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<b>(54) Title:</b> COMPOSITIONS OF FATTY ACID ESTERS OF HEXITANS  <b>(57) Abstract</b>  <p>The invention relates to a composition composed of fatty acid esters of hexitans (sorbitans) and non-esterified polyols, having a content of non-esterified polyols in the range of 6.59 wt. % to 0.05 wt. %, and to a process for purification by removing non-esterified polyols from a composition composed of fatty acid esters of hexitans and non-esterified polyols, which process comprises the steps of: a) dissolving said composition in a solution comprising at least one hydrocarbon and at least one polar organic solvent to produce an organic solution of said composition; b) mixing or contacting said organic solution with an aqueous solution of at least one metal salt under conditions effective to extract said non-esterified polyols; and c) separating said organic solution from the aqueous solution, optionally followed by the recovery of the purified fatty acid esters of hexitans.</p>		

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## COMPOSITIONS OF FATTY ACID ESTERS OF HEXITANS

This invention relates to compositions of fatty acid esters of hexitans (i.e., the fatty acid esters of inner monoethers of hexitols; hexitols are aliphatic, straight-chain, C6 hexahydric alcohols) that are essentially free of non-esterified polyols. In particular the invention relates to compositions of fatty acid esters of sorbitans (i.e., the inner monoethers of sorbitols). In a further aspect, the invention relates to a process for purification by removing non-esterified polyols from compositions composed of fatty acid esters of hexitans and non-esterified polyols.

Compositions of fatty acid esters of hexitans, hereinafter called sorbitan (fatty acid) esters for ease of reference, have wide spread utility in many areas as an emulsifying agent in the formation of water-in-oil and oil-in-water emulsions. They are readily commercially available as e.g., SPAN (registered trademark) emulsifying agents and ALKAMUL (reg. tm) sorbitan esters.

Sorbitan esters can be manufactured by processes described in U.S. patent No. 2,322,820. Generally, to produce sorbitan (hexitan) fatty acid esters, sorbitol (or a similar hexitol) and one or more fatty acids are reacted at a temperature greater than about 200 °C under a flow of inert gas, in the presence of an acidic or basic catalyst. A combination of mono-, di-, tri- and tetra-esters of sorbitan is produced. Also produced are impurities such as non-esterified polyols (hereinafter referred to as polyols) formed by the self-condensation of sorbitan molecules, unreacted sorbitans and sorbides (inner diethers of sorbitols, belonging to the class of hexides). These impurities are also present in commercial sorbitan esters. The washing step mentioned in example 1 of U.S. patent No. 2,322,820 has therefore clearly been disregarded by persons skilled in the art.

It has been found that these polyol impurities tend to form an undesirable sludge during emulsifying processes. Therefore, it is

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desirable to have sorbitan esters essentially free of these polyol impurities in order to avoid the formation of sludge.

It is therefore an object of the present invention to provide compositions of fatty acid hexitan esters, in particular sorbitan esters essentially free of non-esterified polyols, and a process to purify compositions of fatty acid hexitan (sorbitan) esters containing non-esterified polyol impurities. Moreover, such process for purification should be sufficiently selective to separate the closely resembling impurities from the sorbitan esters.

Accordingly, the invention provides compositions of fatty acid esters of hexitans and non-esterified polyols, having a content of non-esterified polyols in the range of 6.59 %wt to 0.05 %wt, preferably in the range of 3.14 %wt. to 0.19 %wt. Further, the invention provides a process for purification by removing non-esterified polyols from a composition composed of fatty acid esters of hexitans and non-esterified polyols, which process comprises the steps of:

- a) dissolving said composition in a solution comprising at least one hydrocarbon and at least one polar organic solvent to produce an organic solution of said composition;
- b) mixing or contacting said organic solution with an aqueous solution of at least one metal salt under conditions effective to extract said non-esterified polyols; and
- c) separating said organic solution from the aqueous solution, optionally followed by the recovery of the purified fatty acid esters of hexitans.

Suitable hydrocarbons are C<sub>5-20</sub> straight or branched alkanes, C<sub>5-20</sub> cycloalkanes and C<sub>6-20</sub> aromatics optionally substituted with inert substituents. Inert substituents are substituents which do not react with either polyols or sorbitan esters such as, for example, alkyl and halo. Examples of such hydrocarbons include, for example, pentane, hexane, heptane, octane, decane, hexadecane, cyclohexane, toluene, ethyl benzene and petroleum ether. The preferred hydrocarbons are pentanes, hexanes, cyclohexane and petroleum ether.

