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Methods of Removal of Pesticide Residues

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RESIDUES from the agricultural use of some pesticides persist for extended periods before becoming completely degraded by natural processes. These long-lived compounds migrate far afield by cycling into plants and animals, and into air, water and soil systems. The extensive production and use of such pesticides on one hand and the economic problems and health hazards of persistent residues on the other, raise the pertinent question: If persisting residues are inevitable, what are the prospects for their removal from foods and other critical sectors? That question is examined in this paper by means of a brief survey of present information on paths to pesticide removals from foods, animals, soils and water.

PROPERTIES OF RESIDUES EXPEDIENT TO THEIR REMOVAL

The persistence of a pesticide residue is a complex matter affected not only by the chemical and physical characteristics of the parent compound and its degradation products, but also by the nature of the formulation applied, the adsorbents or solvents employed, and so on.[1]

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The persistence may also depend to a large degree on characteristics of the host. The waxy surfaces of plants tend to localize and trap many pesticides which thereby become more resistant to removal than they would as true surface residues. Similarly, the fats in milk hold residues during the manufacture of various dairy products. Such localization, however, may often be an advantage. For example, fruits and vegetables with easily peeled skins are more amenable to pesticide removal than are green leafy vegetables.

Its volatility, stability under ultraviolet radiation or the hydrolytic action of acids and alkalis, and relative tendency to bind to or dissolve in plant constituents are important chemical properties of the pesticide that influence residue persistency and therefore ease of removal. Removal procedures must be designed to capitalize on one or more of such properties while maintaining the integrity of the host. Flushing surface residues with water is probably the mildest method that may be effective, while vapour removal by co-distillation with water or some other solvent is extremely effective but generally too severe for many situations. It should be pointed out that these two processes, as well as most other methods, remove pesticides only by physical transfer, thus eventually contributing to the pesticide burdens in air, water and soil.

REMOVALS FROM FOODS

In discussing foods, it is convenient to categorize removal of pesticides as either adventitious or intentional. Adventitious is used here to designate any loss of residue occurring as a by-product of normal processing procedures. This is not to say that such removal is unanticipated, but merely that it is incidental to the purpose of the processing method. The philosophy by which tolerances for pesticide residues on raw agricultural commodities are established in the United States, under the Food, Drug, and Cosmetic Act, expresses an expectation that residues in foods reaching the dinner plate will be but a fraction of those in the raw commodity as a result of normal food-processing methods. As will be illustrated later, washing, peeling, cooking and canning may substantially reduce pesticide residues. Such adventitious removals are therefore not only anticipated, but relied upon, to provide a substantial margin of safety for the food consumer over and beyond the established safe tolerance.

Intentional removal of pesticide residues necessitates processing techniques deliberately designed to remove pesticides and their degradation products. Such techniques would not normally be employed in food processing because they do not enhance the food value. Theoretically feasible methods are available for a number of situations, but, except for a few cases, little serious attention has been given to evaluating potentially effective processes. No doubt the development has been minimal because treatments presently available are too destructive of food quality. Under existing practices of pesticide usage and regulatory controls it is unlikely that intentional removals would be economically advantageous except, perhaps, for the salvage of crops found to contain unlawful residues. Even salvage could probably not be justified unless excessive residues should become unavoidably frequent in major crops. Intentional salvage processing of animal feeds might be justified, however, because of the frequency of residue problems (particularly in dairying), and the relative indestructibility of animal feeds in terms of quality. Indeed, some promising procedures are already being developed. Animal feed processing is also free of what may be another discouragement to intentional decontamination of foods destined for human consumption, namely, the philosophy that underlies the regulation of pesticide residues in raw agricultural commodities. This philosophy calls for identifying food commodities that have excessive residues at their production sites, and a subsequent barring from marketing outlets. In the United States, processing to remove unlawful residues from food crops would be permitted only after petitioning the FDA. That agency would then require such reconditioning to be done under its supervision. It seems obvious that approval would be granted only in unusual cases and that processing for intentional removal would not receive general approval.

FRUITS AND VEGETABLES

Ever since synthetic organic pesticides were first used in agriculture, investigators have been concerned with the fate of the residues during normal food processing. Washing, peeling and cooking were soon recognized as important, but not necessarily reliable, means of reducing residue levels in fruits and vegetables.[2]

Adventitious removals by various unit processes in the food preparation industry and by home preparation of foods have been carefully studied in several recent investigations. Many of these were carried out by the National Canners Association (NCA) in the United States, as well as by various state agricultural experiment stations.

