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DETECTION OF GAS LEAKS

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7 Claims

ABSTRACT OF THE DISCLOSURE

The invention relates to admixing an indicant agent with fuel gases or other non-oxidizing gases so that when the mixture escapes or leaks into the atmosphere it will produce a visible signal or smoke at the locus where leakage is occurring. The smoke is produced by reaction of the indicant with oxygen in the atmosphere. The reaction of some indicants being exothermic produces a heat build-up which is also detectable by infrared photograph or infrared sensors. The indicant agents suitable for admixing with fuel gases are diphosphine; monoalkylphosphines having the structure RPH_2 wherein R is an alkyl group having 1 to 4 carbon atoms; dialkylphosphines having the structure R^1R^2PH wherein R^1 and R^2 are both methyl groups, or wherein R^1 is a methyl group and R^2 is an ethyl group; trifluoromethylphosphines having the structure $(CF_3)_nPH_{2-n}$ wherein n is 1 or 2, e.g. $(CF_3)PH_2$ and $(CF_3)_2PH$. Other suitable agents are silanes having 1 to 6 Si atoms; oxysilane derivatives, e.g. disiloxane; nitrogen silane derivatives, e.g. trisilylamine; silane substituted phosphines, e.g. trisilylphosphine.

The invention also relates to a process of pre-testing pipes, containers, vessels and the like for visually locating leaks, cracks, holes and fissures in such equipment. The interior of such equipment is filled with an inert non-oxidizing gas containing one of the aforementioned indicants, or mixtures thereof, the interior is then sealed and a predetermined superatmospheric pressure is maintained for a suitable period of time so that if any leak is present in the walls of the sealed equipment, the pressurized gas containing the indicant agent will escape into the atmosphere at such locus and the indicant present in the gas can be visually observed so as to precisely locate the leak.

BACKGROUND OF THE INVENTION

This invention relates to visually identifying or detecting gas leaks from a closed vessel, pipe system or conduit containing a normally colorless gas. More specifically, the invention relates to adding an agent or indicant to gases which are substantially colorless to the eye but which will be made visible by the presence of the added agent when the admixed gas escapes into the atmosphere.

Field of the invention

The invention has particular application to all types of natural and synthetic fuel gases which may be used for domestic and industrial purposes. For example, an indicant agent may be added to fuel gases, such as natural gas, coal gas, water gas, producer gas, butane, propane, ethylene, propene, butene and acetylene, so that leakage of such gases may be readily detected in pipeline or storage receptacles. The leakage of such admixed fuel gases can thus be readily observed at any point in the transmission lines without use of expensive equipment. Such admixed fuel gases can also serve as a safety warning to the lay person or consumer upon escape.

The present invention is also useful for pre-testing vessels, pipe systems and the like for visually locating any existing leaks therein. Such vessels or pipe systems are filled with an inert non-oxidizing gas admixed with

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a suitable indicant, or mixtures thereof, and when suitable pressure is applied to the closed vessel or pipe system the escape of the admixed gas and indicant into the atmosphere can be visually detected at the point where any leaks, cracks, or fissures exist.

Description of prior art

The article "Detection, Repair, and Prevention of Gas Leaks" published in American Gas Journal, August 1959, pages 16–28, points out that fuel gas lost through leakage represents a great economic loss today besides potential hazard to public safety. This article contains a survey of ten company practices and refers to 100 literature references. The article refers to the various methods used to detect gas leaks.

In addition, leakage of fuel gas reduces the effective capacity of a distributing system. The detection and location of fuel gas leakage have been previously based on a variety of physical and chemical principles which are costly, require elaborate and expensive equipment and trained technical personnel. The most widely used means is a combustible gas indicator which is based on the controlled aspiration of a sample of the atmosphere to be tested over a hot-wire, catalytic combustion element. The heat of combustion increases the electrical resistance of the wire which is used as a basis for determining the amount of combustible gas present in the sample atmosphere. Infrared gas detectors have also been used which have to be mounted on trucks or automobiles since considerable equipment is involved. Stethoscopes, sonic leak detectors, mass spectrometers and even trained dogs have been used to detect leaks in fuel gas mains. The addition of strong odoriferous compounds to gas mains has also been utilized so that leaks might be detected by smell. Radioactive gases have also been added to gases to serve as leak detectors requiring special detection instruments and trained personnel.

