Removal of Pesticides from Wool Wax by Continuous Countercurrent Dual-Solvent Extraction

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ABSTRACT: Pesticide residues in raw wool wax were removed to below detectable levels by continuous countercurrent extraction with hexane and *N,N*-dimethylformamide (DMF) in a pilot-scale mixer–settler contactor. The disengagement of the phases in the settling compartments was promoted by the addition of a small amount of formic acid (3% vol/vol) to the DMF-rich feed. Empirical equations were developed to predict the effect on the pesticide partition coefficients of the wool wax concentration, the presence of small amounts of water, ethanol, and/or isopropanol in the solvents, and the temperature used in the contactor. These empirical equations were included in equations that describe the concentration of the pesticides in the different stages of the contactor and were used to develop a spreadsheet model that accurately predicted the mixer–settler's performance. The raffinate wool wax produced by this process after conventional neutralization met all BP and USP specifications for pharmaceutical lanolin.
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KEY WORDS: Continuous countercurrent solvent extraction, *N,N*-dimethylformamide, hexane, organochlorides, organophosphates, pesticides, pharmaceutical lanolin, pyrethroids, wool wax.

Pesticide residues in wool wax are a serious impediment to its use in pharmaceutical formulations, cosmetics, and babycare products. A previous paper (1) showed that these residues can be removed to below their detection limits by the batchwise extraction of a wool wax and hexane solution with *N,N*-dimethylformamide (DMF). This procedure produces a high-quality wax but requires the use of considerable quantities of the relatively high-boiling solvent, DMF. To overcome this disadvantage, these solvents have been used in a laboratory pilot-scale continuous countercurrent solvent extractor, and the results are reported in this paper.

MATERIALS AND METHODS

Equipment. A mixer-settler contactor was selected for use in this study because its layout facilitates both the mathematical description of the extraction process and the withdrawal of samples from both phases in each stage for analysis. The 12-stage mixer–settler cascade of 4-L capacity was designed according to the principles formulated by Williams *et al.* (2) and fabricated from mild steel. A flow diagram for the mixer–settler and auxiliaries is shown in Figure 1. The various feeds were pumped from storage to the cascade by magnetically coupled variable-speed gear pumps *via* miniature needle valves and rotameters. The rotameters were calibrated by measuring the volume of appropriate solvent discharged in a fixed time and used to monitor the instantaneous flow of the solutions into, and out of, the contactor. In the operation of the mixer–settler, the two phases were brought into intimate contact in the mixing compartment and passed together as an unstable emulsion into the settling compartment, in which the phases disengaged and passed onto the adjoining mixing compartments. The levels of the interfaces in the settling compartments were determined by the height of the mixed-phase port and the volume fraction of the DMF-rich phase (2), except in the compartment in which the heavy phase was discharged. In this compartment, the level was controlled by a similar geared pump and needle valve.

Reagents. The pesticides used were obtained from ChemService, Inc. (West Chester, PA) or, where indicated in Table 1, were isolated from commercial formulations as described previously (1). To minimize volume changes to the extract and raffinate phases during the extraction process, all solvent feeds were prepared from laboratory-grade solvents as mutually saturated phases. Samples of raw wool wax used as feed were obtained from commercial scouring of a number of different wool types. Their characteristics are given in Tables 2 and 3. Solutions of these waxes in a mixture of laboratory-grade hexane and DMF (10:1, 20–30% wt/vol) were filtered through a bed of diatomaceous earth to remove traces of cellular debris (3) and used as feed to the contactor. To assess the operation of the mixer–settler, the low-pesticide wool wax described previously (1) was spiked with 1000 μg/g of the appropriate pesticide.

Procedure. To commence a run, the mixer–settler was filled with sufficient extraction solvent to immerse the impellers of the mixing compartment stirrers. Agitation was started, and the feed rates of the solvents were adjusted to the desired values. The instantaneous flow rates of the solvents into and out of the apparatus were monitored by the calibrated rotameters. However, the flow rates used in mass-balance cal-

