

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property  
Organization  
International Bureau



WIPO | PCT



(10) International Publication Number  
**WO 2017/176543 A1**

(43) International Publication Date  
12 October 2017 (12.10.2017)

(51) International Patent Classification:

*A01N 43/54* (2006.01)      *A01P 3/00* (2006.01)  
*A01N 43/80* (2006.01)      *A01P 13/00* (2006.01)  
*A01N 25/10* (2006.01)

(21) International Application Number:

PCT/US2017/024953

(22) International Filing Date:

30 March 2017 (30.03.2017)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

62/317,731      4 April 2016 (04.04.2016)      US

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(81) Designated States (*unless otherwise indicated, for every kind of national protection available*): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DJ, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KH, KN, KP, KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States (*unless otherwise indicated, for every kind of regional protection available*): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Published:

— with international search report (Art. 21(3))



WO 2017/176543 A1

(54) Title: SYNERGISTIC COMBINATION OF A LENACIL COMPOUND AND N-BUTYL-1,2-BENZISOTHIAZOLIN-3-ONE FOR DRY FILM PROTECTION

(57) Abstract: A synergistic antimicrobial composition containing lenacil and N-Butyl-1,2-benzisothiazolin-3-one is provided. Also provided is a method of inhibiting the growth of or controlling the growth of microorganisms in a building material, a coating composition containing such a synergistic antimicrobial composition, and a dry film made from such a coating composition.

**SYNERGISTIC COMBINATION OF A LENACIL COMPOUND  
AND N-BUTYL-1,2-BENZISOTHIAZOLIN-3-ONE FOR DRY FILM PROTECTION**

[0001] This invention relates to combinations of antimicrobial compounds and their uses in dry film protection applications, the combinations having unexpectedly greater activity than would be expected for the use of both of the individual antimicrobial compounds.

[0002] Use of combinations of at least two antimicrobial compounds can broaden potential markets, reduce use concentrations and costs, and reduce waste. In some cases, commercial antimicrobial compounds cannot provide effective control of microorganisms, even at high use concentrations, due to weak activity against certain types of microorganisms, e.g., those resistant to some antimicrobial compounds. Combinations of different antimicrobial compounds are sometimes used to provide overall control of microorganisms in a particular end user environment. For example, WO 1998/121962 discloses combinations of 3-iodo-2-propynyl-butylcarbamate and pyriithione, but this reference does not suggest any of the combinations claimed herein. Moreover, there is a need for additional combinations of antimicrobial compounds with relatively low impact on health and/or the environment. The problem addressed by this invention is to provide such additional combinations of antimicrobial compounds.

[0003] Antimicrobial compounds are sometimes included in liquid coating compositions that are applied to a substrate and that become dry films. It is desirable that such dry films control surface fungi and algae and that such dry films also present as little adverse effect as possible on health and the environment.

[0004] In the present invention there is provided a synergistic antimicrobial composition comprising 3-Cyclohexyl-6,7-dihydro-1*H*-cyclopentapyrimidine-2,4-(3*H*,5*H*)-dione hereinafter, "lenacil" (CAS registry number 2164-08-1) and N-Butyl-1,2-benzisothiazolin-3-one, hereinafter "BBIT" (CAS registry number is 4299-07-4).

[0005] The invention further provides a method of inhibiting the growth of or controlling the growth of microorganisms in a building material, the method comprising the step of adding a synergistic antimicrobial composition comprising lenacil and BBIT; wherein the weight ratio of the BBIT to lenacil is from 1:10 to 5:1.

[0006] The present invention further comprises a coating composition comprising a synergistic antimicrobial composition comprising lenacil and BBIT. The coating composition of the present invention may also comprise a synergistic antimicrobial composition comprising lenacil and BBIT; wherein the weight ratio of the BBIT to lenacil is from 1:10 to 5:1.

[0007] Lastly, the present invention provides a dry film made by a process comprising applying a layer of the coating composition onto a substrate comprising a synergistic antimicrobial composition comprising lenacil and BBIT; wherein the weight ratio of the BBIT to lenacil is from 1:10 to 5:1 to a substrate and drying the coating composition or allowing the coating composition to dry.

[0008] The following is a detailed description of the invention.

[0009] As used herein, the following terms have the designated definitions, unless the context clearly indicates otherwise.

[0010] The term "antimicrobial compound" refers to a compound capable of inhibiting the growth of or controlling the growth of microorganisms; antimicrobial compounds include bactericides, bacteriostats, fungicides, fungistats, algaecides, algistats, and herbicides depending on the dose level applied, system conditions and the level of microbial control desired. Such term "antimicrobial compound" as used herein is synonymous with the term "biocide".