Also the use of visual indicants to pre-test leaks in closed systems or vessels offers many advantages over prior methods such as hydrostatic tests, soap test, helium mass spectrometer, etc. Newly laid distribution mains are commonly pressured with gas to test them for tightness prior to being placed in service. Leakage is often detected by the decline in the pressure in the mains as indicated by a recording pressure gage; the dragnet leak detector was developed to facilitate this type of leakage test.

The testing of pipelines is frequently a difficult and expensive task. For economic and safety reasons, pipelines must be leak-free. Hence, each pipeline must be tested for leaks at, or above, its expected operating pressure. In some situations, as in heavily populated areas or where it is required by local regulations, pipelines must be hydrostatically tested.

When a line is hydrostatically tested the presence of leaks is demonstrated by a drop in the line pressure. The location of particular leaks may be indicated by water shows. If a line under test is in an open ditch, leaking water can be easily seen in the bottom of the ditch. Many existing buried lines are tested and leaking water may, or may not, show at the surface of the ground.

In some formations water leaking out of a pipeline will drain down into the ground and will never show above-ground. In others, drainage may be lateral. In swampy areas, leaking water will just mix with the surrounding waters. From the foregoing, it may be seen that hydrostatic testing has some serious limitations.

The oldest and most common method for detecting leaks consists of applying externally a soap solution to the joint or to the point of the apparatus where a leak or crack is believed to be present, and simultaneously introducing a gas into the apparatus under pressure. The appearance of bubbles indicates a leak in the joint or the

presence of a crack. The main drawback is that the bubbles have very short life, in the range of a few seconds, and may not be observed by the operator.

The soap test has many other serious limitations. It is necessary that each valve and joint be painted with soap or a chemical solution to detect leaks. In most cases, it is difficult to get to "hard to get to places." Further, it is difficult to see leaks in the area away from the observer. Further limitations are that in a number of cases there are leaks in the pipe. Thus, it could only be discovered if the leak itself were pinpointed prior to the soap application.

Another method in detecting leaks is the use of hydrogen and helium that is put in the pipeline under pressure and would necessitate the observer to slowly scan the pipe and all the fittings with sensor equipment. This, too, has many serious limitations because there are many areas and surfaces that the observer or technician cannot get to in order to isolate the leak.

SUMMARY OF THE INVENTION

The invention provides a simple means for detecting gas leaks by the addition of an indicant agent to fuel gas. When such admixed fuel gas is stored in vessels or is supplied through gas mains or pipes any leaks which may develop are readily and immediately detected visually by the escaping visible gas without requiring any special equipment of the kind previously in use. Underground or in the dark such leaks can be additionally detected by heat-source infrared photography. A sufficient amount of the indicant agent is added so that the gas will be visible to the eye upon escape into the atmosphere. It is to be understood that when desired the added indicant may be removed from the gas mixture at the delivery end of the supply line prior to use. The indicant agent may be removed by any known methods such as by adsorption in a suitable medium, by reaction with selected reactants, or by differential diffusion.

As previously stated, the invention is particularly useful for detecting leaks in natural fuel gas pipeline systems at various points in route to the final consumer. The invention also serves as a consumer safety warning agent since the lay person can easily identify any leaks visually by observing the escaping admixed gas.

Also according to the present invention, pipes, vessels, closed systems and the like which are intended to contain fluids therein are pre-tested under static or dynamic pressures for leaks which may exist in such items either in the walls thereof or at the joints or connections of process systems in pipelines. A selected indicant carried in a gas vehicle is pumped into the closed or sealed vessel or system to be pre-tested for leaks. A predetermined pressure is maintained for a suitable time in the interior thereof, and if any leaks are present the admixed gas and indicant upon escape into the atmosphere will produce a smoke which can be observed visually, or when using selected indicants by means of infrared equipment for detecting leaks in the dark. Selected sections of a pipeline or system may be closed off and tested for leaks in the same manner.

One of the objects of this invention is to visually identify and locate leakage points in a system or vessel that normally cannot be determined until the system is operating, carrying or containing that material for which it was designed. This invention is effective for pre-testing and visually identifying any existing leak points, such as for pre-testing the piping of aircraft systems, ship systems, missile systems; telephone and cable conduits; gas or liquid systems, such as transmission, distribution lines; pre-testing submarine systems containing gas or liquid; pre-testing of cryogenic containers and transmission lines and the like.