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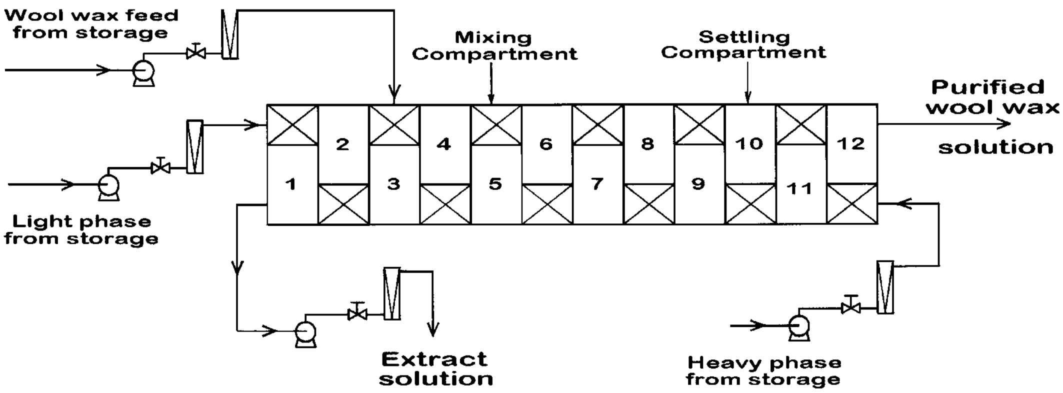

FIG. 1. Flow diagram for mixer–settler contactor.

culations were determined from the measured volume of solvent that was either fed to, or discharged from, the contactor during a given period of equilibrium operation. Although equilibrium conditions were rapidly achieved, the contactor was only sampled after at least 5 L of combined solvent had been discharged from the contactor. The raffinate wool wax and extract waxes were recovered by the batchwise two-stage evaporation of the appropriate phases in a Buchi Rotorvap at 80°C and a reduced pressure of 2 KPa, followed by 125°C and a reduced pressure of 0.1 KPa.

Analytical. Determination of partition coefficients was performed as described previously (1). Pesticide residues in wool waxes were determined after a gel permeation chromatography cleanup followed by gas chromatography (4). Pesticides in the mixer-settler phases were determined by gas chromatography without prior cleanup (1).

TABLE 1
Partition Coefficients for Pesticides between Hexane and Anhydrous DMF at 20°C

Pesticide	P_o	Pesticide	P_o
Bromophos ethyl	0.202	α-BHC	0.136
Bromophos methyl	0.144	β-BHC	0.068
Carbophenothion	0.082	δ-BHC	0.085
Chlorfenvinphos [*E*]	0.044	Aldrin	0.633
Chlorfenvinphos [*Z*]	0.040	Dieldrin	0.170
Chlorpyriphos ethyl	0.163	Endosulfan-α	0.196
Chlorpyriphos methyl	0.055	Endosulfan-β	0.084
Coumaphos	0.019	Endrin	0.202
Diazinon	0.196	Heptachlor	0.333
Dichlofenthion	0.163	Heptachlor epoxide	0.144
Dichlorvos	0.040	Hexachlorobenzene	0.701
Ethion	0.043	Lindane	0.094
Fenchlorphos	0.167	Methoxychlor	0.094
Malathion	0.037	*p,p′*-DDD	0.044
Phosalone	0.021	*p,p′*-DDE	0.212
Pirimiphos ethyl	0.180	*p,p′*-DDT	0.102
Propetamphos	0.049	Tecnazene	0.258
Tetrachlorvinphos	0.068		
Cyhalothrin[a]	0.023		
Cypermethrin	0.024		
Deltamethrin[a]	0.028		
Fenvalerate	0.030		

[a] Isolated from commercial formulations. Abbreviations: BHC, hexachlorocyclohexane; DDD, dichlorodiphenyldichloroethane; DDE, dichlorodiphenyldichloroethylene; DDT, dichlorodiphenyltrichloroethane; DMF, *N,N*-dimethylformamide.

Neutralization. A solution of wool wax (raw or solvent-extracted) in hexane (15–30% wt/vol, 3.5 L) was warmed to 50–60°C and stirred for 5 min with a warm solution of Na_2CO_3 (5% wt/vol) in aqueous isopropanol (35% vol/vol, 150–300 mL). The two phases were allowed to separate, and the aqueous phase was removed. The hexane-rich phase was extracted an additional three times with warm aqueous isopropanol (35% vol/vol, 150–300 mL). If no further processing of the wool wax was to be undertaken, the solvents were removed by two-stage distillation in a Buchi Rotorvap as described previously. If the neutralized wool wax was to be used as feed for the contactor, the small amounts of water and isopropanol present were almost entirely removed by distillation of the hexane/isopropanol/water azeotrope (5).

