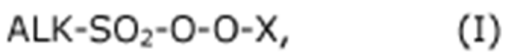
It is the object of the examples disclosed herein to provide an improved process for manufacturing alkanesulfonic acid, especially methanesulfonic acid, allowing improved reaction control. Further, requirements for sulfurtrioxide and alkanes should be not of relevance, meaning that not only absolute pure raw materials might be used, but that impurities do not affect negatively the reaction.

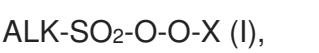
The object of the present disclosure is achieved, by use of a compound of the formula (I)



$$ALK\text{-}SO_2\text{-}O\text{-}O\text{-}X, \qquad (I)$$

wherein ALK is a branched or unbranched alkyl group, especially a methyl, ethyl, propyl, butyl, isopropyl, isobutyl group, or a higher alkyl group, and X = hydrogen, zinc, aluminium, an alkali or alkaline earth metal, as an initiator-precursor for preparing alkanesulfonic acids, especially methanesulfonic acids from alkane, especially methane, and sulfur trioxide, especially pure sulfur trioxide.

In an aspect, there is provided the use of a compound of the formula (I)



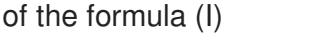
$ALK\text{-}SO_2\text{-}O\text{-}O\text{-}X$ (I),

wherein ALK is a branched or unbranched alkyl group, especially a methyl, ethyl, propyl, butyl, isopropyl, isobutyl group, or a higher alkyl group, and X = hydrogen, zinc, aluminium, an alkali or alkaline earth metal,

as an initiator-precursor for preparing alkanesulfonic acids, especially methanesulfonic acids from alkane, especially methane, and pure sulfur trioxide.

In another aspect, there is provided a mixture containing a compound

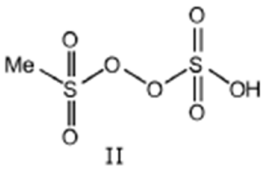
of the formula (I)



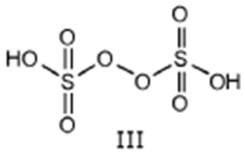
$ALK\text{-}SO_2\text{-}O\text{-}O\text{-}X$ (I),

wherein ALK is a branched or unbranched alkyl group, especially a methyl, ethyl, propyl, butyl, isopropyl, isobutyl group, or a higher alkyl group, and X = hydrogen, zinc, aluminium, an alkali or alkaline earth metal,

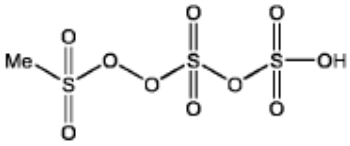
and at least one compound selected from the group consisting of following formulae (II) to (X)



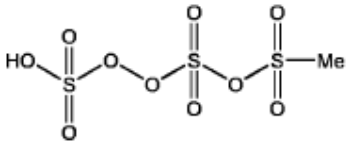

II



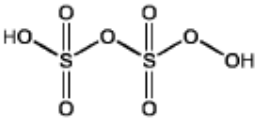

III



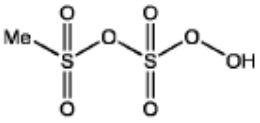

IV




V



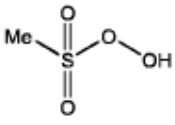

VI



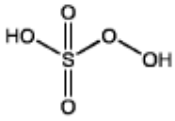

VII




VIII




IX




X

and combinations thereof.

In another aspect, there is provided the compound of the formula (I)

ALK-$SO_2$-O-O-X (I),

wherein ALK is a branched or unbranched alkyl group, especially a methyl, ethyl, propyl, butyl, isopropyl, isobutyl group, or a higher alkyl group, and X = hydrogen, zinc, aluminium, an alkali or alkaline earth metal,

or the mixture as defined above,

in sulfuric acid or alkylsulfonic acid, especially methanesulfonic acid or combinations thereof.

In another aspect, there is provided the use of the mixture as defined above as an initiator-precursor for preparing alkanesulfonic acids, especially methanesulfonic acid, from alkane, especially methane, and pure sulfur trioxide

In a further example, the object of the disclosure is achieved by a process for manufacturing the compound as defined above with X = H, comprising reacting an alkanesulfonic acid, especially methanesulfonic acid, with hydrogen peroxide. It might be followed by the isolation of the compound.

