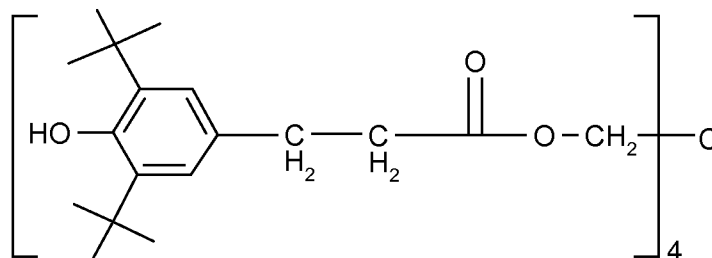


Liquid Stabilizer Mixtures

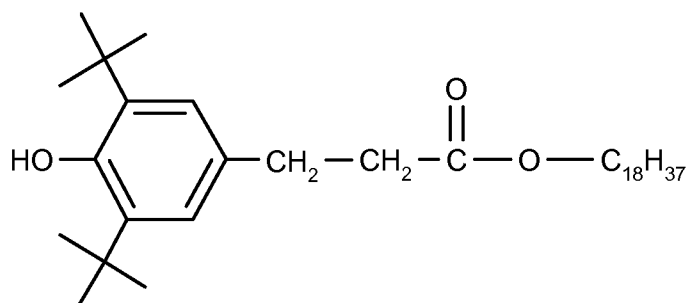
The present invention relates to processes for providing stable liquid blends of a) pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate, b) octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate and c) tris-(2,4-di-tert-butylphenyl)phosphite. The liquid blends of stabilizers are useful as stabilizers for organic materials, for instance synthetic polymer stabilization.

Pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate, octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate and tris-(2,4-di-tert-butylphenyl)phosphite are well known polymer stabilizers, commercially available as IRGANOX 1010, IRGANOX 1076 and IRGAFOS 168 respectively. IRGANOX 1010 and IRGANOX 1076 are hindered phenolic antioxidants and IRGAFOS 168 is a phosphite processing stabilizer.

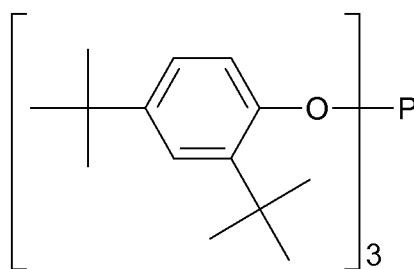
Pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate, IRGANOX 1010 is:



Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate, IRGANOX 1076 is:



Tris-(2,4-di-tert-butylphenyl)phosphite, IRGAFOS 168 is:



The stabilizers are all solids at room temperature. IRGANOX 1010 has a melting point of ca. 120°C. IRGANOX 1076 has a melting point of ca. 56°C. IRGAFOS 168 melts at ca. 185°C.

It would be advantageous for industry to be able to provide a liquid blend of these well known stabilizers. A liquid blend could be pumped to be incorporated into a polymer during melt processing or during solution polymerization. Dosing would be more accurate and incorporation would be easier to perform.

Two processes have now been found for forming a stable liquid blend of a)
pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate),
b) octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate and
c) tris-(2,4-di-tert-butylphenyl)phosphite.

First process.

Disclosed is a process for forming a stable liquid blend of

- a) pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate,
- b) octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate and
- c) tris-(2,4-di-tert-butylphenyl)phosphite,

which process comprises preparing a solid mixture comprising about 2 parts to about 3 parts by weight a), about 2 parts to about 3 parts by weight b) and about 2 parts to about 12 parts by weight c), heating the mixture to 185°C or higher for a sufficient time to obtain a clear liquid blend of a), b) and c), cooling the liquid blend to a temperature of from 90°C to 140°C and maintaining the liquid blend at a temperature of from 90°C to 140°C,

where the liquid blend of a), b) and c) is stable at the temperature at which it is maintained for greater than 120 hours.

Preferably, the process comprises preparing a solid mixture comprising about 5 parts to about 11 parts by weight c).

More preferably, the process comprises preparing a solid mixture comprising about 8 parts to about 11 parts by weight c).

Preferably, the liquid blend of a), b) and c) is maintained at a temperature of from 90°C to 125°C.

More preferably, the liquid blend of a), b) and c) is maintained at a temperature of from 90°C to 110°C.

Second Process.

