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Modified Lanolin

DEFINITION

Modified Lanolin is the purified wax-like substance from the wool of sheep, *Ovis aries* L. (Fam. Bovidae), that has been processed to reduce the contents of free lanolin alcohols and residues of detergent and pesticide. It contains NMT 0.25% of water. It may contain NMT 0.02% of a suitable antioxidant.

IMPURITIES

• **Limit of Free Lanolin Alcohols**

Gel permeation chromatographic cleanup system

Eluant: Methylene chloride

Column: 25-mm × 100-cm; packed with a slurry of styrene–divinylbenzene copolymer beads compressed to a bed length of approximately 77 cm

Flow rate: 4 mL/min

Set up the chromatograph, adjusting to discard the fraction eluting from 0 to 43 min. Collect the fraction eluting from 43 to 60 min, and rinse for 20 min, discarding the rinse fraction.

System suitability

Elution of lanolin alcohols: Melt a suitable quantity of USP Lanolin Alcohols RS, and pass through a fluted filter paper into a container. Transfer 1.0 g to a 10-mL volumetric flask. Dilute with *Eluant* to volume. Transfer 5 mL to the gel permeation chromatographic column, and elute with *Eluant*. Collect 172–240 mL of the column effluent in a suitable evaporator. Evaporate the solvent, cool, weigh the evaporator, and calculate the amount of lanolin alcohols eluted in the evaporator. The column is suitable if NLT 99% of the lanolin alcohols elute in the first 172–240 mL.

Standard solution: 0.5 mg/mL of USP Lanolin Alcohols RS in hexane. Store this solution in a cold, dark place for up to 4 weeks. Before using, warm just sufficiently to dissolve any precipitate if necessary.

Sample solution: Transfer 1 g of Modified Lanolin, previously melted to liquid form by heating on a hot water bath if necessary, to a 10-mL volumetric flask. Dissolve in 7 mL of *Eluant*, dilute with *Eluant* to volume, and filter. Transfer 5.0 mL of this solution to the column, and elute with 320 mL of *Eluant*. Discard the first 172-mL fraction, and collect the next 68-mL fraction (from 172 to 240 mL) in a suitable evaporator. Concentrate by evaporation on a steam bath to 3 mL. Add 50 mL of hexane, and transfer this solution to a 100-mL volumetric flask, adjusting the volume with hexane to 100 mL.

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: GC

Detector: Flame ionization

Columns

Guard: 0.32-mm × 50-cm fused silica uncoated

Analytical: 0.33-mm × 50-m fused silica capillary; bonded with a 0.50-µm layer of phase G2

Temperatures

Detector: 290°

Column: See *Table 1*.

Table 1

Initial Temperature (°)	Temperature Ramp (°/min)	Final Temperature (°)	Hold Time at Final Temperature (min)
210	3	280	—

Flow rate: 7 mL/min

Carrier gas: Nitrogen

Makeup gas: Nitrogen at 50 mL/min

Injection volume: 1 µL

Analysis

Samples: *Standard solution* and *Sample solution*

[NOTE—Allow both the *Standard solution* and the *Sample solution* to elute for NLT 40 min.]

Calculate the percentage of free lanolin alcohols in the portion of Modified Lanolin taken:

$$\text{Result} = (r_U/r_S) \times [(C \times K)/(I \times W)] \times 100$$

r_U = total peak response from the *Sample solution*

r_S = total peak response from the *Standard solution*

C = concentration of USP Lanolin Alcohols RS in the *Standard solution* (mg/mL)

I = volume injected into the gel permeation chromatography column (mL)

W = weight of Modified Lanolin taken (g)

K = corrected fraction of free lanolin alcohols in the USP Lanolin Alcohols RS in the *Standard solution* taken:

$$K = 1 + (0.0062A - 0.0119S)$$

A = acid value of USP Lanolin Alcohols RS

S = saponification value of USP Lanolin Alcohols RS

Acceptance criteria: NMT 6%

• **FOREIGN SUBSTANCES**

Use pesticide-free grade reagents and solvents throughout this test. [NOTE—Reference materials of pesticides for use in the *Standard solution* may be obtained from any commercial source.[1]]

Standard stock solutions: Prepare stock solutions for each reference pesticide containing 100 mg/L in hexane.