Bleaching. A solution of aqueous citric acid (15%, wt/vol, 15–30 mL) was added to the solution of neutralized wool wax (3.5 L), obtained in the previous steps. The mixture was stirred and heated under gentle reflux, while an aqueous solution of Na_2ClO_2 (26% wt/vol, 25–50 mL) was added dropwise over 20 min. The mixture was stirred and heated under reflux for a further 30 min, the phases were allowed to separate, and the bleached wool wax phase was used as feed in the mixer–settler or the wax was recovered as described previously.

RESULTS AND DISCUSSION

Initial experiments with the mixer–settler contactor indicated that by themselves the solvents, hexane and DMF, were not completely satisfactory. In the mixing compartment, a semistable emulsion was formed that did not adequately disengage in the settling compartment. The stability of this emulsion was greatest when the heavy extraction phase was the continuous phase. However, even when the flow rates were adjusted to make the hexane-rich phase the continuous phase, the contactor could only be run for relatively short periods before inadequate separation of the phases occurred. This problem was

TABLE 2
Raw Wool Wax Characteristics

Wool wax	Wool type	Wax characteristics
A	Australian Merino fleece wools, 19μ	Light color with moderate levels of oxidized wool wax.
B	Australian Merino fleece wools, 24μ	Light color with moderate levels of oxidized wool wax.
C	Australian Merino locks and crutchings, 24μ	Dark color with relatively low levels of oxidized wax.
D	Australian Merino bellies, crutchings, locks and stains, 20.5μ	Light color with relatively low levels of oxidized wax.
E	Unknown	Neutralized and bleached wax from a De Smet solvent scour. Contained a significant amount of oxidized wax.
F	Mixture of British, Australian, South African and New Zealand wools	Raw wax recovered by "acid cracking." A dark, odorous wax with high acidity and high levels of oxidized material.
G	New Zealand slipe wools	250 μg/g Ca.
H	New Zealand slipe wools	3140 μg/g Ca.
I	Mixture of Australian and New Zealand wools	A blended commercial wax sample obtained from a commercial lanolin refiner.

TABLE 3
Properties of Raw Wool Waxes

Wool wax	A	B	C	D	E	F	G	H	I
Sulfated ash (%)	0.08	0.08	0.15	0.11	0.14	0.04	0.36	1.04	0.12
Acid value (mg KOH/g)	11.0	6.3	4.0	3.3	1.7	24.6	5.4	8.1	4.8
Saponification value (mg KOH/g)	114	109	98	104	103	113	106	110	99
Peroxide value (meq/kg)	32	22	25	25	24	14	29	23	15
Melting point (°C)	41	40	43	44	44	40	45	46	43
Color[a]									
Yellow	40	30	70	30	6	78	24	31	79
Red	5.1	3.0	5.1	4.2	0.7	18	4.7	7.3	9.1
Free cholesterol (%)	5.5	3.4	2.5	3.6	3.3	4.7	3.9	3.0	2.4
Free total wool wax alcohols (%)	12.4	12.5	11.2	12.6	13.4	11.7	12.8	11.8	11.5
Water content (% w/w)	1.8	1.7	1.1	0.6	0.4	2.5	0.8	0.9	1.6
Pesticide residues (μg/g)									
Propetamphos	—	1.36	29.1	2.9	1.3	36.1	10.2	4.5	5.9
Diazinon	54.3	30.4	46.3	22.5	43.9	—	43.5	80.1	82.5
Dichlofenthion	—	—	—	—	—	0.2	—	125.3	22.5
Chlorfenvinphos	10.5	0.5	1.5	6.3	11.4	24.3	4.2	2.7	16.2
Bromophos ethyl	—	—	—	—	—	—	65.1	—	—
Coumaphos	—	—	—	—	—	—	5.6	12.6	—
Cyhalothrin	1.1	7.1	—	1.4	1.1	1.5	8.9	1.6	—
Cypermethrin	63.2	42.6	65.1	10.4[b]	1.3	3.3	17.4	9.7	21.4
Others						8.8[c]	4.9[d]		

