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(54) Title: FIRE EXTINGUISHING AND FIRE SUPPRESSION COMPOSITIONS COMPRISING 3-CHLORO-1,1,1-TRIFLUOROPROPENE

(57) Abstract: A method of suppressing a flame comprising contacting the flame with a fluid comprising a flame suppression composition, wherein said flame suppression composition comprises 3-Chloro-1,1,1-trifluoropropene and optionally a propellant.



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Fire extinguishing and fire suppression compositions comprising 3-Chloro-1,1,1-Trifluoropropene

5 FIELD OF THE INVENTION

The disclosure herein relates to fire suppression compositions comprising 3-Chloro-1,1,1-trifluoropropene. The disclosure herein further relates to use of the fire suppression compositions for flame suppression, reduction,
10 extinguishment, or inertion.

BACKGROUND OF THE INVENTION

Numerous agents and methods of fire fighting are known and can be
15 selected for a particular fire, depending upon factors such as its size, location and the type of combustible materials involved. Halogenated hydrocarbon fire fighting agents have traditionally been utilized in the fire protection industry, in applications including fire prevention applications, which leave a breathable atmosphere in an enclosed area, total flooding
20 applications, wherein an enclosure is completely filled ("flooded") with an effective amount of the agent (e.g., computer rooms, storage vaults, telecommunications switching gear rooms, libraries, document archives, petroleum pipeline pumping stations, and the like), or in streaming applications wherein the agent is directed towards the location of the fire
25 (e.g., commercial hand-held extinguishers). Such extinguishing agents are not only effective but, unlike water, also function as "clean extinguishing agents", causing little, if any, damage to the enclosure or its contents.

The most commonly-used halogenated hydrocarbon extinguishing agents
30 have been the bromine-containing compounds bromotrifluoromethane (CF₃Br, Halon1301) and bromochlorodifluoromethane (CF₂ClBr, Halon1211). These bromine-containing halocarbons are highly effective in extinguishing fires and can be dispensed either from portable streaming

equipment or from an automatic total flooding system activated either manually or by some method of fire detection. However, due to the presence of Br and Cl atoms within their molecular structure these compounds have been linked to the destruction of stratospheric ozone ("ozone depletion"). The Montreal Protocol and its attendant amendments have mandated that Halon 1211 and 1301 production be discontinued.

Various different fluorinated hydrocarbons have been suggested for use as fire fighting agents, as described by M. L. Robin, "Halogenated Fire Suppression Agents", in Halon Replacements: Technology and Science, A. W. Miziolek and W. Tsang, eds., ACS Symposium Series 611, American Chemical Society, Washington, D.C., August 1994, Chapter 9. For example, hydrobromofluorocarbons (HBFCs) and hydrochlorofluorocarbons (HCFCs) have been proposed as substitutes for the Halon agents. Although effective as fire extinguishing agents, and characterized by lower ODPs compared to the Halons, HBFCs and HCFCs still contribute to the destruction of stratospheric ozone, and as a result their use and production has been slated for phase out.

In U.S. Pat. No. 5,117,917 the use of perfluorocarbons (PFCs), for example perfluoro-n-butane, as fire extinguishing agents is disclosed. The PFCs are efficient fire extinguishing agents and do not contribute to the destruction of stratospheric ozone (i.e., their ODP is equal to zero). However, the extremely high chemical and thermal stability of the PFCs results in their being characterized by very long atmospheric lifetimes. As a result of their long atmospheric lifetimes and their ability to absorb infrared (IR) radiation, the PFCs strongly contribute to global warming, and are characterized by very high GWPs.

In U.S. Pat. No. 5,124,053 the use of hydrofluorocarbons (HFCs) as fire extinguishing agents is disclosed. The HFCs are characterized by efficient fire suppression, zero ODP, low toxicity, and are also "clean" agents, leaving no residues following their use. The HFCs are, however, characterized by moderate GWPs and hence contribute somewhat to

global warming.

