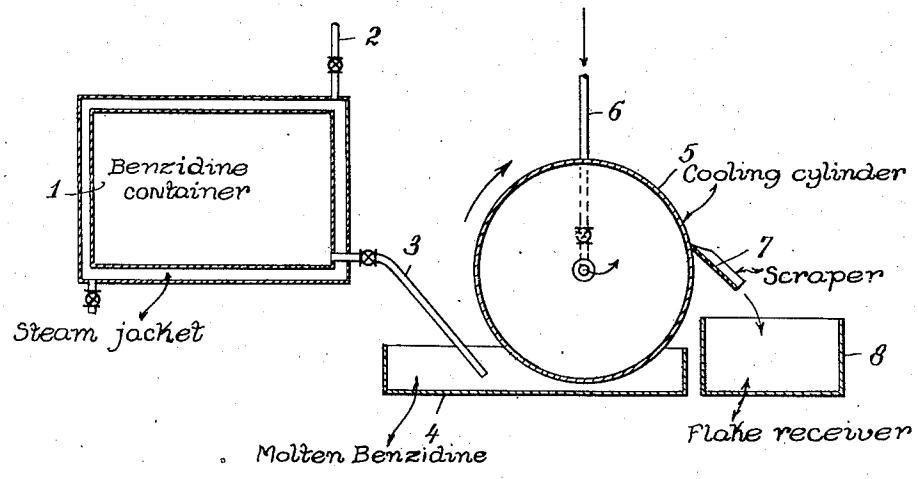
July 6, 1926. 1,591,688

W. L. SPALDING

BENZIDINE FLAKES

Filed June 18, 1923



INVENTOR

William L. Spalding

BY

Pennie, Davis, Marvin & Edmonds

ATTORNEYS

Patented July 6, 1926. 1,591,688

UNITED STATES PATENT OFFICE.

WILLIAM L. SPALDING, OF BUFFALO, NEW YORK, ASSIGNOR TO NATIONAL ANILINE & CHEMICAL COMPANY, INC., OF NEW YORK, N. Y., A CORPORATION OF NEW YORK.

BENZIDINE FLAKES.

Application filed June 18, 1923. Serial No. 646,043.

This invention relates to the production of benzidine in the form of small particles, such as chips or flakes.

In the well-known process of purifying benzidine by distillation either at ordinary or under reduced pressure, the purified benzidine composing the distillate condenses as a liquid which upon being cooled to a temperature at or below its solidifying point hardens or sets to a brittle mass or cake. In commercial practice, the hot benzidine distillate, before solidification, is run into shallow pans or containers where it is allowed to solidify. After solidifying and cooling, the hardened mass is then broken up into lumps which are afterwards pulverized or ground to a powder for storage and subsequent use. This procedure involves considerable inconvenience and difficulty in removing the hardened cake from the pans and also entails considerable expense in that the labor costs and apparatus maintenance charges are relatively high. Further, the operation is a dirty and disagreeable one, and the dust produced more or less attacks the respiratory organs and causes considerable discomfort, if not sickness.

I have found that either purified or crude benzidine can be produced in the form of chips or flakes, in which form it is more suitable for shipping, storing and handling and avoids the losses, inconvenience and difficulties heretofore experienced with this material. It can be produced as chips or flakes at a considerable saving in labor costs and apparatus maintenance charges, and comparatively little dust accompanies its production and handling. The chips or flakes may be of any convenient thickness but I have found that thicknesses ranging from one-sixteenth to one-forty eighth of an inch more or less, are to be desired, preferably about one-thirty second of an inch, since they are especially suitable for use as an intermediate in the manufacture of dyestuffs, or for other uses.

I have found that chips or flakes of benzidine can be produced in a substantially continuous manner from molten benzidine by rotating an interiorly cooled cylindrical drum so that it dips into the molten benzidine to a sufficient extent to form a layer or coating thereon, and by scraping the chilled layer from the rotating drum. While I do not limit myself to this specific procedure and apparatus, yet I have found it a particularly advantageous procedure and apparatus for the manufacture of benzidine chips or flakes when the various conditions of operation, such as temperature of the molten benzidine, temperature of the cooling liquid in the drum, and speed of rotation of the drum, are properly correlated and coordinated.

In the accompanying drawings I have illustrated somewhat diagrammatically an arrangement of apparatus adapted for carrying out the process of the invention and for producing a novel product of the invention.

In said device, as illustrated in the drawing, the benzidine is placed in the steam-jacketed receptacle 1, the jacket of which is supplied by steam by means of pipe 2, the temperature of the steam being such as to furnish sufficient heat to melt and keep the benzidine in the liquid condition, e. g., at a temperature of about 130°–135° C. The liquid benzidine is allowed to flow through pipe 3 into pan 4, in which pan 4 is a smooth cylinder 5 which is revolving in the direction indicated by the arrow. The cylinder 5 may be cooled in any convenient manner, for example by means of cold water furnished by pipe 6 to the interior of the cylinder, the temperature of the water being maintained, for example, at about 30°–35° C. The cylinder 5 which dips into the liquid benzidine in pan 4 to a depth of about one inch, as it revolves picks up on its surface a thin film of liquid benzidine. As the cylinder slowly revolves, for example, at a rate of about twelve revolutions per minute for a cylinder of about four feet in diameter, in the direction of the arrow, the film becomes cool and hardens and is scraped off by means of the scraper 7 and is broken up into small particles which fall and are collected in container 8. It will be noted that the amount of benzidine flowing through pipe 3 into pan 4 can be regulated so as to equal the amount collected in the form of flakes thus making the process substantially a continuous one.

In carrying out the invention by means of this device, the liquid distillate obtained by the distillation of benzidine may be directly charged into or received by receptacle

1. If crude benzidine is to be flaked, it may be added to the receptacle 1 either in the solid or fluid condition.

While it is well known that material other than benzidine has been heretofore produced in the form of chips or flakes, so far as I am aware, benzidine has never been heretofore produced in such form. It will be readily seen from the above description that the production of benzidine in the form of chips or flakes possesses advantages superior to its production in the form of hard masses, cakes, or lumps, and reducing these hard masses or lumps to a powdered condition by grinding, crushing or pulverizing. The cost of preparing the benzidine in the form of flakes is less than when it is run molten into containers, allowed to harden and subsequently broken up. And particularly if it is also subsequently ground or reduced to a powdered state. Further the production in the form of flakes minimizes and practically overcomes the objection caused by its dust.

Throughout the specifications and claims it will be understood that the term "flakes" includes chips, scales or thin pieces of material.

I claim:

1. As a product, benzidine in the form of flakes.

2. As a product, benzidine in the form of flakes having a thickness not to exceed one-sixteenth of an inch.

3. As a product, benzidine in the form of flakes having an average thickness of about one-thirty second of an inch.

In testimony whereof I affix my signature.

WILLIAM L. SPALDING.