Field of the Disclosure

The present disclosure relates to a method for the preparation of Triclopyr acid.

Background

Triclopyr acid is a valuable compound and an intermediate used for extensive applications, amongst which use as a herbicide is a significant one. Triclopyr acid is a systemic and foliar herbicide belonging to the pyridine group that is effective for controlling broadleaf weeds without affecting the grasses and conifers, especially the woody plants that are found in grasslands, uncultivated land, industrial areas, plantation crops and rice fields. Triclopyr acts by getting rapidly absorbed through the roots and foliage, translocating throughout the plant, accumulating in the meristematic tissues and inducing auxin type responses.

The conventional processes for the preparation of Triclopyr acid include hydrolyzing a triclopyr ester with an aqueous alkali to obtain a salt of the acid, followed by acidifying the salt to obtain the product. However, the prior art processes are associated with certain drawbacks such as excess of effluent generation, excess hydrolysis leading to product degradation and loss in the yield and time consuming, multiple step synthetic route.

The inventors of the present disclosure, therefore, envisage a process for the preparation of triclopyr acid that precludes most of the drawbacks associated with the prior art processes.

Objects

Some of the objects of the present disclosure, which at least in one embodiment is adapted to provide, are described herein below:

It is an object of the present disclosure to provide a process for the preparation of triclopyr acid.

It is another object of the present disclosure to provide a process for the preparation of triclopyr acid, which is rapid, economic and environment friendly.

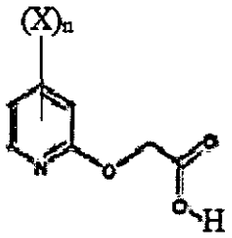
It is still another object of the present disclosure to provide a high yielding process for the preparation of triclopyr acid.

It is yet another object of the present disclosure to provide triclopyr acid with high purity.

Other objects and advantages of the present disclosure will be more apparent from the following description which is not intended to limit the scope of the present disclosure.

Detailed Description

The present disclosure provides a process for the preparation of a compound represented by Formula 1 that includes but is not limited to, initially, dissolving at least one acid in water at a temperature ranging between 30 °C and 90 °C to provide an acid solution. The concentration of the solution typically ranges from 40-90 %.



Formula 1

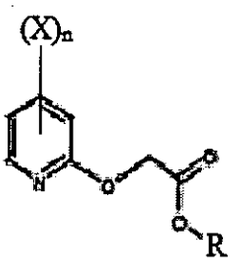
The structure of the compound of Formula 1 is presented hereinabove, wherein:

- X is independently selected from the group that includes but is not limited to Cl, Br and I; and

- n is an integer in the range of 1-4. In one embodiment, the compound represented by Formula 1 is triclopyr acid.

The acid used in the process of the present disclosure is selected from the group that includes but is not limited to hydrochloric acid, sulfuric acid, acetic acid, sulfonic acid, methane sulfonic acid, benzene sulfonic acid, p-chloro benzene sulfonic acid, p-toluene sulfonic acid and sulfuric acid. The acid used in accordance with the present disclosure can be recycled after the completion of the reaction.

Further, an ester represented by Formula 2 is hydrolyzed by the afore-mentioned acid solution at a temperature ranging between 70 °C and 120 °C to obtain a slurry comprising the compound of Formula 1.



Formula 2

wherein:

- R is selected from the group that includes but is not limited to C_1-C_5 alkyl groups;
- X is independently selected from the group that includes but is not limited to Cl, Br and I; and
- n is an integer in the range of 1-4.

The compound of Formula 1 present in the afore-mentioned slurry is isolated by extracting with a solvent or by the process of filtration. The solvent may be an aromatic or an aliphatic solvent and is selected from the group that includes but is not limited to xylene, benzene, ethylbenzene, hexane, cyclohexane and toluene. The compound of Formula 1 is isolated by either extraction or filtration has the same purity.

The compound of Formula 1 synthesized using the process of the present disclosure may be used as such or may be converted to derivatives for different applications. Further, the process of the present disclosure shows 99.0 % recovery and no degradation. Even further, the process yields compound of Formula 1 in just one step, as opposed to the prior art processes; thereby making the overall process time and cost efficient.

The present disclosure is further described in the light of the following non-limiting examples which are set forth for illustration purpose only and not to be construed for limiting the scope of the disclosure.

Example 1

1.0 g mole of Trichlopyr methyl ester solid was added in 1.0 liter of 7.0-7.5 N HCl at 30.0 °C and 80.0 °C. The reaction mixture was then heated under stirring to a temperature ranging between 100 °C and 105 °C for 5.0-8.0 hrs. till the unreacted ester content became less than 0.5 %. The progress of the reaction was monitored by high performance liquid chromatography (HPLC). Further, the resultant mass was cooled to 28-30 °C and the Trichlopyr acid was filtered out. The filtered cake was washed with water to make it free of HCl. The isolated Trichlopyr acid was of 0.98-0.99 moles and the purity was found to be 98.5-99.5 %.

The embodiments herein and the various features and advantageous details thereof are explained with reference to the non-limiting embodiments in the description. Descriptions of well-known components and processing techniques are omitted so as to not unnecessarily obscure the embodiments herein. The examples used herein are intended merely to facilitate an understanding of ways in which the embodiments herein may be practiced and to further enable those of skill in the art to practice the embodiments herein. Accordingly, the examples should not be construed as limiting the scope of the embodiments herein.

The foregoing description of the specific embodiments will so fully reveal the general nature of the embodiments herein that others can, by applying current knowledge, readily modify and/or adapt for various applications such specific embodiments without departing from the generic concept, and, therefore, such adaptations and modifications should and are intended to be comprehended within the meaning and range of equivalents of the disclosed embodiments. It is to be understood that the phraseology or terminology employed herein is for the purpose of description and not of limitation. Therefore, while the embodiments herein have been described in terms of preferred embodiments, those skilled in the art will recognize that the embodiments herein can be practiced with modification within the spirit and scope of the embodiments as described herein.

Any discussion of documents, acts, materials, devices, articles and the like that has been included in this specification is solely for the purpose of providing a context for the disclosure. It is not to be taken as an admission that any or all of these matters form a part of the prior art base or were common general knowledge in the field relevant to the disclosure as it existed anywhere before the priority date of this application.

While considerable emphasis has been placed herein on the particular features of this disclosure, it will be appreciated that various modifications can be made, and that many changes can be made in the preferred embodiments without departing from the principles of the disclosure. These and other modifications in the nature of the disclosure or the preferred embodiments will be apparent to those skilled in the art from the disclosure herein, whereby it is to be distinctly understood that the foregoing descriptive matter is to be interpreted merely as illustrative of the disclosure and not as a limitation.

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