Another object of this invention is to pre-test a ship before a "shake down" cruise in order to eliminate many leaks that normally can be repaired in port, saving time and money. This process has a broad spectrum of utility

in pre-testing for leakage in missile equipment, aircraft systems, leakage pre-testing of gas and liquid lines, chemical lines and containers.

DESCRIPTION OF PREFERRED EMBODIMENTS

According to this invention selected normally gaseous or volatile indicants are added to fuel gases or non-oxidizing gases in amount so that upon escape or leakage of the admixed gas into the normal atmosphere a visible signal or smoke will be produced. These added indicants are preferably substances which are easily volatilized and which can be admixed with the gas and carried along with the gas in a transmission line or admixed with an inert gas in a sealed vessel or pipe system. If a leak develops the added agent reacts with the oxygen in the air to produce a visible smoke.

When vessels, pipe systems and the like are pre-tested for leaks the interior thereof is completely purged or flushed out with an inert vehicle gas, such as nitrogen, hydrogen, helium or carbon dioxide and a selected non-reactive indicant or mixture of indicants is then added in pre-determined amount through a suitably connected inlet. The indicant is admixed with the gas in the interior of the vessel so that the indicant is uniformly distributed therein and the vessel is then sealed. A pre-determined superatmospheric pressure is then maintained in the interior of the vessel and the escape into the atmosphere of the indicant at the locus where a leak may exist can be observed visually. A sufficient amount of the indicant agent is added so that the test gas will be visible to the eye upon escape into the atmosphere. Pressures which are applied to the interior of the vessels and the like to be pre-tested for leaks may vary from slightly above atmospheric pressure to several hundred atmospheres depending upon the character of the vessels being pre-tested, the working pressures under which they may be normally operated, and the character of the leaks to be detected. In the case of relatively large leaks only moderate pressures are necessary to locate them, but as the leaks approach micro-size increasingly greater pressures will be required in order to locate them. The pre-testing pressures may also be maintained at a pre-determined level for a suitable period of time or may be progressively increased to specific levels.

Accordingly to this invention, I admix with fuel gases, or vehicle gases used for pre-testing leaks, one of the following pyrophoric indicant agents or mixtures thereof: diphosphine; monoalkylphosphines having the structure RPH_2 wherein R is an alkyl group having 1 to 4 carbon atoms, for example monomethylphosphine, monoethylphosphine, etc.; dimethylphosphine, methylethylphosphine; monotrifluoromethylphosphine $(CF_3)PH_2$ and ditrifluoromethylphosphine $(CF_3)_2PH$.

The previously described indicant agents are admixed with fuel gases which may be in gaseous or liquid state, or they may be admixed with non-oxidizing vehicle gases for pretesting leaks in vessels and the like, in amounts from about 0.1 gm. to about 2 gms. per 100 cu. ft. of gas, or from about 50 to about 2000 p.p.m. by weight of gas. Other indicant agents such as silanes having 1 to 6 silicon atoms, disiloxane, trisilylamine, trisilylphosphine are suitable. Mixtures of a silane and phosphine are also suitable as indicant agents. The indicant agents are used in sufficient amount so that upon escape into the atmosphere they will react with the oxygen in the atmosphere and produce a visible smoke or signal. In case the vessels and the like or gas lines should contain small amounts of oxygen, the selected indicant agent may react with the oxygen present and thus serve as an oxygen scavenger. Also where pipelines are buried in soil which may contain significant amounts of moisture sufficient indicant is used in the pipelines so that upon leakage through the soil a visible smoke is produced, although some of the indicant may react with the moisture present in the soil. When a substantial leakage occurs the escaping gas tends to produce

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fissures in the soil which tend to dry and harden due to the evaporative effect of the escaping gas, so that the gas containing the indicant will escape into the atmosphere and produce a visible signal.

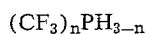
The expression "vessel and the like" used in the appended claims is intended to include pipes, conduits, containers, tanks, vessels, and closed system having spaces for containing fluid therein.

