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# (54) USE OF ISOTHIAZOLONE REDUCTION OF EMISSIONS OF HCN

VERWENDUNG VON ISOTHIAZOLONE ZUR REDUZIERUNG VON EMISSIONEN VON HCN

UTILISATION D'ISOTHIAZOLONE POUR LA REDUCTION DES EMISSIONS DE HCN

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## Description

**[0001]** The invention relates to the use of special additives for reducing the emission of volatile compounds from compositions and products which comprise constituents which can form or release such volatile compounds. In particular, the invention relates to the provision of additives for the reduction of emissions of HCN.

**[0002]** There are many substances and compositions which form or release volatile compounds which have unpleasant odour and are potentially hazardous on accumulation. Depending on the extent of the release or emission of volatile compounds, the unpleasant odour or the potential hazard occurs in a range which extends from "not perceptible" or "not present" to "predominant". In particular, a decrease of the emission is desirable in the case of substances or compositions which release the compounds in question to a relatively great extent. However, even in the case of substances and compounds which release these substances in amounts which are very small and are basically negligible, so that in the case of correct handling, there is no question with regard to odour pollution or safety hazard, a decrease in emissions can also be desirable, however, in order to keep the safety margin, until the individual MAK values are reached, as high as possible.

**[0003]** For example, there are compositions from which, under certain conditions, compounds such as HCN, amines such as $NH_3$ and $CH_3NH_2$ or thio compounds such as $H_2S$, for example, are released. In this case, a decrease in the release, in particular, of HCN is of interest, which can proceed in highly variable extents from, for example, cyanide or thiocyanate ions or compounds having cyanide or thiocyanate substituents or compositions comprising such compounds.

**[0004]** JP-A-63 209 798 discloses a deodoriser composition containing isothiazolone compounds for nitrate reducing bacteria by inhibiting the sulfur compounds emission or the ammonia emission.

**[0005]** JP-A-1 224 098 discloses a deodorising compound for sludge containing isothiazolone.

**[0006]** In addition to the inorganic compounds, in particular, organothiocyanates, which include aromatic and aliphatic organothiocyanates, are potential HCN sources. Examples of aliphatic and aromatic organothiocyanates are thiocyanate-substituted thiazoles, 3-thiocyanato-N-octylacrylamide and methylenebisthiocyanate (MBT). They frequently have a characteristic unpleasant odour and, under certain conditions, also release volatile compounds such as, for example, thio compounds and HCN. The emission values in this case are, at least in the case of approved commercial products, within the permitted limit values (MAK values), but, in principle, can also lie above these. Emissions of this type and, in particular, elevated emissions, represent a potential hazard to persons handling or using the products containing these constituents.

**[0007]** The contents of the individual compounds in question in the surrounding atmosphere serve, for example, as measured parameter for the emission of a product, e.g., for the emission of HCN, the HCN content in the surrounding air serves as indicator, in which case this can be determined using an appropriate test tube (e.g. a Dräger tube: prussic acid 2/a (hydrogen cyanide) (CH 25701)).

**[0008]** Quantitative determination of emissions such as HCN is comparatively complex and frequently cannot be carried out in practice. To remedy this, therefore, subjective organoleptic tests, which give highly meaningful results, are currently customarily also carried out. In this case, the odour impression of the tested compositions is determined, a reference sample being tested in conjunction.

**[0009]** The object therefore underlying the invention is to reduce emissions of volatile compounds such as HCN, which are potentially hazardous or cause unpleasant odour, from compositions and products which comprise constituents which form such compounds which are potentially hazardous or cause unpleasant odour, and, in this regard, to provide low-emission compositions.

**[0010]** This object is achieved by means of the fact that isothiazolone compounds such as N-octylisothiazolone and benzisothiazolone, are used as additive for the reduction of emissions of volatile compounds which are potentially hazardous or cause unpleasant odour from compositions which comprise constituents which form such compounds which are potentially hazardous or cause unpleasant odour.