[a] Lovibond 1/4-inch cell.
[b] α-Isomer of cypermethrin.
[c] α-BHC, 0.4 μg/g; γ-BHC, 7.6 μg/g; δ-BHC, 0.2 μg/g; chlorpyriphos ethyl, 0.1 μg/g; carbophenothion, 0.5 μg/g.
[d] Carbophenothion. For abbreviations see Table 1.

most apparent when neutralized wool waxes were processed and least apparent when wool waxes with high acid values were used as feed. This problem first occurred in the later stages of the mixer–settler, where the concentration of free fatty acids would be expected to be the lowest. The results of test-tube experiments (Table 4) showed that this phase disengagement was strongly promoted by the short-chain carboxylic acids, with small amounts of formic acid being particularly effective. Unlike other polar additives (1), such as water or lower alcohols, formic acid concentrations of up to 6% vol/vol in the DMF-rich phase had no measurable effect on the pesticide partition coefficients. Levels of 3% were enough to promote the rapid disengagement of the phases in the contactor under the different flow rates used (Table 5).

A design feature of the mixer–settler contactor is the separation of the extraction into discrete stages. This makes for easy mathematical modeling of the process. When the wool wax feed is introduced into the mixing compartment of the third stage, the mixer–settler cascade is divided into two sec-

TABLE 4
Effect of Organic Acids on the Rate of Disengagement of Mixer–Settler Phases

Acid added to DMF-rich phase[a] (1% wt/vol)	Time required for phase separation (min)
None	23
Formic acid	0.33
Acetic acid	0.66
Butyric acid	0.75
Lauric acid	2
Stearic acid	15
Citric acid	0.5

[a] For abbreviation see Table 1.

TABLE 5
Feed and Flow Rates Used in the Mixer–Settler Contactor

	Experiment number								
	1	2	3	4	5	6	7	8	9
Wool wax	A	B	C	D	E	F	G	H	I
Wool wax feed (% wt/vol)	25	20	20	20	20	15	25	20	18
Temperature (ºC)	26.0	26.5	23.5	24.0	22.5	21.3	21.8	22.3	24.5
Water (% vol/vol DMF)	0.2	0.5	1.2	1.1	0.75	0.5	0.07	0.07	0.85
Flow rates (mL/min)									
Wool wax feed	60	50	50	50	30	60	40	75	96
Hexane	10	10	10	10	10	10	10	15	18
DMF-rich phase	60	45	45	45	30	50	50[a]	80	95
Yield (%)	85	84	88	89	90[b]	74	84	93[c]	83

[a] 3% citric acid in DMF-rich phase.
[b] Neutralized and bleached wax from solvent scour, overall yield unknown.
[c] Neutralized prior to solvent extraction, overall yield = 77%. For abbreviation see Table 1.

tions whereby stages 1–3 form the enrichment section, and stages 4–12 constitute the extraction section. Assuming that no interaction between different species occurs and that equilibrium partitioning is achieved in all mixer–settler stages, then the individual pesticide concentration in the DMF-rich phases of these sections may be predicted by Equations 1 and 2, respectively.

$$C_n^{\text{enrichment stage}} = \frac{\left(F^{\text{hex}} P^i C_{n-1} + F^{\text{WW}} C^{\text{WW}} - F^L P^i C_\omega\right)}{F^{\text{DMF}}} \quad [1]$$

$$C_n^{\text{extraction stage}} = \frac{\left(F^L P^i C_{n-1} - F^L P^i C_\omega\right)}{F^{\text{DMF}}} \quad [2]$$

The pesticide concentration in the light phase of all of the contactor sections is given by Equation 3,

$$C_n^L = P^i C_n \quad [3]$$

where P^i = the partition coefficient of pesticide i; C_n = concentration of pesticide i in the DMF-rich phase of stage n; C^{WW} = concentration of pesticide i in the wool wax feed; F^{hex} = flow rate of the hexane-rich feed; F^{WW} = flow rate of the wool wax feed; F^L = combined flow rates of the light phases $(F^L + F^{\text{WW}})$; F^{DMF} = flow rate of the DMF-rich phase; n = stage number; and C_ω = concentration of pesticide i in the DMF-rich phase in the last stage of the contactor.

