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Surface Sampling for a Pesticide in Dust and Small Spills of a Solid Dye

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The aim was to determine whether a published sampling technique for loose soil on hard surfaces achieved acceptable efficiency at surface dust/soil coverages of 10–20 mg/100 cm^2. The sampler was a cordless personal sampling pump operated at 4.0 L/min connected to a cassette containing a filter, the cassette being also connected to a Tygon sampling probe that was moved manually on the surface to be sampled. Rhodamine 6G dye dust was evaluated at 10 mg/100 cm^2 coverage at flow rates of 1.0–4.0 L/min. Two National Institute of Standards and Technology (NIST) standard reference materials (SRMs) were used for pesticide experiments: SRM 2711 Montana Soil, and SRM 1649a Urban Dust. The efficiencies for these SRMs were determined uncoated, and coated with 1300 μg chlorpyrifos/g as a Lorsban 4E emulsifiable concentrate formulation. The 3-pass technique sampled both the pesticide-coated and uncoated dust and soil at >79 percent efficiency and at better than 16 percent coefficient of variation (CV) above 15 mg collected mass. Sampling at 20 mg/100 cm^2 coverage lowered the CV to ≤ 7.1 percent. The dye was sampled with similar efficiency ≥ 1.5 L/min, but the CV for the 3-pass technique was <10 percent at 1.5–2.0 L/min, and <20 percent at 1.5–4.0 L/min. The efficiency for a 5-pass technique for the dye exceeded 90 percent at ≥ 1.5 L/min with CVs <10 percent at 1.5, 2.0, 2.5, and 3.5 L/min. The weight change of the sampling probe and the cassette must be measured for accurate determination of low surface coverages rather than just the weight change of the filter alone. The method allows use of personal sampling equipment for aerosols to measure low coverages of loose dust, soil, and chemical solids on hard surfaces.

Keywords Pesticide, Surface Coverage, Chlorpyrifos, Spill, Dust, Soil

Most pure low-volatile pesticides are solid at 25°C and 760 mm Hg.[1] Such pesticides include the major chemical classes of organophosphates, organocarbamates, organochlorines, and inorganics.[1] Since application by using the pure solid pesticide is hazardous, difficult, and uneconomic, commercial pesticides are most often applied over large areas as aerosol sprays using water carrier at the appropriate dilution. The pesticide formulation therefore often has a surfactant to promote pesticide water solubility and a petroleum fraction to modulate droplet size and surface coverage.[1] Such pesticide formulations are called emulsifiable concentrates. The formulation components without the pesticide are the "inert components." Thus a field and workplace application of a pesticide usually involves a complex mixture of chemicals besides the pesticide active ingredient.

After field application in such diverse areas as farms, silos, orchards, parks, gardens, restaurants, golf courses, washrooms, retail stores, and offices, the volatile water carrier evaporates to leave an oily surface film which in soils and in dusty workplaces adsorbs to the outside of dust or soil particles. These particles can then be resuspended by wind, air blasts, and turbulent eddies from moving vehicles, pets, and people to cause exposure by inhalation, oral absorption, and skin exposure.

Many studies have monitored soil pesticide residues.[2–12] The half lives of low-volatile pesticides are often short in the ambient soil environment because of biodegradation (especially in moist, nutrient-rich soils), photodegradation by sunlight, hydrolysis, and water leaching. Conditions on dry, hard surfaces out of sunlight are much more conducive to adsorbed species on dusts being persistent. Most research on such situations has focussed on measuring risk to children via pesticides in room dust [5,8,12–27] rather than to adults, but some worker studies have been done.[29–32]

One of the authors in 1985 developed a general method to sample dust available to children,[33] focusing on lead in dust in a particle size range characteristic of clean room dust (<149 μm). The method consisted of repetitive sampling of a surface area (defined within a template) by a portable cordless pump connected to a cassette with a 0.8 μm mixed cellulose ester filter, and with a 5-cm Tygon or stainless steel sampling appendage

operated at 2.5 L/min. The method eventually became ASTM Method PS 46-96.[34] The objective was not to measure total dust on the surface, but, rather, the dust that was available to children.