[0011] The term "microorganism" includes, for example, fungi (such as yeast and mold), bacteria and algae.

[0012] The following abbreviations are used throughout the specification: ppm = parts per million by weight (weight/weight), mL = milliliter, ATCC = American Type Culture Collection.

[0013] Unless otherwise specified, temperatures are in degrees centigrade (°C), and references to percentages are by weight (wt%). Percentages of antimicrobial compounds in the composition of this invention are based on the total weight of active ingredients in the composition, i.e., the antimicrobial compounds themselves, exclusive of any amounts of solvents, carriers, dispersants, stabilizers or other materials which may be present.

[0014] As used herein, "BBIT" is N-Butyl-1,2-benzisothiazolin-3-one (CAS registry number 4299-07-4).

[0015] When a ratio is the herein to be "X:1 or higher," it is meant that the ratio is Y:1, where Y is X or greater, and when a ratio is the herein to be "X:1 or lower," it is meant that the ratio is Z:1, where Z is X or less. The same logic follows for ratios that are "1:X or higher" and "1:X or lower".

[0016] Lenacil belongs to the class of uracil herbicides, 3-cyclohexyl-1,5,6,7-tetrahydrocyclopentapyrimidine-2,4(3H)-dione (C<sub>13</sub>H<sub>18</sub>N<sub>2</sub>O<sub>2</sub>) (CAS registry number 2164-08-1). Lenacil is a known herbicide that is approved in many jurisdictions as a soil-acting herbicide for the control of grass and broad-leaved weeds in beet and other crops.

[0017] Lenacil has relatively low solubility in water. This relatively low solubility in water is preferred for an antimicrobial material that may be included in a coating composition or other building material, because dried coatings and building materials are exposed to water, which could tend to remove a highly soluble compound from the dried coating or the building material.

[0018] The present invention involves a composition that contains both lenacil and BBIT. It has been surprisingly found that such a composition is synergistically effective as a biocide.

It has been especially surprisingly found that compositions that contain both lenacil and BBIT are synergistically effective as biocides against algae.

[0019] When BBIT is present, preferably the weight ratio of BBIT to lenacil compound is preferably 1:10 to 5:1.

[0020] The mixture of lenacil and BBIT may be included in a coating composition. Lenacil and BBIT may be added to the coating composition separately or as a mixture or any combination thereof. Preferred coating compositions are liquid. Coating compositions may be aqueous or non-aqueous. Aqueous coating compositions generally contain 30% or more water by weight of the mixture, based on the weight of the coating composition.

[0021] Among embodiments in which lenacil and BBIT are included in paint or other coating composition, preferred coating compositions are liquid compositions, especially compositions that contain dispersions of polymers in aqueous media.

[0022] In addition to paints and other coating compositions such as marine anti-fouling, the antimicrobial compound combinations of the present invention are particularly useful in preservation of building materials, e.g., adhesives, caulk, joint compound, sealant, wallboard, etc., polymers, plastics, synthetic and natural rubber, paper products, fiberglass sheets, insulation, exterior insulating finishing systems, roofing and flooring felts, building plasters, bricks, mortar, gypsum board, wood products and wood-plastic composites. When an antimicrobial compound combination of the present invention is present in a building material, it is preferred that some or all of the antimicrobial compound combination is present at the surface of the building material or near enough to the surface of the building material to inhibit microbial growth on that surface.

[0023] In some embodiments, latex paints or other liquid coating compositions are used that contain the antimicrobial compound combinations disclosed herein.

[0024] Coating compositions are designed so that a layer of the coating composition can readily be applied to a substrate and then dried or allowed to dry to form a dry film. Coating compositions contain at least one binder. Binders contain one or more of the following: one or more polymer, one or more oligomer, and/or one or more monomer. Oligomers and monomers in binders are designed to polymerize and/or crosslink during or after the formation of the dry film. Polymers in a binder may or may not be designed to crosslink during or after the formation of the dry film.

[0025] Coating compositions optionally contain one or more pigment. A pigment is a mineral or an organic substance in the form of small solid particles. Pigments provide full or partial opacity to the dry film.