A spreadsheet model was used to assess the efficiency of the contactor using these equations. While general agreement was observed between the predicted and measured pesticide concentrations in the various contactor stages, the results highlighted the need to consider factors that influenced the partition coefficients of the pesticide. As shown previously, the pesticide partition coefficient is affected by the concentration of the wool wax in the hexane-rich phase and by the presence of certain polar additives to the DMF-rich phase.

All waxes, as received, contained varying amounts of entrained water. If not removed, this water migrated into the DMF-rich phase and had a significant adverse effect on the pesticide partition coefficients. Ethanol and isopropanol also adversely affected the partition coefficients, but not to the same extent as did water. These solvents are often used in neutralization processes and therefore have the potential to be present in neutralized wool wax solutions that might be used as feed for this solvent extraction process. These effects can be generalized and predicted empirically by the equations shown in Figures 2–5.

The partition coefficients were also affected by the temperature of the solvents in the mixer–settler. This effect could also be predicted empirically from the results plotted in Figure 6. Values for the empirical constants involved were derived from the average results obtained with 39 different pesticides. The error bars represent the 95% confidence limits.

These effects appear to be additive. The effective partition coefficient, predicted in Equation 4 by the combined equations from Figures 2–6, was in good agreement with the partition coefficients measured at different concentrations and temperatures (Table 6).

$$P^i_{\text{effective}} = \exp\left\{\ln\left(P^i_o\right) + 0.016\times[\text{wool wax}] + 0.106\times[\text{H}_2\text{O}] \times[\text{EtOH}] + 0.06\times[\text{IPA}] - \frac{1627}{273.3 + T_{^\circ\text{C}}} + 5.5433\right\} \quad [4]$$

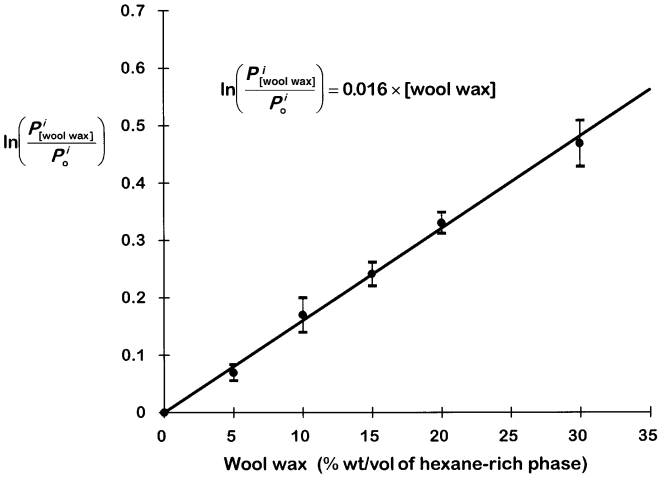

FIG. 2. The effect of the wool wax concentration in the hexane-rich phase on the pesticide partition coefficients. $P^i_{[\text{wool wax}]}$ = the partition coefficient of species i with various concentrations of wool wax in the hexane-rich phase, and P^i_o = the partition coefficient of species i in the absence of wool wax.

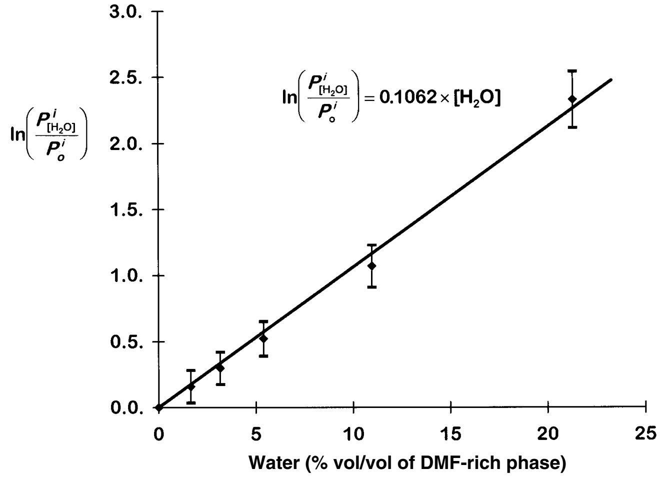

FIG. 3. The effect of water in the dimethylformamide (DMF)-rich phase on the pesticide partition coefficients. $P^i_{[H_2O]}$ = the partition coefficient of species *i* with various concentrations of water in the DMF-rich phase, and P^i_o = the partition coefficient of species *i* in the absence of water.