To assess potential dust resuspendability in air, the total amount of dust in defined particle size ranges must be known. This aim was accomplished for loose soil by using a higher flow rate of 4.0 L/min, obtaining a better understanding of the ergonomics of the sampling process, while still retaining the cordless advantage.[35] Soil particle sizes of 63–180 μm were sampled with efficiencies between 83–85 percent (coefficient of variation CV = 11%). The technique performed better for a rough surface than a smooth one of the same surface material for the first two passes, but the type of surface made no statistical difference at $p \leq 0.05$ by the end of the third pass. The method was validated over the soil coverage range 100–1500 $\mu g/cm^2$ or 10–150 mg/100 cm^2 using a 100-cm^2 template. The sampling of polydisperse surface soil particles was more efficient than expected from the efficiencies for monodisperse particles.[35]

The new technique has not been demonstrated to apply for all chemical species of interest, nor for dust, or for small spills of solid chemicals. The present work demonstrates how the new technique performed for a model pesticide adsorbed on soil and dust, and for small spills of a model dye.

METHODS

Rationale for the Selected Pesticide

The organic pesticide that has been detected in dusts inside homes in 25 percent of the situations sampled is the chlorinated organophosphate chlorpyrifos (Dursban, Lorsban).[22] The maximum literature levels are: a concentration of 1300 $\mu g/g$ in soil or dust[22]; a surface dust/soil coverage of 6.4 $\mu g/m^2$[5]; and a surface deposition pad coverage of 990 $\mu g/m^2$.[26] Chlorpyrifos has been used for structural, crack, crevice, and outdoor turf and perimeter treatments for the control of pests in the urban environment.[26] The compound has been the focus of attempts to assess if wipe sampling can provide estimates of skin exposures.[36–38]

Chlorpyrifos is O,O-diethyl-3,5,6-trichloro-2-pyridyl phosphorothioate, and was first patented in 1966 by the Dow Chemical Company.[39] Its melting point is 43°C, and at 25°C it has a vapor pressure of 2.5 mPa and a water solubility of 2 mg/L.[40] It is relatively persistent in soils with a $t_{0.5}$ of 60 to 120 days.[40]

The 1999 air threshold limit value (TLV®) is 0.2 mg/m^3, with a "skin" notation.[41] The pure chemical is not genotoxic, teratogenic, or mutagenic unlike some of its formulations and technical grades, which may contain the more genotoxic sulfotepp and trichloropyridinol impurities. On June 8, 2000, the Environmental Protection Agency (EPA) banned uses in areas where children could be exposed.

Use of a liquid formulation in spraying, rather than a pure solid pesticide, is expected to cause a difference in sampling parameters due to greater stickiness and tackiness of the contaminated soil and dust relative to without pesticide formulation. The part of the formulation that persists after volatilization of the water carrier and the lower boiling fraction of the formulation is the pesticide plus the surfactant in a thin film of the high boiling compounds of the petroleum fraction. The sampling situation which would cause the lowest sampling efficiency would be just after spraying since the dust would be damp. However, most people do not come willingly into freshly sprayed rooms or fields because of odors and wet surfaces, and are generally not exposed to this situation.

Thus, the practical situation that would be the worst sampling situation relative to exposure is after a reentry interval, when a site is reoccupied the next morning after overnight spraying operations (as is common for restaurants), or when odors cease. This situation was simulated by loading the dust or soil at the highest surface coverage and/or concentration observed so far in field studies, that is, 990 $\mu g/m^2$[26] or 1300 $\mu g/g$.[22] The 990 $\mu g/m^2$ coverage corresponds to 990 $\mu g/g$, assuming 10 mg is sampled from 100 cm^2. Thus, the concentration maximum of 1300 $\mu g/g$ is the worst case scenario that might influence practical sampling after reentry. This loading concentration was therefore selected for investigation with a Lorsban 4E formulation containing 40.7 percent (w/w) active ingredient,[38] as obtained from Dow Elanco.

Thus, 10 g samples of each soil and dust were coated uniformly by first solubilizing 32 mg of the formulation in 20 mL of acetonitrile, adding the solution to the soil or dust in a round-bottom flask with a ground glass joint, and then evaporating the solvent by rotary evaporation. The solid residue was held in a vacuum desiccator until constant weight (2–3 days). Gas chromatography/mass spectrometry (GC/MS) of the Lorsban 4E formulation was done for quality control purposes using conditions published elsewhere.[38]

Rationale for Solid Pure Chemical to Be Used

Many dry solid chemicals are often spilled in small amounts, for example, drugs in pharmacies, chemicals in laboratories, waste materials by technicians, powders by workers, and food ingredients in the home. The solid organic chemical chosen was to be as nontoxic as possible for health and safety reasons. Thus, pesticides, drugs, and irritant chemicals were not considered. Rhodamine 6G or basic red 1 dye (99% w/w) was chosen since it is often used in tunable dye lasers, for example, for laser eye and other surgery,[42,43] for fluorescent labeling purposes in biochemistry,[44] as a fluorescent tracer in environmental studies,[45] and also to dye silk, cotton, wool, leather, plastic, paper, and bast fiber.[45] This xanthene dye, 2-(6-ethylamino)-3-(ethylimino)-2,7-dimethyl-3H-xanthen-9-yl)benzoic acid ethyl ester monohydrochloride, is moderately soluble in water, and has a low vapor pressure because it is a salt. Even so, it has measurable volatilization at 200°C.[45]