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Suitable polar organic solvents include oxygenated hydrocarbons such as straight, branched, cyclic or aromatic  $C_1$ - $C_{11}$  alcohols, aldehydes, ketones, esters and ethers; and nitrogen bases having a pKa value for their conjugate acids of less than about 10, preferably less than about 8. Examples of such alcohols include, for example, methanol, ethanol, n-propanol, isopropanol, butanol, pentanol, hexanol, cyclohexanol, benzyl alcohol and decanol. Examples of such aldehydes include, for example, formaldehyde, acetaldehyde and benzaldehyde. Examples of such ketones include, for example, acetone, methyl ethyl ketone, methyl phenyl ketone, and diethyl ketone. Examples of such esters include, for example, ethyl acetate, methyl isobutyrate and 2-ethylhexyl acrylate. Examples of such ethers include, for example, diethyl ether, dioxane and tetrahydrofuran. Examples of such nitrogen bases include, for example, acetonitrile, aniline, allyl amide and pyridine. Other suitable polar organic solvents include inert sulphur-containing solvents that do not react with polyols or sorbitan esters such as, for example, dimethyl sulphoxide and tetramethylene sulphone.  $C_1$ - $C_5$  alcohols, particularly isopropanol, are preferred because of the sorbitan ester solubility and ease of removal.

The impurity-containing (polyol-containing) sorbitan ester is first dissolved in a solution of a hydrocarbon and a polar organic solvent to produce a sorbitan ester solution (hereinafter organic solution). Lower boiling solvents such as hydrocarbons and polar organic solvents with less than about carbon atoms are preferred for ease of removal of the solvents after treatment. The organic solution preferably contains hydrocarbon and polar organic solvent, preferably in a ratio within the range of about 1:1000 to about 5:1, more preferably about 1:100 to about 3:1, most preferably about 1:10 to about 2:1.

The organic solution is then mixed or contacted with an aqueous metal salt solution. The salt solution contains water and from about 1 to about 20 weight percent, preferably, from about 5 to about 10 weight percent, based on the metal salt solution, of a

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metal salt. Suitable metal salts include, for example, metal halide salts, metal nitrate salts, metal acetate salts and metal sulphate salts. The preferred metal salt is a metal halide salt. The metal salt solution can optionally contain alcohols.

5        Suitable metal halide salts include, for example, alkali metal halides, alkaline earth metal halides, and trivalent metal halides. Examples of such halide salts include, for example LiCl, NaBr, KI, CaBr<sub>2</sub>, MgI<sub>2</sub>, NaCl, KCl, CaCl<sub>2</sub>, MgCl<sub>2</sub>, AlCl<sub>3</sub> and FeCl<sub>3</sub>. The preferred metal halide salts are alkali metal halides and alkaline  
10        earth metal halides, particularly, NaCl, KCl, CaCl<sub>2</sub> and MgCl<sub>2</sub>. Suitable sulphate salts are alkali metal sulphates, alkaline earth metal sulphates, and trivalent metal sulphates. Examples of such sulphate salts include, for example, sodium sulphate, calcium sulphate, aluminium sulphate and ferric sulphate. Suitable acetate  
15        salts are alkali metal acetates, alkaline earth metal acetates, and trivalent metal acetates. Examples of such acetate salts include, for example, sodium acetate, lithium acetate, magnesium acetate and aluminium acetate. Suitable nitrate salts are alkali metal ni-  
20        trates, alkaline earth metal nitrates, and trivalent metal nitrates. Examples of such nitrate salts include, for example, sodium nitrate, calcium nitrate, aluminium nitrate and ferric nitrate.

For convenience, the inventive process will further be described in terms of its preferred embodiment, in which a metal  
25        halide salt solution is used. In a typical process, the metal halide salt solution is added and mixed into the organic solution to produce a combined solution. The ratio of the organic solution to the metal halide salt solution is preferably within the range of about 10:1 to about 1:10, more preferably about 5:1 to about 1:5.  
30        Then, the combined solution is allowed to phase separate into an organic phase containing the hydrocarbon, the polar organic solvent and sorbitan ester and an aqueous phase containing water, metal halide salt and polyol impurities. The metal halide salt solution is preferably heated to a temperature within the range of about  
35        25 °C to about 90 °C, more preferably about 45 °C to about 80 °C,

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with the upper limit below about the boiling point of the organic solvents used. For a hexane/isopropanol solution, the metal halide salt solution is preferably heated at a temperature within the range of about 50 °C to about 55 °C, to hasten the phase separation. Subsequently, the aqueous phase is separated from the organic phase and then the purified sorbitan ester is recovered by removing the organic solvents by flashing, distilling or other means for removing the organic solvents including hydrocarbons and the polar organic solvents. At least a portion of the separated aqueous phase can be recycled to the mixing stage and used for the further removal of polyol impurities.