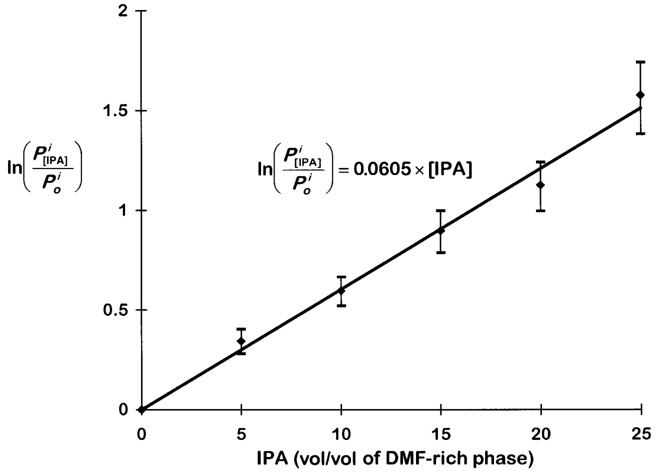

FIG. 5. The effect of isopropanol (IPA) in the DMF-rich phase on the pesticide partition coefficients. $P^i_{[IPA]}$ = the partition coefficient of species *i* with various concentrations of IPA in the DMF-rich phase, and P^i_o = the partition coefficient of species *i* in the absence of IPA.

where [IPA] is the isopropanol concentration. Accurate modeling of the mixer–settler was obtained by using the effective partition coefficient defined in Equation 4 in Equations 1–3. This is demonstrated graphically in Figure 7 which shows that the predicted and measured levels of pesticides in each stage of the contactor were in good agreement.

Processing of wool waxes. A number of commercial wool waxes obtained from different wool types and with relatively high levels of pesticide residues were used to evaluate this solvent extraction process. In spite of the quality range of these raw wool waxes, all were satisfactorily processed in the mixer–settler contactor. The most difficult waxes to treat were those derived from New Zealand slipe wools. The presence of calcium soaps in these waxes gave rise to more stable emulsions that were not as readily broken by the presence of formic acid. For these samples, citric acid was slightly more effective, but prior neutralization or dilution with other waxes was a better solution.

Although these raw waxes contained relatively high pesticide levels no residues could be detected in the raffinate waxes after processing. The spreadsheet model of the process predicted that all pesticide residues except diazinon would be reduced to levels below 0.001 µg/g, or more than an order less than their detection limits. The levels predicted for diazinon, a pesticide that was present in most waxes and often at the highest concentrations, are given in Table 7. These levels are much less than the detection limit of 0.07 µg/g for diazinon, based on use of the sensitive thermionic specific detector (4).

During wax recovery from the raffinate phase, it is important to remove the last traces of DMF. DMF has the ability to penetrate intact skin easily; accordingly, the threshhold limit value–time-weighted average (TLV–TWA) [skin] is reason-

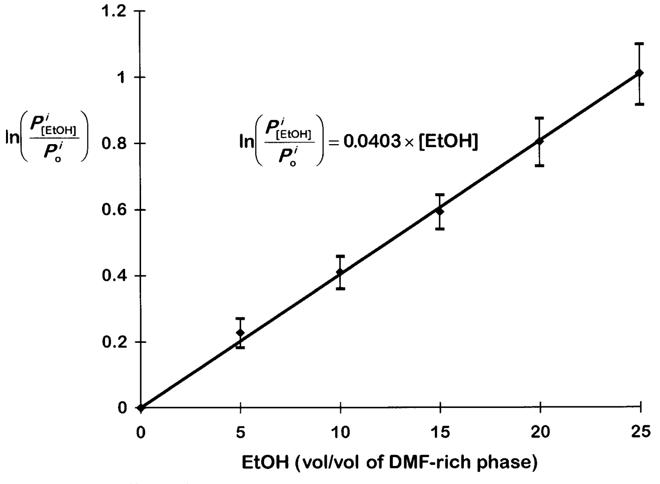

FIG. 4. The effect of ethanol in the DMF-rich phase on the pesticide partition coefficients. $P^i_{[EtOH]}$ = the partition coefficient of species *i* with various concentrations of ethanol in the DMF-rich phase, and P^i_o = the partition coefficient of species *i* in the absence of ethanol. For abbreviation see Figure 3.

