CS_2$, $C_2N_2$, EF, $CH_3Br$, SF, and PPO) and 5 mg/L ($PH_3$). After 48 h of exposure, the desiccators were opened and aired for 24 h in a fumehood to obtain samples containing residual fumigant.

**Preparation of Standards and Recovery Studies.** Diluted gas standards were prepared by first removing the same volume of air as the known volume of concentrated fumigant to be injected into an Erlenmeyer flask (1 L) containing two glass beads (2−3 mm o.d). Volumes of concentrated gas fumigant used were 5 mL of COS, $C_2N_2$, SF, and $CH_3Br$ and 0.5 mL of $PH_3$. Liquid fumigants were added in small quantities, e.g., 10 $\mu$L of $CS_2$, EF, and PPO. In spiking studies, for the investigation of recovery of spiked samples, a known amount of fumigant (**Table 2**) was added into sealed flasks (250 mL) containing hay (5 g) 10 min before microwaving. The reported analyses are the mean of duplicate samples.

**Microwave Procedure.** The hay sample to be analyzed (5 g) was transferred to a bottle (250 mL) and sealed with a Mininert valve equipped with a septa for gas sampling. The bottle was placed in the microwave oven. For evaluation of the procedure, microwave irradiation was performed in stages (**Table 1**). The defined microwave stages were used both to compare efficiency of release of different fumigants and to reduce problems of increased pressure, due to increased heat in a sealed system. The power settings recorded are the manufacturer's rated power output, e.g., the "medium high" and "medium" setting on the oven has a rated output of 630 and 495 W (**Table 1**), respectively. Fumigant in the headspace was determined by GC, and the system was left for 2 min (for cooling) before further irradiation. The process of irradiation, analysis, and leaving for 2 min was repeated until the amount of fumigant in the headspace either remained constant or started to decline.

**Evaluation of Fumigant Stability under Microwave Irradiation.** A known amount of fumigant (**Table 2**) was added to sealed flasks (250 mL) containing distilled water (0.5 g), which was equivalent to the amount of water in 5 g of hay sample. The flasks were then treated with microwave irradiation following the stages shown in **Table 1**. The measurement of fumigants in the headspace and the process of irradiation are the same as above. The concentrations of fumigant were calculated on the basis of peak areas, which were calibrated periodically using a gas standard. The data recorded in the figures are the mean of duplicate samples.

**Precautions for Handling Fumigants.** Fumigant transfer, fumigation, aeration, and all containers containing fumigant were conducted or placed in the fumehood. Airtight syringes were used for the transferring and injection of fumigants.

## RESULTS AND DISCUSSION

**Interaction between Fumigant and Water under Microwave Irradiation.** During all four stages of microwave irradiation (180 s), the fumigants of $CS_2$, SF, PPO, and $PH_3$ were very stable, the variations of their concentration were <5%, and interaction between the fumigants and the water did not occur at either 495 or 630 W power settings (**Figure 1**). COS and $CH_3Br$ were relatively stable, and the variations of their