Any of the known methods in the art to separate organic/aqueous liquid phases can be utilized in the invention. The mixing and the phase separation steps can be carried out in either a single vessel or multiple vessels. For example, the steps may be conducted in one agitated vessel or other suitable mixing device, and phase separation can be carried out in separate gravity settling vessel or other suitable mechanical separation device. These steps also can be repeated in multiple stages either batch or continuous mode. For example, multiple stage mixer-settlers, gravity extraction column, centrifugal extractor or other device known to those skilled in the art.

In another embodiment, the organic sorbitan ester solution can be contacted with the metal salt solution to remove the polyol impurities. In the inventive process, the polyols can be substantially extracted (at least about 60%, preferably at least about 70%, more preferably at least about 90%) into the metal salt solution.

For example, the polyols can be extracted by contacting the organic solution with the metal salt solution in a liquid-liquid two-phase contactor such as described in U.S. Pat. No. 3,351,434, the disclosure of which is herein incorporated by reference. In general, the organic solution and the metal salt solutions are pumped through a two-phase contactor tank in their different strata. The polyol impurities will be transferred to the metal salt

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solution when a disk wettable by the organic solution rotates between the two solution layers. The disk is such that it is only wettable by the organic solution containing the sorbitan ester. The purified sorbitan fatty acid esters may then be recovered from the treated organic solution.

The organic sorbitan ester solution can also be contacted with the metal salt solution to remove the polyol impurities by a dispersion-free solvent extraction method such as, for example, by a FIBER-FILM contactor (FIBER-FILM is a trade mark) (from Merichem Company) and hollow-fibre membrane technology. The FIBER-FILM contactor is described in U.S. Pat. Nos. 3,754,377; 3,758,404; and 3,839,487, the disclosure of which is herein incorporated by reference. In general, the aqueous metal salt solution is introduced onto the outside surface of continuous, small diameter fibres (e.g. glass fibres and metal fibres) placed inside a retained cylinder. The metal salt solution adheres to the fibre surface in preference to the sorbitan ester solution which flows through and parallel to the aqueous-wetted fibre material.

The hollow-fibre membrane technology is described in U.S. Pat. Nos. 5,053,132; 4,997,569; 4,921,612 and 4,789,468, the disclosure of which is herein incorporated by reference. The hollow-fibre membrane technology is also described in Prasad, R. and Sirkar, K.K., "Dispersion-Free Solvent Extraction with Microporous Hollow-Fibre Modules," AIChE J., 34(2), 177 (1988). A hydrophilic microporous hollow-fibre module, a hydrophobic microporous hollow-fibre module or a membrane having both a hydrophilic side and a hydrophobic side can be used in the inventive process. In general, a hydrophilic microporous hollow-fibre module is preferred over a hydrophobic module because the solute distribution coefficient between organic and aqueous phases are less than 1. A hydrophilic microporous hollow-fibre module can be a regenerated cellulose hollow-fibre or CUPROPHAN type hollow-fibre (from ENKA, FRG) for example. The two phases can be flowing concurrently or flowing in the opposite direction at either tube side or shell side. The aqueous phase flow (metal salt solution) can be tube side or shell



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side, but preferably shell side with the organic phase (sorbitan ester solution) kept at a pressure higher than the aqueous phase pressure to oppose the tendency of the aqueous phase which preferentially wets the hydrophilic membrane to flow through the membrane and disperse in the organic phase. Alternatively, an asymmetrical-ly-wettable, porous membrane having a hydrophilic side and a hydrophobic side can be used for the extraction. These membranes can be a composite structure having a hydrophilic layer such as, for example, porous regenerated cellulose, porous cellulose-  
5 acetate, microporous glass and porous porcelain and a hydrophobic layer such as, for example, porous polyethylene, porous polypropylene and porous polytetrafluoroethylene. The treated-sorbitan esters are recovered from the extracted-sorbitan ester solution.  
10

The inventive process removes non-esterified polyols while leaving the sorbitan esters undisturbed. Further, the inventive process is very efficient and effective in removing non-esterified polyols. Also, it has been found that the process of this invention has short phase separation time.  
15

#### Illustrative Embodiment

20 The following illustrative embodiments describe the process of the invention and are provided for illustrative purposes and are not meant as limiting the invention.