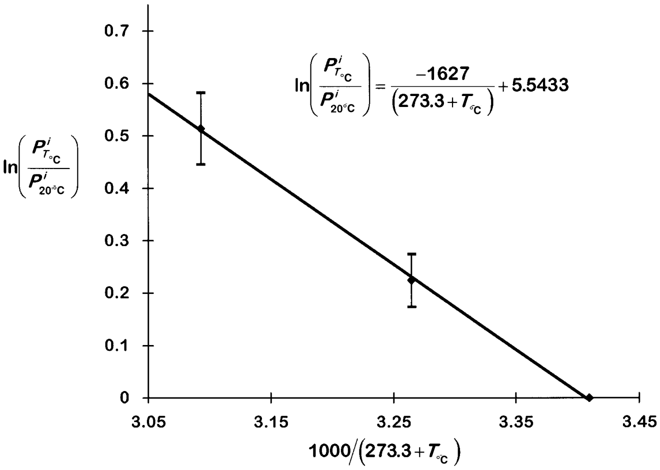

FIG. 6. The effect of temperature on the pesticide partition coefficients. $P^i_{T°C}$ = the partition coefficient of species *i* at various temperatures, and $P^i_{20°C}$ = the partition coefficient of species *i* at 20°C.

TABLE 6
Comparison Between Predicted and Measured Partition Coefficients

Pesticide	Wool wax (%)[a]	Temperature (°C)	Water (%)[b]	Predicted	Measured
Diazinon	25	31	2.1	0.44	0.45
Diazinon	32.5	52.2	4.0	0.87	0.91
Propetamphos	25	31	2.1	0.11	0.11
Propetamphos	32.5	52.2	4.0	0.22	0.27
Cypermethrin	25	31	2.1	0.054	0.051
Cypermethrin	32.5	52.2	4.0	0.11	0.12
Dieldrin	32.5	52.2	4.0	0.39	0.40
Carbophenothion	25	31	2.1	0.19	0.18
Chlorfenvinphos	32.5	52.2	4.0	0.18	0.18
Coumaphos	25	31	2.1	0.043	0.045

[a] wt/vol of hexane-rich phase.
[b] wt/vol of DMF-rich phase. For abbreviation see Table 1.

ably low at 10 ppm (6). In the laboratory, simple distillation left levels of about 2000 µL/kg of DMF in the wax. This could be reduced to acceptable levels of below 10 µL/kg by the use of a thin-film evaporator or by vacuum steam stripping. However, a much simpler approach was to schedule neutralization and bleaching steps after the solvent extraction process, in which the use of aqueous alcohols conveniently removed the last traces of DMF to below detectable levels in the wax.

As shown in Table 7, this extraction process produced high-quality waxes from all raw waxes tested; the waxes, except for the acid values and the sulfated ash values of those produced from slipe wools, met all BP and USP specifications for the highest-quality pharmaceutical lanolin. The required

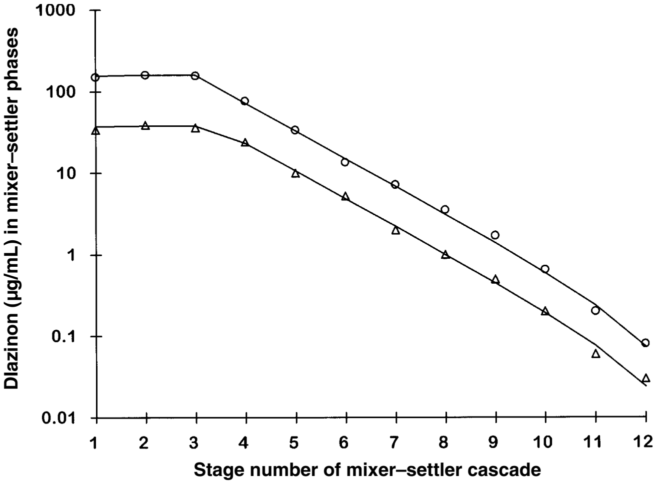

FIG. 7. Predicted and measured levels of diazinon in the phases in each stage of the mixer–settler cascade. Solid lines represent the predicted performance, while the points, (○) hexane-rich phase and (△) DMF-rich phase, are the experimental values. The flow rates used were DMF feed, 60 mL/min; hexane feed, 10 mL/min, and wool wax feed (25% wt/vol), 75 mL/min. The temperature of the contactor was 27.5°C. The DMF contained 0.1% vol/vol of water. The wax was spiked with 500 mg/g of diazinon and contained 1.1% vol/wt of water. For abbreviation see Figure 3.