There are no workplace guidelines. The chemical is in the EPA inventory under the Toxic Substances Control Act (TSCA). There is no evidence that it is carcinogenic in two-year studies

in mice of either gender, but there is some equivocal evidence in rats of either gender relative to site-specific tumor induction in the adrenal gland medulla (pheochromocytomas) at the highest dose of 250 ppm in the diet.[45] The chemical tends to cling to surfaces since it is slightly deliquescent, and it is hard to remove from hands once they are contaminated. There was a need to develop a sampling/cleaning method that involved minimum skin exposure.

Rationale for Dusts and Soils Selected

The previous optimization study[35] utilized fractions of sieved local soil for method development purposes. Transfer of technology is best done with standard reference materials (SRMs), for example, from the National Institute for Standards and Technology (NIST).[46] NIST SRM 2711 is Montana II till soil of particle size <74 μm of about 1.5–2.2 percent water and about 2 percent carbon content with 24 elements defined.[47] SRM 1649a is an Urban Dust atmospheric particulate of particle size <125 μm. The volume distribution has an arithmetic mean particle diameter in μm of 34.6 ° 0.4, and the median is 24 μm. It has water content of 1.23 ° 0.07 percent and a carbon content of 16 ° 5 percent. It is certified at the ng/g level for 22 polyaromatic hydrocarbons (PAHs), 35 polychlorinated biphenyl (PCB) congeners, and 8 chlorinated pesticides. Reference values for another 22 PAHs, another chlorinated pesticide, 32 inorganic constituents, total organic carbon, and carbon composition are also provided.[48]

These SRMs were sieved to check particle sizes Labline Instruments (Dubuque, IA, Minisieves, Catalog No. 4610), and also resieved after coating with the chlorpyrifos formulation since particle agglomeration was expected.

Sampling Method

The components of the surface sampling apparatus were a portable, cordless pump of 4.0 L/min flow rate (SKC, Eighty-Four, PA, Airchek Model 224), connected by 6.4 mm ID Tygon tubing to a 37-mm polystyrene sampling cassette with a mixed cellulose ester filter of 0.80 μm pore size or a Teflon filter of 5.0 μm pore size. The entrance port of the cassette had a sampling probe of Tygon tubing of 6.4 mm I.D. × 5.0 cm length with the sampling end cut at a 45° angle.[35] A 10 cm × 10 cm sampling template (SKC West, Fullerton, CA) was taped with masking tape to the sampling surface at the inner margin of the template. The sample was applied over the surface by pouring it through a sieve of size 180 μm that was moved slowly about 3 cm above the template surface. Although the 100-cm^2 smooth surface was not covered uniformly, the applied mass was accurately known and was varied between 10 mg to 20 mg. A smooth surface used previously[35] was utilized as it produced the lowest dust recoveries.[35]

The sampling technique involved up to five passes over the sampling area proceeding from the template inner margin into the center as described elsewhere.[35] The sampling probe was tilted as appropriate, with regular inversion accompanied by finger tapping to ensure no blockages and to facilitate dust collection on the filter. Weights were determined on a four-decimal place balance by subtracting cassette/sampling probe weight before sampling from that after sampling, as well as weighing each component part of the sampling train before and after sampling. All experiments were done at least in triplicate.

RESULTS

Chlorpyrifos

The results for the surface sampling of coated and uncoated SRM 2711 and SRM 1649a at 4.0 L/min for up to three sampling passes are provided in Table I. The uncoated SRM 2711 soil at 10.5 mg/100 cm^2 coverage was sampled at better than 90 percent efficiency and CV 5.3 percent by even one sampling pass. The coated soil showed collection efficiencies for 3 sampling passes of >80% at ≥ 15 mg/100 cm^2 for coated soil, whereas 10 mg/100 cm^2 uncoated soil and 20 mg/100 cm^2 of coated soil were sampled at >90 percent efficiency. At 9.9 mg/100 cm^2 coated soil, the 3-pass efficiency was 59 percent with CV 27 percent, being inefficient and imprecise. The proportion of coated soil in the sampling probe and in the cassette was about 1:1 at about 10 mg/100 cm^2. The cassette always contained >50 percent beyond 15 mg/100 cm^2 for the coated soil.