**[0011]** Preferred embodiments are the subject-matter of the subclaims.

**[0012]** According to the invention, it has been found that emissions which are potentially hazardous or have unpleasant odour may be greatly reduced or eliminated by addition of the abovementioned additive compounds. The extent of the emission reduction may be set via the concentration of the additives. In the case of products which already comply with the legally prescribed limit values (MAK values), according to the invention, a further reduction or elimination of such emissions can increase the safety margin until the MAK values are reached, whereas in the case of substances and compositions whose emission lies above this, a decrease or elimination of the emissions can lead to the corresponding commercial products being approved or being able to be put on the market.

**[0013]** The reduction can appear in advantages with respect to odour versus a comparison product which contains no additive according to the invention. The advantage achieved is, for example, a reduction of the odour strength and/or a change of the odour note or type of odour.

**[0014]** The following description, to illustrate the invention, is essentially concerned with reducing the emission of

HCN from organothiocyanate compounds. However, the invention is not restricted thereto, as the further qualitative results for emissions of thio compounds show. To illustrate the reduction of HCN emissions, the test substances selected were firstly an aromatic thiocyanatothiazole or 3-thiocyanato-N-octylacrylamide and secondly aliphatic methylenebisthiocyanate (MBT).

**[0015]** Suitable emission-reducing additives according to the invention are, for example, the following isothiazolones and mixtures of the same: N-octylisothiazolone, 5-chloro-N-methylisothiazolone, N-methylisothiazolone, a mixture of 5-chloro-N-methylisothiazolone and N-methylisothiazolone, 1,2-benzisothiazolone, 5,6-dihydro-2-methyl-2H-cyclopent(d)isothiazol-3(4H)-one.

**[0016]** The preferred emission-reducing additive is N-octylisothiazolone. In the examples, the embodiments according to the invention prepared are preparations which comprise the emission-reducing additive, in particular an isothiazolone, which reduces the emission of the preparations and, inter alia, also acts as an HCN trap.

**[0017]** The invention makes it possible to reduce the release of HCN both in aqueous systems over a wide pH range, such as pH 1 to pH 12, e.g. by adding water-soluble isothiazolones, such as N-methylisothiazolone, benzisothiazolone or 5,6-dihydro-2-methyl-2H-cyclopent(d)isothiazol-3(4H)-one, and in lipophilic media, e.g. by adding N-octylisothiazolone.

**[0018]** According to the invention, therefore, in particular HCN emissions can be reduced from primarily liquid products (solutions, dispersions, preferably aqueous dispersions).

**[0019]** Since substances and compositions having high HCN emissions are not suitable for the market, according to the invention, the possibility is provided of reducing their emissions so greatly that marketing is thus possible. Moreover, according to the invention, by adding the emission-reducing additives, emissions which are already acceptable and comply with current safety regulations can further be decreased, for example the organoleptic properties can also be improved, which can lead to an increased market acceptance.

**[0020]** Furthermore, it is important that the action, e.g. a biocidal action, ascribed to the treated compositions is not impaired by the emission-reducing additives.

**[0021]** The invention can be used, for example, in the area of solutions or dispersions for a fungicidal and algistatic finishing of materials and technical preservation. Concrete areas of application and user products are, for example, preservatives, disinfectants, impregnating agents, paints, sizings and adhesives, primers, coatings, lubricants and plasters.

**[0022]** The emission-reducing additives according to the invention are used at least in a total amount of 0.01% by weight, preferably 0.01 to 10.0% by weight, in particular 0.1 to 7.5% by weight, and preferably 0.5 to 5.0% by weight, based on the composition. When a plurality of emission-reducing compounds are used, the proportion of each of these additive components can be set according to the specific product requirements.

**[0023]** Experimental results are given below which indicate the HCN emission from aromatic organothiocyanate-containing compositions or which confirm the emission-reducing action of the said additive compounds.