TABLE 7
A Comparison of the Properties of the Raffinate Wool Wax with US and British Pharmacopoeia Specifications

	Pharmacopoeia specification		Raffinate wool waxes/Experiment number								
	BP	USP	A/1	B/2	C/3	D/4	E/5	F/6	G/7	H[b]/8	I/9
Sulfated ash (%)	0.15	n.s.	0.01	0.005	0.008	0.007	0.04	0.04	0.29 (0.03)	0.08	0.03
Acid value (mg KOH/g)	1.0	0.9	2.6 (0.6)	2.3 (0.7)	1.9 (0.6)	1.4 (0.6)	0.8	6.2 (0.9)	1.3 (0.8)	1.0	1.1 (0.8)
Saponification value (mg KOH/g)	90–105	n.s.	103	107	105	101	103	103	102	104	99
Peroxide value (meq/kg)	<20	n.s.	5.7	4.2	3.5	5.1	7.2	3.2	3.5	4.7	4.6
Melting point (°C)	38–40	n.s.	39	38	40	40	41	38	39	40	38
Water-soluble											
Acid substances	P/F	P/F	P	P	P	P	P	P	P	P	P
Alkaline substances	P/F	P/F	P	P	P	P	P	P	P	P	P
Oxidizing substances	P/F	n.s.	P	P	P	P	P	P	P	P	P
Water absorption capacity (%)	>200	n.s.	265	270	290	>300	>300	>300	280	>300	>300
Color[a]											
Yellow	ns	n.s.	10 (4)	9 (3)	17 (5)	6 (2)	4	52 (9)	9 (5)	17 (7)	10 (5)
Red	ns	n.s.	2.0 (0.3)	1.0 (0.2)	2.1 (0.5)	1.1 (0.3)	0.4	6.2 (1.2)	3.1 (0.9)	3.2 (0.8)	2.1 (0.3)
Free cholesterol (%)	ns	n.s.	0.9	1.1	1.0	1.2	1.2	1.7	1.6	1.5	0.9
Free total wool wax alcohols (%)	ns	<6[c]	4.0	5.5	6.1	5.3	6.1	6.3	5.2	5.4	3.3
Total pesticide residues (µg/g)	ns	<3 µg/g[c]	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Predicted diazinon in raffinate wax (µg/g)			0.002	0.003	0.005	0.002	0.002	—	<0.001	<0.001	0.001

[a] Lovibond 1/4-inch cell.
[b] Raw slipe wax neutralized before extraction.
[c] USP Modified Lanolin. Abbreviations: n.s. = not specified; n.d. = none detected; P = Pass, F = Fail. Values in parentheses are after neutralization and bleaching.

acid and sulfated ash values were easily achieved by conventional neutralization of the wool wax, either prior to or after solvent extraction.

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REFERENCES

1. Jones, F.W., The Removal of Pesticide Residues from Wool Wax by Solvent Extraction, *J. Am. Oil Chem. Soc. 74*:1241–1245 (1997).
2. Williams, J.A., L. Lowes, and M.C. Tanner, The Design of a Simple Mixer Settler, A Method of Design by Calculation Giving Automatic Interface Control, *Trans. Instn. Chem. Engrs. 36*:464–475 (1958).
3. Orwin, D.F.G., and J.L. Woods, Cellular Debris in the Grease of Wool Fibres, *Text. Res. J. 55*:84–92 (1985).
4. Jones, F.W., Multi-Residue Analysis of Pesticides in Wool Wax and Lanolin Using Gel Permeation and Gas Chromatography, *J. Agric. Food Chem. 44*:3197–3201 (1996).
5. Horsley, L., Azeotropic Data—III, *Advances in Chemistry Series 116,* edited by R.F. Gould, American Chemical Society, Washington, DC, 1973, pp. 31, 197, 464–465.
6. American Conference of Governmental Industrial Hygienists Inc., *Documentation of the Threshold Limit Values and Biological Exposure Indices,* 5th edn., Cincinnati, 1986, p. 209.

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