The sampling efficiencies of the 3-pass technique for 20 mg/100 cm^2 and 15 mg/100 cm^2 SRM 1649a dust were not statistically different from each other at $p \leq 0.05$ within the coated and uncoated classifications. Both classifications also

TABLE I

Efficiency of sampling coated and uncoated Montana soil (SRM 2711) and urban dust (SRM 1649a) using different surface loadings over the 100-cm^2 smooth surface and a three-pass sampling technique at 4.0 L/min

			Cumulative efficiency[B] for pass		
SRM	Coated	Loading[A]	1	2	3
2711	–	10.5(0.6)	94(5.0)	92(11)	93(10)
	+	9.9(0.5)			59(16)
		15.3(0.4)	83(9)	85(7)	80(13)
		16.8(0.3)	82(1)	80(2)	79(4)
		19.6(0.6)	91(5)	90(5)	90(5)
1649a	–	10.2(0.6)	72(9)	72(3)	70(4)
		15.3(1.0)	73(8)	76(5)	79(9)
		20.5(1.1)	77(6)	78(6)	79(5)
	+	10.2(1.1)	91(3)	92(2)	93(3)
		15.0(0.5)	94(5)	97(4)	94(4)
		21.4(0.3)	86(5)	86(5)	84(6)

The quantities in parentheses are standard deviations of triplicate measurements.

[A]mg/100 cm^2.

[B]in percent.

had CVs <11 percent at these coverages. However, the coated dust was sampled significantly more efficiently than uncoated dust at $p \leq 0.05$ at all coverages, though sampling efficiencies still exceeded 70 percent at all coverages whether the dust was coated or not. Coating the dust improved recoveries at lower coverages, the reverse of the findings for soil. The uncoated particles passed completely through a 230 mesh sieve (<62 μm). Only 95 percent of the coated particles came through the 230 mesh sieve, but all passed through a 170 mesh sieve (<84 μm). Thus, the small increase in particle size caused by coating may help increase sampling efficiency, a phenomenon also observed for sampling sieved local soil.[35]

Dust and soil, coated or uncoated, were sampled by the 3-pass technique with an efficiency above 79 percent at 15 mg/100 cm^2 or above with CV ≤ 16 percent Thus, 15 mg/100 cm^2 coverage rather than 10 mg/100 cm^2 is nearer the least quantifiable limit [CV of 10.6%, preferred inaccuracy of ° 10%, and minimum inaccuracy of ° 25% according to NIOSH [51]] for these coated and uncoated dusts and soils.

Sampling of Rhodamine 6G

Since this system was so different relative to coated and uncoated dusts and soils, a complete flow rate dependence study was performed at 10 mg/100 cm^2 (Table II).

Evaluation of different flow rates using 5-sampling passes over a surface coverage of 10 mg/cm^2 showed that flow rates ≥ 1.5 L/min were necessary for sampling efficiencies of >90 percent. The corresponding efficiencies for three sampling passes were not statistically significant from those for five passes at $p \leq 0.05$. For flow rates ≤ 2 L/min, all the collected sample resided in the sampling probe. Even at 4.0 L/min flow rate, a substantial proportion (about 20%) of the sampled mass did not reach the filter cassette.

Residual color showed that a small amount of sample (generally <1%) still remained on the surface. This residue could be removed by wet swabs (water solvent) using at least three fresh swabs per single sampling pass for a total of 3 sampling passes. The dye on each swab was desorbed by ultrasonication in 1 mL of water. Absorbances were read at 528 nm in a 1-cm Suprasil cuvet. The linear range was 1.0–10 μg dye/mL. The measured molar absorptivity was 82,727 ° 200 M^{-1} cm^{-1} [literature, >81,400 M^{-1} cm^{-1}[49]].

DISCUSSION

There are no previous reports on sampling efficiencies for spilled Rhodamine 6G dye, or for pesticide-containing dusts on hard surfaces, for the cordless vacuum technique. The results for the dye in Table II showed that the whole cassette and sampling probe rather than just the material on the filter had to be weighed before and after sampling at 10 mg/100 cm^2 coverage to determine the surface coverage accurately. Thus, while the mass collected on the filter could be analyzed to determine analyte concentration in the dust/dirt, the surface coverage could not be assessed accurately from weights collected on the filter or cassette, even when the proportion collected there increased with increasing flow rate. The use of filter mass changes alone leads to a bias towards low coverages. The slight deliquescence of Rhodamine 6G is probably responsible for the greater imprecision observed for the 5-pass sampling technique versus the 3-pass technique, and observed for high flow relative to the lower flow rates for 1.5–4.0 L/min.