[NOTE—Concentrated stock solutions may be stored in glass-stoppered containers in a dark refrigerator at 2°–5° for up to 1 year. Most pesticides may be dissolved directly in hexane; however, the hexachlorocyclohexane isomers and the DDT group of pesticides may require initial dissolution in the minimum volume of acetone followed by dilution with hexane to the specified concentration.]

Standard solution: Dilute volumes of the *Standard stock solutions* quantitatively with hexane, and combine to obtain a composite *Standard solution* having the concentrations indicated in *Table 2*. Store the composite *Standard solution* in a glass-stoppered glass container in the dark at 2°–5°, and replace it every 2 months. [NOTE—Two or more separate composite *Standard solutions*, each preferably containing NMT 8 reference pesticides, may be prepared if needed. Reference pesticides should be selected for composite *Standard solutions* on the basis that relative retention times (see *Table 2*) differ sufficiently so that peaks in chromatograms will not be expected to overlap, and they should be selected and combined appropriately for the chromatographic system and detector used.]

Table 2

Reference Pesticide[a]	Standard Solution (Concentration in µg/mL)		Relative Retention Times (Relative to 1.0 for Chlorpyrifos)	
	Electron-Capture Detector	Flame-Photometric Detector	System I	System II
Tetrachloronitrobenzene (TCBN)	0.05	—	0.29	0.24
alpha-Hexachlorocyclohexane (alpha BHC)	0.05	—	0.40	0.35

Reference Pesticide[a]	Standard Solution (Concentration in µg/mL)		Relative Retention Times (Relative to 1.0 for Chlorpyrifos)	
	Electron-Capture Detector	Flame-Photometric Detector	System I	System II
beta-Hexachlorocyclohexane (beta BHC)	0.30	—	0.43	0.56
Hexachlorobenzene (HCB)	0.05	—	0.45	0.33
gamma-Hexachlorocyclohexane (lindane)	0.05	—	0.48	0.41
Propetamphos	—	0.30	0.48	0.42
Diazinon	—	0.20	0.52	0.40
Dichlofenthion	0.10	0.20	0.67	0.56
Ronnel	0.30	0.40	0.81	0.66
Heptachlor	0.10	—	0.83	0.60
Malathion	—	0.40	0.91	1.05
Chlorpyrifos	0.30	0.30	1.00	1.00
Aldrin	0.20	—	1.05	0.76
Pirimiphos ethyl	—	0.40	1.14	1.14
Chlorfenvinphos Z	0.40	0.40	1.17	1.40
Heptachlor epoxide	0.20	—	1.29	1.17
Chlorfenvinphos E	0.40	0.50	1.30	1.51
Bromophos ethyl	0.40	0.50	1.51	1.45
1,1-Dichloro-2-(2-chlorophenyl)-2-(4-chlorophenyl)ethene (*o,p*-DDE)	0.30	—	1.55	1.51
1,1-Dichloro-2-(4-chlorophenyl)-2-(4-chlorophenyl)ethene (*p,p*-DDE)	0.30	—	1.88	1.86
Stirophos	0.60	0.80	1.58	1.97
alpha-Endosulfan	0.40	—	1.63	1.47

Reference Pesticide[a]	Standard Solution (Concentration in µg/mL)		Relative Retention Times (Relative to 1.0 for Chlorpyrifos)	
	Electron-Capture Detector	Flame-Photometric Detector	System I	System II
1,1-Dichloro-2-(2-chlorophenyl)-2-(4-chlorophenyl)ethane (*o,p*-TDE)	0.40	—	1.90	2.19
Dieldrin	0.30	—	1.91	1.84
Endrin	0.40	—	2.13	2.29
beta-Endosulfan	0.40	—	2.19	2.77
1,1-Dichloro-2,2-bis(4-chlorophenyl)ethane (*p,p*-TDE)	0.40	—	2.41	2.87
1,1,1-Trichloro-2-(2-chlorophenyl)-2-(4-chlorophenyl)ethane (*o,p*-DDT)	0.40	—	2.55	2.70
Ethion	1.00	0.40	2.56	3.36
Carbophenothion	0.80	1.00	2.94	3.70
1,1,1-Trichloro-2,2-bis(4-chlorophenyl)ethane (*p,p*-DDT)	0.50	—	3.13	3.50
Methoxychlor	0.60	—	4.70	7.20
Carbophenothion sulfone	5.00	—	5.10	9.20
Carbophenothion sulfoxide	5.00	—	5.40	10.00

[a] Suitable materials may be obtained from either Chem Service, 660 Tower Lane, P.O. Box 3108, Westchester, PA 19381-3108 or Greyhound, 88 Grange Road West, Birkenhead, Merseyside, L43 4XF, England U.K.