[0026] The antimicrobial compound combinations are useful for preservation of the dry film coating resulting after application of paint or other liquid coating composition. Preferably, the antimicrobial composition is an aqueous latex paint comprising one or more of the antimicrobial compound combinations disclosed herein, or the dry film coating resulting from application of the paint to a surface. An aqueous latex paint is an aqueous liquid coating composition in which the binder is a polymer in the form of a latex (i.e., in the form of polymer particles dispersed throughout the water). More preferred are aqueous latex paints in which the binder contains one or more acrylic polymer.

[0027] Typically, the amount of the antimicrobial compound combinations of the present invention to control the growth of microorganisms is from 100 ppm to 40,000 ppm active ingredient. For example, in the present invention, lenacil plus BBIT is present in an amount from 100 ppm to 10,000 ppm. The antimicrobial combinations of the composition are present in an amount of at least 100 ppm and no more than 10,000 ppm, alternatively no more than 8,000 ppm, alternatively no more than 6,000 ppm, alternatively no more than 5,000 ppm, alternatively no more than 3,000 ppm, alternatively no more than 2500 ppm, alternatively no

more than 2,000 ppm, alternatively no more than 1,000 ppm, and alternatively no more than 500 ppm. Concentrations mentioned above are in a liquid coating composition containing the antimicrobial compound combinations; antimicrobial compound combination levels in the dry film coating will be higher.

[0028] The present invention also encompasses a method for preventing microbial growth in building materials, especially in dry film coatings, by incorporating any of the claimed antimicrobial compound combinations into the materials.

[0029] Typically, the antimicrobial compositions are used to inhibit growth of algae and/or fungi.

[0030] The composition of the present invention contains lenacil and BBIT. It is contemplated that some embodiments may contain one or more additional antimicrobial compound.

[0031] The following are examples of the present invention.

[0032] Sample preparation for antimicrobial testing was performed as follows:

[0033] Sample preparation:

[0034] A solution containing 97% biocide active ingredient for BBIT and a dispersion containing 30% biocide active ingredient for lenacil was post added into white, acrylic/silicone based outdoor paint free of biocides to give a total active ingredient concentration of 10,000 and 1,000ppm respectively. These paints were then diluted with a biocide free acrylic/silicone based paint and mixed to prepare targeted concentrations of biocide combinations for the testing. The total biocides concentrations obtained were 125, 250, 500, 1000, 2000 and 5000 ppm. After biocides addition or dilution, the paints were mixed 90 seconds with the horse power shaker (AXEL 75M3372/ Agitateur SO-10MI) until uniformity was achieved. The obtained paints containing BBIT and containing lenacil were mixed together to obtain paints containing the two actives in the desired ratios and amounts.

After one day, the paints were applied to Schleicher & Schuell filter paper at 280µm wet film thickness and dried for 3 days at room temperature avoiding direct exposure to sunlight.

Square discs (1.8 cm x 1.8 cm) were cut out from each panel and were used as the substrate for algal efficacy tests. This sample size allowed for an agar border when the sample disc was placed into the well of the test plate.

**[0035]** Algal Efficacy Testing:

**[0036]** Algal efficacy was tested according to modified ASTM 5589 which is a standard accelerated test method for determining resistance of various coatings (including paints) to algal defacement. To accommodate for high-throughput screening, this method was scaled down from petri plates to 6-well plates. Bold Modified Basal Freshwater Nutrient Solution was used as growth medium for the algal culture and Bold's agar was used for the efficacy testing. A single coupon was placed with a pair of sterile forceps at the center of the agar plug (on top) with the painted surface facing upwards. The algal inoculum was prepared by diluting an exponentially growing culture of *Chlorella sp.* to  $1 \times 10^6$  cfu/ml.

[0037] Algal inoculum:

Organism	Strain No.	Type	Medium for testing
<i>Chlorella sp.</i>	ATCC 7516	Unicellular Chlorophyte	Bold Modified Freshwater Solution

[0038] Each well that contains a tested coupon was inoculated with 1750  $\mu$ l of algal suspension ( $1 \times 10^6$  cfu/ml) making sure that the whole surface (paint film as well as the agar surrounding it) was evenly covered. The plates were incubated at room temp (21°C - 25°C) with cyclic exposure to 14 hours fluorescent light followed by 10 hours darkness, for a period of three weeks.

