



Naphthalene emissions from moth repellents or toilet deodorant blocks determined using head-space and small-chamber tests

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Abstract

The present study investigated the emissions of naphthalene and other compounds from several different moth repellents (MRs) and one toilet deodorant block (TDB) currently sold in Korea, using a headspace analysis. The emission factors and emission rates of naphthalene were studied using a small-scale environmental chamber. Paper-type products emitted a higher concentration of the total volatile organic compounds (VOCs) (normalized to the weight of test piece) than ball-type products, which in turn emitted higher concentration than a gel-type product. In contrast, naphthalene was either the most or the second highest abundant compound for the four ball products, whereas for paper and gel products it was not detected or was detected at much lower levels. The abundance of naphthalene ranged between 18.4% and 37.3% for ball products. The results showed that the lower the air changes per hour (ACH) level was, the higher the naphthalene concentrations became. In general, a low ACH level suggests a low ventilation rate. The emission factor for naphthalene was nearly 100 times higher for a ball MR than for a gel or a paper MR. For the ball MR, the lower ACH level resulted in higher emission rate.

Key words: volatile organic compounds (VOCs); chemical composition; ball-type; gel-type; paper-type

Introduction

Exposure to naphthalene is a recent subject of concern because of prevalence of this compound in the environment and its adverse health effects. Since it is a white solid that evaporates easily (ATSDR, 2005), naphthalene is present as a vapor under typical atmospheric conditions, and has been prevalent in both indoor and outdoor air (Van Winkle and Scheff, 2001; Batterman *et al.*, 2002; Edward *et al.*, 2005; Li *et al.*, 2005; Reisen and Arey, 2005; Zuraimi *et al.*, 2006). Recently, Li *et al.* (2005) has reported that naphthalene was the most abundant polycyclic aromatic hydrocarbons (PAHs) in residential air of ten Chicago area homes. A long-period inhalation exposure to naphthalene may cause kidney and liver damage, retinal damage, and central nervous system damage (USEPA, 2003a). Exposure to high concentrations of naphthalene can damage or destroy some red blood cells, thereby causing hemolytic anemia (USEPA, 2003a). Since inhalation of naphthalene has caused cancer in animal tests, this compound has been classified as possibly carcinogenic to humans (IARC, 2002; USEPA, 2003a).

The major commercial use of naphthalene is in the manufacture of polyvinyl chloride plastics (ATSDR, 2005). Its major consumer use is in moth repellents (MRs) and toilet deodorant blocks (TDBs). Like many other countries,

mothballs or other MRs are employed in clothes chests in many Korean homes and in toilets in many public access buildings. According to personal communication at the marketing department in one of the largest companies for MRs and TDBs in Korea, the yearly marketing amount in Korea is about 1.0×10^7 dollars. Although there is a perceived benefit of MRs or TDBs, there might also be health risks from the inhalation exposure to naphthalene because of its various adverse health effects. Nevertheless, limited information is available about the inhalation exposure to naphthalene associated with the use of MRs or TDBs. Such data are needed to properly link environmental exposure with health effects.

Although direct-indoor monitoring data can be employed to properly estimate the inhalation exposure of building occupants, emissions data obtained under laboratory conditions can provide valuable information for semiquantitative estimation of inhalation exposure. A headspace measurement method has been employed regarding the semiquantitative determination of volatile components that are emitted from consumer products (Colombo *et al.*, 1991). A quantitative analysis in estimating the volatile organic compounds (VOCs) from the emission of consumer products can be carried out by using small environmental test chambers (Kemmlin *et al.*, 2003; Wilke *et al.*, 2004; Singer *et al.*, 2006). Consequently, this study investigated the emissions of naphthalene and

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other compounds from several different MRs and one TDB currently sold in Korea, using headspace analysis (USEPA, 2003b). Furthermore, this study estimated the emission rates of naphthalene from selected MRs by using a small-scale environmental chamber.

