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CRYSTALLIZATION OF DDT

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This invention relates to improvements in the manufacture of technical or commercial dichlorodiphenyl trichloroethane, the insecticidal material designated as DDT.

Technical DDT is a solid material at ordinary temperatures, but readily melts at below 100° C., usually at about 90° C. Pure DDT melts at about 106° C. or somewhat higher.

In the usual process of manufacture, it is first obtained in molten form, the molten material is allowed to cool and solidify, and the solid product so obtained is ground for use as a powder. When DDT is solidified by this procedure, the solid material obtained is somewhat gummy due to incomplete crystallization. It is, therefore, common practice to age it for substantial periods of time, for example 16 to 40 hours, before grinding.

In some cases, the molten material is solidified on a cooled, rotating drum and produced as flakes of the solid product. Where the product is solidified on a flaking drum, the drum must be rotated very slowly, thus requiring a large capital investment in the solidifying equipment for a given production.

With the foregoing procedures, there is a definite tendency for the production of a material with segregated isomers or crystals, which is undesirable.

The present invention provides improvements in the conversion of the molten DDT to the solid form which gives a product which is free from gummy characteristics, which has no segregation of isomers or crystals, which is more easily ground than the previous products, is more brittle and is somewhat different in appearance, having a chalky white appearance. It may be ground without aging, and the storage bins required for such aging are thus unnecessary. Furthermore, the invention permits the production of the solid material at a substantially higher rate from given equipment than has heretofore been possible.

In accordance with the present invention, the molten DDT is supercooled below its normal crystallization temperature and after such supercooling, it is permitted to solidify without further cooling. Advantageously, the supercooling is such that the temperature of the material, before crystallization begins, is between 45° and 60° C. but it may be below 45°, or somewhat above 60° C. It is important, in order that supercooling may be obtained, to cool the material quickly and with as little agitation as possible and also to avoid or minimize contact of the material with solidified DDT.

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The procedure may be carried out in various ways, as by the use of film type shell and tube heat exchangers, heat exchangers with a rotating blade or blades to keep the film of DDT in motion and thereby effecting rapid heat transfer, by the use of rotating drums internally cooled which dip into a body of the molten DDT and have the film scraped off after cooling but prior to solidification, or in other forms of apparatus.

Advantageously, a conventional flaking machine, operated with rapid rotation of the drum so that supercooled liquid rather than a solidified product is produced, is used. The important factor in carrying out the process is the cooling at a rate sufficient to produce a supercooled liquid, such cooling being from the temperature of the molten DDT, say around 100° C., to a temperature below its normal solidification point, advantageously from about 50 to about 60° C., at a rate sufficient to avoid crystallization during the cooling operation. The supercooled liquid then is allowed to stand with resultant solidification to form the easily ground product of the invention. The invention will be illustrated by the following examples but it is not limited thereto.

Example I.—100 grams of DDT (technical) were melted and heated to 100° C. and poured into a prewarmed porcelain dish. It was then allowed to cool to 55° C. without agitation, by exposure to room atmosphere. At this time the liquid was stirred vigorously, whereupon it crystallized into a hard, brittle, uniform mass, the solidification being accomplished by a rise in temperature to about 64° C.

Example II.—DDT at 100° C. was fed to the inside surface of a vertical jacketed stainless steel pipe, 3/4" inside diameter and 4' long, used as a film type cooler. The rate of feed was 147 grams per minute. Cooling water was circulated through the jacket. The DDT was discharged at 53 to 59° C., and was then placed in a container and allowed to cool. It crystallized without stirring to a hard, brittle, uniform mass free from the gummy characteristics of DDT as ordinarily crystallized, and of a nature such as to permit ready grinding.

Example III.—DDT at a temperature of 100° C. was placed in a pan into which dipped the bottom portion of the circumference of a rotating cylindrical drum internally cooled with water. The DDT liquid film picked up from the pan was scraped off by a knife set 3/4 of the way around the drum from the point where the drum surface left the liquid in the pan. The drum was 8.25" long by 6.25" in diameter and was rotated

at the rate of 8 revolutions per minute. The product was discharged at the rate of .84 lb. per minute at 50 to 52° C. and delivered to a container where it cooled to room temperature and crystallized into a hard, brittle, uniform mass.

The apparatus in accordance with Example III is, of course, of the type of conventional flaking machines. The important distinction in operation as compared with flaking machines heretofore used for the solidification of DDT is the rapid rate of rotation of the drum with the product of a supercooled liquid, as distinguished from a flaked solid.

Example IV.—Molten DDT at 100 to 102° C. was cooled as in Example III with the supercooled liquid discharged on to a belt 8" wide moving at a linear speed of 3 ft./min. The supercooled liquid solidified on the belt and was discharged at the idler pulley. The cooling drum rotated at 30 R. P. M.; the liquid was discharged from the drum at 55° to 58° C. In 4 minutes, 16 lbs. of solid, brittle DDT was obtained.

We claim:

1. The process of solidifying DDT (dichlorodiphenyl trichloroethane) in hard crystalline form which includes cooling molten DDT to a temperature not above about 60° C. without solidification, whereby a supercooled liquid is obtained, and permitting the supercooled liquid to crystallize.

2. The process as in claim 1 where the supercooling is carried out by transferring heat from a film of the molten DDT to a cooled surface.

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REFERENCES CITED

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

Callaham, "Chemical and Metallurgical Engineering," vol. 5, page 114 (1944). (Copy in Patent Office Scientific Library.)