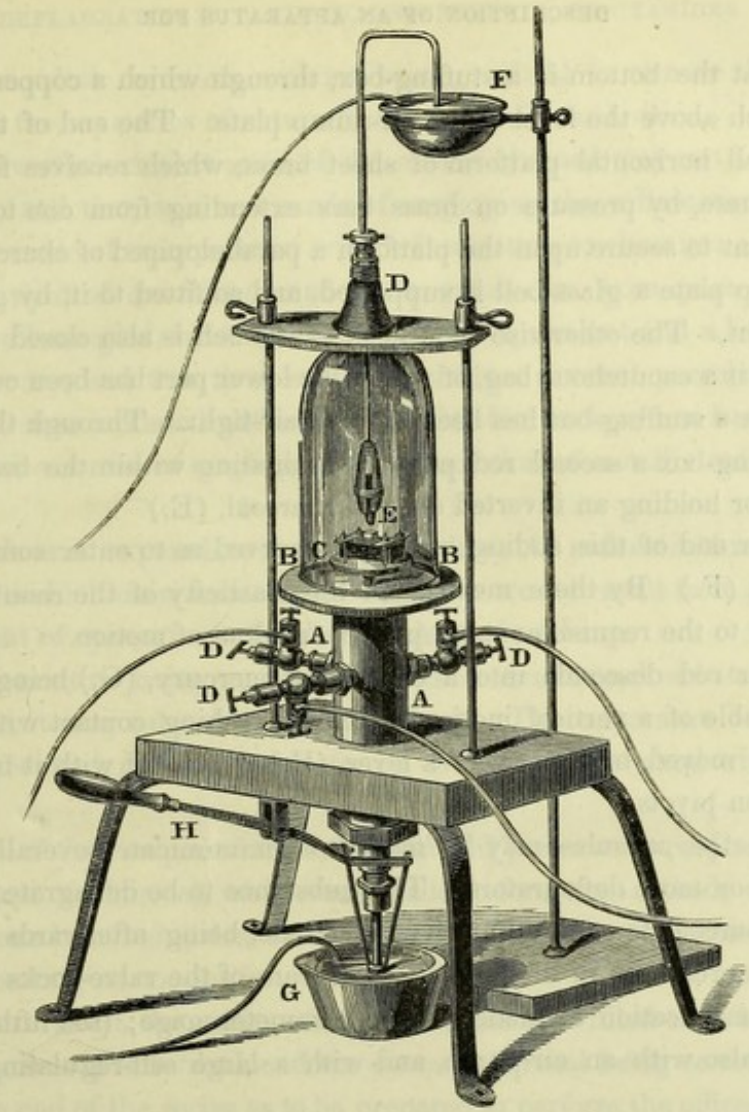
Description of an apparatus for deflagrating cabinets, phosphurets, or cyanides, in vacuo or in an atmosphere of hydrogen : with an account of some results obtained by these and by other means, especially the isolation of calcium / by Robert Hare.



ARTICLE V.

Description of an Apparatus for Deflagrating Carburets, Phosphurets, or Cyanides, in Vacuo or in an Atmosphere of Hydrogen, with an account of some Results obtained by these and by other means; especially the Isolation of Calcium. *By Robert Hare, M.D.* *Read October* 18, 1839.

Upon a hollow cylinder of brass (A A) an extra air-pump plate (B B) is supported. The cylinder is furnished with three valve cocks, (D D D,) and

terminates at the bottom in a stuffing-box, through which a copper rod slides so as to reach above the level of the air-pump plate. The end of the rod supports a small horizontal platform of sheet brass, which receives four upright screws. These, by pressure on brass bars extending from one to the other, are competent to secure upon the platform a parallelopiped of charcoal. Upon the air-pump plate a glass bell is supported, and so fitted to it, by grinding, as to be air-tight. The otherwise open neck of the bell is also closed air-tight by tying about it a caoutchouc bag, of which the lower part has been cut off, while into the neck a stuffing-box has been secured air-tight. Through the last mentioned stuffing-box a second rod passes, terminating within the bell in a kind of forceps, for holding an inverted cone of charcoal, (E.)

The upper end of this sliding rod is so recurved as to enter some mercury in a capsule, (F.) By these means and the elasticity of the caoutchouc bag, this rod has, to the requisite extent, perfect freedom of motion.

The lower rod descends into a capsule of mercury, (G,) being, in consequence, capable of a vertical motion, without breaking contact with the mercury. It is moved by the aid of a lever, (H,) connected with it by a stirrup working upon pivots.

Of course the capsules may be made to communicate severally with the poles of one or more deflagrators. The substance to be deflagrated is placed upon the charcoal forming the lower electrode, being afterwards covered by the bell, as represented in the figure. By means of the valve-cocks and leaden pipes a communication is made with a barometer gage; (see fifth volume of this work;) also with an air-pump, and with a large self-regulating reservoir of hydrogen.

The air being removed by the pump, a portion of hydrogen is admitted, and then withdrawn. This is repeated, and then the bell glass, after as complete exhaustion as the performance of the pump will render practicable, is prepared for the process of deflagration in vacuo. But, if preferred, evidently hydrogen or any other gas may be introduced from any convenient source by a due communication through flexible leaden pipes and valve-cocks.