Washing and Blanching

Loosely held residues of several pesticides on various fruits and vegetables are removed with reasonable efficiency by types of washing processes normally used in home or commercial preparation (Table I). This was the characteristic fate of residues of DDT applied as wettable powder to tomatoes where cold water washing by commercial techniques removed approximately 90% and home-type washing some 78% of the total DDT residues.[3] Residual DDT on spinach, which is more closely associated with waxes, was removed less well, even by warm water washing. This removal was considerably improved by adding detergent to the wash water.[3] Detergents also improved the removal of parathion from spinach,[3] carbaryl from tomatoes,[3] DDT from potatoes,[3] and carbaryl and DDT from apricots.[4] However, in some cases detergent washing was decidedly less effective than plain water washing.

Washing was ineffective in removing DDT residues from market samples of green beans.[5] This was attributed to the residues being in the inner tissue of the bean and not on the surface. The deeper location may have been related to the formulation used and/or to "field weathering" of the original DDT deposit, which could greatly reduce the surface deposits.

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TABLE I.—PESTICIDE REMOVAL FROM RAW VEGETABLES DURING COMMERCIAL AND HOME PROCESSING, AS PERCENTAGE LOSS (WET BASIS)*

TOMATOES

		Unwashed	*Washed*	*Wash+ detergent*	*Peeled*	*Stewed*	*Canned juice*
		ppm	−%	−%	−%	−%	−%
DDT	Commercial..	7.7	89	85	99+		99+
	Home........	4.4	78		99+		99+
		3.7				85	
Carbaryl	Commercial..	5.2	83	96			98
	Home........	8.4	77		92	69	77
Malathion	Commercial..	15.9	91	83			99+

GREEN BEANS

		Unwashed	*Washed, trimmed*	*Blanched 3 min.*	*Boiled 12 min.*	*Canned*
		ppm	−%	−%	−%	−%
DDT	Commercial..	4.08		50		95
	Home........	0.67	9		46	
		12.70	71	80		89
Carbaryl	Commercial..	11.0	52	81		
	Home........	8.0	41		77	
Malathion	Commercial..	1.12	96	99+		99+

SPINACH

		Unwashed	*Washed*	*Wash+ detergent*	*Blanched*	*Canned*
		ppm	−%	−%	−%	−%
DDT	Commercial..	27.3	48	73	55	95
	Home........	20.2	47		69	55 (cooked)
Parathion	Commercial..	1.5	47	57	66	83
	Home........	1.7	57		35	29 (cooked)

*From National Canners Association[3] and Hemphill *et al.*[5]

Blanching effectively removes certain residues, although it may not remove much more than will a thorough prewashing. In the NCA studies, blanching of green beans removed substantial portions of DDT, carbaryl and malathion; water blanching was generally superior to steam.[3]

Peeling

Peeling (and trimming) obviously reduce residues. Compounds that penetrate the epidermis are not completely removed by washing, and peeling may completely remove the remaining residue. The recent NCA studies showed peeling to be very effective in the removal of carbaryl, malathion and DDT from tomatoes, and of DDT from potatoes.[3] Similar observations have been made with other vegetables and fruits.[2]

Cooking

The effects of cooking on pesticide residues are extremely varied, but may contribute greatly to residue removal. DDT partially converts to DDD (a less hazardous compound) during canning of several fruits and vegetables.[3, 5] Metal ions, especially iron, are involved in the conversion, which does not occur when the canning is done in glass.

The NCA studies reported substantial loss of malathion during canning of tomatoes and green beans, but such cooking loss was not apparent for carbaryl in green beans or parathion in spinach and broccoli.[3] Major reductions in residues of other compounds have been reported to be effected by cooking.[2]

No valid generalizations can be drawn from this short review except that food preparative methods apparently do appreciably reduce pesticide residues. The NCA study involving DDT, malathion, parathion and carbaryl residues in five different vegetables[3] concluded with the opinion that normal processing satisfactorily removed all these pesticides, except parathion, from every commodity. The total diet studies of the Food and Drug Administration (United States) provide evidence that the residues being contributed to the diet from normally processed foods are a fraction of levels predicted on the basis of foods bearing residues at tolerance values.[6] Adventitious removal of residues during food processing and preparation certainly contributes to that important difference.