## Aromatic organothiocyanate + HCN trap

### Example I

**[0024]** Aqueous dispersions which comprised 7.8% by weight of aromatic organothiocyanate (in the form of 26% by weight of a 30% strength dispersion), based on the weight of the dispersions, were admixed with various amounts of N-octylisothiazolone (45% strength solution) (0.5%, 1.0%, 2.0%, 3.0% or 5.0%, based on the weight of the dispersion) and were tested for compatibility and stability. The procedure and results of "HCN" measurements are reported below.

Appearance after storage at room temperature for 7 days:

**[0025]** After 7 days, homogeneous yellowish-white dispersions were present unchanged.

### Example II

HCN measurements using a Dräger test tube for HCN (see above) (5 strokes) for the dispersions of Example I:

**[0026]** In this test, the Dräger test tube was used together with a pump. Both tips of the tube were broken off and one end of the tube was then inserted tightly into the pump. The HCN content was tested by holding the other end of the tube in the air space over the surface of the respective sample in a 50 ml wide-neck sample vial which was filled with 50 g of the sample. The temperature was 23.5°C. The sample vials had been sealed for 4 weeks. For measurement, the air was sucked through the tube. The measurement period was about 60 seconds for 5 strokes and correspondingly about 24 seconds for 2 strokes. The result was then determined on the basis of the coloration (reaction of HCN with

$HgCl_2$ and methyl red). The measurement method corresponds to the Dräger instruction 234-257 of 1995.)
**[0027]** Blank value (without additive, aromatic organothiocyanate-containing aqueous dispersions) → approx. 1.5 ppm.

Example I: + 5% N-octylisothiazolone → 0 ppm (45% strength solution)

HCN measurements using Dräger tubes for HCN (see above) (5 strokes) for the dispersions according to Example I in a 250 ml wide-neck bottle which contained approximately 40 g of the respective dispersion:

**[0028]** Blank value: approximately 1 - 2 ppm
All other samples: no HCN detectable!!

Result:

**[0029]** An addition of N-octylisothiazolone (45% strength solution) significantly decreases the HCN emission of aromatic-organothiocyanate-containing dispersions.

**Example III**

**[0030]** Aqueous dispersions were produced which comprised 7.8% by weight of aromatic organothiocyanate (in the form of 13% by weight of a 60% strength dispersion) based on the weight of the dispersions. The dispersions were homogeneous and whitish-yellow (very high quality).

**Example IV**

HCN measurements using Dräger tubes (5 strokes) of the dispersions of Example III (aromatic organothiocyanate without and with 1.0%, 2.0%, 3.0% and 5.0% N-octylisothiazolone (45% strength solution):

**[0031]** The dispersions were measured after storage at room temperature for 24 hours in a 250 ml wide-neck bottle which contained approximately 100 g of the respective sample:

| | Zero value | After 1 week at room temperature |
|---|---|---|
| Blank: without additive | >> 30 ppm | > 30 ppm |
| + 1% Kathon 893 | approx. 25 ppm | 8 ppm |
| + 2% Kathon 893 | approx. 10 ppm | 5 ppm |
| + 3% Kathon 893 | approx. 5 ppm | 2-3 ppm |
| + 5% Kathon 893 | approx. 2 ppm | 2 ppm |

**[0032]** The dispersions remained unchanged with respect to homogeneity and whitish-yellow colour. There was a marked odour improvement with respect to the blank value.
**[0033]** The aromatic organothiocyanate content of the dispersions containing 0 or 1% N-octylisothiazolone (45% strength solution) was:

| | |
|---|---|
| without N-octylisothiazolone | 7.1% |
| with 1% N-octylisothiazolone | 7.1% |

Result:

**[0034]** An addition of N-octylisothiazolone significantly decreases the HCN emission of aromatic-organothiocyanate-containing dispersions. The odour of such dispersions is improved in this case. There is no decomposition of the organothiocyanate active compound.