Having described the apparatus, I will give an account of some experiments, made with its assistance, which, if they could have illuminated science as they did my lecture room, would have immortalized the operator. But, alas, we may be dazzled, and almost blinded by the light produced by the hydro-oxygen

blow-pipe, or voltaic ignition, without being enabled to remove the darkness which hides the mysteries of nature from our intellectual vision.

I hope, nevertheless, that some of the results attained may not be unworthy of attention; and that, as a new mode of employing the voltaic circuit, my apparatus and mode of manipulation will be interesting to chemists.

An equivalent of quicklime, made with great care from pure crystallized spar, was well mingled, by trituration, with an equivalent and a half of bicyanide of mercury, and was then enclosed within a covered porcelain crucible. The crucible was included within an iron alembic, such as has been described by me, in this volume, as employed for the isolation of metallic radicals. (See page 38.)

The whole was exposed to heat approaching to redness. In two experiments the residual mass had such a weight as would result from the union of an equivalent of cyanogen with an equivalent of calcium.

A similar mixture being made, and, in like manner, enclosed in the crucible and alembic, it was subjected to a white heat. The apparatus being refrigerated, the residual mass was transferred to a dry glass phial with a ground stopper.

A portion of the compound thus obtained and preserved was placed upon the parallelopiped of charcoal, which was made to form the cathode of two deflagrators of one hundred pairs, each of one hundred square inches of zinc surface, co-operating as one series.

In the next place, the cavity of the bell-glass was filled with hydrogen, by the process already described, and the cone of charcoal being so connected with the positive end of the series as to be prepared to perform the office of an anode, was brought into contact with the compound to be deflagrated. These arrangements being accomplished, and the circuit completed by throwing the acid upon the plates, the most intense ignition took place.

The compound proved to be an excellent conductor; and during its deflagration emitted a most beautiful purple light, which was too vivid for more than a transient endurance by an eye unprotected by deep-coloured glasses. After the compound was adjudged to be sufficiently deflagrated, and time had been allowed for refrigeration, on lifting the receiver minute masses were found upon the coal, which had a metallic appearance, and which, when moistened,

produced an effluvium, of which the smell was like that which had been observed to be generated by the silicuret of potassium.

Similar results had been attained by the deflagration, in a like manner, of a compound procured by passing cyanogen over quicklime, enclosed in a porcelain tube, heated to incandescence.*

Phosphuret of calcium, when carefully prepared, and, subsequently, well heated, was found to be an excellent conductor of the voltaic current evolved from the apparatus above mentioned. Hence it was thought expedient to expose it in the circuit of the deflagrator, both in an atmosphere of hydrogen and in vacuo. The volatilization of phosphorus was so copious as to coat nearly all the inner surface of the bell-glass with an opake film, in colour resembling that of the oxide of phosphorus, generated by exposing this substance under hot water to a current of oxygen.†

The phosphuret at first contracted in bulk, and finally was, for the most part, volatilized. On the surface of the charcoal, adjoining the cavity in which the phosphuret had been deflagrated, there was a light pulverulent matter, which, thrown into water, effervesced, and, when rubbed upon a porcelain tile, appeared to contain metallic spangles, which were oxydized by the consequent exposure to atmospheric oxygen.

In one of my experiments with the apparatus above described, portions of the carbon forming the anode appeared to have undergone complete fusion, and to have dropped in globules upon the cathode. When rubbed, these globules had the colour and lustre of plumbago, and, by friction on paper, left traces resembling those produced by that substance. They were susceptible

* After the above mentioned experiments were made, I was led to believe that the compound, obtained as above described by heating lime with bicyanide of mercury, contained fulminic acid, or an analogous substance. The mass being dissolved in acetic acid, and the filtered solution subjected to nitrate of mercury, a copious white precipitate resulted. This, being desiccated, proved to be a fulminating powder. It exploded, between a hammer and anvil, with the sharp sound of fulminating silver.

† The compound usually designated as the phosphuret of calcium consists, according to Thomson, of one atom of phosphate of lime, as well as two atoms of pure phosphuret. Hence it is easy to see that the oxygen which enters into the constitution of the oxide, deposited, as above mentioned, upon the interior surface of the bell-glass, is derived from the phosphate.

of reaction neither with chloro-hydric nor with nitric acid, neither separately nor when mixed. They were not in the slightest degree magnetic.

About 1822, Professor Silliman obtained globules, which were at first considered as fused carbon, but were subsequently found to be depositions of that substance transferred from one electrode to the other. Several of these globules were, by him, sent to me for examination, of which none, agreeably to my recollection, appeared so much like products of fusion as those lately obtained.

Formerly plumbago was considered as a carburet of iron, but latterly, agreeably to the high authority of Berzelius, has been viewed as carbon holding iron in a state of mixture, not in that of chemical combination. It would not, then, be surprising if the globules which I obtained consisted of carbon converted from the state of charcoal into that of plumbago.