VEGETABLE OILS

Commercial processing of oils may adventitiously reduce chlorinated pesticide residue levels to below detection limits.[7, 8] The key process is in deodorization of the oil by steam stripping under high temperature—low pressure conditions; this amounts to a forced volatilization of the pesticide contaminants. Virtually all the commonly used organochlorine pesticides have been removed from vegetable oils in this manner. The extent to which this type of processing is employed is not known, but monitoring data show that "refined" vegetable oils have substantially lower average residue levels than do crude oils.[9]

DAIRY AND OTHER ANIMAL PRODUCTS

Surveys have consistently shown this food group to be the major source of pesticide residues in human diets.[6, 10] The residues in animal products are concentrated in fats and fatty tissues, and adventitious decontamination occurs whenever fat is removed during cooking or other processing.

It was established quite early that processing raw milk into various products, including pasteurized milk, cream, butter and cheese, distributed DDT according to the fat content, with its concentration remaining fairly constant in the fat.[11] This generalization has since been verified in several studies with other organochlorine pesticides (Telodrin, DDT, lindane, aldrin, dieldrin, endrin).[12, 13] Subsequent research, however, showed that milk-drying processes at elevated temperatures remove substantial amounts of some persistent organochlorine insecticides.[12-15] These removals are primarily the result of co-distillation with water vapour, and it is apparent that not all compounds are suitably volatile under the conditions used. In contrast to dried milk, evaporated milk usually contained a higher proportion of the residue, while residues in condensed milk were only slightly lower than those in raw milk. Intentional processing of milk into dried whole milk or dried non-fat milk, therefore, seems to be a feasible means of salvaging this valuable commodity in some high residue situations. Direct steam injection under a partial vacuum, a process for expelling volatile off-flavours, removed from milk only small proportions of DDT, dieldrin and heptachlor, but a considerable quantity of the more volatile lindane (Table II).[12-16] Steam deodorization under a

TABLE II.—PESTICIDE REMOVAL FROM MILK DURING MANUFACTURING PROCESSES, AS PERCENTAGES (ALL VALUES ON THE FAT BASIS)*

Pesticide	*Drum dried whole milk*	*Spray dried whole milk*	*Evaporated milk*	*Condensed milk*	*Commercial steam distillation—vacuum processing*
	—%	—%	—%	—%	—%
p,p'-DDT		63			8
Lindane	63	81			24
Dieldrin	53	56		41	3
Heptachlor	94	95		0	
Heptachlor epoxide	62	65		48	
Chlordane	50	25	45	11	
Endosulfan	50	38	39		
Telodrin	10-20	10-20	40-50		
Methoxychlor		15	19	8	

*Adapted from references 12 - 16.

high vacuum, however, completely removed dieldrin and heptachlor epoxide from butter oil.[17] The application of forced volatilization by co-distillation with water vapour is apparently as effective here as in the purification of vegetable oils.

Ultra-violet irradiation has been reported to lower some organochlorine residues in milk.[18] A single irradiation of milk that was flowing over a surface cooler reduced methoxychlor by 33% and DDT by 17%. No other organochlorine pesticides were removed to an extent greater than 10 to 11%.

With meats, the only potential for residue removal lies in trimming away fat and/or its rendering during cooking. Several studies have shown that organochlorine insecticide residues in meat (chicken) are generally present in rendered fat at approximately the same concentration as those in tissue fat.[19, 20]

Pesticide reduction in chicken during cooking was found to depend upon cooking temperature. Cooking in water at 190 to 200° F. for three hours removed about 45% of the DDT, dieldrin and heptachlor residues; lindane removal was more complete. Autoclaving for three hours at 15 psi (temperature equivalent 250° F.), however, removed over 95% of the DDT, dieldrin and lindane and 90% of the heptachlor residues.[19] Frying and baking of chicken were also evaluated for DDT and lindane removal; 50 to 75% losses were obtained.[21] Some conversion of DDT to DDD occurred with each of the cooking methods, and this increased during the higher temperature methods.[22] With other pesticides, cooking in water at 121° C. removed 63% of chlordane residues and all of the Telodrin, but Ovex residues were not affected.[20]

In contrast to these findings with chicken, cooking of beef under various conditions was not very effective in reducing DDT residues.[23] With the exception of frying and pressure cooking (35 and 50%, respectively), relatively little removal occurred. One imagines this might reflect the smaller relative loss of fat during the cooking of beef in comparison to chicken. The higher temperatures associated with frying and pressure cooking, by causing greater fat removal, may have contributed to the more substantial losses of residue associated with such methods.

