Jan. 10, 1950 A. J. RITCHIE 2,494,454

METHOD OF FILLING A CONTAINER BY VACUUM AND THEN PLACING THE CONTAINER UNDER PRESSURE

Filed Aug. 10, 1944

FIG. 1

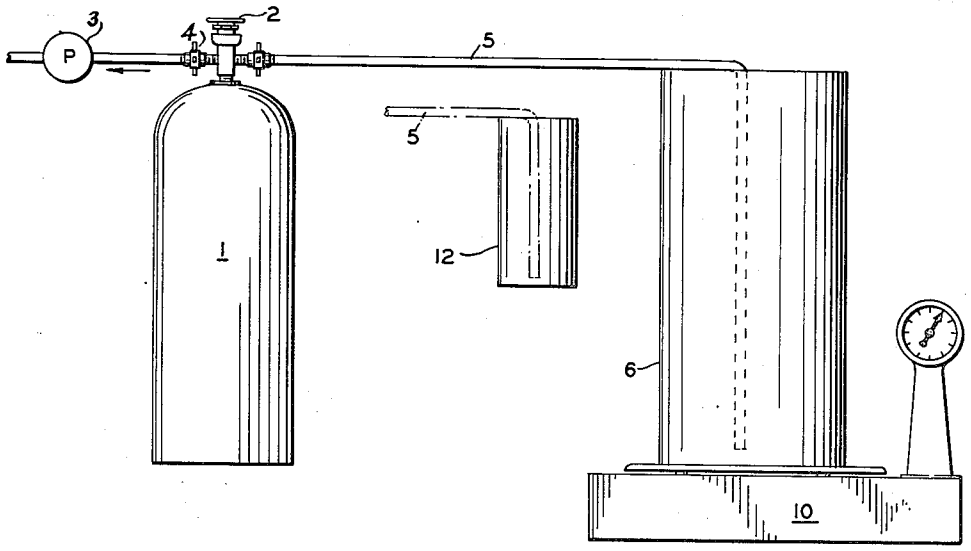
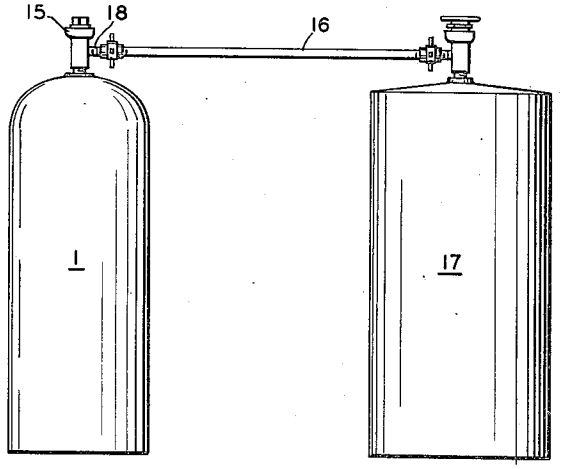


FIG. 2



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Patented Jan. 10, 1950 2,494,454

UNITED STATES PATENT OFFICE

2,494,454

METHOD OF FILLING A CONTAINER BY VACUUM AND THEN PLACING THE CONTAINER UNDER PRESSURE

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Application August 10, 1944, Serial No. 548,937

2 Claims. (Cl. 226—116)

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This invention relates to synthetic smoke or fog, and has for its object the provision of an improved method of filling containers with smoke compositions comprising titanium tetrachloride as the principal component and charging the container with a pressure-creating medium. The invention is especially concerned with the filling of containers with smoke compositions comprising titanium tetrachloride, phosphorus and carbon bisulfide as the smoke-forming materials, and carbon dioxide under pressure as the pressure medium. Smoke compositions of the character described in my copending application Serial No. 471,079, filed January 1, 1943, now Patent No. 2,407,384, of September 10, 1946, may advantageously be charged into containers and placed under a suitable working pressure by the method of this invention. The invention is directed to the filling of the metallic container (usually steel cylinders with closed ends) in which the composition is kept until discharged in creating a smoke. The invention also includes improvements in applying and using valves in connection with such containers during the filling thereof with the composition and carbon dioxide.