1 Materials and methods

1.1 Test products

A marketing survey regarding the MR distribution was performed in three largest supermarkets in Korea. There were 11 different MR products, and seven of them were selected for headspace tests on the basis of sales figures (i.e., the number of items sold during the previous year). These products occupied more than 80% of the yearly marketshare in Korea. There were four ball products, two paper products, and one gel product. One of the four ball products was a TDB, whereas other products were all for clothes chests. One of each type was further investigated to estimate chemical emission rate using a small-scale chamber. All products were purchased from supermarkets less than one year after being manufactured.

1.2 Headspace tests

Headspace analysis was conducted to determine tentatively the VOCs emitted from six MRs of different brands and one TDB by applying USEPA method (2003b). For the ball and gel MRs and the ball TDB, sample materials occupied just roughly 40% of the total volume of the 40-ml glass bottles, which was fitted with Teflon-septum top screw-caps. For the paper MRs, a whole piece of paper (2.5 cm × 4.5 cm × 0.03 cm) was placed in the bottle. The weights of test pieces were as follows: 10.08–11.24 g ball products; 0.23–0.25 g paper products; and 12.15 g for gel product. The glass bottles were placed in a water bath for 90 min at 60°C to allow VOCs evolution. One milliliter of the gaseous sample was drawn into a 10-ml pressure lock syringe, and transferred to a gas chromatograph (HP 5890II) and mass spectrometer (HP MSD5973) (GC/MS) system (Hewlett Packard, USA). A 30-m long fused silica column with a 0.32-mm inside diameter and a 1- μ m film thickness (HP-5, Agilent Technologies, USA) was programmed initially for 5 min at 40°C, and it increased at a rate of 5°C/min up to 250°C, and held for 5 min. The concentrations of the total VOCs (integrated chromatographic peak areas of analytes, converted to toluene-equivalent concentration) for analytes in the headspace gas phase of each product were determined for this experiment. Analytes that were detected in insignificant amounts (chromatographic peak area 10^4) were not included in the current paper.

1.3 Small-scale chamber tests

One ball product (BT1), which provided the highest headspace concentration of the total VOCs and naphthalene, one gel (GT) product, and one paper (PT1) product were selected for chamber study. The weights of test pieces were as follows: 32.4–32.7 g ball products; 5.4 g paper

products; and 32.5 g for gel product. An electropolished stainless steel (SS) test chamber (40 cm × 25 cm × 50 cm) was employed to determine the emission rates of naphthalene from selected products. The top of the chamber acts as a door, the sealing being made with a silicon gasket. Eight holes (each 0.635 cm) were drilled through the SS walls for air inlet and outlet, instrument connections, and sampling ports. The holes were blocked with Swagelok valves or Teflon tape. The clean air for the chamber is supplied from a zero-grade air cylinder. The air leakage of the chamber was determined by measuring the flow rate at the chamber outlet, and this amount was compared with the supplied airflow rate. The chamber temperature ranged between 21 and 26°C. Relative humidity (RH) was measured between 20% and 25% just prior to the chamber inlet and outlet using a humidity meter (Thermo Recorder TR-72S, T & D Co., Taiwan, China). The air in the chamber was mixed by a metal fan (14-cm diameter). Homogeneity within the chamber was tested by the simultaneous collection of samples at two different ports. The dispersion of the results obtained from three trials was within the precision of the measurements (less than 15%). The outlet-flow rate was within 5% of the inlet-flow rate. Since the velocity near the surface of the test products can affect the mass transfer coefficient, the products were tested for emissions under a typical indoor velocity (5–10 cm/s) (ASTM, 1998). The ventilation rate was 1 ± 0.05 air changes per hour (ACH). This was tested with a digital bubble flow meter and was comparable to that suggested by the ASTM (1998). Since ACH indicates the amount of dilution and flushing that occurs in indoor environments (ASTM, 1998), two other ventilation rates (0.5 ± 0.03 and 2 ± 0.05 air changes per hour) were further tested for the emissions from one selected MR (BT1). The background concentrations in the empty chamber were either undetected or just trace amounts.