The object of the disclosure is achieved by a process for manufacturing alkanesulfonic acids, especially methanesulfonic acid, comprising the following steps:

- providing sulfur trioxide;

- reacting the sulfur trioxide with an alkane, especially methane, in a high-pressure autoclave or laboratory reactor;

- setting a pressure of from 1 to 200 bar;

- preparing the initiator-precursor according to formula (I), by reacting an alkanesulfonic acid or a solution containing the alkanesulfonic acid with a 30 % to 100 % (w/w) hydrogen peroxide solution;

- adding the initiator-precursor according to formula (I) or a solution thereof to the reactor;

- controlling the temperature of the reaction mixture at 0 °C to 100 °C;

- if necessary purifying the reaction product, for example, by distillation or extraction.

In another aspect, there is provided a process for manufacturing alkanesulfonic acids, especially methanesulfonic acid, comprising the following steps:

- providing pure sulfur trioxide;

- reacting the pure sulfur trioxide with an alkane, especially methane, in a high-pressure autoclave or laboratory reactor;

- setting a pressure of from 1 to 200 bar;

- preparing the compound of the formula (I)

ALK-$SO_2$-O-O-X (I),

wherein ALK is a branched or unbranched alkyl group, especially a methyl, ethyl, propyl, butyl, isopropyl, isobutyl group, or a higher alkyl group, and X = hydrogen, zinc, aluminium, an alkali or alkaline earth metal,

or the mixture as defined above,

as initiator-precursor by reacting an alkanesulfonic acid or a solution containing the alkanesulfonic acid with a 30 % to 100 % (w/w) hydrogen peroxide solution;

- adding the initiator-precursor or a solution thereof to the reactor;

- controlling the temperature of the reaction mixture at 0 °C to 100 °C;

- if necessary purifying the reaction product, for example, by distillation or extraction.

In a further aspect, there is provided the use of a compound of the formula (I)

ALK-$SO_2$-O-O-X (I),

wherein ALK is a branched or unbranched alkyl group, especially a methyl, ethyl, propyl, butyl, isopropyl, isobutyl group, or a higher alkyl group, and X = hydrogen, zinc, aluminium, an alkali or alkaline earth metal, or the mixture as defined above in a device for performing the process as defined above, wherein the device comprises:

- a reactor (1) in which pure sulfur trioxide reacts with the compound of formula (I) as initiator-precursor to form an initiator; and reaction of said "in-situ"-built initiator with an alkane, especially methane;

- a distillation means (2) for distilling the product formed in the reactor (1) ; and

- a filling means (3); as well as

- connection means to connect the reactor (1) with the distillation means (2) , and the filling means (3) with the distillation means (2).

After the reaction has taken part, the reaction mixture contains essentially of the respective alkanesulfonic acid, especially methanesulfonic acid, as well as sulfuric acid. This mixture of alkanesulfonic acid, especially methanesulfonic acid (MSA), and $H_2S0_4$ might afterwards be used as the respective mixture. The combination of an alkanesulfonic acid, especially methanesulfonic acid, and sulfuric acid provides a strong acid in which even gold might be dissoluted enabling different fields of technical applicability.

Alternatively, the alkanesulfonic acid, especially MSA, might be separated i.e. the method of the invention comprises the optional step of the purifying the reaction product, which might be done by distillation or extraction.

But also alkanesulfonic acids, and specially methanesulfonic acids, might be used in different technical fields, i.e. as cleaning agent (cleaning comprising the area of

cleaning and caring, home care as well as industrial and institutional cleaning of hard and soft surfaces, i.e. in dishwashing, commercial laundry, cleaning and sanitation, vehicle and transportation care, concrete cleaning, membrane cleaning, and others), for regeneration of ion exchange resins, in galvanic proceedings, in the area of oil, gas, mining, treatment of metals and/or their surfaces, in different areas of pharmaceutical, chemical and argro-chemical industry or in the production of biodiesel. MSA might also be used in galvanization process of plastics, the broad area of batteries, such as lead

-

# C L A I M S

1. Use of a compound of the formula (I)

ALK-$SO_2$-O-O-X (I),

wherein ALK is a branched or unbranched alkyl group, especially a methyl, ethyl, propyl, butyl, isopropyl, isobutyl group, or a higher alkyl group, and X = hydrogen, zinc, aluminium, an alkali or alkaline earth metal,

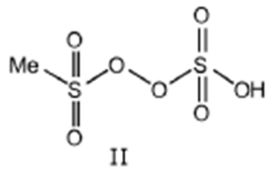
as an initiator-precursor for preparing alkanesulfonic acids, especially methanesulfonic acids from alkane, especially methane, and pure sulfur trioxide.

