J. VOIGT.

MANUFACTURE OF ACETYLENE TETRACHLORID.

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908,051. Patented Dec. 29, 1908.

Fig. 1.

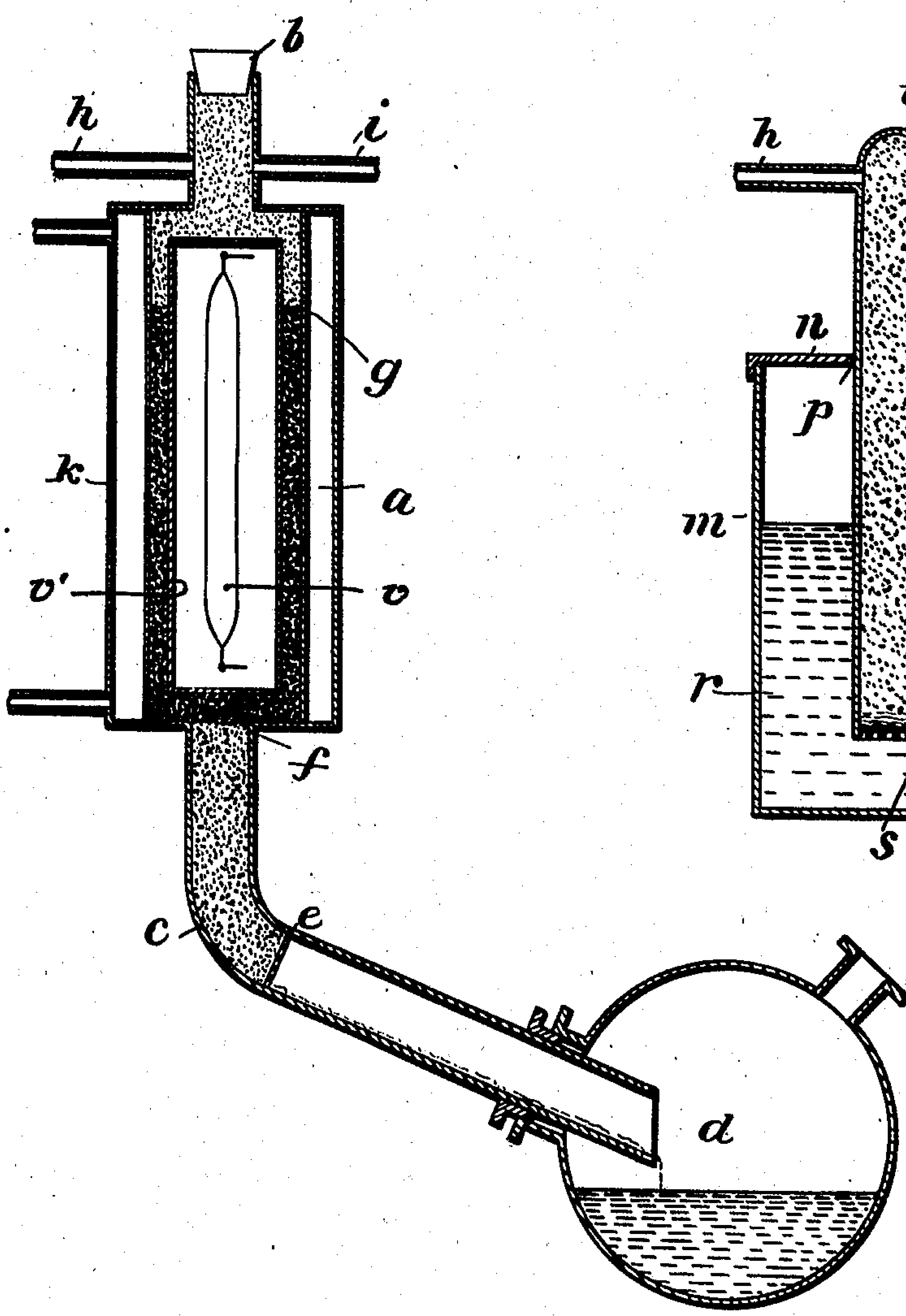
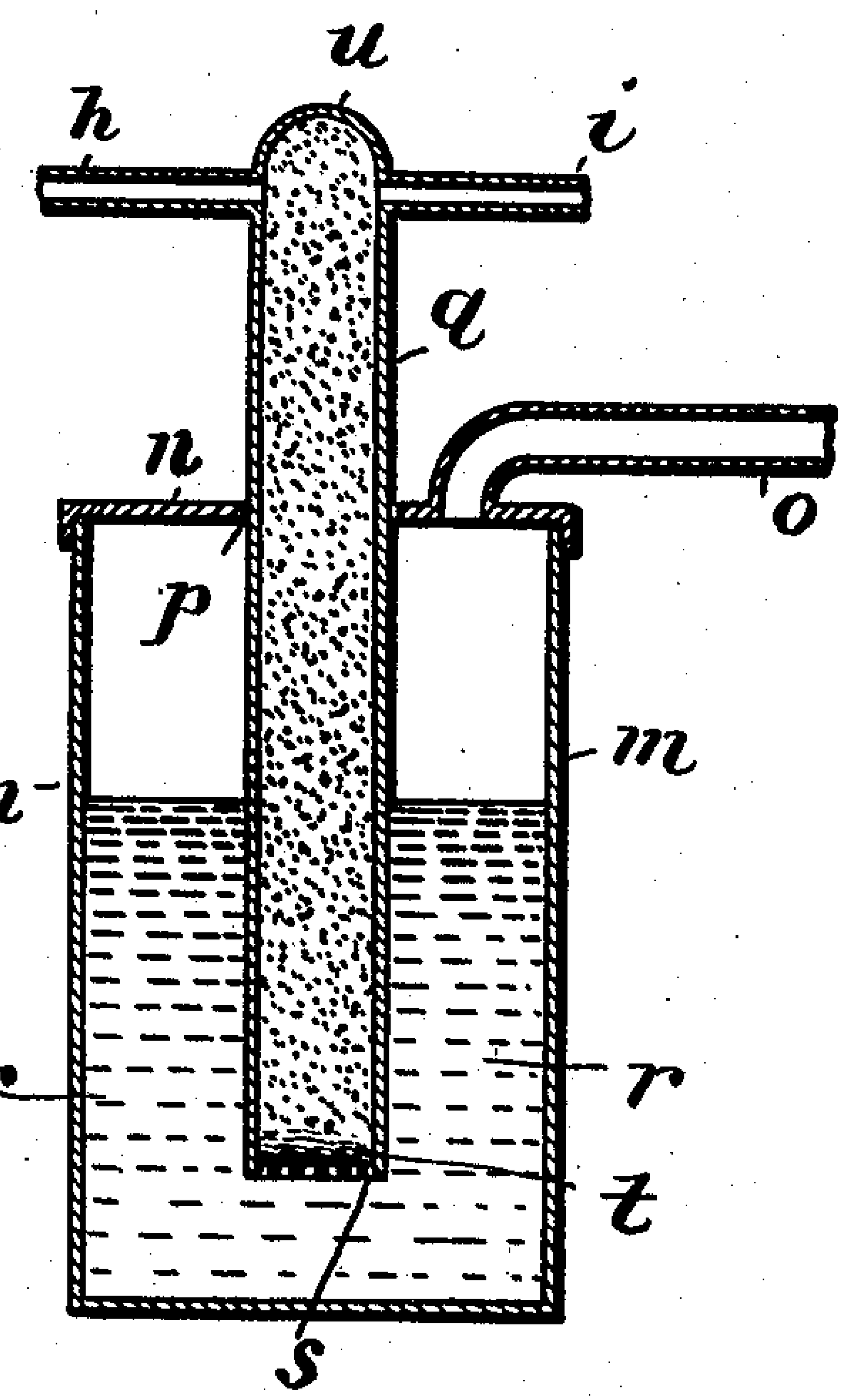


Fig. 2



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MANUFACTURE OF ACETYLENE TETRACHLORID.