I have discovered a method of filling containers with the components of a smoke composition that results in greater efficiency of time, material and energy and which minimizes contamination of the composition. The components of the smoke composition react with and are impaired by water. I have found that moisture-laden air in the container is objectionable. I have found that the removal of the air sufficiently to result in a high vacuum effectively removes the moisture and that the vacuum may be used very advantageously in filling the container. I have found that the smoke-producing composition comprising titanium tetrachloride and a solution of phosphorus in carbon bisulfide may be introduced into a substantially dry container by means of the vacuum with a minimum of contamination and with the utmost safety to the workmen.

In accordance with the method of my invention, I place the container under a high vacuum eliminating most of the air and its contained moisture, and, by means of an appropriate valve apparatus, I suck the liquid smoke-producing composition into the container, either as a mixture or as separate components, and then charge compressed carbon dioxide into the container to subject the composition to a high pressure. It is advantageous to keep the titanium tetrachloride and the solution of phosphorus in carbon bisulfide separated until they are charged into the container, and I, accordingly, use the vacuum to suck the components in separately. I prefer to suck the titanium tetrachloride in first

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and in an amount insufficient to fill the container leaving an appreciable space and a residual vacuum of sufficient sub-atmospheric pressure to suck in the solution of phosphorus and carbon bisulfide. The container is accordingly charged with the three components titanium tetrachloride and the solution of phosphorus in carbon bisulfide by means of the suction of the vacuum. While I prefer to suck in the titanium tetrachloride first, largely because of its greater volume, I may reverse the order and suck in the solution of phosphorus in carbon bisulfide first, and then admit the titanium tetrachloride. I may also suck them both in together. The container filled with the three components, as herein described, is then charged with carbon dioxide under a high pressure, such as results from introducing liquid carbon dioxide.

I have found it very advantageous to provide the container with a special valve which is used when the container is under a vacuum. This valve is removed after the smoke-forming composition has been introduced into the container. It is the usual practice to provide the charged container with a permanent valve that is used to retain the composition in the container for the discharge of the liquid in forming the smoke. In accordance with that preferred aspect of my invention in which I charge the titanium tetrachloride and solution of phosphorus and carbon bisulfide in separately, I remove the special valve after charging the phosphorus and carbon bisulfide, thereby breaking the vacuum and then attach the permanent valve. A source of carbon dioxide under high pressure such as a container of liquid carbon dioxide is connected to the permanent valve and the desired quantity of carbon dioxide is charged into the smoke composition in the container.

In the accompanying drawings:

Figs. 1 and 2 are diagrammatic illustrations, in side elevation, of arrangements of apparatus used in the invention.

In carrying out a method of my invention in the filling of steel cylinders, the preliminary step is to remove any moisture adhering to the inside of the cylinder 1. This may be done by means of a blast of air, preferably dry, warm or hot air. Those cylinders which have a threaded connection, or the like, for the attachment of the permanent or discharge valve are provided with a temporary or filling valve 2. The filling valve 2 is connected to a vacuum pump 3, and, when a vacuum of around 28 inches of mercury is reached, the valve is closed and disconnected from the vacuum pump by the coupling 4. The removal of air in creating the vacuum effectively diminishes the gaseous water in the container. A pipe 5 leading to a body of liquid titanium tetrachloride in the container 6 (under a pressure