Further disclosed is a process for forming a stable liquid blend of

- a) pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate,
- b) octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate and

c) tris-(2,4-di-tert-butylphenyl)phosphite,

which process comprises preparing a solid mixture comprising about 2 parts to about 3 parts by weight a) and about 2 parts to about 3 parts by weight b), heating the mixture to 90°C or higher for a sufficient time to form a clear liquid mixture of a) and b), adding about 2 parts to about 12 parts by weight c) thereto to obtain a clear liquid blend of a), b) and c) and maintaining the liquid blend at a temperature of from 90°C to 140°C,

where the liquid blend of a), b) and c) is stable at the temperature at which it is maintained for greater than 120 hours.

Preferably, the process comprises adding about 5 to about 11 parts by weight c) to the liquid mixture of a) and b).

More preferably, the process comprises adding about 8 to about 11 parts by weight c) to the liquid mixture of a) and b).

Preferably, the liquid blend of a), b) and c) is maintained at a temperature from 90°C to 125°C.

More preferably, the liquid blend of a), b) and c) is maintained at a temperature from 90°C to 110°C.

Cooling may be active cooling or allowing slowly to cool at ambient conditions.

Stable means no observed precipitation or solids at a certain temperature for greater than a certain length of time.

Example 1

10 gram samples of IRGANOX 1010/IRGANOX 1076/IRGAFOS 168 are dry mixed and placed in a glass vial in a 200°C oven for half an hour. Clear liquid blends are achieved. The clear liquid blends are then transferred to ovens set at 150°C, 140°C

and 135°C to observe if any precipitation occurs at these temperatures at 24, 48, 72, 96 and 120 hour time periods.

The following results are observed at 120 hours:

Formulation	1	2	3	4	5
IRGANOX 1010 wt.%	9	11	13.8	16	18
IRGANOX 1076 wt.%	22	20	17.2	15	13
IRGAFOS 168 wt.%	69	69	69	69	69
Precipitation Temp. (°C)	>140	>140	<135	<135	<135

Formulations 3-5 are very stable liquid blends. Higher levels of IRGANOX 1010 are advantageous even though its melting point is much higher than IRGANOX 1076.

Example 2

Example 1 is repeated, except that in this case, glass vials containing mixtures of IRGANOX 1010 and IRGANOX 1076 are placed in an oil bath and heated to greater than 90°C to obtain a clear liquid. IRGAFOS 168 is slowly added to the liquid with stirring to obtain a clear liquid blend. The liquid blends are transferred to ovens set at 150°C, 140°C and 135°C to observe if any precipitation occurs at these temperatures at 24, 48, 72, 96 and 120 hour time periods.

Claims

1. A process for forming a stable liquid blend of

- a) pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate,
- b) octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate and
- c) tris-(2,4-di-tert-butylphenyl)phosphite,

which process comprises preparing a solid mixture comprising about 2 parts to about 3 parts by weight a), about 2 parts to about 3 parts by weight b) and about 2 parts to about 12 parts by weight c), heating the mixture to 185°C or higher for a sufficient time to obtain a clear liquid blend of a), b) and c), cooling the liquid blend to a temperature of from 90°C to 140°C and maintaining the liquid blend at a temperature of from 90°C to 140°C,

where the liquid blend of a), b) and c) is stable at the temperature at which it is maintained for greater than 120 hours.

2. A process according to claim 1 which comprises preparing a solid mixture comprising about 5 parts to about 11 parts by weight c).

3. A process according to claim 1 which comprises preparing a solid mixture comprising about 8 parts to about 11 parts by weight c).

4. A process according to claim 1 where the liquid blend of a), b) and c) is maintained a temperature of from 90°C to 125°C.

5. A process according to claim 1 where the liquid blend of a), b) and c) is maintained a temperature of from 90°C to 110°C.

6. A process for forming a stable liquid blend of

- a) pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate,
- b) octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate and
- c) tris-(2,4-di-tert-butylphenyl)phosphite,

which process comprises preparing a solid mixture comprising about 2 parts to about 3 parts by weight a) and about 2 parts to about 3 parts by weight b), heating the mixture to 90°C or higher for a sufficient time to obtain a clear liquid mixture of a) and b), adding about 2 parts to about 12 parts by weight c) thereto to obtain a clear liquid blend of a), b) and c) and maintaining the liquid blend at a temperature of from 90°C to 140°C,

where the liquid blend of a), b) and c) is stable at the temperature at which it is maintained for greater than 120 hours.

7. A process according to claim **6** which comprises adding about 5 to about 11 parts by weight c) to the liquid mixture of a) and b).

8. A process according to claim **6** which comprises adding about 8 to about 11 parts by weight c) to the liquid mixture of a) and b).

9. A process according to claim **6** where the liquid blend of a), b) and c) is maintained at a temperature from 90°C to 125°C.

10. A process according to claim **6** where the liquid blend of a), b) and c) is maintained at a temperature from 90°C to 110°C.