#### Example 1

This example demonstrates purification of impurity-containing sorbitan esters according to the invention.  
25

50 Millilitres of SPAN 20 emulsifying agent (SPAN is a trade mark) (sorbitan monolaurate from Imperial Chemical Industries, ICI) was dissolved in a mixture of 50 ml of hexanes and 50 ml of isopropyl alcohol. The solution was poured into a 250 ml separatory  
30 funnel and 50 ml of 10 %wt aqueous sodium chloride solution was added. The funnel was stoppered, shaken, then placed upright in a ring-stand to allow phase separation to occur. The salt solution was at ambient temperature. Total phase separation was complete in 10 minutes. The organic phase was collected and the organic  
35 solvent was flashed off. The recovered sorbitan monolaurate was

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analyzed by gas chromatography and compared to an identical sample of unwashed SPAN 20 (tm) emulsifying agent. The results are shown in Table 1. The results show a decrease in the amount of polyols present.

Table 1

<u>SAMPLE</u>	<u>Sorbitol</u>	<u>Total FA **</u> (%wt)	<u>Sorbide ***</u> (%wt)	<u>Sorbitan***</u> (%wt)
Unwashed	ND*	2.58	7.34	5.99
Washed	ND	2.67	0.19	ND

- 5     \*     ND denotes not detected, i.e., below detection limit of method  
          at less than 0.05 %wt or less than 0.3 %wt for total sorbitol.  
      \*\*   FA denotes fatty acids, i.e., lauric acid.  
      \*\*\*   non-esterified polyols (condensed and unreacted)

Example 2

- 10       This example demonstrates another embodiment of the inventive process.

- 50 Millilitres of SPAN 20 (tm) emulsifying agent (sorbitan monolaurate from ICI) was dissolved in a mixture of 50 ml of hexanes and 50 ml of isopropyl alcohol. The solution was poured  
15   into a 250 ml separatory funnel and 50 ml of 10 %wt aqueous sodium sulphate solution was added. The funnel was stoppered, shaken, then placed upright in a ring-stand to allow phase separation to occur. The salt solution was at ambient temperature. Total phase separation was complete in 8 minutes. The organic phase was  
20   collected and the organic solvent was flashed off. The recovered sorbitan monolaurate was analyzed by gas chromatography. The results are shown in Table 2.

Example 3

- 25       This example demonstrates another embodiment of the inventive process.

      The purification was carried out in a similar manner to Example 2 except petroleum ether (a low boiling fraction, 30-60 °C,

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of petroleum consisting chiefly of hydrocarbons of the methane series, principally pentanes and hexanes from Baxter Co.) was substituted for hexanes. The phase separation was complete in 7.5 minutes. The results are shown in Table 2.

#### 5 Comparative Example 1

This example demonstrates a purification method where neat sorbitan monolaurate was washed with aqueous sodium sulphate solution as disclosed in US 2,322,820.

10 50 Millilitres of SPAN 20 (tm) emulsifying agent (sorbitan monolaurate from ICI) was added to a 250 ml separatory funnel. 50 ml of a 10 %wt solution of aqueous sodium sulphate solution was added. The salt solution was at ambient temperature. The funnel was stoppered and shaken whereupon a thick white emulsion formed. After 12 hours, approximately 30% phase separation had occurred. 15 After 4 days approximately 60% phase separation had occurred. Total phase separation required 1-2 weeks. The results are shown in Table 2.

Table 2

<u>SAMPLE</u>	TOTAL		
	<u>FA*</u>	<u>SORBIDE**</u>	<u>SORBITAN**</u>
	(%wt)	(%wt)	(%wt)
Untreated SPAN 20 (tm)	2.23	6.94	5.86
Comparative Example 1	2.21	3.56	3.03
Example 2	2.43	1.74	1.21
Example 3	2.47	1.99	1.15

\* FA denotes fatty acids.