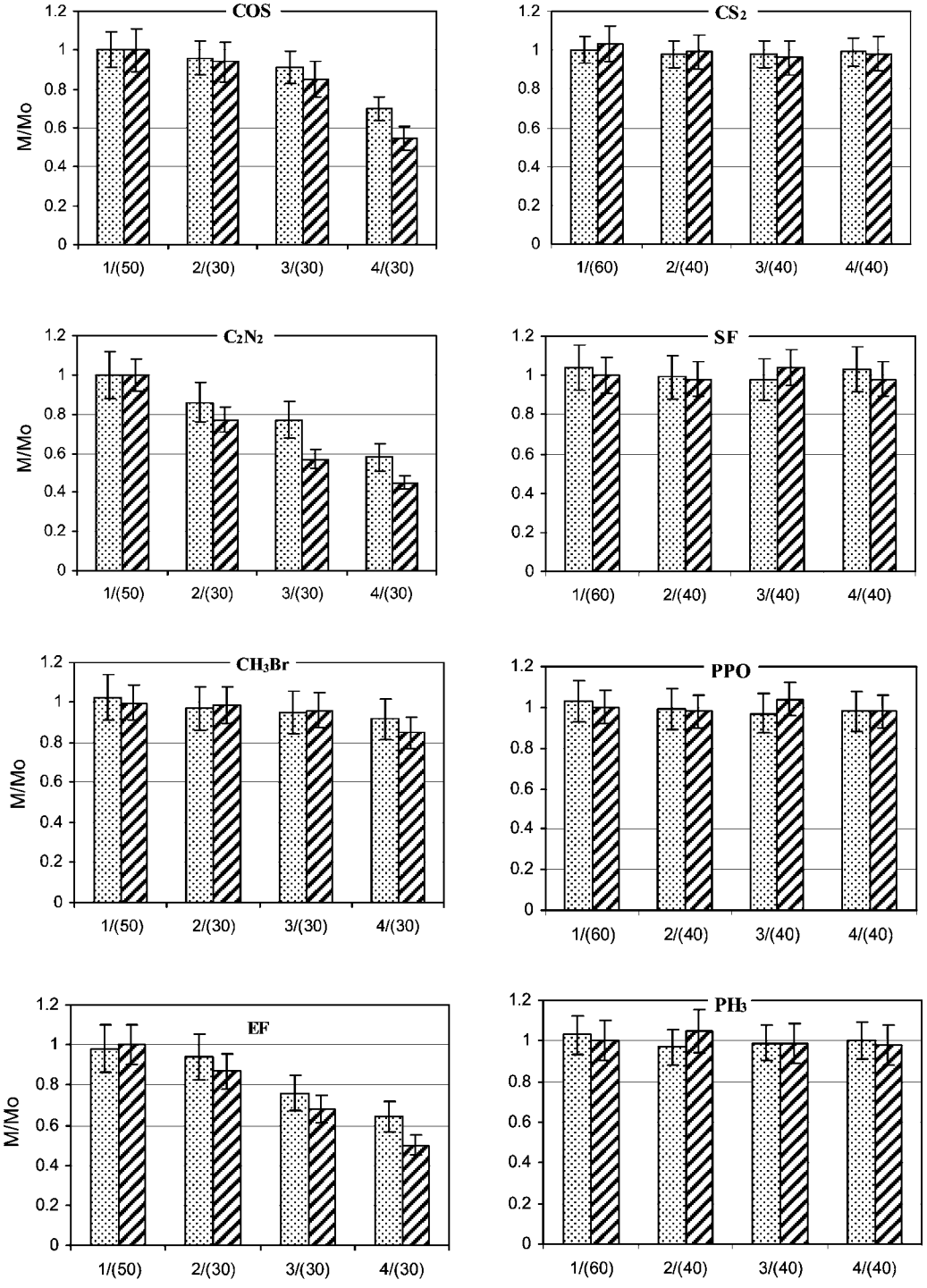


**Figure 1.** Stability of fumigant concentrations (means of duplicates) in the headspace after microwave heating, where $M/M_0$ is the ratio of mass of fumigant ($M$) in the headspace to total applied mass ($M_0$) (dotted bars, 495 W; and slashed bars, 630 W).

concentration were <16% during the first three stages of microwave irradiation (110 s) at both 495 and 630 W power settings. However, 5 and 13% of COS were converted to hydrogen sulfide ($H_2S$) at third- and fourth-stage treatments, respectively. $C_2N_2$ and EF were relatively stable, and the variations of their concentration were <20% during the first two stages of microwave irradiation (80 s), but levels of $C_2N_2$ and EF rapidly declined after the second treatment at both 495 and 630 W power settings. The major part of the lost $C_2N_2$ was converted to hydrogen cyanide (HCN), and similarly, EF was converted to formic acid and ethanol. Therefore, for analysis of COS, $C_2N_2$, EF, and $CH_3Br$, the time and power setting should be carefully selected to reduce overheating and the conversion of fumigant.

**Time and Power Setting of Microwave Irradiation for Release of Fumigants from the Hay.** The release of COS, $CS_2$, $C_2N_2$, EF, $CH_3Br$, SF, PPO, and $PH_3$ after each irradiation stage (**Table 1**) is shown in **Figure 2**: Maximum release of COS, $C_2N_2$, and EF was obtained by microwave irradiation at 630 W for 80 s and a second-stage treatment at 630 W for 110 s or a