### Example V

### Solutions containing aromatic organothiocyanate + N-octylisothiazolone:

**[0035]** Liquid preparations based on aromatic organothiocyanate (60% strength solution) and N-octylisothiazolone (45% strength solution) (data in parts by weight), HCN emission measurement (see above):

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| N-octylisothiazolone (45% strength solution) | 0 | 90 | 80 | 70 | 60 | 50 | 40 | 30 | 20 | 10 |
| Organothiocyanate (60% strength solution) | 100 | 10 | 20 | 30 | 40 | 50 | 60 | 70 | 80 | 90 |

**[0036]** HCN emission in ppm, measured using Dräger tubes at 22°C, measurement vessel 100 ml wide-neck bottle filled with 100 g of solution:

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| After 48 h | >> 30 | | | | | < 1 | < 1 | < 1 | < 1 | 2 |
| After 96 h | | < 1 | < 1 | < 1 | < 1 | | | | | |
| After 1 week | | | | | | < 1 | < 1 | 1 | 1 | 2 |

**[0037]** The solutions had not changed after storage at room temperature for 2 months.

### Example VI

### Solutions containing aromatic organothiocyanate + N-octylisothiazolone:

**[0038]** Liquid preparations based on aromatic organothiocyanate (60% strength solution) and N-octylisothiazolone (45% strength solution) (data in parts by weight), HCN emission measurement (see above):

| | | | | |
|---|---|---|---|---|
| N-octylisothiazolone (45% strength solution) | 8 | 6 | 4 | 2 |
| Organothiocyanate (60% strength solution) | 92 | 94 | 96 | 98 |

**[0039]** HCN emission in ppm, measured using Dräger tubes at 22°C, measurement vessel 100 ml wide-neck bottle filled with 100 g of solution:

| | | | | |
|---|---|---|---|---|
| After 48 h | 1-3 | 4-5 | 17 | > 30* |
| After 1 week | 2-3 | 5 | 7 | 17-18 |

* 2 strokes

Result:

**[0040]** By adding N-octylisothiazolone to aromatic-thiocyanate-containing solutions, homogeneous mixtures which have a significantly decreased HCN emission are obtained in all mixing ratios.

### Further additives decreasing the HCN emission of aromatic organothiocyanate

**[0041]** In the studies, further HCN traps were found, where in each case homogeneous dispersion comprising 7.8% by weight of aromatic organothiocyanate was prepared containing 2% by weight of active compound of the respective additive (in the case of $H_2O_2$ (30% strength) it was therefore 6.66% by weight), and the HCN emission was measured as a function of time using a Dräger tube.

**[0042]** HCN emission in ppm, measured using Dräger tubes at 22°C, measurement vessel 50 ml wide-neck bottle filled with 25 g of dispersion:

| HCN trap | HCN after 48 h in ppm | after 1 week ppm | pH after 1 week |
|---|---|---|---|
| Without addition | > 30 | 20 | 4.5 |

(continued)

| HCN trap | HCN after 48 h in ppm | after 1 week ppm | pH after 1 week |
|---|---|---|---|
| A) N-Methylisothiazolone | 2-3 | 2 | 4.0 |
| B) 5,6-Dihydro-2-methyl-2H-cyclopent(d) isothiazol-3(4H)-one** | 0 | 0 | 4.1 |

*** 50% active content, initial concentration = 4%

Result: The further additives studied lead to a reduction of the HCN emission, which reduction, in comparison with the isothiazolones, is somewhat lesser.

**[0043]** Preferred examples of these further additives lead to a comparatively high reduction (to < 20 ppm) of the HCN emission of aromatic-organothiocyanate-containing compositions.

## HCN emission and stability of commercial compositions which comprise aromatic organothiocyanate and N-octylisothiazolone:

**[0044]** A test was made of whether instabilities or reductions in active compound occur during storage of commercial compositions which comprise aromatic organothiocyanate and N-octylisothiazolone.

Result:

**[0045]** An addition of N-octylisothiazolone to, for example, film preservatives based on compositions which contained aromatic organothiocyanate, led to optically clear, homogeneous solutions. The HCN emission was significantly reduced. The active compound content is virtually unchanged after storage at room temperature or +40°C for 1 month.

**[0046]** The same applies to the HCN emission of pure acrylic masonry paint which was treated in a corresponding manner.

