

Impact of the Purification Process on the Niclosamide Quality in Commercial Production

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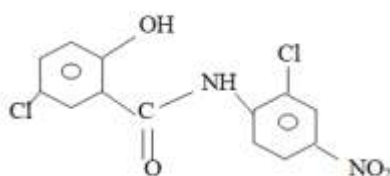
ABSTRACT-Niclosamide is an anthelmintic drug used to treat tapeworm infestations, including diphyllorhynchiasis, hymenolepiasis, and taeniasis in animals. Quality of the drug is the highest priority in commercial manufacturing. Though different processes parameters play important role the final quality/purity of the drug mainly depends on the purification process. Though there are number of different ways to purify the compound. Solvent based purification process is the found to be most effective and commercially viable as the solvent can be recovered and reused

Index Terms- OCPNA, PCL₃, Methanol, HPLC

I. INTRODUCTION

The current research guides a stepwise walkthrough of the solvent based purification method and its advantage over other purification methods. In the solvent based purification method methanol is used as the solvent to remove impurities that might be a leftover of unreacted OCPNA or 5-Chloro salicylic acid.

Formula	C ₁₃ H ₈ Cl ₂ N ₂ O ₄
Molecular weight	327.119 g/mol
CAS No	50-65-7



Niclosamide is commercially manufactured mainly by reacting two chemical compounds C₇H₅ClO₃ and C₆H₅ClN₂O₂.

Though different manufacturing methods yield various purities and yield the purification process of

finished compound has a profound impact on the quality & purity of the drug.

Definition

Purification of a compound usually refers to increasing its quality by eliminating impurities by various methods

Purification method

Various methods used for the purification of complex organic compounds are discussed below in this article.

- Sublimation
- Crystallization
- Distillation
- Fractional Distillation
- Vacuum Distillation
- Steam Distillation
- Differential Extraction
- Chromatography

In this research vacuum distillation is used to purify the compound.



Vacuum distillation setup used for the experiment

As gravity filtration is often a slow and tedious process, vacuum filtration allows this process

to be sped up. By connecting a vacuum pump to the filtration machinery, you can create an environment

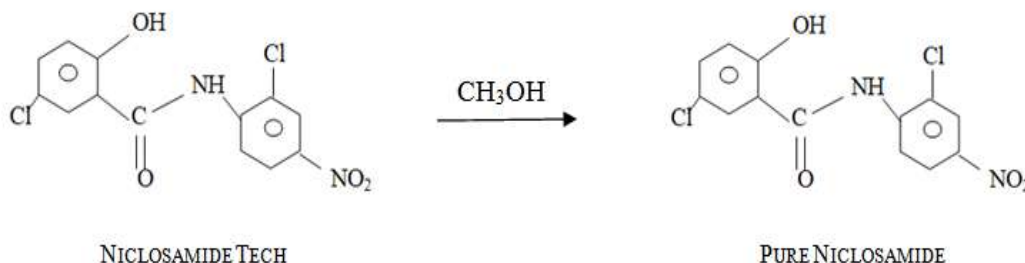
with a reduced pressure. Reduced pressure will cause the solution to be sucked through the filter rapidly.

II. COMPARATIVE ANALYSIS

S.NO	Name of Solvent	Chemical Formula	Commercial Viability	Purity Achieved
1	Methanol	CH ₃ OH	yes	In Range
2	Ethyl Acetate	C ₄ H ₈ O ₂	No	Not in range
3	Acetone	C ₃ H ₆ O	No	Not in range
4	Acetonitrile	CH ₃ CN	No	Not in range

III. PURIFICATION PROCESS

- Charge 50 gms of Niclosamide tech and 250 ml of methanol into RBF
- Heat the mixture to 60- 65 °C
- Maintain the temperature of 60 – 65 ° C for 1 hour
- Cool the RT to 30 – 35 ° C
- Stir the RT for 60 minutes at 30-35 °C
- Filter the compound through funnel & wash with methanol and hot water
- Dry at 50 – 55 ° C



IV. PURITY BY HPLC

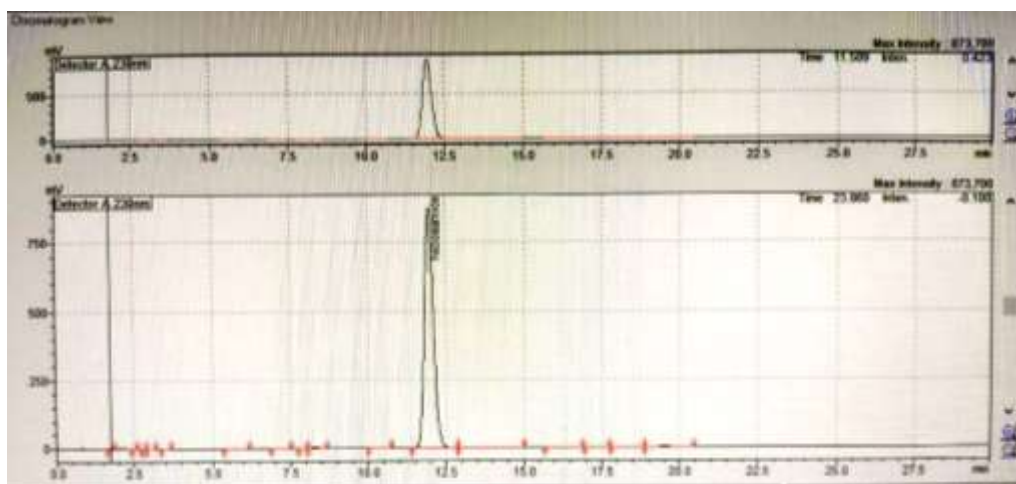
HPLC stands as an abbreviation for the High Performance Liquid Chromatography is an effective way to check the purity of the final compound

Test solution

Dissolve 50 mg of the substance to be examined in methanol, heating gently, cool and dilute to 50.0 ml with the same solvent.



HIGH PERFORMANCE LIQUID CHROMATOGRAPHY USED FOR THE EXPERIMENT



Chromatograph obtained from HPLC

Purity achieved - 99.56 %

V. CONCLUSION

Several methods of purification are being tried out using Ethanol, Acetone, Aceto nitrile and Methanol but the highest purity and commercial advantage lies in using Methanol as it is a polar solvent and is available at competitive prices.

Crucial points

- Methanol is suitable for purity
- Temperature is crucial (Reflux is required)
- Maintenance of reflux is crucial
- Wet case wash with hot water is important
- Drying at 50- 55 ° C is very important

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