In U.S. Pat. No. 6,478,979 the use of perfluorinated ketones as fire
extinguishing agents is disclosed. These compounds are characterized by
5 efficient fire suppression, zero ODP and low GWP. However, the
perfluorinated ketones are also characterized by high chemical reactivity
(cf. N. P. Gambarayan, et. al., *Angew. Chemie Intern. Ed.*, 5(11), 947
(1966); A. M. Lovelace, et. al., *Aliphatic Fluorine Compounds*, ACS
Monograph Series, 1958, p. 180.). For example, the ketone
10 $\text{CF}_3\text{CF}_2\text{C}(\text{O})\text{CF}(\text{CF}_3)_2$ reacts with water to form the highly acidic, highly
toxic, and corrosive perfluoroacid perfluoropropionic acid, $\text{CF}_3\text{CF}_2\text{COOH}$,
this hydrolysis reaction also occurring when the compound is absorbed
across the lung/air interface.

15 SUMMARY OF THE INVENTION

Thus, there is a need in this field for substitutes or replacements for the
commonly-used, bromine-containing fire extinguishing agents. Such
substitutes should have a low ozone depletion potential (ODP); should
20 have the ability to efficiently extinguish, control, and prevent fires, e.g.,
Class A (trash, wood, or paper), Class B (flammable liquids or greases),
and/or Class C (energized electrical equipment) fires; and should be
"clean extinguishing agents", i.e., be electrically non-conducting, volatile or
gaseous, and leave no residue following their use. Preferably, substitutes
25 will also be low in toxicity, not form flammable mixtures in air, and have
acceptable thermal and chemical stability for use in extinguishing
applications. In addition, suitable Halon replacements should exhibit a
minimum impact on climate change, i.e., they should not contribute
significantly to global warming, being characterized by a low global
30 warming potential (GWP).

In one aspect, the invention provides a flame suppression
composition including 3-Chloro-1,1,1-trifluoropropene ($\text{CF}_3\text{CH}=\text{CHCl}$), or

HCFO-1233zd). The compositions of the present disclosure may comprise 3-Chloro-1,1,1-trifluoropropene as a single component, or in combination with one or more of a compound selected from HFCs, HFEs, HFOs, HCFOs, HBFOs, fluorinated ketones, and iodofluorocarbons. A further aspect provides for a method of reducing the flammability of a fluid comprising adding the flame suppression composition described above to the fluid.

Another aspect is for a method of suppressing a flame comprising contacting the flame with a fluid comprising the flame suppression composition described above.

Another aspect is for a method of extinguishing or suppressing a fire in a total-flood application comprising: (a) providing an agent comprising the flame suppression composition described above; (b) disposing the agent in a pressurized discharge system; and (c) discharging the agent into an area to extinguish or suppress fires in that area.

A further aspect is for a method of inerting an area to prevent a fire or explosion comprising: (a) providing an agent comprising the flame suppression composition described above; (b) disposing the agent in a pressurized discharge system; and (c) discharging the agent into the area to prevent a fire or explosion from occurring. The present invention further relates to a method for replacing or substituting for the fire extinguishing agent having a GWP of about 150 or more, or a high GWP agent in a fire protection system, with a composition having a lower GWP.

The present invention will provide compositions that have zero or low ozone depletion potential and low global warming potential (GWP). One aspect of the present invention is to provide an agent with a global warming potential of less than 1. Another aspect of the present invention is to reduce the net GWP of fire protection agents by adding 3-Chloro-1,1,1-trifluoropropene to said agents.

In a preferred embodiment, compounds of the present disclosure are useful in flame suppression, reduction, extinguishment, or inertion (collectively flame suppression compositions).

5

One aspect provides methods for reducing the flammability of a fluid, said methods comprising adding a flame suppression composition of the present disclosure to said fluid. The flammability associated with any of a wide range of flammable fluids may be reduced according to the present disclosure. For example, the flammability associated with fluids such as ethylene oxide, flammable hydrofluorocarbons, and hydrocarbons including, for example, 1,1-difluoroethane (HFC-152a), 1,1,1-trifluoroethane (HFC-143a), difluoromethane (HFC-32), propane, hexane, octane, and the like can be reduced according to the present disclosure.

For the purposes of the present disclosure, a flammable fluid may be any fluid exhibiting flammability ranges in air as measured via any standard conventional test method, such as ASTM E-681, and the like.