2. A mixture containing a compound of the formula (I)

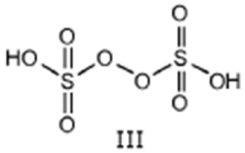
ALK-$SO_2$-O-O-X (I),

wherein ALK is a branched or unbranched alkyl group, especially a methyl, ethyl, propyl, butyl, isopropyl, isobutyl group, or a higher alkyl group, and X = hydrogen, zinc, aluminium, an alkali or alkaline earth metal,

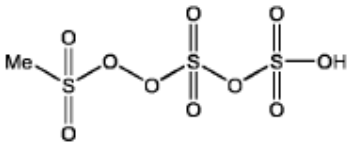
and at least one compound selected from the group consisting of following formulae (II) to (X)



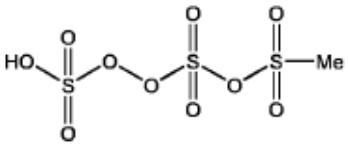

II



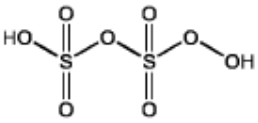

III



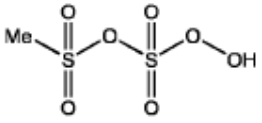

IV




V



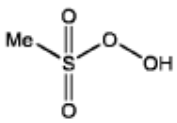

VI



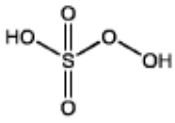

VII

$H_2O_2$

VIII




IX




X

-

and combinations thereof.

3. The compound of the formula (I)

ALK-$SO_2$-O-O-X (I),

wherein ALK is a branched or unbranched alkyl group, especially a methyl, ethyl, propyl, butyl, isopropyl, isobutyl group, or a higher alkyl group, and X = hydrogen, zinc, aluminium, an alkali or alkaline earth metal,

or the mixture of claim 2,

in sulfuric acid or alkylsulfonic acid, especially methanesulfonic acid or combinations thereof.

4. Use of the mixture according to claim 2 or 3 as an initiator-precursor for preparing alkanesulfonic acids, especially methanesulfonic acid, from alkane, especially methane, and pure sulfur trioxide.

5. A process for manufacturing alkanesulfonic acids, especially methanesulfonic acid, comprising the following steps:

- providing pure sulfur trioxide;

- reacting the pure sulfur trioxide with an alkane, especially methane, in a high-pressure autoclave or laboratory reactor;

- setting a pressure of from 1 to 200 bar;

- preparing the compound of the formula (I)

ALK-$SO_2$-O-O-X (I),

wherein ALK is a branched or unbranched alkyl group, especially a methyl, ethyl, propyl, butyl, isopropyl, isobutyl group, or a higher alkyl group, and X = hydrogen, zinc, aluminium, an alkali or alkaline earth metal,

or the mixture of claim 2 or 3,

-

as initiator-precursor by reacting an alkanesulfonic acid or a solution containing the alkanesulfonic acid with a 30 % to 100 % (w/w) hydrogen peroxide solution;

- adding the initiator-precursor or a solution thereof to the reactor;

- controlling the temperature of the reaction mixture at 0 °C to 100 °C;

- if necessary purifying the reaction product, for example, by distillation or extraction.

6. Use of a compound of the formula (I)

ALK-$SO_2$-O-O-X (I),

wherein ALK is a branched or unbranched alkyl group, especially a methyl, ethyl, propyl, butyl, isopropyl, isobutyl group, or a higher alkyl group, and X = hydrogen, zinc, aluminium, an alkali or alkaline earth metal, or

the mixture of claim 2 or 3

in a device for performing the process according to claim 5, wherein the device comprises:

- a reactor (1) in which pure sulfur trioxide reacts with the compound of formula (I) as initiator-precursor to form an initiator; and reaction of said "in-situ"-built initiator with an alkane, especially methane;

- a distillation means (2) for distilling the product formed in the reactor (1) ; and

- a filling means (3); as well as

- connection means to connect the reactor (1) with the distillation means (2) , and the filling means (3) with the distillation means (2).