[0039] At the end of the incubation period the samples were scored for percent inhibition regarding color intensity compared to the blank sample

[0040] The Synergy Index calculation was performed as follows:

[0041] The SI is calculated based on F.C.Kull et. Al. method (Applied Microbiology, Vol. 9 (1961). In this study, SI was calculated based on the following formula with the minimum inhibitory concentration chosen based on the percent inhibitory exhibited by the individual antimicrobial against each microorganism tested.

$$SI = \frac{Qa}{QA + Qb/QB}$$

Qa = the concentration of Antimicrobial A in the blend

QA = the concentration of Antimicrobial A as the only biocide

Qb = the concentration of Antimicrobial B in the blend

QB = the concentration of Antimicrobial B as the only antimicrobial

[0042] SI value of < 1 in the formula indicates a synergism of the blended biocides exists.

[0043] Note: If any of the active with maximum concentration tested did not exhibit some inhibition, this maximum tested concentration is used to calculate the estimated SI and a sign of less than (<) is included to take into account that higher concentration of the active (e.g. Lenacil) is needed to achieve the targeted inhibition. The minimal targeted inhibition was set at 75%, meaning a coupon with at least 75% algal growth inhibition was considered as a pass.

[0044] Compositions listed below that contain both lenacil and BBIT are examples of the present invention.

[0045] Test Results for BBIT with lenacil at three weeks were as follows:

[0046] Pass level  $\geq 75\%$  inhibition

Pass level > 75% inhibition	
BBIT/Lenacil	
	Chlorella
BBIT	
Total conc, ppm	5000
% inhibition	85
Lenacil	
Total conc, ppm	1,000
% inhibition	90
BBIT/Lenacil 1/10	
Total conc, ppm	250
% inhibition	95
SI	0.23

BBIT/Lenacil 1/5	
Total conc, ppm	250
% inhibition	90
SI	0.22
BBIT/Lenacil 1/2	
Total conc, ppm	500
% inhibition	100
SI	0.37
BBIT/Lenacil 1/1	
Total conc, ppm	500
% inhibition	100
SI	0.30
BBIT/Lenacil 2/1	
Total conc, ppm	1000
% inhibition	90
SI	0.47
BBIT/Lenacil 5/1	
Total conc, ppm	2000
% inhibition	100
SI	0.67

[0047] BBIT + Lenacil at ratios of 1:10 to 5:1 exhibited a synergy.

## CLAIMS

1. A synergistic antimicrobial composition comprising lenacil and N-Butyl-1,2-benzisothiazolin-3-one.
2. The synergistic antimicrobial composition of claim 1, wherein the weight ratio of the N-Butyl-1,2-benzisothiazolin-3-one to lenacil is from 1:10 to 5:1.
3. A method of inhibiting the growth of or controlling the growth of microorganisms in a building material, the method comprising the step of adding the synergistic antimicrobial composition of claim 1 to the building material.
4. A method of inhibiting the growth of or controlling the growth of microorganisms in a building material, the method comprising the step of adding the synergistic antimicrobial composition of claim 2 to the building material.
5. A coating composition comprising the synergistic antimicrobial composition of claim 1.
6. A coating composition comprising the synergistic antimicrobial composition of claim 2.
7. A dry film made by a process comprising applying a layer of the coating composition of claim 5 to a substrate and drying the coating composition or allowing the coating composition to dry.
8. A dry film made by a process comprising applying a layer of the coating composition of claim 6 to a substrate and drying the coating composition or allowing the coating composition to dry.

**INTERNATIONAL SEARCH REPORT**

International application No  
PCT/US2017/024953

**A. CLASSIFICATION OF SUBJECT MATTER**  
 INV. A01N43/54      A01N43/80      A01N25/10      A01P3/00      A01P13/00  
 ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)  
 A01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)  
 EPO-Internal, CHEM ABS Data, WPI Data

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	page 10, lines 14-21 page 11, lines 21-26 page 15, lines 19-24 page 19, line 30 page 33, lines 17-28 claim 9	2,4,6,8
A	----- WO 2014/085738 A1 (ROHM & HAAS [US]; DOW GLOBAL TECHNOLOGIES LLC [US]) 5 June 2014 (2014-06-05) paragraphs [0046], [0049] claims 1-7	1-8
A	----- EP 1 772 055 A1 (ROHM AND HAAS FRANCE SAS [FR]) 11 April 2007 (2007-04-11) page 7 - page 55; tables 1-54 claims 1, 2 -----	1-8

Further documents are listed in the continuation of Box C.       See patent family annex.

\* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"E" earlier application or patent but published on or after the international filing date	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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"O" document referring to an oral disclosure, use, exhibition or other means	"&" document member of the same patent family
"P" document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search  30 May 2017	Date of mailing of the international search report  12/06/2017
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Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer  Davies, Maxwell
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