Chamber tests started within 10 min after the placement of the full-size test products in the chamber. Chamber-air samples were collected at average elapsed times of 5, 25, 45, 65, 85, 105, 180, 240, and 300 min after initiating the test. Air samples were collected by passing air through adsorbent tubes, containing Tenax-TA. The tubes are connected to be calibrated constant flow pumps for 10-min sampling periods. The sampling pump was calibrated by a digital flow meter prior to and following the collection of each sample. The average of these two rates was used as the sample-flow rate in all volume calculations. The flow rate was adjusted between 150 and 350 ml/min, depending upon the ventilation rates. The sampling flow rate did not exceed 50% of the inlet flow rate, as suggested by the ASTM (1998).

Samples of naphthalene, collected in the Tenax TA trap, were analyzed by the coupling of a thermal desorption system (TDS, Model Aerotrap 6000, Tekmar, USA) to a GC (HP 6890, Hewlett Packard, USA), with a flame ionization detector. A fused silica column (0.32 mm i.d., 60 m, and 1 μ m film thickness) (SPB-5, Supelco Co., USA) was programmed from 35 to 220°C at 4°C/min. The adsorbent trap was thermally desorbed at 250°C for 10 min, and the

target compound was cryofocussed at 120°C on a cryo trap (a 15.2-cm-long, 0.32-cm-o.d. tube packed with glass beads). The cold trap was rapidly heated to 250°C, then the contents were flushed into the Cryofocusing Module (CM) of the TDS and cooled to 120°C to refocus the target compound. The CM was heated to 225°C and flushed to transfer the target compound to the GC. The initial oven temperature was set at 35°C for 5 min and ramped at 4°C/min to 200°C for 5 min.

The quality control program for the measurements of naphthalene included laboratory- and field-blank Tenax traps, and spiked samples. At the beginning of each day, the laboratory- and field-blank Tenax traps were analyzed to check for any contamination during sampling and analytical procedures. To check the quantitative response, a known standard was directly injected into a trap and the target compound was transferred to the GC. When the quantitative response differed by more than 15% from that predicted by a specified calibration equation, a new calibration equation was determined. The precision (within 15%) and method detection limits ($1.1 \mu\text{g}/\text{m}^3$) of naphthalene were determined by seven repeated analyses of one of the calibration standards.

2 Results and discussion

2.1 Constituents in headspace samples

The abundance of constituents of VOC emissions from 6 MRs and one TDB were determined from the headspace gas phase (Table 1). Thirty-six compounds were emitted from at least one of the seven products, at chromatographic peak areas above 10^4 and at a spectral search quality of greater than 70%. Of the 36 compounds, only four compounds (1,2-bis(trimethylsilyl)acetylene, chloroform, pentane, and toluene) were emitted from all products. It is noteworthy that limonene consisted of 61.3% of peak area for BT1.

The chemical composition and concentrations of the total VOCs varied broadly within the products. Paper products emitted a higher concentration of the total VOCs (normalized to weight of test piece) than ball products, which in turn emitted higher concentration than a gel product. BT1 emitted the highest concentration of the total VOCs among the four ball products. Meanwhile, a ball TDB (BT4) emitted somewhat lower levels of the total VOCs, as compared with the three other ball MRs. In contrast, naphthalene was either the most or the second highest abundant compound for the four ball products, whereas for paper and gel product it was either undected or at trace level. The abundance of naphthalene ranged between 18.4% and 37.3% for the ball products. For example, of the 20 compounds detected in the headspace sample of BT1, naphthalene was the most abundant compound. Considering both the abundance and the concentrations of the total VOCs, BT1 emitted the highest emission concentration of naphthalene among the four test products. It is also noteworthy that limonene was the most abundant compound for BT1 (61.3%), and paper (PT2) (17.4%).

