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STABILIZATION OF PENTACHLOROPHENOL

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ABSTRACT OF THE DISCLOSURE

Hydroxybenzaldehydes such as salicylaldehyde, when present in a stabilizing amount, are useful in inhibiting degradation of pentachlorophenol.

This application is a continuation of copending application Ser. No. 777,167, filed Nov. 19, 1968 and now abandoned.

This invention relates to the stabilization of pentachlorophenol. More particularly, this invention relates to pentachlorophenol containing a stabilizing amount of a hydroxybenzaldehyde such as o-hydroxybenzaldehyde (salicylaldehyde) wherein degradation of freshly chlorinated "penta" is substantially arrested.

Pentachlorophenol is commonly prepared by direct chlorination of phenol or low molecular weight chlorophenols in the presence of an aluminum or aluminum chloride catalyst without subsequent refining. Following chlorination, the molten "penta" is transferred to temporary storage pending flaking or prilling operations which are necessary to transform the product to a state convenient for end use.

It is desirable to produce pentachlorophenol of high quality. It is particularly desirable that the product assay approximately 90% or more pentachlorophenol while, at the same time, contain minimal amounts of high boiling materials. By following the teaching of the prior art, it has been possible to optimize process conditions in such a manner that pentachlorophenol having the aforementioned quality can be prepared. Unfortunately, high "penta" assay attained at the chlorination end point can rarely, if ever, be retained if the product is held molten at approximately 200° C. for even short periods of time. As previously indicated, such short term storage is frequently necessary prior to finishing steps. Degradation during the period from the end of chlorination step to commencement of finishing is reflected in a loss of "penta" assay that may be as great as six percentage points.

This loss is accompanied by a concomitant increase in quantity of high boiling compounds which, aside from the loss of pentachlorophenol, are detrimental to product quality in that they frequently contain significant amounts of alkali insoluble materials and color bodies.

Accordingly, it is an object of this invention to provide stabilized pentachlorophenol. More particularly, it is an object of this invention to provide a method of stabilizing freshly chlorinated pentachlorophenol wherein the loss in "penta" assay is minimized and the formation of additional high boiling compounds is significantly reduced during storage temperatures above the crystallizing point of the pentachlorophenol.

According to the present invention, contacting pentachlorophenol with a stabilizing amount of a hydroxybenzaldehyde, preferably salicylaldehyde, following the chlorination step is highly effective in preventing degradation. While the stabilizer may be added at various times such as during the final period of chlorination, after chlorination prior to storage, during storage, or a combination of these, the most effective results are obtained by adding all of the stabilizer immediately after chlorination and agitating the mixture moderately. The character and duration of agitation are not critical.

The amount of hydroxybenzaldehyde employed can be varied; however, for efficient treatment amounts vary from about 0.15 to about 0.5% by weight based on the weight of the pentachlorophenol will provide excellent results. A significant improvement in stabilization can be effected, however, by the use of small amounts of at least 0.01%. Much larger amounts as high as 1.0% can be used but are generally not needed, and the use of such amounts is therefore wasteful.

The following examples illustrate the advantageous and unexpected results which are achieved by the use of the treatment agents of this invention, but it is not intended that this invention be limited by or to the examples.

EXAMPLE 1

A penachlorophenol product prepared by the chlorination of phenol having a chlorination end point assay of 90.0% is treated with 0.025% by weight salicylaldehyde based on the weight of the pentachlorophenol. The treated sample and an untreated sample of the pentachlorophenol are held at a temperature of 190–200° C. for a period of two hours. Analysis of the treated and untreated samples by gas-liquid chromatography indicates a residual assay of the pentachlorophenol as follows:

treated sample: 88.0
untreated sample: 86.8

EXAMPLES 2–9

Following the procedures of Example 1, a series of pentachlorophenol products are treated with a hydroxybenzaldehyde as summarized below in Table I.

TABLE I

Example number	Initial "penta" assay	Percent treating agent[1]	Hours at 190–200° C.	Final assay
2	92.9	None	6	86.6
	92.9	0.15	6	89.8
3	92.4	None	4.5	87.8
	92.4	0.20	4.5	90.7
4	94.0	None	4.5	89.6
	94.0	0.20	4.5	92.1
5	92.6	None	4.5	86.4
	92.6	0.20	4.5	90.0
6	94.1	None	4.5	88.6
	94.1	0.20	4.5	91.7
7	93.8	None	4.5	89.3
	93.8	0.20	4.5	91.6
8	87.6	None	6	84.5
	87.6	0.21	6	87.6
9	90.5	None	4.5	87.4
	90.5	0.25	4.5	89.9

[1] Salicylaldehyde.

Similar results are obtained upon replacing the o-hydroxybenzaldehyde in the foregoing examples with m or p hydroxybenzaldehyde.

While this invention has been described with respect to certain embodiments, it is not so limited, and it is to be understood that variations and modifications thereof may be made which are obvious to those skilled in the art

without departing from the spirit or scope of this invention.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A stabilized pentachlorophenol composition consisting essentially of molten pentachlorophenol containing a stabilizing amount of hydroxybenzaldehyde.

2. The composition of claim **1** wherein a hydroxybenzaldehyde is present in the amount from 0.01 percent to 1.0 percent by weight of pentachlorophenol.

3. The composition of claim **1** wherein hydroxybenzaldehyde is present in the amount from 0.15 percent to 0.50 percent by weight of pentachlorophenol.

4. The composition of claim **1** wherein the hydroxybenzaldehyde is o-hydroxybenzaldehyde.

References Cited

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