Result:

**[0047]** At the application concentration, no HCN is detectable and the stability was excellent.

## Example VII

### 3-Thiocyanato-N-octylacrylamide:

Preparation of 3-thiocyanato-N-octylacrylamide and N-octyl-3-thiocyanatoacrylamide from N-octylisothiazolone (NOITZ, extracted from N-octylisothiazolone (45% strength solution) by shaking with toluene and water) and KCN:

**[0048]** 213 g (1 mol) of NOITZ + 65 g (1 mol) of KCN were stirred together in 600 ml of water. 10% strength $H_2SO_4$ was added dropwise with stirring and cooling (maximum temperature 30°C). The mixture was stirred for 3 hours. The precipitate was filtered off by suction, taken up in 500 ml of warm DIPE, dried over $Na_2SO_4$ and crystallized out with addition of approximately 1.5 l of PE. The crystals were filtered off by suction and washed with PE. After drying, 145.4 g of N-octyl-3-thiocyanatoacrylamide (60.5% yield) resulted, in the form of yellowish crystals, F: 81°C.

**[0049]** The 3-thiocyanato-N-octylacrylamide which was isolated in crystalline form is virtually insoluble in water (< 0.01%) and dissolves at about 2.5 to 10% in glycols or glycol ethers. The 2.5 to 10% strength solutions of 3-thiocyanato-N-octylacrylamide in glycols or glycol ethers emit HCN, that is to say approximately 30 ppm (1 stroke) after storage at room temperature for 1 week, whereas the crystalline material had an HCN emission of approximately 25 ppm at 5 strokes (Dräger tube, see above).

| Solvent: | | | HCN (1 stroke) |
|---|---|---|---|
| 1,2-Propyl glycol | approx. 2.5% | completely soluble | |
| Dipropyl glycol | 10% | completely soluble | > = 40 |
| Triethylene glycol | 8% | completely soluble | approx. 30 |
| Phenoxypropanols | 10% | completely soluble | approx. 30 |

(continued)

| Solvent: | | | HCN (1 stroke) |
|---|---|---|---|
| Phenoxyethanol | 10% | completely soluble | approx. 30 |
| 1-Methoxy-2-propanol | 10% | completely soluble | approx. 30 |
| Butyl diglycol | 10% | completely soluble | approx. 30 |
| Ethylene glycol | << 1% | completely soluble | approx. 30 |
| Deionized water | virtually insoluble (< 0.01%) | | |

**[0050]** All solutions, after storage at room temperature for 2 months were without change, clear and slightly yellowish.

**[0051]** A 5% strength aqueous dispersion of 3-thiocyanato-N-octylacrylamide is almost white, homogeneous and has an HCN emission of 13 ppm (5 strokes) (Dräger tube, see above).

**[0052]** Adding, for example, N-octylisothiazolone considerably decreased the HCN emission. The results were roughly as for the organothiocyanate tested.

## Claims

1. Use of isothiazolone compounds, as additive for the reduction of emissions of HCN, from compositions which comprise compounds which have cyanide or thiocyanate ions or cyanide or thiocyanate substituents, in particular organothiocyanate ions or organothiocyanate substituents.

2. Use according to Claim 1, **characterized in that**, isothiazolone compounds are selected from N-octylisothiazolone, 5-chloro-N-methylisothiazolone, N-methylisothiazolone, a mixture of 5-chloro-N-methylisothiazolone and N-methylisothiazolone, 1,2-benzisothiazolone or 5,6-dihydro-2-methyl-2H-cyclopent(d)isothiazol-3(4H)-one.

3. Use according to Claim 2, **characterized in that**, isothiazolone compounds is N-octylisothiazolone.

4. Use according to one of Claims 1 to 3 of isothiazolone compounds, as additive for the reduction of emissions of HCN, from liquid compositions, in particular from solutions and dispersions, preferably from aqueous dispersions.

5. Use according to Claim 4, **characterized in that** the liquid composition is selected from cleaning agents, disinfectants, preservatives, lubricants, impregnating agents, paints, coatings and plasters.