at least equal to the pressure of the atmosphere) is connected to the valve 2 and the valve 2 is opened. Any suitable means of measuring the amount of titanium tetrachloride sucked into the container may be used. A convenient procedure is to place the container on a scales 10 and let the titanium tetrachloride be sucked in until the desired weight has been reached. The introduction of titanium tetrachloride reduces the amount of vacuum and I, accordingly, shut valve 2 to interrupt the flow at a point where there is an appreciable vacuum left, sufficient at least, to suck in the carbon bisulfide and phosphorus solution and leave room for charging the carbon dioxide. The pipe 5 leading to the source of titanium tetrachloride is removed from the titanium tetrachloride container 6 and inserted into the carbon bisulfide and phosphorous solution in container 12, as shown in broken lines. Of course, the pipe 5 may be disconnected from the valve and another pipe connected for receiving the solution of phosphorus and carbon bisulfide. The valve 2 is again opened and the desired quantity of the carbon bisulfide and phosphorous solution is sucked into the container 1. The tank is agitated to mix the titanium tetrachloride, carbon bisulfide and phosphorous solution together and facilitate their intersolution. The pipe 5 and filling valve 2 are now removed, and, of course, the vacuum is broken and the unfilled space in the tank is under atmospheric pressure. The permanent or discharge valve 15 (Fig. 2) is now securely attached to the container and a pipe 16 is attached to the valve leading to a source of carbon dioxide under a high pressure, advantageously a container 17 of liquid carbon dioxide. The valve is opened and the carbon dioxide is run in until the desired pressure is attained. Ordinarily, carbon dioxide n an amount equal to about 5% of the weight of titanium tetrachloride is sufficient for the purpose. The amount of carbon dioxide is not critical, and more or less may be used as conditions dictate. For normal conditions, a sufficent amount of carbon dioxide should be charged into the container to provide a pressure of around 150 to 180 pounds per square inch at room temperature.

In order to reduce the fire hazard in the handling of the phosphorous-carbon-bisulfide solution, I add carbon tetrachloride to the solution before mixing the solution with the titanium tetrachloride. From about ½ to 1 part (by weight) of carbon tetrachloride per part of carbon bisulfide is sufficient for the purpose.

The following is an example of a typical operation for filling a steel container having a capacity of about 278 pounds of water. The dried container 1 with the filling valve 2 attached is placed under a vacuum of about 28 inches of mercury, and then the valve is connected to pipe 5 which is inserted into tank 6 of liquid titanium tetrachloride. The valve is opened and 308 pounds of titanium tetrachloride (sp. gr. 1.74) are run into the container 1. The valve is closed and the pipe is removed from the titanium tetrachloride and dipped into vessel 12 having a solution comprising 2 pounds of carbon bisulfide, 8 ounces of phosphorus and 1.5 pounds of carbon tetrachloride. The valve 2 is opened and this solution is sucked into the container. The container is agitated to facilitate intermixing

and solution of the materials. The pipe 5 and valve 2 are disconnected from the container 1, exposing the contents to atmospheric pressure, and the permanent valve 15 is securely attached by means of threads to the container. The threaded discharge end of the valve is connected to pipe 16 leading to tank 17 of liquid carbon dioxide. The valve is opened, 16 pounds of carbon dioxide are charged into the container, and the valve 15 is closed tightly. The pipe is removed, a protecting cap not shown is placed over the threaded discharge end 18, and the charged container is ready for shipment to the armed forces. It is only necessary to attach a suitable nozzle to the threaded discharge end of the valve and open the valve to discharge the smoke composition and make the smoke.

I claim:

1. The method of charging a container with a smoke composition comprising titanium tetrachloride, carbon bisulfide and phosphorus which comprises placing the container in a substantially dry condition under a vacuum, using the vacuum to suck titanium tetrachloride into the container, stopping the flow of titanium tetrachloride into the container leaving an appreciable space and a partial vacuum therein, then using the remaining vacuum to suck a solution of phosphorus in carbon bisulfide into the container, and then charging liquid carbon dioxide into the container.

2. The method of charging a container with a smoke composition comprising titanium tetrachloride, carbon bisulfide and phosphorus which comprises attaching to the container a filling valve, placing the container in a substantially dry condition under a vacuum, attaching a duct leading to a source of titanium tetrachloride to the valve and opening the valve to suck the charge of titanium tetrachloride into the container, closing the valve before the container is filled with titanium tetrachloride whereby an appreciable space and a partial vacuum remain in the container, connecting the valve by a duct to a solution of phosphorus in carbon bisulfide, removing the filling valve and attaching a discharge valve to the container, connecting the discharge valve to a source of carbon dioxide under pressure and charging the carbon dioxide into the container, and closing the discharge valve and disconnecting it from the source of carbon dioxide.

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