No. 908,051. Specification of Letters Patent. Patented Dec. 29, 1908.

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To all whom it may concern:

Be it known that I, JULIUS VOIGT, doctor of philosophy, residing at Schwanheim-on-the-Main, Germany, have invented certain new and useful Improvements in the Manufacture of Acetylene Tetrachlorid, of which the following is a specification.

This invention has for its object to provide an improved commercial safe, and economical, process of manufacture of acetylene tetrachlorid, a product which has of late been frequently employed as a solvent, and as an extracting agent. The separation of this compound directly from chlorin and acetylene is attended with danger in consequence of these substances being very liable to explode when they come into contact.

Mouneyrat has, in 1898, (*Bulletin de la Societe Chimique de Paris* (3). 19. 1898 page 448 ff) stated that acetylene and chlorin, when mixed in a gaseous condition, can enter into reaction without explosion occurring if air be entirely excluded from the operation. The observation of Mouneyrat does not however permit of manufacturing acetylene chlorid on a commercial scale and its application has, so far, been confined to the laboratory.

In an article by J. Nieuwland of Notre Dame, Indiana, U. S. A., in the "*Journal für Gas Beleuchtung*", Keppler, 1904, experiments are described in which acetylene and chlorin have been combined to form acetylene tetrachlorid in open vessels without any explosion occurring. These experiments have decreased the importance of Mouneyrat's observations concerning the necessary exclusion of the air, but Nieuwland has not described any conditions which have to be observed to invariably, and with certainty, prevent the explosions and ignitions peculiar to a gaseous mixture of acetylene and chlorin. Nieuwland has stated that he often observed in his experiments ignitions under circumstances which he could not account for, and this fact can be verified without difficulty by repeating his experiments, and it is evident that the commercial manufacture of acetylene tetrachlorid can only be safely undertaken if the main difficulty and danger attendant upon it be entirely removed.

The present invention is based on the observation, made by me that it is possible to safely manufacture acetylene tetrachlorid and obtain good quantitative yields if the chlorin and acetylene are, before they come into contact, disseminated through porous or comminuted material which cannot itself enter into reaction with the components, and the mixture of gases thus obtained can by properly prolonging the portion of the tube filled with sand, etc. be safely conveyed to the place of reaction and then the reagents be combined to form acetylene chlorid (with, or without, the porous or comminuted material being present) by suitable contact substances, or by the action of light.

The solid bodies which can be used to prevent explosion and into which each gas is caused to flow, may be of the most varied nature, such as sand, pulverized earthenware, infusorial earth, or other pulverulent material, or it may consist of porous clay, or the like. The size of the grains, or pores, may vary from the finest river sand up to grains of 5 millimeters diameter. A mixture of different sizes may also be used. Grains having a diameter of from 1½ to 2 millimeters are well suited for carrying out the invention. If a porous material is used the pores preferably should not be more than 3 to 4 millimeters in diameter, as otherwise a carbon deposit may be formed by the reactions. Their maximum size should be such that they do not permit the gases igniting, and their minimum size should be such that they do not oppose too great a resistance to the flow of the gases. The conversion of the gas so obtained into acetylene tetrachlorid can be effected by various means. Natural and artificial sources of light, containing chemically active rays, are both suitable, and simple contact substances, such as iron, antimony, and the like, or liquid intermediaries, or vehicles, (for instance, antimony pentachlorid), are very suitable.

In the accompanying drawing, Figure 1 is a vertical sectional view through an apparatus adapted to carry out the present invention; Fig. 2 is a similar view of a slightly different form of apparatus.

Referring to Fig. 1, *a* designates a tube, for example of lead, having its upper end closed by a plug *b* and bent at *c* so as to provide an inclined section which extends into a receiver *d*. A sieve *e* is arranged within the lower inclined portion of the tube, being secured in position by solder or other suitable means, and from said sieve to the point *f* the tube is filled with grains of gravel and from the point *f* to the point *g* with a mixture of powdered iron and sand. The remaining section of the tube, above the mixture of sand and iron is filled with pure sand. With this upper section of the tube *a* communicate two nipples or pipes *h*, *i* through which, respectively, acetylene and chlorin gases are supplied to the tube. Surrounding a portion of the tube *a* is a device *k* for cooling the tube and its contents.