I claim:

1. The process of detecting gas leaks from a conduit through which fuel gas is flowing which comprises the steps of admixing an indicant agent with a fuel gas, said indicant agent selected from the group consisting of diphosphine; a monoalkylphosphine having the structure RPH_2 wherein R is an alkyl having 1 to 4 carbon atoms; dimethyl phosphine; methylethylphosphine; and trifluoromethylphosphine having the structure $(\text{CF}_3)_n\text{PH}_{3-n}$, wherein n is 1 or 2; and mixtures thereof, and transmitting said admixed gas through a conduit, said indicant agent being present in said fuel gas in amount to be visible when said admixed gas escapes or leaks into the atmosphere.

2. The process of locating leaks in a vessel and the like for containing a fluid which comprises the steps of filling said vessel with a gas containing an indicant agent being selected from the group consisting of diphosphine; a monoalkylphosphine having the structure RPH_2 wherein R is an alkyl having 1 to 4 carbon atoms; dimethyl phosphine; methylethylphosphine; and trifluoromethylphosphine having the structure $(\text{CF}_3)_n\text{PH}_{3-n}$, wherein n is 1 or 2; and mixtures thereof, sealing said vessel, maintaining a pre-determined superatmospheric pressure in the interior of said vessel and observing the escape into the atmosphere of said indicant at the locus of said equipment where a leak is present.

3. The process of locating leaks in a vessel and the like which comprises the steps of purging the interior of vessel with an inert gas, admixing an indicant agent with said gas until the indicant agent is uniformly distributed therein, said indicant agent being selected from the group consisting of diphosphine; a monoalkylphosphine having the structure RPH_2 wherein R is an alkyl having 1 to 4 carbon atoms; dimethylphosphine; methylethylphosphine, and trifluoromethylphosphine having the structure



wherein n is 1 or 2; and mixtures thereof, sealing said vessel containing said indicant agent, and maintaining a pre-determined superatmospheric pressure in the interior of said vessel and observing the escape into the atmosphere of said indicant agent at the locus of said equipment where a leak is present.

4. The process of locating leaks in a vessel and the like which comprises the steps of flushing out all the air present in the interior of said vessel with a gas contain-

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ing an indicant, said indicant agent being selected from the group consisting of diphosphine; a monoalkylphosphine having the structure RPH_2 wherein R is an alkyl having 1 to 4 carbon atoms; dimethylphosphine; methylethylphosphine; and trifluoromethylphosphine having the structure $(\text{CF}_3)_n\text{PH}_{3-n}$, wherein n is 1 or 2; and mixtures thereof, sealing said vessel while filled with said gas, and maintaining a pre-determined superatmospheric pressure in the interior of said vessel and observing the escape into the atmosphere of said indicant at the locus of said equipment where a leak is present.

5. The process of detecting fuel gas leaks from a container which comprises the step of admixing an indicant agent with a fuel gas, said indicant agent being selected from the group consisting of diphosphine; a monoalkylphosphine having the structure RPH_2 wherein R is an alkyl having 1 to 4 carbon atoms; dimethylphosphine; methylethylphosphine; and trifluoromethylphosphine having the structure $(\text{CF}_3)_n\text{PH}_{3-n}$, wherein n is 1 or 2; and mixtures thereof, and storing said admixed fuel gas in a container, said indicant being present in said fuel gas in amount to be visible when said admixed gas escapes into the air.

6. Fuel gas admixed with a leakage indicant agent, said indicant agent being selected from the group consisting of diphosphine; a monoalkylphosphine having the structure RPH_2 wherein R is an alkyl having 1 to 4 carbon atoms; dimethylphosphine; methylethylphosphine; and trifluoromethylphosphine having the structure $(\text{CF}_3)_n\text{PH}_{3-n}$, wherein n is 1 or 2; and mixtures thereof, said indicant being present in amount from about 50 to about 2000 p.p.m. by weight, said indicant upon leakage into the atmosphere reacting with oxygen present to produce a visible signal.

7. Fuel gas admixed with a leakage indicant agent selected from the group consisting of a silane having 1 to 4 silicon atoms; disiloxane; trisilylamine; and trisilylphosphine, said indicant agent upon leakage into the atmosphere reacting with oxygen present to produce a visible smoke.

References Cited

UNITED STATES PATENTS

1,967,871	7/1934	Dantsizen	252—408 XR
2,353,287	7/1944	Benesh	48—193 XR
3,003,349	10/1961	Sullivan et al.	73—40.7
3,085,423	4/1963	Champion	73—40.7 XR
3,361,547	1/1968	Packo	73—40.7 XR

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