All uncoated and coated particles used in the present study had sizes well below the 180 μm upper diameter limit of the optimized method validated with local soil.[35] In the latter study, the mass of soil retained in an unsaturated probe was consistently about 0.7 mg. This implied that 12 mg or more should be collected to define the method's least quantifiable limit. The present data are in good agreement. Use of a presaturated probe lowers this limit, but then cross contamination may occur. We recommend that a wide enough defined area be sampled to ensure masses ≥ 15 mg are collected with this technique to have a representative filter mass for analysis and to provide accurate data at low surface coverages. Use of a cordless balance is indicated in the field if available. In addition, if analyte specific

TABLE II
Dependence of flow rate and number of sampling passes for 10 mg of Rhodamine 6G deposited on a smooth 100 cm^2 surface

Flow rate (L/min)	Cumulative efficiency for pass					In-probe (% recovered)
	1	2	3	4	5	
1.0	58(11)	70(17)	70(21)	69(25)	64(25)	102(3)
1.5	82(9)	92(9)	91(9)	96(13)	93(9)	106(3)
2.0	79(10)	94(8)	93(5)	98(15)	94(8)	98(4)
2.5	68(14)	78(8)	85(13)	86(12)	90(5)	80(5)
3.0	96(10)	117(18)	117(23)	112(28)	118(27)	60(7)
3.5	96(20)	101(14)	102(11)	99(6)	102(1)	32(10)
4.0	89(13)	100(10)	100(15)	98(20)	96(21)	20(5)

The quantities in parentheses are standard deviations of triplicate measurements.

concentrations are also desired, a mixed cellulose ester filter is recommended for collection of inorganics and metal compounds since these filters facilitate nitric acid digestions,[33] and a Teflon filter should be used for organics since this filter material is inert and suitable for liquid/solid extractions.

Lewis et al.[52] showed that the pesticides bendiocarb, carbaryl, chlordane, chlorpyrifos, DDT, diazinon, dichlorvos, heptachlor, methyoxychlor, permethrin, *o*-phenylphenol, and propoxur as well as 10 PAHs were enriched in the fraction <106 μm relative to the 106–2000 μm fraction of carpet dust. About 60 percent of the carpet dust mass was of size <150 μm. Since carpet dust is a major reservoir of house dust on hard surfaces, the focus on particle sizes <180 μm is also justified in the present study.

The prior methods that have been utilized for hard surfaces to sample dust and soils containing pesticides have been of much greater flow rate or vacuum such as a vacuum cleaner.[8,19,22,25] These methods depend on a source of electrical power. The major aim is to clean the surface at high dust surface coverages. Such cleaners are difficult to use in low coverage situations since there is much solid holdup in the nonbag sampling path of vacuum cleaners, and a large proportion of sampled surface solids may not be collected in the collection bag for weighing. Clearly, more work is required to define the low loading collection efficiency cutoffs for vacuum cleaners on hard and carpeted surfaces. The HVS-3 vacuum system used in many recent studies for carpet and floor dust loadings[8,22,25] has been evaluated in the ASTM D5483-93 method for carpet.[50] The sampling efficiency on hard floors for low dust coverages is unknown. Another type of noncordless vacuum cleaner to sample inorganic dusts in carpets that employs a nozzle with teeth has also been reported.[53–55]

The present cordless technique does not require a local source of power, though requirements include effective chargers, battery packs, an accurate mass balance, pre- and post-sampling pump calibration, and a performance-based validation of the sampling technique of any human operator.[35] Nevertheless, the present technique is more flexible than techniques dependent on plug-in power sources, and can perform acceptably in situations of low dust coverage, a situation where vacuum cleaners may not be suited for dual use as cleaners and to measure low surface coverage. Low dust coverages can be measured by the present cordless vacuum technique because the area of exposure to dust in the dust collection path is minimal. A stainless steel sample sampling probe as used originally in 1985[33] may further minimize sampling probe holdup at low dust coverage but at more expense. In addition, the same pumps used to take personal aerosol samples can be used for surface dust monitoring: industrial hygienists are already familiar with pump use and calibration. The application of the optimized method for lead and other inorganic chemical species relative to other methods is discussed elsewhere.[35] Further studies to increase confidence in its general applicability would be helpful.

CONCLUSIONS

At least 15 mg of sample must be collected with use of the appropriate filter (mixed cellulose ester of pore size 0.8 μm for inorganics and a 5.0 μm Teflon filter for organics) and by 3–5 sampling passes. The probe and cassette must be preweighed and these components reweighed after sampling to obtain accurate low surface coverages of 10–20 mg/100 cm^2. Use of a cordless field balance is recommended. Sufficient mass must be accumulated on the filter to allow the filter analysis to be representative of the sample.

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