Gel permeation chromatography cleanup system

Eluant: Methylene chloride and hexane (1:1)

Column: 25-mm × 50-cm; packed with a slurry of 35 g of styrene–divinylbenzene copolymer beads compressed to a bed length of about 20 cm

Operating pressure: 8–11 psi

Flow rate: 5 mL/min. Set up the chromatograph, adjusting to discard the fraction eluting from 0 to 12 min. Collect the fraction eluting from 12 to 32 min, and rinse for 2 min, discarding the rinse fraction.

System suitability

Elution of lanolin: Melt a suitable quantity of Lanolin, and pass through a fluted filter paper into a container. Transfer 6.0 g to a 50-mL volumetric flask. Dilute with *Eluant* to volume, and filter. Transfer 5.0 mL of this solution to the gel permeation chromatographic column, and elute with *Eluant*. Collect 100 mL of the column effluent in tared beakers in 10-mL increments. Evaporate the solvent, cool, weigh the beakers and contents, and calculate the amount of lanolin eluted in each 10-mL increment. The column is suitable if NLT 96% of the lanolin elutes in the first 60 mL.

Elution of pesticide from lanolin: Dissolve suitable quantities of diazinon, diclofenthion, bromophos ethyl, lindane, and dieldrin in hexane to obtain a *Standard solution* having concentrations of 0.4, 0.4, 1.0, 0.1, and 0.6 µg/mL, respectively. Transfer 5.0 mL of this solution to a 10-mL volumetric flask containing 1 g of USP Lanolin RS. Dilute with methylene chloride to volume. Transfer 5 mL of this solution to the gel permeation chromatographic column, and elute with 160 mL of *Eluant*. Discard the first 60-mL fraction, and collect the next 100-mL fraction (from 60 to 160 mL). Transfer this collection fraction to a concentrator fitted with a graduated collection flask, add 50 mL of hexane, and concentrate by evaporation to 5 mL. Inject this fraction into the chromatographs described in *Chromatographic system I* and *Chromatographic system II*. Record the chromatograms, and measure the heights of the peaks obtained from the five pesticides in the *Standard solution*. Calculate the recoveries of each of the five pesticides used in the fortified USP Lanolin RS solution.

Prepare a test solution by mixing hexane with the *Standard solution* (1:1). Inject into the chromatographs described in *Chromatographic system I* and *Chromatographic system II*. Record the chromatograms, and measure the peak heights of the five pesticides in the chromatogram of the *Sample solution*. Compare the peak heights from the fraction of the *Standard solution* to the peak heights of the corresponding pesticides from the *Sample solution*: NLT 85% of the added amounts of each of the five pesticides is recovered.

Sample solution: Transfer 6 g of Lanolin, previously melted to liquid form by heating on a hot water bath if necessary, to a 50-mL volumetric flask. Dissolve in 25 mL of *Eluant*, dilute with *Eluant* to volume, and filter. Transfer 5.0 mL of this solution to the column, and elute with 160 mL of *Eluant*. Discard the first 60-mL fraction, and collect the remaining fraction in a suitable evaporator. Concentrate by evaporation on a steam bath to 3 mL, add 50 mL of hexane, and evaporate again to remove all traces of methylene chloride, adjusting the volume with hexane to 3.0 mL.

Chromatographic system I

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: GC

Detector: Electron capture

Column: 0.53-mm × 30-m fused silica capillary; bonded with a 1.5-µm layer of phase G1, and a 0.53-mm × 6-m fused silica uncoated guard column connected to a modified packed column-type injector system

Column temperature: 200°. [NOTE—The initial temperature of the column may be adjusted so that the retention times of ethion and *p,p'*-DDT are 2.56 and 3.1, respectively, relative to chlorpyrifos.]