Limonene is a terpene, which is potentially reactive with ozone to generate secondary pollutants with oxidants such as ozone, hydroxyl radicals, and nitrogen oxides (Wolkoff *et al.*, 1998; Weschler and Shields, 1999; Finlayson-Pitts and Pitts Jr, 2000; Atkinson and Arey, 2003). The addition of this compound to household products is most likely due to its favorable odor and solvent properties (Nazaroff and Weschler, 2004). Household products, which are reported to contain limonene (in Korea and other countries), include air freshener (Cooper *et al.*, 1995; Salthammer, 1999), furniture polish (Colombo *et al.*, 1991), disinfectants, general purpose cleaners, and glass cleaners (Zhu *et al.*, 2001). For BT2, toluene (21.4%) was the second highest abundant compounds. This compound was also identified in other household products: lemon fresh and antibacterial spray (Zhu *et al.*, 2001); household cleaners and polishes (Sack *et al.*, 1992); paste wax, detergents; and liquid-floor waxes (Knöppel and Schauenberg, 1989). In contrast to the ball products, naphthalene was not a main compound in the headspace gas phase of the gel and paper MRs. Moreover, naphthalene was undetected in the headspace sample of PT2.

2.2 Characterization of concentration-time profiles for chamber tests

To determine quasi-steady-state emission conditions, the procedure included the determination of the time profiles of the naphthalene concentration, which were developed after the introduction of the sample products into the chamber. Fig. 1a presents the time-series concentrations

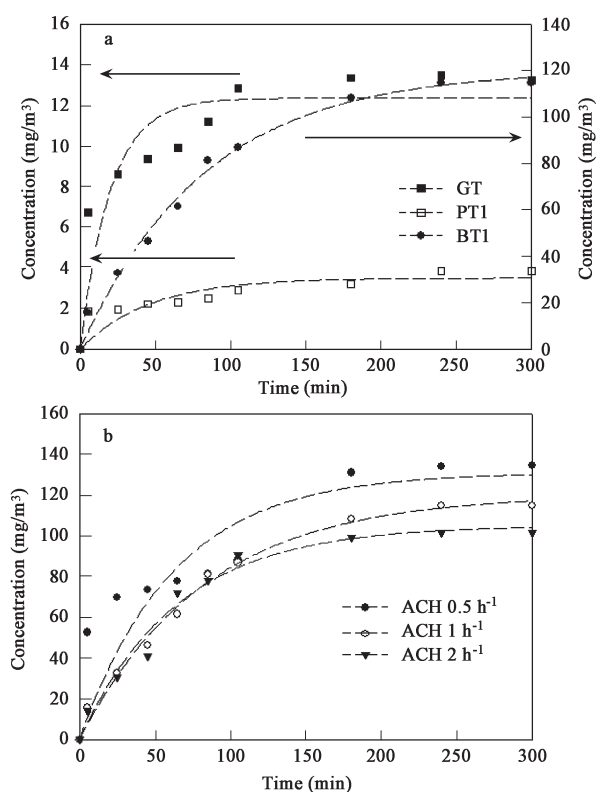


Fig. 1 Time-series concentrations of naphthalene emitted from three products (BT1, GT, and PT1) at ACH 1 h⁻¹ (a) and one BT1 according to the ACH levels (b). ACH: air changes per hour.

Table 1 Abundance of constituents of the volatile organic compounds (VOCs) emission from products determined by headspace analysis