It may be concluded that appreciable reduction of most organochlorine residues contributed to the diet by meats might be achieved through intensive cooking. The effectiveness depends upon whether the fat removal is substantial and whether fat drippings are used later.

REMOVAL FROM ANIMALS AND ANIMAL FEEDS

A practical means of intentionally removing pesticide residues from animals would be advantageous to the livestock industries, which have been frequently beset by problems involving persistent pesticides. The natural loss from the body of many organochlorine insecticides, for example, is a relatively slow process. As a rule, meat, milk or eggs will bear residues whenever the producing animals encounter these substances. Potential ways to change the dynamics of such compounds in animals include reducing the size of fat depots, while stimulating metabolic activity, altering the absorption of the compounds from the gut, and increasing their metabolic degradation.

Investigations have been made along each of these lines. Body fat reserves can be mobilized under the combined influence of restricted feeding and administered thyroid-stimulating substances. With the body fat utilized more rapidly, residual pesticides may be metabolized more quickly and eliminated. Removal of DDT from the tissues of hens, and indirectly from eggs, was expedited by starvation[24] or by feeding

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severely imbalanced diets.[25] Both of these methods brought about rapid weight loss. Similarly, studies have shown some acceleration of DDT and heptachlor depletion in dairy cattle on low energy rations and/or thyroprotein.[26, 27] Generally, however, the small gains in pesticide depletion rates were offset by associated unfavourable physiological effects (moult in chickens and reduced production in both species).

Reducing pesticide absorption in the digestive tract by feeding charcoal appears to be an effective and easily managed means to minimize residue storage. Recent study showed that charcoal feeding also accelerated dieldrin depletion in cattle, apparently by retarding absorption of the tissue dieldrin that is normally re-cycled into the digestive tract.[28] The potential effectiveness of this procedure seems great and it warrants more intensive study with various activated adsorbents.

Increasing depletion rates by accelerating the metabolic degradation of residual pesticides in animals also presents interesting possibilities for development. Various investigators have obtained an increased elimination of dieldrin and other organochlorine insecticides in rats treated with various additional pesticides or drugs.[29-32] This response is associated with induction of hepatic mixed-function oxidases, enzymes that are involved in the metabolic degradation of many lipoid-soluble chemicals. Excretion of dieldrin metabolites, for example, is markedly greater when rats are treated with DDT[33] or barbiturates[31] to obtain enzyme induction. Such agents can therefore be utilized both to minimize accumulation of organochlorine residues and to increase their depletion rates. In the case of dieldrin-treated rats, both phenomena occur to a very high degree (Fig. 1). Species other than rats also show this response, but it is far from certain that mammals do so generally. That point needs more investigation. Several compounds that combine low toxicity with high potency (equal or much superior to DDT or the barbiturates) have recently been studied. Some of these may have real potentials for use in the intentional removal of pesticide residues from living animals.

Minimizing exposure to pesticides and pesticide residues is the most direct and easily achieved way to control the residue levels in food products from animals. Animal feeds, which undoubtedly represent the prime source of unwanted pesticide exposure in the livestock industries, are more durable than human foods and the quality criteria for their palatability are much less stringent. Feeds, therefore, could be subjected to relatively severe processes for intentional removal of residues without undue losses in value.