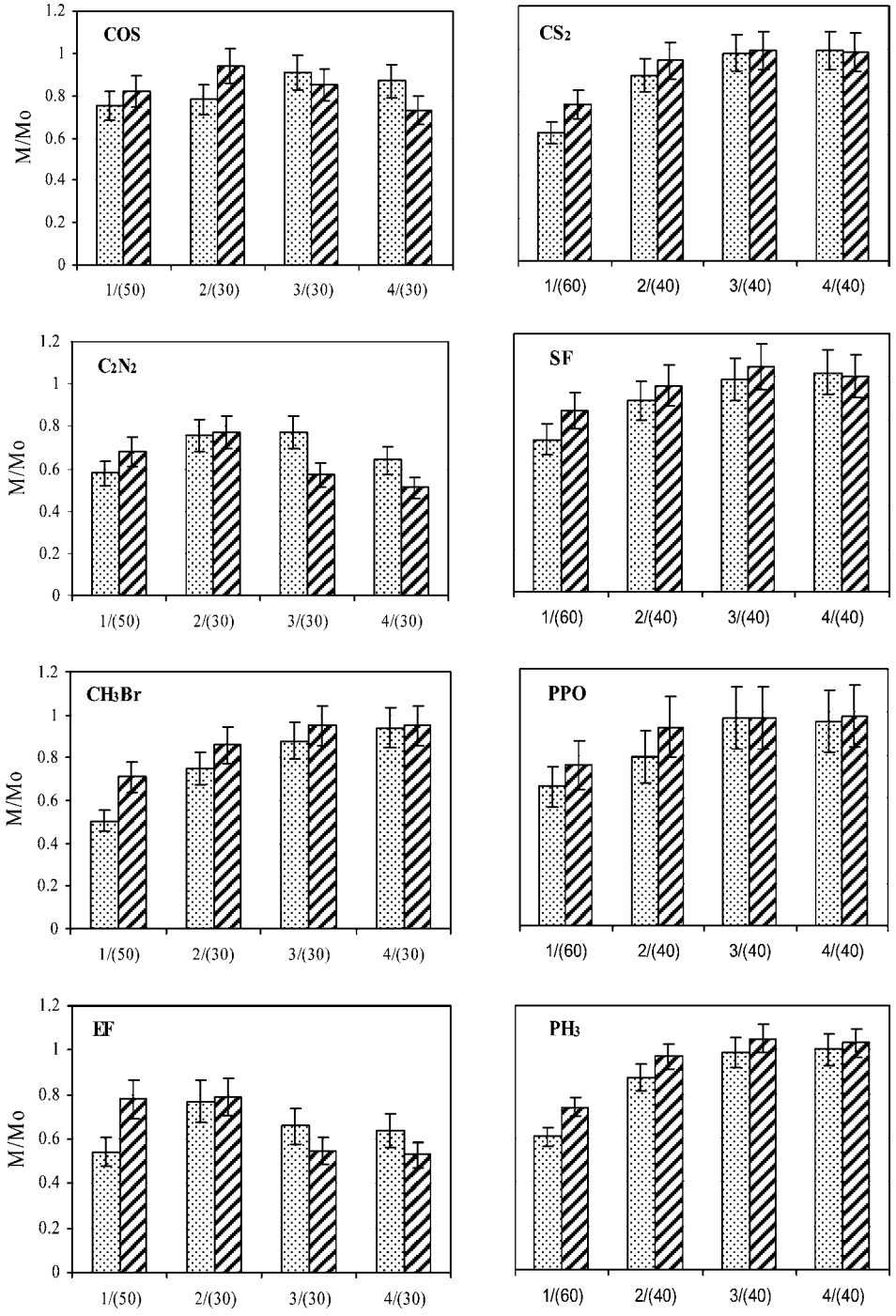


**Figure 2.** Fumigant residues (means of duplicates) in fumigated hay released into the headspace by microwave heating, where $M/M_0$ is the ratio of mass of fumigant ($M$) in the headspace to total applied mass ($M_0$) (dotted bars, 495 W; and slashed bars, 630 W).

third-stage treatment at 495 W; maximum release of $CH_3Br$ was obtained by irradiation at 630 W for 110 s and three stage treatments or at 495 W for 140 s and four stage treatments; and maximum release of $CS_2$, SF, PPO, and $PH_3$ was obtained by irradiation at 630 W for 140 s and three stage treatments or at 495 W for 180 s and four stage treatments.

The naturally occurring COS, $CS_2$, and EF were released from untreated hay at a level of quantification (LOQ < 0.01 mg/kg) after first- or second-stage treatment at 630 or 495 W, respectively. However, natural levels of COS, $CS_2$, and EF were 0.5−1.0 mg/kg after third-stage treatment at 630 W or fourth-stage treatment at 495 W. That is, the natural levels of COS, $CS_2$, and EF did not cause a significant interference when use of a first- or second-stage treatment for maximum release of fumigants. No interfering $C_2N_2$, $CH_3Br$, SF, PPO, and $PH_3$ was released from the control (unfumigated) hay at LOQ (LOQ < 0.01 mg/kg).