A further aspect provides methods of suppressing a flame, said methods comprising contacting a flame with a fluid comprising a flame suppression composition of the present disclosure. Any suitable methods for contacting the flame with the present composition may be used. For example, a flame suppression composition of the present disclosure may be sprayed, poured, and the like onto the flame, or at least a portion of the flame may be immersed in the flame suppression composition. In light of the teachings herein, those of skill in the art will be readily able to adapt a variety of conventional apparatus and methods of flame suppression for use in the present disclosure.

A further embodiment provides methods of extinguishing or suppressing a fire in a total-flood application comprising providing an agent comprising a flame suppression composition of the present disclosure; disposing the agent in a pressurized discharge system; and discharging the agent into an area to extinguish or suppress fires in that area.

Another embodiment provides methods of inerting an area to prevent a fire or explosion comprising providing an agent comprising a flame suppression composition of the present disclosure; disposing the agent in
5 a pressurized discharge system; and discharging the agent into the area to prevent a fire or explosion from occurring.

DETAILED DESCRIPTION

2-Chloro-1,1,1-trifluoropropene as used herein in the present
10 disclosure is intended to include all single configurational isomers, single stereoisomers, or any combination thereof. For example HCFO-1233zd is meant to represent the E-isomer, Z-isomer, or any combination or mixture of both isomers in any ratio. The term "extinguishment" is usually used to denote complete elimination of a fire; whereas, "suppression" is
15 often used to denote reduction, but not necessarily total elimination, of a fire or explosion. As used herein, terms "extinguishment" and "suppression" will be used interchangeably in appropriate context. There are four general types of halocarbon fire and explosion protection applications.

20 First, in total-flood fire extinguishment and/or suppression applications, the agent is discharged into an enclosed space to achieve a concentration sufficient to extinguish or suppress an existing fire. This is often, though not always, done by an automatic system, which detects the fire and then automatically discharges the extinguishing agent to fill the
25 space with the concentration of a gaseous or an evaporated volatile liquid agent to the concentration needed to suppress or extinguish the contained fire. Total flooding use includes protection of enclosed, potentially occupied spaces such, as computer rooms as well as specialized, often unoccupied spaces such as aircraft engine nacelles and engine
30 compartments in vehicles.

Second, in streaming applications, the agent is applied directly onto a fire or into the region of a fire. This is usually accomplished using

manually operated wheeled or portable units. A second method, included as a streaming application, uses a "localized" system, which discharges agent toward a fire from one or more fixed nozzles. Localized systems may be activated either manually or automatically.

5 Third, in explosion suppression, a fluorocarbon or hydrofluorocarbon of the present disclosure is discharged to suppress an explosion that has already been initiated. The term "suppression" is normally used in this application because the explosion is usually self-limiting. However, the use of this term does not necessarily imply that the
10 explosion is not extinguished by the agent. In this application, a detector is usually used to detect an expanding fireball from an explosion, and the agent is discharged rapidly to suppress the explosion. Explosion suppression is used primarily, but not solely, in defense applications.

Fourth, in inertion, a fluorocarbon or hydrofluorocarbon of the
15 present disclosure is discharged into an enclosed space to prevent an explosion or a fire from being initiated. Often, a system similar or identical to that used for total-flood fire extinguishment or suppression is used. Usually, the presence of a dangerous condition (for example, dangerous concentrations of flammable or explosive gases) is detected, and the
20 fluorocarbon or hydrofluorocarbon of the present disclosure is then discharged to prevent the explosion or fire from occurring until the condition can be remedied. In addition, in fire prevention applications for extinguishing agents, the agent is directed to an enclosed area upon detection of a potential hazard, such as a smoldering ember or a fire near
25 to but not within an enclosed area. In these applications, the atmosphere in the enclosed area will not sustain or initiate combustion but remains breathable.

The extinguishing method can be carried out by introducing the
30 composition into an area surrounding a fire. Any of the known methods of introduction can be utilized provided that appropriate quantities of the composition are metered into the enclosed area at appropriate intervals. For example, a composition can be introduced by streaming, e.g., using

conventional portable (or fixed) fire extinguishing equipment; by misting; or by total flooding, e.g., by releasing (using appropriate piping, valves, and controls) the composition into an enclosed area surrounding a fire. The composition can optionally be combined with an inert propellant, e.g.,
5 nitrogen, argon, decomposition products of glycidyl azide polymers or carbon dioxide, to increase the rate of discharge of the composition from the streaming or flooding equipment utilized.

