

March 25, 1952

J. L. KALLOK

2,590,544

PROCESS FOR THE CRYSTALLIZATION OF DDT

Filed April 19, 1949

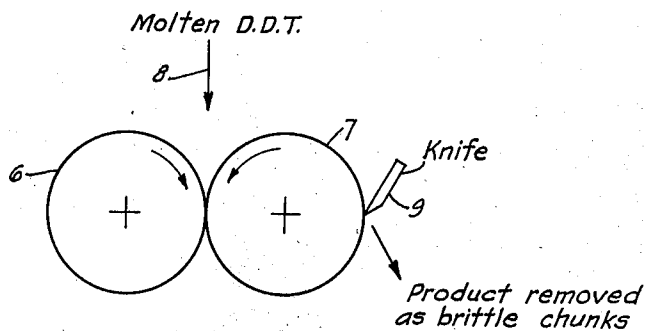


Fig. 1

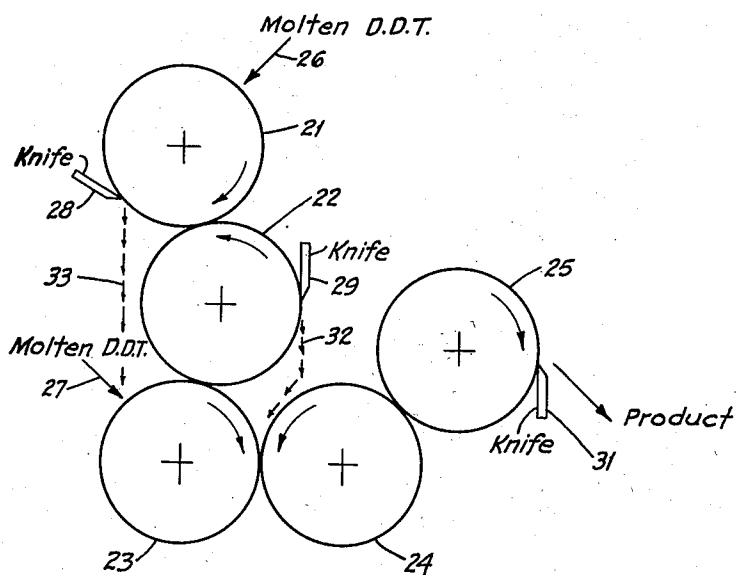


Fig. 2

INVENTOR.
John L. Kallok

BY

E. C. Hoff & Slick
ATTORNEYS

UNITED STATES PATENT OFFICE

2,590,544

PROCESS FOR THE CRYSTALLIZATION
OF DDT

John L. Kallok, Torrance, Calif., assignor to
Montrose Chemical Corporation of California,
Los Angeles, Calif., a corporation of Delaware

Application April 19, 1949, Serial No. 88,489

3 Claims. (Cl. 18—57)

1

This invention relates to an improvement in the manufacture of dichlorodiphenyl trichloroethane. This material is widely used as an insecticide, its so-called para-para isomer (2,2 bis (p-chlorophenyl) 1,1,1-trichloroethane) having unique properties in this respect. The material is produced as a mixture of several isomers and the most commonly employed form is known as "technical DDT." This is a solid material at ordinary temperatures, but melts readily at below 100° C., usually at about 90° C. The effective ingredient in the DDT, the so-called para-para isomer, melts at about 106° C.

For convenience and simplicity in expression, dichlorodiphenyl trichloroethane will be referred to hereinafter as DDT.

To employ DDT as an insecticide it is usual to place it in solution or else to sub-divide it to finely divided form and disperse it in a dust-like carrier. Since DDT exhibits many characteristics which can be compared to those of sugar or sucrose in that if a DDT solution is cooled it forms a liquid mass comparable to molasses and which solidifies only after passage of such an extremely long period of time as to be uneconomical in commercial practice; even when solidified the material may be gummy and sticky and is difficult to grind. It has been suggested that DDT solutions should be cooled to a temperature not above about 60° C., without solidification, to produce a supercooled liquid, the latter being set aside and permitted to crystallize; the rate of cooling is sufficient to avoid crystallization during the cooling operation, it being essential to produce a supercooled liquid. This practice, I have found, is not feasible in commercial operation because of the tendency to produce a molasses-like material in which the DDT forms a solid so slowly as to be uneconomical in commercial practice, the solidification requiring a matter of weeks. The other commercial practices used at present are such as to make the cooling operation extremely difficult and expensive and to give a final product containing gummy constituents which make the solid difficult and expensive to grind, and which prevent the material from being free-flowing and non-caking.

I have found that if one cools the DDT solution under such conditions that a minimum of supercooling is effected and so that many fine crystal nuclei are formed in the mass, then a solid DDT is produced which is hard, and is free of gummy constituents and which can be readily reduced to a finely divided form by grinding or

2

other suitable comminution methods. More particularly, I have found that DDT should be cooled in film form and under such conditions that it is worked with great vigor during the cooling operation. If this is done, then a hard product results, as compared to one which has the molasses-like properties which characterizes the product produced with super-cooling of liquid DDT.