\*\* Polyols (condensed and unreacted)

#### 20 Example 4

This example demonstrates another embodiment of the inventive process.

25 Salt solutions of 10 %wt concentration were prepared for calcium chloride, sodium chloride and sodium sulphate. These salt solutions were kept at room temperature (25 °C). Solutions of

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SPAN 20 (tm) emulsifying agent (sorbitan monolaurate from ICI) were prepared by dissolving 80 ml of the sorbitan ester in 200 ml of a 1:1 mixture of isopropanol and toluene. To each of 4 separatory funnels was added 50 ml of the SPAN 20 (tm) solution. Each of the  
5 funnels were then charged with 50 ml of a salt wash solution. The fourth flask was charged with 50 ml of deionized water. Each funnel was then stoppered, shaken vigorously 15 seconds, vented, uprighted, then unstoppered. Bulk phase separation occurred most quickly for calcium chloride, in less than 10 minutes. Sodium  
10 chloride and sodium sulphate bulk separated within 15 minutes. The solution washed with deionized water did not separate after 1 week: incomplete separation was evident after 2 weeks.

#### Example 5

15 This example demonstrates another embodiment of the inventive process.

Solutions of calcium chloride were prepared at 1, 3, 5 and 10 %wt concentration. These solutions were warmed to 50 °C by placing in an oven. 100 g of SPAN 20 (tm) emulsifying agent (sorbitan monolaurate from ICI) was dissolved in 270 g of a 1:1  
20 solution of isopropanol and toluene. The concentration of the sorbitan ester was 27%. Four 125 ml separatory funnels were used, each was charged with 50 ml of the sorbitan ester solution. Additionally, each funnel was charged with the warmed salt solution, each at a different concentration. Each flask was stoppered and  
25 shaken vigorously for 1 minute, vented, then uprighted and unstoppered. Bulk phase separation occurred most quickly for the sample washed with 10% salt solution, less than 10 minutes. The other samples bulk phase separated in greater than 10 minutes, but less than 2 hours. Total separation occurred in less than 2 hours for  
30 all samples.

#### Example 6

Timed experiments were conducted using 10% sodium chloride solutions and 33% SPAN 20 (tm) emulsifying agent solutions in 1:1 isopropanol:toluene.

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150 Millilitres of the emulsifying agent solution was added to a 500 ml separatory funnel. 100 ml of the salt wash solution was added, either warmed at 60 °C or at room temperature (25 °C). The flask was stoppered, shaken vigorously 10 seconds, vented, upright-  
5 ed and unstoppered. Bulk phase separation occurred in 5:00 minutes for salt wash at room temperature and 2:56 minutes for warmed salt solution.

Example 7

10 The temperature effect on the inventive process is illustrated.

10 Percent by weight salt solutions of potassium chloride, sodium chloride, calcium chloride and sodium sulphate were prepared. Each salt solution was divided into two portions. One portion was warmed at 60 °C. The other portion was kept at room  
15 temperature. A solution of SPAN 20 (tm) emulsifying agent was prepared by dissolving 200 ml of surfactant in 200 ml of isopropanol and 200 ml of toluene. The solution was stirred for 30 minutes to completely dissolve the material. To each of 4 separatory funnels was added a 50 ml charge of the surfactant solution. 50 ml  
20 of salt wash solution was then added. Each flask was stoppered, shaken vigorously for 10 seconds, vented, uprighted and then unstoppered. The time for bulk phase separation to occur was measured. The time was seen to vary with the temperature of the wash solution, faster bulk phase separation occurring  
25 when the salt wash solution was warmed. The results are shown in Table 3.

Table 3

<u>Salt Solution</u>	<u>Room Temperature</u>	<u>Warmed</u>
NaCl	1:40 (minutes:seconds)	1:08
KCl	1:42	1:04
CaCl <sub>2</sub>	1:17	1:00
NaSO <sub>4</sub>	1:58	1:18

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Example 8

This example demonstrates further embodiments of the inventive process.

5 A 10 %wt solution of sodium chloride was warmed at 60 °C by placing in a warming oven. In a typical run, 20 ml of SPAN 20 (tm) emulsifying agent (sorbitan monolaurate from ICI) was dissolved in a mixture of 20 ml of isopropanol and 20 ml of a cosolvent. Cosolvents were hexane, petroleum ether, cyclohexane and hexadecane. The sorbitan ester solutions were placed in 250 ml separa-  
10 tory funnels. 50 ml of warmed salt solution was added to each funnel, which was then stoppered, shaken and vented. The funnels were uprighted, stopper removed, and the time for bulk phase separation to occur measured. Results are shown in Table 4 below.