Preferably, the extinguishing process involves introducing a flame
10 suppressant of the present disclosure to a fire or flame in an amount sufficient to extinguish the fire or flame. One skilled in this field will recognize that the amount of flame suppressant needed to extinguish a particular fire will depend upon the nature and extent of the hazard. When the flame suppressant is to be introduced by total flooding, cup burner test
15 data is useful in determining the amount or concentration of flame suppressant required to extinguish a particular type of fire.

Laboratory tests useful for determining effective concentration ranges of flame suppression compositions when used in conjunction with
20 extinguishing or suppressing a fire in a total-flood application or fire inertion are described, for example, in U.S. Pat. No. 5,759,430, which is hereby incorporated by reference. The present flame suppressants may be utilized additionally in combination with a propellant (e.g., for expelling a liquid flame suppressant from a sealed vessel), where the propellant can
25 be moderately flammable or flammable, provided that the resultant composition comprising flame suppressant and such propellant is non-flammable.

All of the compositions and methods disclosed and claimed herein can be
30 made and executed without undue experimentation in light of the present disclosure. While the compositions and methods of this disclosure have been described in terms of preferred embodiments, it will be apparent to those of skill in the art that variations may be applied to the compositions and methods and in the steps or in the sequence of steps of the method

described herein without departing from the concept, spirit, and scope of the present disclosure. More specifically, it will be apparent that certain agents which are chemically related may be substituted for the agents described herein while the same or similar results would be achieved. All
5 such similar substitutes and modifications apparent to those skilled in the art are deemed to be within the spirit, scope, and concept of the present disclosure as defined by the appended claims.

EXAMPLES

10

The present disclosure is further defined in the following Examples. It should be understood that these Examples, while indicating preferred embodiments, are given by way of illustration only. From the above discussion and these Examples, one skilled in the art can ascertain the
15 preferred features, and without departing from the spirit and scope thereof, can make various changes and modifications to adapt it to various uses and conditions.

Example 1

20

Dynamic extinguishing concentration data for E-1233zd were obtained using the standard cup burner test procedure (Appendix B, NFPA 2001, 2012 Edition), in which air and n-heptane fuel are continuously supplied to a flame produced in a glass cup burner. Vapor of the agent to
25 be tested was then mixed with air and introduced to the flame, with the concentration of agent being slowly increased until the flow was just sufficient to cause extinction of the flame. Agent concentration at extinguishment (the extinguishing concentration) was then determined via GC analysis of a sample taken near the edge of the cup in the cup burner
30 apparatus.

The extinguishing concentration for n-heptane fuel was determined to be 4.8% v/v E-1233zd.

Example 2

The experimental procedure of Example 1 was repeated with E-
5 1233zd, replacing the cup burner with a solid rod of PMMA plastic. The
extinguishing concentration for PMMA was determined to be 3.7% v/v E-
1233zd.

Example 3

10 A 0.53 m³ cubic test enclosure was constructed for static flame
extinguishment tests (total flooding). The enclosure was constructed from
Lexan® plastic and equipped with a hinged door to allow access, an inlet
in the center of the enclosure ceiling for agent introduction and a small
vent, also located on the ceiling of the test enclosure. A 0.75 in diameter
15 ceramic bowl was located in the center bottom of the enclosure and filled
with 20 drops of heptane. The heptane fuel was ignited, the enclosure
access door closed, and the fire allowed 30 second pre-burn before
introduction of the agent. An amount of E-1233zd (stored in a small metal
cylinder equipped with a dip tube and valve and pressurized to 110 psig
20 with nitrogen), predetermined to produce a concentration of 4.8% v/v was
then delivered into the enclosure through a nozzle located in the center of
the enclosure ceiling. The heptane flame was rapidly extinguished.

Example 4

The experimental procedure of Example 1 was repeated with E-
25 1233zd, replacing the n-heptane fuel with several small pieces of PMMA.
The PMMA was ignited, the enclosure access door closed, and the fire
allowed 30 second preburn before introduction of the agent. An amount
of E-1233zd (stored in a small metal cylinder equipped with a dip tube and
valve and pressurized to 110 psig with nitrogen), predetermined to
30 produce a concentration of 3.7% v/v was then delivered into the enclosure

through a nozzle located in the center of the enclosure ceiling. The PMMA flame was rapidly extinguished .

