1,077,674

PATENT SPECIFICATION

NO DRAWINGS

1,077,674

Date of Application and filing Complete Specification: July 14, 1964.

No. 29026/64.

Application made in United States of America (No. 298,523) on July 23, 1963.

Complete Specification Published: Aug. 2, 1967.

© Crown Copyright 1967.

Index at acceptance:—F4 P1A1A; C1 H(D3, DX)

Int. Cl.:—F 25 j//C 04 b

COMPLETE SPECIFICATION

Acetylene Container Filler Composition

We, AMERICAN CRYOGENICS, INC., a Corporation organised and existing under the laws of the State of Delaware, United States of America, of 601 California Street, San Francisco, California, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a filler composition of the type adapted for holding dissolved acetylene in containers at elevated pressures.

The need for filler composition in acetylene containers is created by properties peculiar to acetylene gas. It is well known that acetylene gas, even in very small amounts, may become violently unstable at pressures in excess of 15 psi, and almost with certainty above 50 psi. This condition is substantially overcome by dissolving the acetylene in acetone. The acetone dissolves as much as six times its own weight of acetylene. Pressures up to 300 psi or more of dissolved gas may be obtained in this manner if the acetone container is filled with a highly porous material of which the individual pores are minute.

In order to be useable for the above purpose, the highly porous material must be such that it will not settle, shrink or disintegrate so as to leave voids within which acetylene gas under pressure may collect and become unstable. Thus, in addition to the essential characteristic of high porosity, it is most important that the porous material have high compressive (resistance to crushing) strength and dimensional stability.

A number of materials have been developed for use as a filler for the above purpose. The most important of these developments is represented by several recent United States patents which disclose the use of filler compositions of charcoal and fibrous asbestos dispersed and set in a silica-lime product. In particular, it has been taught that certain ratios of the ingredients are critical, and when preferred limits are observed outstanding products adapted for the above purpose are obtained. In addition, these patents disclose certain methods for compounding the product and for using the product in filling suitable containers for holding the acetylene gas and carrier fluid.

The present invention aims to improve upon these disclosures by way of providing a product having significantly improved crushing strength.

In accordance with the present invention, it has been discovered that if the filler composition is made according to the following formula, this advantageous and essential property is obtained:

	Ratio by Weight
Water to solids	3.45
Silica to lime	1.75
	Percent of total solids in the composition by weight
Asbestos	18.40
Charcoal	18.40

In addition, it has been discovered that these advantageous properties are enhanced and time saved in preparation by making a number of modifications in the process for preparing the composition. These changes will be discussed in conjunction with the following detailed description of the practice of the present invention.

As in prior procedures, the acetylene container filler is prepared by initially pre-treating charcoal and lime.

Pulverized lime (any high calcium, commercially available pulverized lime) is hydr-

[Price 4s. 6d.]

ated with water to obtain a slurry comprising about 20% by weight of the calcium oxide.

Charcoal, preferably screened to pass through a 1/2" mesh, is pre-soaked. The pre-soaking procedure utilized herein represents a significant departure from prior practices. Whereas it has previously been the practice to soak the charcoal in water for at least 12 hours, the present method comprises soaking the charcoal in water for not more than about two hours. During the shortened pre-soaking period, the charcoal-water mixture is subjected to a vacuum for at least part of the pre-soaking period. Preferably, the vacuum is applied to the soaking charcoal for 10 minutes under a pressure of about 28—30" of mercury.

The order and method of combination of the charcoal, lime, asbestos, and water, and the conditions for the combination in accordance with the present invention also represent significant departures from prior procedures. Previously, it has been the practice to add the asbestos first to water and mix the materials until all visible air bubbles are gone. Then pre-soaked charcoal was added with agitation. Following this, silica was mixed into the formula for about 15 to 30 minutes. Finally, the hydrated lime was added and mixed for about 15 minutes following which the formula was ready for charge into suitable cylinders.

In the present improved method, the pre-soaked charcoal (prepared as described above by the new technique) is first added to water, and silica added thereto with agitation. A vacuum is then applied to the charcoal-silica mixture while the lime slurry is added, also with agitation. Following the lime-slurry addition, the vacuum is removed and the asbestos is added. Agitation and mixing is continued for about a half hour or until the entire mass is homogeneous. The improved formula is now ready for charge into suitable cylinders.

In preparing the present formula, the materials are used in the relative quantities as given in the table above. For example, sufficient water is added at the various stages so that the final water-to-solid ratio is 3.45. In particular, the percent of asbestos is controlled to be 18.40.

The present filling procedure represents a substantial improvement in product and time saving over prior techniques. Whereas the present procedure is accomplished virtually in one operation and over a relatively short period of time, prior techniques have required complete initial filling of the cylinders, vibration, long settling periods of many hours, subsequent vibration and refilling, and repetition of the entire cycle.

In the present improved method, the completed filler slurry is introduced into suitable empty commercial acetylene containers. In the preferred procedure, the slurry is placed in a hopper from which it is introduced into the cylinders. Preferably, one of the top fuse plugs of each of the cylinders is removed and a vacuum line attached so that filling is carried out while placing a vacuum on the interior of the cylinder. Sufficient slurry is then introduced into each cylinder to completely fill it. The cylinders are vibrated during filling, and while being vibrated additional slurry is added to thereby complete the filling operation in a single rapid step. The cylinders are then indurated in the manner previously employed.

Induration is suitably accomplished by applying heat to the containers while preventing loss of water from the slurry. Preferably the induration is performed promptly after filling of the cylinders. Since water expands during the heating process and should not be allowed to escape, suitable hydraulic expansion heads for the containers are provided.