Table 4

<u>Cosolvent</u>	<u>Time</u>
hexane	0:54 (minutes:seconds)
petroleum ether	1:10
cyclohexane	1:19
hexadene	12:00

Example 9

15 This example demonstrates another embodiment of the inventive process.

In a vial, 5 ml of SPAN 20 (tm) emulsifying agent (sorbitan monolaurate from ICI) was dissolved in a mixture of 5 ml of hexane and 5 ml of the following organic solvents and shaken. The organic  
20 solvents were acetone, benzaldehyde, dioxane, tetrahydrofuran, ethyl acetate, pyridine, acetonitrile, methylene chloride, tetramethylene sulphone, and diethyl amine. 10 ml of 10% aqueous NaCl was then added and the vials were vigorously shaken. Phase separation for acetone, benzaldehyde dioxane, tetrahydrofuran, ethyl  
25 acetate, pyridine and acetonitrile occurred within 2-24 hours.

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Methylene chloride and tetramethylene sulphone did not phase separate readily. A chemical reaction occurred with diethyl amine.

Example 10

5 This example demonstrates another embodiment of the inventive process.

A 10 %wt solution of aluminium sulphate, ferric chloride and ferric nitrate was prepared. A sorbitan ester solution was prepared by dissolving 50 ml of SPAN 20 (tm) emulsifying agent (sorbitan monolaurate from ICI) in 50 ml of isopropanol and 50 ml of hexane. The mixture was stirred to give a homogeneous solution. The solution of aluminium sulphate, ferric chloride and ferric nitrate were each placed in a 250 ml separatory funnel. 50 ml of the sorbitan ester solution was also placed in each of the three 250 ml separatory funnels. The funnels were stoppered, shaken, 15 vented, then uprighted to allow phase separation to occur. For each solution, phase separation occurred within 10 minutes. For ferric chloride and ferric nitrate solutions, the salt solutions were orange.

Example 11

20 This example demonstrates another embodiment of the inventive process.

SPAN 20 (tm) emulsifying agent (sorbitan monolaurate from ICI) was dissolved in mixtures of isopropanol (IPA)/hexane in various ratios:

	<u>Combination</u>	<u>Ratio</u>	<u>SPAN 20 (tm) (ml)</u>
1.	5 ml IPA/25 ml hexane	1:5	30
2.	5 ml IPA/50 ml hexane	1:10	55
3.	25 ml IPA/5 ml hexane	5:1	30
4.	50 ml IPA/5 ml hexane	10:1	55

25 The above combinations were stirred to homogeneous solutions. 1 and 2 gave cloudy pale yellow liquids. 2 was almost opaque. 4 was transparent. The contents of each flask were poured into each of 4

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separatory funnels. To 1 and 3 was added 30 ml of 10% aqueous calcium chloride. To 2 and 4 was added 55 ml of the same. Each flask was stoppered, shaken, vented, then uprighted to allow phase separation to occur.

- 5           Phase separation for 3 and 4 was rapid, within 5 minutes. Phase separation for 1 and 2 was much slower, required 18 hours or more. The water used in the wash step was recovered to determine if IPA dissolved in it. Based on these results, it appears that IPA does not significantly dissolve in the salt aqueous phase.

#3	30 ml wash used	30 ml recovered
#4	55 ml wash used	-60 ml recovered

- 10           The aqueous layers of 1 and 2 remained thick and milky after 18 hours. When recovered, these layers were much greater in volume than the amount used in the wash. It appears that phase separation was not complete after 18 hours, and organic phase was emulsified in the wash phase.

15   Example 12

This example demonstrates another embodiment of the inventive process.

- 20           A solution of SPAN 20 (tm) emulsifying agent (sorbitan mono-laurate from ICI) was prepared by dissolving 150 ml of SPAN 20 (tm) emulsifying agent in 150 ml hexane and 150 ml isopropanol. The mixture was stirred until the solution was homogeneous. The resulting solution was washed with 10% aqueous solution of sodium sulphate.

1.           aqueous:organic                   10:1

- 25           Ten parts of aqueous phase were used to wash 1 part of the SPAN 20 (tm) solution. Phase separation occurred overnight, approximately 18 hours. Phases were clear after this time.