Tubes *h*, *i* connected with the main tube *a* serve respectively to conduct acetylene and chlorin gases to said main tube. The gases mix in the upper, sand-filled section of the tube and as they pass downwardly through the intermediate section, filled as aforesaid with a mixture of sand and powdered iron, they unite to form acetylene tetrachlorid. This mixing of the gases generates heat and in order to maintain the apparatus below the boiling point of acetylene tetrachlorid it is necessary to employ the cooling apparatus *k* by which the temperature is preferably maintained at from fifteen to twenty degrees centigrade.

Acetylene tetrachlorid formed as above described percolates through the gravel-filled section of the tube *a* and passes into the receiver *d* which is provided with an outlet. Any suitable regulating means may be employed for controlling the supply of the chlorin and acetylene gases.

As before stated a porous material may be substituted for the sand in the apparatus. If the desired reaction is produced by rays of light the apparatus must be made of glass and great care taken to so arrange the tube that the entire length thereof is exposed to the action of the light rays. In such a construction the section *f g* of the tube is filled with pure sand. If the tube *a* is of metal it is necessary to arrange a mercury vapor lamp *v* in a transparent cylinder *v'* within the section *f g* of the tube.

By the means above described it is possible to effect a mixture of acetylene and chlorin in a single tube without danger. Said gases may also be mixed by means of such an apparatus as is illustrated in Fig. 2, referring to which *m* indicates a container or vessel of any suitable size, form and material, which is provided with a lid or cover *n*. A tube *o* leading through the cover *n* provides an escape for any excess gas which may be formed in the vessel, and the lid or cover is also provided with an opening *p* through which extends a tube *q*, of glass or lead, which tube extends downward below the body of liquid *r* in the vessel *m*. The liquid *r* may be any suitable solvent by which chlorin and acetylene may be chemically united, for instance, antimony pentachlorid, or a mixture of antimony pentachlorid and antimony trichlorid, or a solution of these substances in acetylene tetrachlorid or other suitable solvent.

The tube *q* is entirely filled with porous or comminuted material which is supported by a sieve *s* at the lower end of the tube, a body of asbestos fibers *t* being arranged between the sieve. The upper end *u* of the tube is closed and with said tube communicate branch tubes *h*, *i*, adapted to respectively admit acetylene and chlorin gases to the tube. The gases mix within the tube and after passing through the sieve *s* come into contact with the liquid *r* by which they are absorbed and caused to unite, forming acetylene tetrachlorid.

In the form of apparatus shown in Fig. 2 it will be seen that the reaction takes place within the solution and outside of the tube through which the gases pass, whereas with the apparatus shown in Fig. 1 the reaction occurs in the body of the tube.

Having thus described the invention, what is claimed is:

1. The herein described process of manufacturing acetylene tetrachlorid, consisting in causing acetylene and chlorin gases to unite while contained in a body of granular non-catalytic material.

2. The herein described process of manufacturing acetylene tetrachlorid, consisting in separately introducing chlorin and acetylene gases into a body of porous or comminuted non-catalytic material, and causing said gases to react one on the other while passing through such material.

3. The herein described process of manufacturing acetylene tetrachlorid, consisting in separately introducing chlorin and acetylene gases into a body of porous or comminuted non-catalytic material containing a substance which by contact with said gases causes them to react one on the other.

4. The herein described process of manufacturing acetylene tetrachlorid, consisting in separately introducing chlorin and acetylene gases into a mixture of granular non-catalytic material containing powdered iron.

In testimony whereof, I have signed my name to this specification in the presence of two subscribing witnesses.

JULIUS VOIGT.

Witnesses:
FRANZ HASSLACHER,
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