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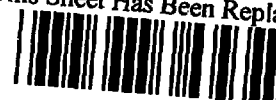
| REPLACED SHEETS                                                                                        | SG |
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**IMPROVED PROCESS FOR THE MANUFACTURE OF CYCLO-  
PROPYLNITRILE**

**ABSTRACT OF INVENTION**

An improved manufacturing procedure for the preparation and isolation of cyclopropylnitrile is provided. 4-Halobutyronitrile is reacted with an alkali metal base in the presence of an aprotic polar solvent, an inorganic salt and a catalytic amount of water at an elevated temperature. The reaction product cyclopropylnitrile is isolated by azeotropic distillation.

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32345-00

IMPROVED PROCESS FOR THE MANUFACTURE OF CYCLO-  
PROPYLNITRILE

Cyclopropyl nitrile is a vital raw material in the manufacture of herbicidal agents. It is particularly useful in the manufacture of herbicidally potent, yet environmentally benign 1-(o-cyclopropylcarbonyl)-phenylsulfamoyl urea derivatives. The preparation of cyclopropyl nitrile from 4-chlorobutyronitrile is described in U. S. 3,843,709. However, when this preparation is applied to a manufacturing scale, an unstirrable mass forms causing decreased reaction yield, broken or damaged equipment and problematic product isolation.

Therefore it is an object of this invention to provide an improved process for the manufacture of cyclopropyl nitrile which is suitable for large scale production with increased isolated product yield.

It is another object of this invention to provide an efficient and effective method of isolation of cyclopropyl nitrile in high purity and with essentially no water content.

It is a further object of this invention to provide a convenient and economic source of essential starting material in the production of herbicidal sulfamoyl urea derivatives.

Herbicidal agents and in particular 1-(o-cyclopropylcarbonyl)phenylsulfamoyl urea derivatives are prepared using cyclopropyl nitrile

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as the basic starting material. It has now been found that cyclopropyl nitrile can be prepared on a commercial manufacturing scale without the formation of an unstirrable mass and accompanying equipment damage or breakage and decreased product yield by reacting 4-halobutyronitrile with an alkali metal base in the presence of an aprotic polar solvent, an inorganic salt and a catalytic amount of water at a temperature of about 50°-100°C. The preparation is shown in flow diagram I wherein X is halogen, M is an alkali metal and R is hydrogen or C<sub>1</sub>-C<sub>6</sub>alkyl.

Flow Diagram I



Methods available in the art to prepare cyclopropyl nitrile, upon scale-up, give the formation of an unstirrable mass. The inability to stir or agitate a reaction mixture results in incomplete reaction, hazardous reaction conditions and damaged or broken equipment. Surprisingly, it has been found that the addition of at least 0.25 mole, preferably about 0.4 to 1.5 mole and more preferably about 0.5 to 1.0 mole of an inorganic salt eliminates the formation of an unstirrable mass thus preventing agitation problems, poor yields and a broken or damaged agitator. Advantageously, it has been found that the addition of a catalytic amount of water to the reaction mixture is critical to the initiation of the reaction. Predictable and reproducible reaction initiation prevents the potential hazard of large scale run-



away reaction rates and sudden uncontrollable exotherms.

In general, the reaction rate increases as the temperature increases, however it has now  
5 been found that under large scale conditions, reaction temperatures greater than about 110°C give mainly amide side-product formation and little or no cyclopropyl nitrile product. Elevated  
10 temperatures of about 50°- 100°C, preferably about 60°- 90°C are suitable in the inventive process.

Aprotic polar solvents suitable for use in the inventive process are sulfoxides, sulfones, carboxylic acid amides, pyrrolidones and the like. Sulfoxides and carboxylic acid amides are  
15 preferred, dimethylsulfoxide and dimethyl formamide are more preferred and dimethylsulfoxide is most preferred.

Alkali metal bases useful in the process of the invention are any alkali metal hydroxides or  
20 alkoxides or mixtures thereof. Preferable alkali metal bases are monovalent bases such as NaOR, KOR or LiOR, more preferred are the hydroxides of sodium or potassium and most preferred is NaOH. Stoichiometric amounts of the alkali metal base may  
25 be used in the process of the invention. In a preferred embodiment of the invention, the alkali metal base is added to the reaction mixture incrementally over a period of time.

Inorganic salts suitable for use in the  
30 present invention are metal halides, metal sulfates or metal carbonates, preferably metal halides such as sodium halide or potassium halide, more preferably sodium halide and most preferably sodium chloride.

35 The product cyclopropyl nitrile, in order to be suitable for use in manufacturing 1-(o-cyclopropyl-carbonyl)phenylsulfamoyl urea



derivatives, must be essentially free of water and minor components. It has now been found that highly pure and essentially anhydrous cyclopropyl nitrile product can be isolated directly from the crude reaction mixture by direct azeotropic distillation thus avoiding the use of large amounts of extraction solvents and tedious and costly fraction distillation procedures. Upon completion of the reaction, the reaction mixture is cooled to about room temperature, neutralized to a pH range of about 4.0 - 9.0, preferably about 6-8, to avoid amide by-product formation, diluted with water to aide the azeotropic removal of the cyclopropyl nitrile, and azeotropically distilled using a Dean Stark trap to give high purity, essentially anhydrous product cyclopropyl nitrile. Advantageously, this procedure allows the isolation of pure cyclopropyl nitrile on a manufacturing scale without the use and handling of extraction solvents and without the need for costly fraction distillation columns.

For a more clear understanding of the invention, specific examples thereof are set forth below. These examples are merely illustrative and are not to be understood as limiting the scope and underlying principles of the invention in any way.

