



A.D. 1854 N° 2161.

Manufacture of Sulphuric Acid.

LETTERS PATENT to James Shanks, of Saint Helens, in the County of Lancaster, Manufacturing Chemist, for the Invention of “**AN IMPROVED MODE OF MANUFACTURING SULPHURIC ACID.**”—A communication from Robert Von Seckendorf, of Gera, in the State of Reuss, in Germany.

Sealed the 15th December 1854, and dated the 9th October 1854.

PROVISIONAL SPECIFICATION left by the said James Shanks at the Office of the Commissioners of Patents, with his Petition, on the 9th October 1854.

I, JAMES SHANKS, of St. Helens, in the County of Lancaster, Manufacturing
5 Chemist, do hereby declare the nature of the said Invention for “**AN IMPROVED MODE OF MANUFACTURING SULPHURIC ACID**” to be as follows:—

This Invention for the manufacture of sulphuric acid consists in certain means used for the separation or extraction of sulphuric acid contained in gypsum or sulphate of lime, either in a native or artificial state.

10 It is necessary, in the first place, to provide a large vessel or receptacle, which I call a decomposing vessel or vat, made of wood, stone, or lead, or of any other suitable material, and of sufficient size to produce a large quantity of sulphuric acid at one operation; I then take 86 parts by weight, or one equivalent, of native gypsum, or 68 parts by weight of anhydrous sulphate of lime, in a finely
15 pulverized state, and place it in the decomposing vessel or vat, and add to

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it 140 parts by weight of chloride of lead; I then add a large quantity of water, at the temperature of 130° to 180° of Fahrenheit's thermometer, and then stir the whole well together. An immediate decomposition takes place, sulphate of lead immediately subsides, and chloride of calcium is held in the supernatant liquid. 5

In order to save trouble and expedite the process, it is desirable that the solution of chloride of calcium should indicate only 4° on Twaddle's hydrometer. The heavy white precipitate which now remains in the decomposing vessel is sulphate of lead. This substance is to be treated with hydrochloric acid, and in order to get a full decomposition of the sulphate of lead, I find it advisable 10 to use more than its equivalent of the hydrochloric acid.

By adding hydrochloric acid to the sulphate of lead, and mixing them well together by stirring at a temperature of about 150° Fahrenheit, an immediate change takes place, and chloride of lead is formed, which falls to the bottom of the vessel, while the liquid sulphuric acid is now set free. The sulphuric 15 acid being thus liberated, is now to be withdrawn from the decomposing vessel in the usual manner, and evaporated to the strength required for manufacturing or other purposes. A further quantity of water may be added to the decomposing vessel for washing out more of the sulphuric acid from the chloride of lead, and which further quantity may be evaporated or used for washing 20 out further quantities of chloride of lead. In performing this cycle of operations, I have now arrived at the point at which I began. Another similar quantity of gypsum in a finely divided state, as herein-before described, is again to be added in mixture with the chloride of lead in the decomposing vessel, with the required quantity of water at the temperature of 150° Fahren- 25 heit, and by these means an indefinite quantity of sulphuric acid may be produced by the agency of a comparatively small quantity of lead.

SPECIFICATION in pursuance of the conditions of the Letters Patent, filed by the said James Shanks in the Great Seal Patent Office on the 7th April 1855. 30

TO ALL TO WHOM THESE PRESENTS SHALL COME, I, JAMES SHANKS, of Saint Helens, in the County of Lancaster, Manufacturing Chemist, send greeting.

WHEREAS Her most Excellent Majesty Queen Victoria, by Her Letters Patent, bearing date the Ninth day of October, in the year of our Lord 35

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One thousand eight hundred and fifty-four, in the eighteenth year of Her reign, did, for Herself, Her heirs and successors, give and grant unto me, the said James Shanks, Her special license that I, the said James Shanks, my executors, administrators, and assigns, or such others as I, the
5 said James Shanks, my executors, administrators, and assigns, should at any time agree with, and no others, from time to time and at all times thereafter during the term therein expressed, should and lawfully might make, use, exercise, and vend, within the United Kingdom of Great Britain and Ireland, the Channel Islands, and Isle of Man, an Invention for
10 "**AN IMPROVED MODE OF MANUFACTURING SULPHURIC ACID,**" being a communication from Robert Von Seckendorff, of Gera, in the State of Reuss, in Germany, upon the condition (amongst others) that I, the said James Shanks, by an instrument in writing under my hand and seal, should particularly describe and ascertain the nature of the said Invention, and in what manner
15 the same was to be performed, and cause the same to be filed in the Great Seal Patent Office within six calendar months next and immediately after the date of the said Letters Patent.