Carrier gas: Helium

Flow rate: 25 mL/min. Adjust so that the retention time of chlorpyrifos is 4 min.

Makeup gas: Nitrogen, 40 mL/min

Injection volume: 5 µL

Chromatographic system II

Mode: GC

Detector: Flame photometric

Column: 0.53-mm × 30-m fused silica capillary; bonded with a 1.0-µm layer of phase G3, and a 0.53-mm × 6-m fused silica uncoated guard column connected to a modified packed column-type injector system

Column temperature: 200°. [NOTE—The initial temperature of the column may be adjusted so that the retention time of ethion is 3.36 relative to that of chlorpyrifos.]

Carrier gas: Helium

Flow rate: 25 mL/min. Adjust so that the retention time of chlorpyrifos is 4 min.

Makeup gas: Nitrogen, 40 mL/min

Injection volume: 5 µL

Analysis

The following procedure is to be followed for *Chromatographic systems I* and *II*.

Samples: *Standard solution* and *Sample solution*

Inject the appropriate composite *Standard solution* and the *Sample solution* into the gas chromatograph, record the chromatograms, and measure the areas of all the peaks observed in the chromatograms. Compare the peak areas of any of the pesticide residues in the *Sample solution* from each chromatographic system with the peak areas that correspond to the retention times in the appropriate composite *Standard solution* from each corresponding chromatographic system.

Calculate the quantity, in ppm, of the individual specified residue found in the sample taken:

$$\text{Result} = (r_U/r_S) \times (C/W) \times 30$$

r_U = peak area of each residue from the *Sample solution*

r_S = peak area of each residue from the *Standard solution*

C = concentration of the reference pesticide in the *Standard solution* (mg/L)

W = weight of Lanolin taken (g)

Acceptance criteria

Individual specified residue: NMT 1 ppm

Total specified residues: NMT 3 ppm

SPECIFIC TESTS

- **Fats and Fixed Oils,** *Acid Value (Free Fatty Acids)*⟨401⟩

Sample: 12.5 g

Acceptance criteria: The free acids obtained from the *Sample* require NMT 2.0 mL of 0.10 N sodium hydroxide for neutralization.

- **Alkalinity**

Sample: 2.5 g

Analysis: Dissolve the *Sample* in 10 mL of ether, and add 2 drops of phenolphthalein TS.

Acceptance criteria: No red color is produced.

- **Water-Soluble Acids and Alkalies**

Sample: 12.5 g

Analysis: Warm the *Sample* with 50 mL of water on a steam bath, constantly stirring the mixture until the Lanolin is melted.

Acceptance criteria: The fat separates completely on cooling, leaving the water layer nearly clear and neutral to litmus. Retain the water layer for the test for *Ammonia*.

- **Ammonia**

Sample solution: 10 mL of the solution from *Water-Soluble Acids and Alkalies*

Analysis: Add 1 mL of 1 N sodium hydroxide to the *Sample solution*, and boil.

Acceptance criteria: The vapors do not turn red litmus to blue.

- **Water Determination,** *Method I*⟨921⟩

Solution A: Chloroform and methanol (3:2)

Sample solution: 250 mg/mL of Modified Lanolin in *Solution A*

Analysis: Determine the water content of a 10.0-mL portion of the *Sample solution*. Perform a blank determination on 10.0 mL of *Solution A*, and make any necessary correction.

Acceptance criteria: NMT 0.25%

- **Petrolatum**

Sample: 3 g

Analysis: Heat the *Sample* on a steam bath, with frequent stirring, until it loses about 0.25% of its weight. Boil 40 mL of dehydrated alcohol with 500 mg of the dried lanolin so obtained.

Acceptance criteria: The solution is clear or NMT opalescent.

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in tight, preferably rust-proof containers, preferably at controlled room temperature.
- **USP Reference Standards** ⟨11⟩

USP Lanolin RS

USP Lanolin Alcohols RS

[1] Suitable materials may be obtained from either Chem Service, 660 Tower Lane, P.O. Box 3108, Westchester, PA 19381-3108 or Greyhound, 88 Grange Road West, Birkenhead, Merseyside, L43 4XF, England U.K.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
MODIFIED LANOLIN	Documentary Standards Support	SM32020 Small Molecules 3
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

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