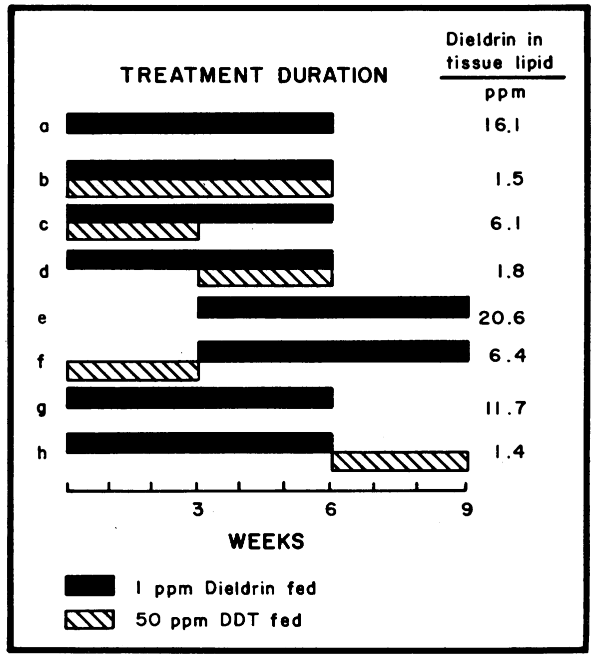

Fig. 1.—Effects of time and duration of DDT administration on the storage dieldrin in rat adipose tissue. Values listed are means for groups of five rats. All DDT treatments caused highly significant reductions in dieldrin storage. The DDT effect persisted for at least three weeks after discontinuing DDT treatment, but was definitely weakened (cf. a, b and c). DDT treatment for the final three weeks of a six-week dieldrin exposure was as effective as DDT given continuously (b and d). Pretreatment with DDT for three weeks was effective throughout the following six weeks of dieldrin exposure (e and f). Administration of DDT after the dieldrin treatment was highly effective in reducing residual dieldrin in fat (g and h). The rats in groups a, b, c and d were sacrificed at the end of the sixth week and those in groups e, f, g and h at the end of the ninth week.[34]

Dehydration in a rapid high-temperature dryer is a common means of processing forage, especially alfalfa. Early literature recorded substantial removal of malathion,[35] parathion and aldrin, chlordane and toxaphene[36] during dehydration. The proportions removed varied from 60 to over 80%, which seems remarkable in view of the high residue levels that were then being studied (45 to >200 ppm on the dry basis).

Recent studies have shown that the principle holds also at low-residue levels (under 1 ppm). Both DDT and heptachlor residues in alfalfa were reduced by over 50% during dehydration in commercial equipment.[37, 38] Saturating low-moisture hay with water and then dehydrating it was also highly effective and 86% of the total DDT present was removed. This is another example of the utility of co-distillation with water vapour for pesticide removal.

Recent studies at the University of California have shown that low DDT residues in alfalfa hay could be almost completely removed by "washing" with hot vapours of several different organic solvents; efficiencies ranged from 73 to 97%.[37] An interesting contrast is the observation that water vapour was itself very effective and removed over 85% of the total residue. Similar results were obtained with residual endrin.[39] It was suggested that artificial dehydration processes, particularly if preceded by steam vapour treatment or organic solvent washing, might have practical applications in cleaning hays and other forages. Such processing might be especially advantageous if applied to the "by-product" feedstuffs derived from processing vegetables, sugar beets and various fruits. These peels, pulps and pomaces frequently contain significant residues of the more persistent pesticides, which are then passed on to the animal.

At least one intentional removal has been worked out based upon chemical destruction of the pesticide.[40] Seeds treated with compounds such as Difolatan may be salvaged for animal feeding by treatment with an aqueous solution of an ionizable sulfide, inorganic sulfite or thiosulfite. Effectiveness is claimed for removing residues of pesticides containing halogen on a saturated carbon, which is α or β to an S or O atom (as in Difolatan and Captan), and those having a methylphosphate group. Applications other than seed salvage may be possible using this principle.

REMOVAL FROM WATER AND SOILS

The earth's soils and waters are the ultimate repositories for most of the persisting pesticide residues. In both cases, associations with organic matter or adsorptive surfaces are important in holding the pesticides.

Leaching and sediment transport by water redistribute soil pesticides and may, in a local sense, represent removal. Vaporization, degradation by soil organisms or inorganic reactants, and uptake by plants are also important in soil pesticide dynamics. Recently instituted massive research and monitoring programs are now beginning to provide reliable data that define the magnitude of these various processes as they occur under diverse conditions of soil types and climates. This important information is providing few leads, however, that might be exploited for intentional removal of pesticides from soils.