**Recovery of Fumigant.** Recoveries of fortified levels (0.5−2.0 ng/L or 0.025−0.1 mg/kg) of each fumigant at the power settings outlined in **Table 1** are shown in **Table 2**. The fortified levels were much lower than maximum residue levels (MRLs) of $CH_3Br$ and $PH_3$ (no MRLs for other fumigants). Recoveries

of COS, $CS_2$, $CH_3Br$, SF, PPO, and $PH_3$ were 91−102%. Although recoveries of COS and $CH_3Br$ were high (>91 and >94%, respectively) at the second- or third-stage treatment, continued treatment, such as at the fourth-stage treatment, resulted in a significant decline of the fumigants. Recoveries of $C_2N_2$ and EF were relatively low (76 and 78%, respectively). Both $C_2N_2$ and EF could be broken down under microwave heating, particularly after third-stage irradiation treatment (**Figures 1** and **2**), 35−45% of $C_2N_2$, and EF was reduced in the headspace at both 630 and 495 W power settings. These results are consistent with the stability of fumigant under microwave irradiation (**Figure 1**). The effectiveness of microwave irradiation extraction was evaluated from residues in microwaved hay, which were determined after transfer to another empty flask, and then, a further microwave irradiation was preformed. Residues determined after a second microwave irradiation were low (2.1−4.4% of those determined from the first heating). The amount of fumigant residue was not increased by further "extraction" (irradiation) indicating that completeness of extraction was achieved at above the recommended power regimes. It should be noted that the small volume occupied by the hay (5 g), relative to the flask volume (250 mL), was chosen to minimize the proportion of fumigant retained by the hay. It is possible to increase the sensitivity of the method by increasing the proportion of hay to headspace, but such increases may decrease the proportion of the fumigant in the headspace.

**Advantage of Microwave Irradiation for Releasing Fumigant from Hay.** The LOQs by the microwave irradiation method were <0.1 mg/kg for COS, $CS_2$, $C_2N_2$, EF, and $PH_3$ and <0.5 mg/kg for $CH_3Br$, SF, and PPO, which are much lower than those reported for purge and trap procedures (*6*) and for solvent extraction (*14*, *15*), where comparable data are available. This is principally because (i) the fumigants extracted by microwave irradiation are partitioned between two phases—solid (hay) and air (headspace). However, the fumigants extracted by solvent extraction are partitioned between three phases—solid (hay), air (headspace), and liquid (solvent). The partitioning coefficients of fumigant concentration in solvents such as acetone, ethanol, and other organic solvents are greater than in the air or headspace (fumigation concentration in solvent [$C_{solvent}$] > fumigation concentration in air [$C_{air}$]). That is, in the same volume of solvent or air, the larger fraction of the fumigant will be dissolved into the solvent rather than the headspace. This will increase the LOQs for headspace analysis. (ii) As compared with the solvent extraction method, microwave irradiation is without the interference or contamination associated with solvents. (iii) In addition, gas volumes that can be injected into a GC are much larger (100−250 $\mu$L) than solvent volumes (1−3 $\mu$L), especially for packed or wide-bore capillary ("megabore") columns. This is especially important for determination of sulfur gases using an FPD detector, where peak height is proportional to the square of the injected mass.

In summary, the microwave irradiation satisfies the requirements (*24*) for validation of procedures for extraction of fumigant residues. For example, recoveries of fortified samples were adequate and a completeness of extraction was achieved. Use of microwaves to release fumigants into the headspace involves the safety issue of increased pressure leading to bursting (although no such burstings have occurred in our laboratory). Optimal power settings need to be determined on individual ovens, as they may vary with equipment. However, the method has the following advantages over existing procedures for multiresidue determination of fumigants. First, results are obtained very quickly; second, problems caused by solvents, such as contamination and disposal, are avoided; third, the cost of a domestic microwave oven can be readily recovered from the reduced use of solvents. The microwave method described in this publication does not have the versatility of microwave-assisted solvent extraction (*19*, *20*), especially in analysis of nonvolatile chemicals. However, it has the potential to be useful in analysis of volatile chemicals, such as fumigants.

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