| Compound | Peak area percentage (%) | | | | | | |
|----------------------------------------------------------------|--------------------------|-------|-------|-------|-------|--------|-------|
| | BT1 | BT2 | BT3 | BT4 | PT1 | PT2 | GT |
| Acetone | 1.4 | 1.8 | 2.4 | 2.8 | 4.5 | nd | nd |
| Azulene | nd | nd | nd | nd | nd | 12.4 | nd |
| Benzene | nd | 0.1 | nd | nd | 0.1 | nd | 0.2 |
| 1,2-Bis(trimethylsilyl)acetylene | 2.4 | 4.5 | 23.5 | 27.3 | 15.7 | 14.3 | 16.3 |
| 2-(2-Butoxyethoxy)ethanol | nd | nd | 1.9 | nd | 3.8 | nd | 5.4 |
| Carbon tetrachloride | 0.1 | 0.1 | nd | nd | nd | 0.1 | nd |
| Chloroform | 0.1 | 0.1 | 0.2 | 0.2 | 0.2 | 0.3 | 0.3 |
| Decadien | nd | nd | nd | nd | nd | 4.5 | 9.3 |
| Decanal | 2.8 | 2.4 | 7.0 | 6.5 | nd | nd | 11.5 |
| Decane | nd | nd | nd | nd | nd | 9.8 | nd |
| 1,4-Dioxane | nd | nd | nd | nd | nd | 2.4 | nd |
| Dodecane | 0.9 | 1.8 | 3.4 | 2.0 | nd | nd | 9.5 |
| 2,3-Dimethyl cyclohexylamine | nd | 3.8 | nd | nd | nd | nd | nd |
| Ethylbenzene | 0.1 | 0.1 | 0.2 | nd | 0.2 | nd | 0.4 |
| 4-Ethyltoluene | nd | nd | nd | nd | 0.1 | nd | 0.2 |
| Hexadecane | nd | nd | 2.8 | nd | nd | 4.0 | nd |
| Hexyl acetate | 1.0 | nd | nd | nd | nd | nd | nd |
| Limonene | 61.3 | nd | 2.4 | nd | nd | 17.4 | nd |
| <i>d</i> -Manitol | nd | nd | nd | nd | nd | nd | 14.8 |
| 2-Methyldecane | nd | nd | nd | nd | 13.3 | nd | nd |
| 3-Methylhexane | nd | 1.7 | nd | nd | nd | nd | nd |
| Myrcene | 1.9 | 8.7 | nd | nd | nd | nd | nd |
| Naphthalene | 18.4 | 30.3 | 37.3 | 22.8 | 0.9 | nd | 2.4 |
| Nonadecane | 2.1 | 1.3 | 1.7 | 19.9 | nd | 11.3 | nd |
| Nonanal | 1.3 | 3.2 | 3.3 | 2.8 | 31.5 | nd | nd |
| Nonane | nd | nd | nd | nd | nd | nd | 10.2 |
| Pentadecane | nd | 1.9 | 2.4 | 5.3 | 6.1 | nd | 5.5 |
| Pentane | 1.2 | 6.4 | 2.2 | 2.9 | 6.5 | 4.8 | 6.6 |
| α -Pinene | 0.8 | 5.4 | nd | nd | nd | nd | nd |
| Styrene | nd | 0.1 | nd | nd | 0.2 | 0.1 | 0.1 |
| Tetradecane | 0.8 | 1.1 | 1.9 | nd | nd | 2.8 | nd |
| Toluene | 2.2 | 21.4 | 4.5 | 5.4 | 8.5 | 2.1 | 6.7 |
| Tetramethylsilane | 1.1 | 3.6 | 2.4 | 2.1 | 7.5 | 11.2 | nd |
| 1,2,4-Trimethylbenzene | nd | nd | 0.1 | nd | 0.2 | 0.2 | 0.1 |
| 1,3,5-Trimethylbenzene | nd | nd | 0.1 | nd | 0.2 | 0.1 | 0.1 |
| <i>m,p</i> -Xylene | 0.1 | 0.2 | 0.3 | nd | 0.5 | 0.4 | 0.4 |
| Conc. of total VOCs (mg/m ³) | 12,063 | 8,165 | 7,233 | 5,445 | 1,803 | 2,623 | 3,648 |
| Norm conc. of total VOCs (mg/(m ³ ·g)) ^a | 1,194 | 726 | 689 | 509 | 7,839 | 10,492 | 300 |

BT: ball products; PT: paper products; GT: gel products; nd: not detected. ^a Concentrations of total VOCs were normalized to the weights of test pieces.

of naphthalene emitted from the three products (BT1, GT, and PT1) under the conditions of ACH (1 h⁻¹). A quasi-steady state condition was achieved around 105 min after initiating the chamber test for GT and PT1, and around 180 min for BT1. The emission concentrations were in descending order of BT1 > GT > PT1. This is consistent with the amounts of naphthalene estimated in the headspace gaseous phase of these products. By multiplying the abundance of naphthalene by the concentrations of the total VOCs, the amounts of naphthalene in the headspace phase were estimated to be 2,698, 88, and 16 mg/m³, for BT1, GT, and PT1, respectively.