In general, the persisting pesticides are degraded more rapidly under conditions favouring high microbial activity. This activity can be enhanced by manures or cover crops, which provide readily available energy sources.[41] The organic matter must be mixed into the soil by suitable cultivation, however, since plant matter on the soil surface appears to reduce volatilization and hold residues in the soil environment.[42] No doubt some balance in soil organic matter is optimal for pesticide degradation. Persistence is greater in soils with excessive organic matter, as in muck soils.[43] Additional methods of stimulating soil microbial activity should be actively sought. Anaerobic biodegradation of DDT in soil was rapid in controlled experiments.[44] However, field application of this finding would require establishing suitable anaerobic conditions, perhaps by flooding.[45]

Other practices[41] that might facilitate removal of residues in soil include:

(a) Frequent cultivation. This might improve volatilization and photodecomposition.

(b) Cropping with plants that accumulate the pesticide. This, of course, merely transfers the problem to a different sector, but might help to salvage fields needed for special uses.

(c) Addition of adsorbents such as activated carbon to soil to lower the availability of the pesticide residues. The technique has been successfully used to eliminate phytotoxic effects of residual herbicides and should be thoroughly investigated with other pesticides. This would not constitute removal, however, and the pesticide's total residence time in the soil would be lengthened.

(d) Intensified leaching, perhaps with facilitating additives in the water.

Some of these methods may become valuable aids in rehabilitating soils in small areas that support intensive agriculture. None of them, however, greatly improve the outlook for intentional removal of pesticides from soils on a large scale.

Water could conceivably be cleaned up by procedures for removal of the sediments carrying adsorbed pesticides and by use of activated media to adsorb the free compounds.[2, 46, 47] Sedimentation processes already employed in purification of municipal waters undoubtedly serve this function to some degree. Activated carbon will remove organochlorine pesticides from water, but large quantities of carbon are required to achieve maximum removals.[2, 48] The oxidants, ozone and potassium permanganate, have not produced promising results relative to removal of organochlorine residues.[48] Biological trapping of residues has also been suggested for water purification.[2] This probably occurs in sewage lagoons through the growth of algae, but the algae would have to be harvested and destroyed elsewhere in order to achieve actual removal. Ion-exchange resins could be used to reduce the content of ionic pesticides, while solvent extraction techniques could remove nonionic compounds. All of these processes require extensive engineering and chemical evaluation to be properly judged.

Summary Available information on paths to pesticide removals from foods, animals, soils and water is reviewed in assessing the prospects that effective removal of persistent residues may succeed. Conclusions are that: (a) The normally low and legally regulated residues in raw agricultural commodities are reduced during many of the steps associated with preparation for consumption. These adventitious removals appear to be substantial for many pesticides in fruits, vegetables and vegetable oils, but dairy and other animal products are little influenced. Effective procedures for intentional removals have not been developed. (b) Methods to minimize residues in animal products by cleaning animal feeds and by altering the dynamics of pesticide residues within the animal are being probed with promising results. Their practical values, however, have not been assessed. (c) No generally effective methods to clean up the soils and water are known, and little attention is being given to their development. It is obvious that cleaning such pesticide repositories, even on a small local scale, would prove immensely difficult.

Résumé L'auteur passe en revue tous les moyens actuellement connus pour débarrasser les aliments, les animaux, le sol et l'eau de pesticides persistants et se demande s'il est possible d'obtenir un succès complet. Conclusions: (a) les résidus minimes et légalement contrôlés qui se trouvent sur les produits agricoles sont encore réduits au cours de la préparation des aliments pour le consommateur. Cette diminution paraît importante dans les fruits, les légumes et les graisses végétales mais elle est pratiquement nulle dans les produits laitiers et animaux. Aucune méthode pour enlever efficacement les résidus de pesticides n'a encore été mise au point. (b) Des méthodes visant à diminuer la teneur en pesticides des produits animaux sont à l'étude; il s'agit de nettoyer la nourriture donnée aux bêtes ou de modifier la dynamique des pesticides dans le corps de l'animal. Les résultats semblent encourageants; reste à savoir si ces méthodes seront applicables en pratique. (c) On ne connaît pas de méthode efficace et universelle pour enlever les pesticides du sol et de l'eau et on s'inquiète fort peu d'en chercher. Il est évident que même à une échelle locale restreinte, la tâche serait extrêmement difficile.

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