The invention is conveniently practiced by feeding the liquified DDT onto the first of a plurality of rollers, the several rollers being set so closely together that a minimum thickness film is carried upon each roll; the respective rolls are cooled to a temperature approximating a normal atmospheric temperature, that is, preferably below 30° C., and preferably between 5° and 30° C. and usually about 10° C. Crystallization can be carried on continuously, the DDT being fed onto one roll and cut off the final roll of the series. The rolls are preferably differentially driven so that the DDT film on any given roll is torn apart and worked vigorously by the roll which rotates substantially in contact with it. Differential driving is not necessary if the rolls are so set with respect to one another as to effect a squeezing together and a pulling apart of the material. In this manner, millions of fine crystal nuclei are formed fractially instantaneously in the freshly fed and filmed DDT liquid. The vigorous working carried these fine crystal nuclei to all parts of the liquid film while it undergoes cooling so that crystallization takes place with great rapidity and a solid, hard mass results.

The invention will be further illustrated by the following examples, but is not limited to these in the drawing accompanying and forming a part hereof, Figures 1 and 2 are diagrammatic representations of the apparatus utilized respectively in Examples 1 and 2, as follows:

Example 1.—Two 8" rolls, indicated at 6 and 7 in Figure 1 of the drawing, were driven respectively at 20 R. P. M. and 25 R. P. M. DDT was fed as at 8 onto the first roll 6 to cover the roll completely and to provide a slight excess to work in and fill the bite between the rolls. After a few minutes' operation, the DDT liquid coating on the rolls began to crystallize and as soon as crystallization was complete, the solid DDT was removed continuously with a doctor knife 9 as brittle chunks from the final roll 7.

Example 2.—To illustrate further the practice of the present invention, a five-roll device was set up as in Figure 2, including rolls 21, 22, 23, 24 and 25. Molten DDT at 100° to 120° C. was fed

3

onto roll 21 from feeder 26 and onto roll 23 from feeder 27. A doctor knife 28 was so positioned with respect to roll 21 that the material cut therefrom dropped onto roll 23. Similarly, a doctor knife 29 was so positioned that material cut from roll 22 dropped between rolls 23 and 24. The material from roll 21 which was still liquid but cool, was cut off by the knife 28 and dropped onto roll 23, as indicated at 33. The material transferred to roll 22 was also cut off by doctor knife 29 and fell into the bite between rolls 23 and 24, as indicated at 32. The material from roll 22 was partially crystallized and, by the time it had transferred from between roll 23 and 24 and between rolls 24 and 25, crystallization was complete. The DDT was cut off the final roll 25 by knife 31 at the rate of 1,000 pounds per hour and was dry and brittle.

The condition of the DDT on each roll, its temperature and speed of rotation are given in the following table:

Roll	Condition	Temperature	Speed-R. P. M.
21	Scraped clean	°C. 10	23
22	Scraped, leaving a thin film of D. D. T.	30-40	45
23	1/8" thick film	10	57
24	do.	30-40	92
25	Scraped, leaving a thin film of D. D. T.	10	155

To produce a relatively soft, flaky DDT which can be ground easily, the molten DDT is agitated violently in a water cooled vessel to about 75° C. During the cooling, DDT crystals are formed and are broken up by the violent agitation so that a partially crystallized, mushy mixture is formed. This mushy mixture is then fed onto the multiple chilled rolls, as in Example 2. The product is softer than that produced when the molten DDT is fed directly to the rolls, but it is still flaky and is particularly suited for grinding in certain types of mills.

I claim:

1. The process of solidifying DDT to a hard crystalline form which consists essentially in continuously cooling molten DDT in the form of a film from a temperature above its setting point

4

to a final temperature between 5° C. and 30° C., to effect, initially, formation of a multitude of crystal nuclei in the liquid and, finally, formation of a firm solid and, simultaneously and continuously during such continuous cooling, working the DDT in the form of a film until the DDT attains said final temperature and is of a hard crystalline form.

2. The process of solidifying DDT to a hard crystalline form which consists essentially in continuously cooling molten DDT in the form of a film from a temperature above its setting point to a final temperature of about 10° C. to effect, initially, formation of a multitude of crystal nuclei in the liquid and, finally, formation of a firm solid and, simultaneously and continuously during such continuous cooling, working the DDT in the form of a film until the DDT attains said final temperature and is of a hard crystalline form.

3. The process of solidifying DDT to a hard crystalline form which consists essentially in first cooling a mass of molten DDT from a temperature above its setting point to about 75° C. while violently agitating the mass to produce a mushy mixture, and then cooling the mushy mixture continuously in the form of a film from said temperature of about 75° C. to 30° C. and during such continuous cooling working the DDT continuously as a film until the film is a solid and the DDT is at a final temperature between 5° C. and 30° C.

JOHN L. KALLOK.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,883,112	Tiers	Oct. 18, 1932
2,200,774	Halliday	May 14, 1940
2,453,076	Little	Nov. 2, 1948
2,491,160	Bruce	Dec. 13, 1949
2,555,309	Beam	June 5, 1951

FOREIGN PATENTS

Number	Country	Date
455,683	Germany	Feb. 6, 1928