Figure 1b exhibits the time series concentrations of naphthalene, emitted from one ball product (BT1), according to ACH. For most of the time periods, the lower the ACH level was, the higher the naphthalene concentrations became. In general, a low ACH level suggests a low ventilation rate. The time needed to reach a quasi-steady state condition was similar for the three ACH levels (around 180 min after initiating the test).

2.3 Emission factors and emission rates

Assuming perfect mixing within the chamber and no net loss of naphthalene from air, the emission factors were calculated from the observed concentrations by following equation (CDHS, 2004):

$$EF_t = Q \times (C_t - C_0) / W$$

where, EF_t (mg/(g·h)) is the source emission factor, C_t (mg/m³) is the concentration of naphthalene at time t , C_0 (mg/m³) is the background concentration in the chamber, Q (m³/h) is the measured flow rate of air into the chamber, and W (mg) is the mass of MRs employed in this chamber test. The concentrations measured for 300 min (5 h) after initiating the chamber test were employed as the equilibrium concentration (C_t). This condition satisfies with 3 cycles of residence time for ACHs is 1 and 2 h⁻¹. The emission rate (mg/h), which is defined as the mass of naphthalene emitted from the MRs per unit time, was calculated by multiplying the emission factor of W . Since

Table 2 Emission rates and emission factors for naphthalene according to moth repellent products or the ACH levels

| Product | ACH (h ⁻¹) | Emission rate (mg/h) | Emission factor (mg/(g·h)) |
|---------|------------------------|----------------------|----------------------------|
| BT1 | 0.5 | 6.292 | 0.1924 |
| | 1 | 5.847 | 0.1794 |
| | 2 | 5.274 | 0.1628 |
| GT | 1 | 0.056 | 0.0017 |
| PT1 | 1 | 0.008 | 0.0014 |

the background concentrations in the empty chamber were either undetected or trace amounts, C_0 was ignored. The flow rates (Q) were 0.025, 0.05, and 0.1 m³/h for the ACHs of 0.5, 1, and 2 h⁻¹, respectively.

The calculated emission factors and emission rates are presented in Table 2. The BT1 exhibited a much higher emission factor and emission rate, as compared to GT or PT1. The emission factor was nearly 100 times higher for the ball MR than for the gel or the paper MR. This finding helps consumers in selecting safer MRs to minimize any potential health effects from the inhalation of naphthalene.

Table 2 also presents the emission rates and emission factors estimated for BT1, under three ACH conditions (0.5, 1, and 2 h⁻¹). For evaporation-associated household emission sources, the emission factor of a given compound is influenced by the mass transfer coefficient (Zhu *et al.*, 1999). In general, the higher the velocity and the level of turbulence were, the greater the mass transfer coefficient became (ASTM, 1998), thereby elevating the emission factor or emission rate of the compound. Although greater velocity and turbulence can cause higher evaporation or sublimation from indoor emission sources, higher ventilation rate can also occur, thereby lowering indoor air levels of emitted compounds. Regarding BT1, a low ACH level result in a high emission rate and emission factor became. This result suggests that the ventilation effect which decreases the emission concentrations under high ACHs exceeded the mass transfer effect which increases the emission rates under high ACHs on the emission rate of the compounds in the chamber. Meanwhile, it is noteworthy that considering the typical storage of mothballs or other MRs in clothing chests where the air turbulence is generally lower than other open spaces, the emission factors and emission rates presently obtained would be overestimated. Other parameters such as room temperature, and relative humidity would also be important for the emission of naphthalene from MR.

3 Conclusions

The current study evaluated the emissions of naphthalene and other compounds from several different MRs or one TDB currently sold in Korea with headspace analysis and small-scale environmental chamber. The chemical composition and concentrations of the total VOCs varied broadly with products or product types. Naphthalene was either the most- or the second-highest abundant compound for ball products. A quasi-steady state condition was achieved for naphthalene concentrations at certain times

after initiating the chamber test. The emission factor was substantially higher for a ball MR than for a gel or a paper MR. This finding helps consumers in selecting safer MRs to minimize any potential health effects from the inhalation of naphthalene.

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