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(54) **TRICHLORURE DE MONOBUTYLETAIN STABILISE**

(54) **STABILIZED MONOBUTYLTIN TRICHLORIDE**

(57) The invention relates to monobutyltin trichloride which, as a stabilizer, comprises one or more glycerol esters of optionally unsaturated aliphatic carboxylic acids having 1 to 18 carbon atoms.

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ABSTRACT OF THE DISCLOSURE

The invention relates to monobutyltin trichloride which, as a stabilizer, comprises one or more glycerol esters of optionally unsaturated aliphatic carboxylic acids having 1 to 18 carbon atoms.

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STABILIZED MONOBUTYLTIN TRICHLORIDEFIELD OF THE INVENTION

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The invention relates to a monobutyltin tri-chloride (MBTC) which is particularly suitable for hollowware coating and which is stabilized against the influence of moisture and darkening.

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BACKGROUND OF THE INVENTION

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It is known to apply coatings of all sorts of metal oxides, in particular of tin dioxide, to glass containers in order to improve their resistance to impact and abrasion. This tin dioxide coating acts as a primer for the so-called cold end coating applied after the annealing process.

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Customarily, tin compounds are brought into contact with the hot glass surface in vapor or spray form in the so called hot end coating, a thin tin dioxide coating being produced pyrolytically. On account of their physical properties, such as water solubility, vaporizability and the like, and of their low toxicity, monoorganotin trichlorides are, in particular, employed for this purpose (DE-C-25 41 710).

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Depending on the purity and quality of the products, however, it has been shown in the processing of these compounds that due to the occurrence of solid particles, in particular after long storage,

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1 significant trouble can occur with the glass coating
process.

5 According to EP-A-0 132 024, to avoid the
occurrence of solid particles, dopants of all sorts of
types, in particular alcohols, are added to the
monoalkyltin trihalides.

10 A further problem which occurs with
monobutyltin trichloride after long storage is
darkening, which can lead at its most pronounced to a
dark brown coloration. This dark coloration of the
product can likewise significantly adversely affect
the working process, in particular in measuring and
metering operations. Furthermore, with monobutyltin
15 trichloride visible crystallizations very rapidly
occur under the influence of moisture; on longer
action, presumably caused by hydrate formation, the
formation of a liquid multiphase system can even
20 occur.

BRIEF SUMMARY OF THE INVENTION

25 It has been found that a monobutyltin
trichloride composition stabilized against the effects
of moisture and darkening is obtained when one or more
glycerol esters of aliphatic carboxylic acids are
added to it. Suitable esters are the glycerol mono-,
di- and triesters of aliphatic, optionally unsaturated
30 carboxylic acids having 1 to 18 carbon atoms. Suitable

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1 glycerol esters are, for example, acetates,
propionates, 2-ethylhexanoates, valerates, caprylates,
dodecanoates and octadecanoates. Particularly active
5 here are glycerol mono- and diacetates or mixtures of
the two, and 2-ethylhexanoic acid esters.

Amounts of additive from 0.5 to 10% by
weight can be employed; as a rule 0.5 to 1.5% by
weight is adequate.

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DETAILED DESCRIPTION OF THE INVENTION

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The stabilizers employed according to the
invention are in this case surprisingly considerably
more active than the dopants of the prior art. Thus,
for example (see examples), when using 1% glycerol
monoacetate crystallization only occurs to 15%, while
in the case of the dopants according to EP-A-0 132 024
20 crystallization is present on the entire surface after
the same time.

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The activity of the stabilizers employed is
explained in greater detail by means of the following
examples.

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Testing of MBTC for stability to environmental
moisture

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In order to test the effects of
environmental moisture on MBTC, MBTC samples standing

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1 open are observed and the time is determined after
which noticable changes in the product can be recorded
in each case.

5 For this purpose, clock glasses of 10 cm
diameter are provided with 2 ml each of MBTC sample
and are exposed next to one another to the surrounding
air while standing open in a hood.

10 The hood front flap is kept open 15 cm above
the bottom; the center of the sample glasses is
located at a distance of 10 cm from the opening.

15 The tests are repeated using different
positions of the clock glasses relative to one another
and the test results meaned in order to compensate for
any possible test scatter.

In the experimental arrangement, this
crystallization begins at the edge of the liquid
surface and then continues inwards.

20 The table indicates the proportion of the
surface crystallized relative to the total surface
area. The lower the proportion of the surface
crystallized, the better the stabilizing action.

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Glmono: glycerol monoacetate
Gldiac: glycerol diacetate
Gltriac: glycerol triacetate
Gl-2eth-mono glycerol 2-ethylhexanoic acid monoester
30 Gl-2eth-di: glycerol 2-ethylhexanoic acid diester

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Test period (Hours)	MBTC without additions	MBTC +1% Ethanol *	MBTC +0.5% Glmono	MBTC +1% Glmono	MBTC +1.5% Glmono	MBTC +0.5% Gldiac	MBTC +1% Gldiac	MBTC +1.5% Gldiac
1	100%	15%	10%	5%	2%	10%	5%	1%
2	-----	50%	15%	10%	5%	15%	10%	5%
3	-----	100%	50%	15%	10%	40%	15%	10%

* Comparison experiment

Test period (Hours)	MBTC + 0.5% Gltriac	MBTC + 1% Gltriac	MBTC + 1.5% Gltriac	MBTC + 1% Gl-2eth-mono	MBTC + 1% Gl-2eth-di
1	15%	10%	5%	5%	5%
2	20%	15%	10%	5%	5%
3	50%	25%	15%	10%	10%

5 Testing of MBTC for stability to darkening

MBTC darkens on standing in the light, which leads to the initially almost water-clear liquid assuming a dark-brown appearance after some time.

10 An additional effect which is seen, in particular with the mono/diesters of glycerol, is a stabilization against this darkening, so that this process is considerably slowed.

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	Starting color	Stored 1 week	Stored 1 month	Stored 3 months
MBTC + 1% Ethanol	almost water-clear	yellow	light brown	dark brown
MBTC + 1% Glmono	almost water-clear	almost water-clear	almost water-clear	light yellow
MBTC + 1% Gldiac	almost water-clear	almost water-clear	almost water-clear	light yellow

* Comparison experiment

1 WHAT IS CLAIMED IS:

5 1. A composition of monobutyltin
trichloride, stabilized against crystallization and
against darkening, comprising monobutyltin trichloride
and an effective amount of a stabilizer selected from
the group consisting of glycerol esters of optionally
10 unsaturated aliphatic carboxylic acids having 1 to 18
carbon atoms, and mixtures thereof.

15 2. A stabilized composition of monobutyltin
trichloride according to Claim 1, characterized in that
the stabilizer is present in an amount from 0.5 to 10%
by weight of the composition.

20 3. A stabilized composition of monobutyltin
trichloride according to Claim 1 wherein the stabilizer
is glycerol monoacetate, glycerol diacetate or a
mixture of both.

25 4. A stabilized composition of monobutyltin
trichloride according to Claim 2 wherein the stabilizer
is glycerol monoacetate, glycerol diacetate or a
mixture of both.

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