Comparison of 1233zd performance with Halon 1301

Based on NFPA 2001 (2012 edition) the minimum design concentration for Class C fires, the most commonly encountered fire types in IT and telecommunication facilities or any facilities containing electrically energized equipment, is defined as the minimum Class A extinguishing concentration times a safety factor of 1.35. For fluorinated compounds, the Class A extinguishing concentration is the agent concentration required to extinguish a PMMA fire. Hence, the design concentration for HCFO-1233zd, based on Examples 2 and 4, would be $3.7 \times 1.35 = 5.0\%$ v/v. The amount of agent required by weight to provide a concentration, C, is found from the equation (NFPA 2001):

$$W = (V/S) \times C/(100 - C)$$

where W is the weight of agent required in pounds, S is the specific volume of the agent at 70 F and 1 atmosphere.

Table 1 compares the mass efficiency of HCFO-1233zd to that of Halon 1301, where it can be seen that HCFO-1233zd is more efficient than Halon 1301 on a mass basis. This is a very surprising result as the industry has sought an agent matching the weight effectiveness of Halon 1301 for the past 25 years with no success.

TABLE 1

	Class C, minimum design concentration, % v/v	S, ft ³ /lb	Mass of agent per 100 m ³ , Class C hazard kg
HCFO-1233zd	5.0	0.1782	29.5
Halon 1301	5.0	0.15915	33.1

Claims

What is claimed:

- 5 1. A method of suppressing a flame comprising contacting the flame with a fluid comprising a flame suppression composition, wherein said flame suppression composition comprises 3-Chloro-1,1,1-trifluoropropene and optionally a propellant.
- 10 2. The method of claim 1 wherein said flame suppression composition comprises E- 3-Chloro-1,1,1-trifluoropropene.
3. The method of claim 1 wherein said flame suppression composition comprises Z- 3-Chloro-1,1,1-trifluoropropene.
4. The method of claim 1 wherein said flame suppression composition
15 comprises Z- 3-Chloro-1,1,1-trifluoropropene and E-Chloro-1,1,1-trifluoropropene.
5. The method of Claim 1 wherein the propeelant is selected from one of nitrogen, argon, carbon dioxide, helium, CF₃I, or combinations thereof.
- 20 6. A method of extinguishing or suppressing a fire in a total-flood application comprising: (a) providing an agent comprising a flame suppression composition; (b) disposing the agent in a pressurized discharge system; and (c) discharging the agent into an area to extinguish or suppress fires in that area; wherein said flame suppression composition
25 comprises 3-Chloro-1,1,1-trifluoropropene.
7. A method of extinguishing or suppressing a fire in a streaming application comprising: (a) providing an agent comprising a flame suppression composition; (b) disposing the agent in a pressurized
30 discharge system; and (c) discharging the agent towards the location of a fire to extinguish or suppress the fire; wherein said flame suppression composition comprises 3-Chloro-1,1,1-trifluoropropene.

8. A method of inerting an area to prevent a fire or explosion comprising:
(a) providing an agent comprising a flame suppression composition; (b)
disposing the agent in a pressurized discharge system; and (c)
5 discharging the agent into the area to prevent a fire or explosion from
occurring; wherein said flame suppression composition comprises 3-
Chloro-1,1,1-trifluoropropene.
9. A method of preventing a fire in an enclosed area, comprising: (a)
10 detecting a potential fire or ignition source, (b) discharging an agent
comprising a flame suppression composition into the enclosed area
thereby preventing the fire, wherein the resultant atmosphere within the
enclosed area will not sustain or initiate combustion but remains habitable;
wherein said flame suppression composition comprises 3-Chloro-1,1,1-
15 trifluoropropene. .

INTERNATIONAL SEARCH REPORT

International application No

PCT/US2014/057943

A. CLASSIFICATION OF SUBJECT MATTER

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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)

A62D

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, WPI Data, CHEM ABS Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

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Name and mailing address of the ISA/

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INTERNATIONAL SEARCH REPORT

International application No
PCT/US2014/057943

C(Continuation). 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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INTERNATIONAL SEARCH REPORT

Information on patent family members

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