NOW KNOW YE, that I, the said James Shanks, do hereby declare the nature of the said Invention, and in what manner the same is to be
20 performed, to be particularly described and ascertained in and by the following statement (that is to say):—

This Invention for the manufacture of sulphuric acid consists in certain means used for the separation or extraction of sulphuric acid contained in gypsum or sulphate of lime, either in a native or artificial state.

25 It is necessary, in the first place, to provide a large vessel or receptacle (the form I prefer to be of great horizontal area, but of small depth), and which I call a decomposing vessel or vat, made of bricks, stone, lead, or any other suitable material, and of sufficient size to produce a large quantity of sulphuric acid at one operation; I then take eighty-six parts by weight, or one equivalent,
30 of native gypsum, or sixty-eight parts by weight of anhydrous sulphate of lime, in a finely pulverized state, and place it in the decomposing vessel, and add to it one hundred and forty parts by weight of chloride of lead; I then add a large quantity of water, at a temperature of about one hundred and thirty degrees Fahrenheit's thermometer; or the water may be added at the
35 atmospheric temperature, and brought up afterwards by the application of steam or external heat. The whole is then to be thoroughly mixed and well stirred. An immediate change takes place; sulphate of lead speedily subsides, and chloride of calcium is held in solution in the supernatant liquid. In order to save trouble and expedite the process, it is desirable that the solution

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of chloride of calcium should indicate only four degrees specific gravity on Twaddle's hydrometer.

It is also desirable that no loss of lead should take place in this process; therefore the solution of chloride of calcium now in the decomposing vessel should be examined as to whether it contains any traces of lead in solution. 5 This may be done either while it remains in the decomposing vessel, or by transferring it by means of a syphon to another vessel large enough to contain the whole. A few drops of this solution should then be taken in a glass tube, and any of the usual re-agents for ascertaining the presence of salts of lead, such as a solution of sulphuretted hydrogen, or any of the soluble sulphurets, 10 may be dropped into the tube containing the solution of chloride of calcium. A black or dark brown precipitate will appear if any lead be present, in which case a small quantity of hydrate of lime or any other neutralizing agent may be added to the solution of chloride of calcium till no blackness is to be observed. When this point is attained, and the oxide of lead thus separated 15 from the solution is precipitated, the solution of chloride of calcium may be run away, or disposed of in any other way which may be thought proper, while the precipitated compound of lead may be added from time to time to the salts of lead in the first-named decomposing vessel.

The heavy white precipitate which now remains in the decomposing vessel 20 is sulphate of lead. This substance is now to be treated with hydrochloric acid. And in order to get a full decomposition of the sulphate of lead, I find it desirable to use rather more than its equivalent of hydrochloric acid, and at a specific gravity not less than twenty degrees to thirty degrees on Twaddle's 25 hydrometer.

By adding the hydrochloric acid to the sulphate of lead, and mixing well together, while the whole ingredients are raised to a temperature of one hundred and forty degrees Fahrenheit's thermometer, or thereabouts, by the application of steam or external heat, an immediate change takes place, and chloride of lead is formed, which falls to the bottom of the vessel, while 30 sulphuric acid is set free as a liquid.

The sulphuric acid being thus liberated, is now to be withdrawn from the decomposing vessel, and evaporated to the strength required for manufacturing or other purposes. A further quantity of water may be added to the decomposing vessel for washing out more of the sulphuric acid from the chloride of 35 lead, and which further quantity may be evaporated or used for elutriating further quantities of chloride of lead. In performing this cycle of operations, I have now arrived at the point at which I began. Another similar quantity of gypsum in a finely pulverized state, as herein-before described, is again to

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be added in mixture with the chloride of lead in the decomposing vessel, with the required quantity of water, at the temperature and under the conditions already mentioned. By these means an indefinite quantity of sulphuric acid may be produced by the agency of a comparatively small quantity of lead.

: 5 Having thus described the nature of the Invention of "An Improved Mode of Manufacturing Sulphuric Acid," as communicated by my foreign correspondent, and explained the manner of carrying the same into effect, I would have it understood that I do not confine myself to the exact details either of proportions of materials used, or description of apparatus employed, in these
10 processes; but what I do claim is,—

First, the decomposition of sulphate of lime, whether native or artificial, by means of chloride of lead, in the manner herein-before described, for the purpose of obtaining sulphate of lead.

And, secondly, I claim the decomposition of sulphate of lead, so obtained by
15 the use of hydrochloric acid, for the purpose of obtaining free sulphuric acid, also herein-before described.

In witness whereof, I, the said James Shanks, have hereunto set my hand and seal, the Sixth day of April, in the year of our Lord One thousand eight hundred and fifty-five.

20 JAMES SHANKS. (L.S.)

CLAUDIUS BUCHANAN, Witness,
of Liverpool,
Ironfounder.

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