Heating of the containers may be carried out in an oven maintained at a temperature range of from about 350°—375°F. during the heating period. Treatment generally continues over a period of about 15—16 hours after the oven reached the 350° mark.

Following the induration period, the filler is dried and the water contents driven off. The containers are cooled and are then ready for filling with the acetylene fluid.

To illustrate the advantages of the present product and method, the following test was made. A filler made in accordance with the present invention was analysed and found to have a crushing strength of 337 psi. At the same time, it had a porosity of 90.6% and a density of 0.247 grams per cc.

For comparison, a filler made in accordance with the prior procedures noted herein was analysed. It was found to have a crushing strength of 210 psi, a porosity of 90.7% and a density of 0.257 grams per cc.

It will therefore be seen that the present material exhibits a rather overwhelming advantage in crushing strength while still retaining the desirable high porosity and low density of the prior products.

WHAT WE CLAIM IS:—

1. An acetylene container filler composition consisting essentially of charcoal and fibrous asbestos dispersed and set in a silica-lime, the composition comprising the following:—

	Ratio by Weight
Water to solids	3.45
Silica to lime	1.75

	Percent of total solids in the composition by weight
Asbestos	18.40
Charcoal	18.40

ated with water to obtain a slurry comprising about 20% by weight of the calcium oxide.

Charcoal, preferably screened to pass through a 1/2" mesh, is pre-soaked. The pre-soaking procedure utilized herein represents a significant departure from prior practices. Whereas it has previously been the practice to soak the charcoal in water for at least 12 hours, the present method comprises soaking the charcoal in water for not more than about two hours. During the shortened pre-soaking period, the charcoal-water mixture is subjected to a vacuum for at least part of the pre-soaking period. Preferably, the vacuum is applied to the soaking charcoal for 10 minutes under a pressure of about 28—30" of mercury.

The order and method of combination of the charcoal, lime, asbestos, and water, and the conditions for the combination in accordance with the present invention also represent significant departures from prior procedures. Previously, it has been the practice to add the asbestos first to water and mix the materials until all visible air bubbles are gone. Then pre-soaked charcoal was added with agitation. Following this, silica was mixed into the formula for about 15 to 30 minutes. Finally, the hydrated lime was added and mixed for about 15 minutes following which the formula was ready for charge into suitable cylinders.

In the present improved method, the pre-soaked charcoal (prepared as described above by the new technique) is first added to water, and silica added thereto with agitation. A vacuum is then applied to the charcoal-silica mixture while the lime slurry is added, also with agitation. Following the lime-slurry addition, the vacuum is removed and the asbestos is added. Agitation and mixing is continued for about a half hour or until the entire mass is homogeneous. The improved formula is now ready for charge into suitable cylinders.

In preparing the present formula, the materials are used in the relative quantities as given in the table above. For example, sufficient water is added at the various stages so that the final water-to-solid ratio is 3.45. In particular, the percent of asbestos is controlled to be 18.40.

The present filling procedure represents a substantial improvement in product and time saving over prior techniques. Whereas the present procedure is accomplished virtually in one operation and over a relatively short period of time, prior techniques have required complete initial filling of the cylinders, vibration, long settling periods of many hours, subsequent vibration and refilling, and repetition of the entire cycle.

In the present improved method, the completed filler slurry is introduced into suitable empty commercial acetylene containers. In the preferred procedure, the slurry is placed in a hopper from which it is introduced into the cylinders. Preferably, one of the top fuse plugs of each of the cylinders is removed and a vacuum line attached so that filling is carried out while placing a vacuum on the interior of the cylinder. Sufficient slurry is then introduced into each cylinder to completely fill it. The cylinders are vibrated during filling, and while being vibrated additional slurry is added to thereby complete the filling operation in a single rapid step. The cylinders are then indurated in the manner previously employed.

Induration is suitably accomplished by applying heat to the containers while preventing loss of water from the slurry. Preferably the induration is performed promptly after filling of the cylinders. Since water expands during the heating process and should not be allowed to escape, suitable hydraulic expansion heads for the containers are provided.

Heating of the containers may be carried out in an oven maintained at a temperature range of from about 350°—375°F. during the heating period. Treatment generally continues over a period of about 15—16 hours after the oven reached the 350° mark.

Following the induration period, the filler is dried and the water contents driven off. The containers are cooled and are then ready for filling with the acetylene fluid.

To illustrate the advantages of the present product and method, the following test was made. A filler made in accordance with the present invention was analysed and found to have a crushing strength of 337 psi. At the same time, it had a porosity of 90.6% and a density of 0.247 grams per cc.

For comparison, a filler made in accordance with the prior procedures noted herein was analysed. It was found to have a crushing strength of 210 psi, a porosity of 90.7% and a density of 0.257 grams per cc.

It will therefore be seen that the present material exhibits a rather overwhelming advantage in crushing strength while still retaining the desirable high porosity and low density of the prior products.

WHAT WE CLAIM IS:—

1. An acetylene container filler composition consisting essentially of charcoal and fibrous asbestos dispersed and set in a silica-lime, the composition comprising the following:—

	Ratio by Weight
Water to solids	3.45
Silica to lime	1.75

	Percent of total solids in the composition by weight
Asbestos	18.40
Charcoal	18.40

2. An acetylene container filler composition substantially as herein described.

For the Applicants:
LLOYD WISE, BOULY & HAIG,
Chartered Patent Agents,
Norman House,
105—109 Strand,
London, W.C.2.

Leamington Spa: Printed for Her Majesty's Stationery Office by the Courier Press.—1967
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.