2.           aqueous:organic                   1:1

- 30           1 part aqueous phase was used to wash 1 part of the SPAN 20 (tm) solution. Phase separation was complete in 1 minute, 15 seconds.



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3.           aqueous:organic                           1:10

1 part of aqueous phase was used to wash 10 parts of the SPAN 20 (tm) solution. The water phase disappeared, apparently dissolving up into the organic phase. Salt crystals formed and  
5       adhered to the sides of the separatory funnel.

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C L A I M S

1. A composition composed of fatty acid esters of hexitans and non-esterified polyols, having a content of non-esterified polyols in the range of 6.59 %wt to 0.05 %wt.
2. A composition as claimed in claim 1, having a content of non-esterified polyols in the range of 3.14 %wt to 0.19 %wt.
3. A composition as claimed in claim 1 or 2, wherein the fatty acid esters of hexitans are fatty acid esters of sorbitans.
4. A process for purification by removing non-esterified polyols from a composition composed of fatty acid esters of hexitans and non-esterified polyols, which process comprises the steps of:
  - a) dissolving said composition in a solution comprising at least one hydrocarbon and at least one polar organic solvent to produce an organic solution of said composition;
  - b) mixing or contacting said organic solution with an aqueous solution of at least one metal salt under conditions effective to extract said non-esterified polyols; and
  - c) separating said organic solution from the aqueous solution, optionally followed by the recovery of the purified fatty acid esters of hexitans.
5. A process as claimed in claim 4, wherein the fatty acid esters of hexitans are fatty acid esters of sorbitans.
6. The process of claim 4 or 5, wherein the salt solution is heated to a temperature within the range of about 20 °C to about 90 °C prior to step b).
7. The process of any one of claims 4-6, wherein the metal salt is a metal halide salt.
8. The process of claim 7, wherein the metal halide salt is selected from the group consisting of alkali metal halides and alkaline earth metal halides.

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9. The process of claims 8, wherein the metal halide salt is selected from the group consisting of LiCl, NaBr, KI, CaBr<sub>2</sub>, MgI<sub>2</sub>, NaCl, KCl, CaCl<sub>2</sub> and MgCl<sub>2</sub>.
10. The process of claim 9, wherein the metal halide salt is present in an amount of about 5 to 10 weight percent based on the salt solution.
11. The process of claim 10, wherein the metal halide salt is selected from the group consisting of NaCl, KCl, CaCl<sub>2</sub> and MgCl<sub>2</sub>.
12. The process of claim 11, wherein the ratio of the organic solution to the aqueous metal salt solution is within the range of about 10:1 to about 1:10.
13. The process of any one of claims 4-12, wherein the hydrocarbon is selected from the group consisting of C<sub>5-20</sub> alkanes, C<sub>5-20</sub> cycloalkanes, C<sub>6-20</sub> aromatics.
14. The process of claim 13, wherein the hydrocarbon is selected from the group consisting of pentane, hexane, heptane, octane, decane, hexadecane, cyclohexane, toluene, ethyl benzene, and petroleum ether.
15. The process of claim 14, wherein the hydrocarbon is pentane or hexane.
16. The process of any one of claims 4-15, wherein the polar organic solvent is selected from the group consisting of oxygenated hydrocarbons, nitrogen bases having a pKa of their conjugated acids of less than about 10 and inert sulphur-containing solvents.
17. The process of claim 16, wherein the polar organic solvent is selected from the group consisting of C<sub>1</sub>-C<sub>11</sub> alcohols, aldehydes, ketones, esters and ethers.
18. The process of claim 17, wherein the organic solvent is an alcohol.
19. The process of claim 18, wherein the alcohol is selected from the group consisting of methanol, ethanol, n-propanol, isopropanol, butanol, pentanol, hexanol, cyclohexanol, benzyl alcohol and decanol.
20. The process of claim 19, wherein the alcohol is a C<sub>1</sub>-C<sub>5</sub> alcohol.

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21. The process of any one of claims 4-20 in which the organic solution is contacted with the aqueous metal salt solution in a liquid-liquid two-phase contactor.
- 5 22. The process of claim 21 in which the organic solution is contacted with the aqueous metal salt solution in a FIBER FILM contactor.
23. The process of claim 21 in which the organic solution is contacted with the metal salt solution through a hollow-fibre membrane.