6. Use according to one of the preceding claims, **characterized in that** the isothiazolone compounds is used at least in an amount of 0.01% by weight, preferably 0.01 to 10.0% by weight, in particular 0.1 to 7.5% by weight and especially 0.5 to 5.0% by weight, based on the composition.

## Patentansprüche

1. Verwendung von Isothiazolonverbindungen als Additiv zur Verminderung von Emissionen von HCN aus Zusammensetzungen, die Verbindungen enthalten, die Cyanid- oder Thiocyanationen oder -substituenten aufweisen, insbesondere Organothiocyanationen oder -substituenten.

2. Verwendung nach Anspruch 1, **dadurch gekennzeichnet, daß** die Isothiazolonverbindungen unter N-Octylisothiazolon, 5-Chlor-N-methylisothiazolon, N-Methylisothiazolon, Mischung von 5-Chlor-N-methylisothiazolon und N-Methylisothiazolon, 1,2-Benzisothiazolon oder 5,6-Dihydro-2-methyl-2H-cyclopent(d)isothiazol-3(4H)on ausgewählt werden.

3. Verwendung nach Anspruch 2, **dadurch gekennzeichnet, daß** als Isothiazolonverbindungen N-Octylisothiazolon verwendet wird.

4. Verwendung von Isothiazolonverbindungen nach einem der Ansprüche 1 bis 3 als Additiv zur Verminderung von Emissionen von HCN aus flüssigen Zusammensetzungen, insbesondere aus Lösungen und Dispersionen, bevor-

zugt aus wäßrigen Dispersionen.

5. Verwendung nach Anspruch 4, **dadurch gekennzeichnet, daß** die flüssige Zusammensetzung unter Reinigungsmitteln, Desinfektionsmitteln, Konservierungsmitteln, Schmiermitteln, Imprägniermitteln, Farben, Lacken und Putzen ausgewählt wird.

6. Verwendung nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, daß** die Isothiazololverbindungen bezogen auf die Zusammensetzung mindestens in einer Menge von 0,01 Gew.-%, bevorzugt 0,01 bis 10,0 Gew.-%, insbesondere 0,1 bis 7,5 Gew.-% und bevorzugt 0,5 bis 5,0 Gew.-% eingesetzt werden.

## Revendications

1. Utilisation de composés isothiazolone, en tant qu'additif pour la réduction d'émissions d'HCN, à partir de compositions comprenant des composés renfermant des ions cyanure ou thiocyanate ou des substituants cyanure ou thiocyanate, en particulier des ions organothiocyanate ou des substituants organothiocyanate.

2. Utilisation selon la revendication 1, **caractérisée en ce que** les composés isothiazolone sont choisis parmi la N-octylisothiazolone, la 5-chloro-N-méthylisothiazolone, la N-méthylisothiazolone, un mélange de 5-chloro-N-méthylisothiazolone et de N-méthylisothiazolone, la 1,2-benzisothiazolone ou la 5,6-dihydro-2-méthyl-2H-cyclopent(d)isothiazol-3(4H)-one.

3. Utilisation selon la revendication 2, **caractérisée en ce que** le composé isothiazolone est la N-octylisothiazolone.

4. Utilisation selon l'une des revendications 1 à 3 de composés isothiazolone, en tant qu'additif pour la réduction d'émissions de HCN, à partir de compositions liquides, en particulier à partir de solutions et de dispersions, de préférence à partir de dispersions aqueuses.

5. Utilisation selon la revendication 4, **caractérisée en ce que** la composition liquide est choisie parmi des agents nettoyants, des désinfectants, des conservateurs, des lubrifiants, des agents d'imprégnation, des peintures, des revêtements et des plâtres.

6. Utilisation selon l'une des revendications précédentes, **caractérisée en ce que** le composé isothiazolone est utilisé au moins en une quantité de 0,01 % en poids, de préférence de 0,01 à 10,0 % en poids, en particulier de 0,1 à 7,5 % en poids et particulièrement de 0,5 à 5,0 % en poids, par rapport à la composition.