The term GLC designates gas liquid chromatography.

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EXAMPLE 1Preparation of cyclopropyl nitrile (2 molar scale)

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A stirred mixture of 4-chloro-butyronitrile (213.4g, 2.0 mole), sodium chloride (58.0g, 0.5 mole) and 2.0g of water in 200g of dimethylsulfoxide is heated to 80°C, treated with  
 5 ground solid sodium hydroxide (88g, 2.2 mole) over a 3 hour period and held at 80°C for about 1 additional hour. The reaction mixture is cooled to room temperature neutralized to about pH6.8 with concentrated HCl (37% aqueous solution), diluted  
 10 with 200ml water (readjusted pH to 6.8 with 37% HCl) and azeotropically distilled with a Dean Stark trap to remove the cyclopropyl nitrile/water azeotrope. The aqueous layer is continuously returned to the distillation pot to give the  
 15 initial product as the distillate cyclopropyl nitrile, 133.5g. A 121.5g sample of this cyclopropyl nitrile is azeotropically distilled using a Dean Stark trap (to remove water) and continuous return of the organic layer to give the  
 20 final product cyclopropyl nitrile, 106.6.g, 87.8% yield, 98% pure by GLC analysis and 0.04% H<sub>2</sub>O by Karl Fischer titration.

## EXAMPLE 2

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### Preparation of cyclopropyl nitrile (30 molar scale)



30

A stirred mixture of 4-chloro-butyronitrile (3.17kg, 30 mole), sodium chloride (0.87kg, 15 mole) and .03kg water in 3.0kg of dimethylsulfoxide is heated to 80°C, treated with  
 35 NaOH pellets (1.24kg, 30.9 mole) over a 2 hour period and held at 80°C for about 1 additional



hour. (Another 0.012kg of NaOH was added during the hold period.) The reaction mixture is cooled to room temperature, neutralized to about pH 6.8 - 7.0, diluted with 3.0 L of water (readjusted pH to about 7.0) and azeotropically distilled with a Dean Stark trap to remove the cyclopropyl nitrile/water azeotrope. The aqueous layer is continuously returned to the distillation pot to give the initial product cyclopropyl nitrile distillate, 1.811kg. A 1.72kg sample of this cyclopropyl nitrile is azeotropically distilled using a Dean Stark trap (to remove water) and continuous return of the organic layer to give the final product cyclopropyl nitrile, 1.63kg, 85% yield, 97.1% pure by GLC analysis and 0.28% H<sub>2</sub>O by Karl Fischer titration.

### EXAMPLE 3

#### 20 Preparation of cyclopropyl nitrile (Plant scale)



25 A)

Using essentially the same procedure described in Examples 1 and 2, cyclopropyl nitrile is prepared on a 111kg scale (1.071 kmole) in a pilot plant using 100 gallon and 500 gallon glass-lined reactors to give the initial cyclopropyl nitrile product distillate, 59.3kg, 82.48% yield, 95.3% purity and 4.4% water content. This cyclopropyl nitrile distillate is combined with another pilot plant batch and azeotropically dried as described in Examples 1 and 2 to give the final



cyclopropyl nitrile product, 95.1% recovery, 98.3% pure and 0.4% water content.

B)

5           Using the essentially the same procedure described above, cyclopropyl nitrile is prepared on a plant scale of 1,021kg(9.86 kmole), using a 2,000 gallon reactor to give the initial cyclopropyl nitrile distillate, 592kg, 89.5% yield,  
10 95.2% pure and 4.4% water content. This product is combined with another plant run and azeotropically distilled to give the final cyclopropyl nitrile product, 95.5% recovery, 96.4% pure and 0.4% water content.

15

#### EXAMPLES 4-8

#### Comparative preparation of cyclopropyl nitrile

##### General Procedure

20

A stirred mixture of 4-chlorobutyro-nitrile and NaOH in dimethylsulfoxide, with and without NaCl and with and without a catalytic amount of water, is heated to 80°C and stirring  
25 observations are made and recorded in Table I.

As can be seen from Table I, the absence of the addition of NaCl to the reaction mixture leads to an unstirrable mass and the absence of a catalytic amount of water may induce hazardous  
30 reaction conditions wherein the reaction rate is uncontrollable and large exotherms occur. When the stirring is poor or completely stopped, the reaction remains incomplete and product yields and purity are diminished.



Table I

|    | 4-CBN <sup>1</sup> | NaOH         | Water        | NaCl         | DMSO         |                         |
|----|--------------------|--------------|--------------|--------------|--------------|-------------------------|
| 5  | <u>Example</u>     | <u>moles</u> | <u>moles</u> | <u>moles</u> | <u>grams</u> | <u>Stirring</u>         |
|    | 4                  | 1.0          | 1.1          | 0.0          | 0.0          | 200.0 Poor <sup>2</sup> |
|    | 5                  | 1.0          | 1.1          | 0.0          | 1.0          | 200.0 Good              |
|    | 6                  | 2.0          | 2.0          | 0.1          | 1.0          | 200.0 Good              |
|    | 7                  | 6.0          | 6.6          | 0.05         | 3.0          | 600.0 Good              |
| 10 | 8                  | 1.0          | 1.1          | 0.0          | 0.5          | 200.0 Good <sup>3</sup> |

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<sup>1</sup>Chlorobutyronitrile

<sup>2</sup>Stirring completely impeded by reaction mass

<sup>3</sup>Hang-fire conditions (delayed reaction initiation

15 followed by uncontrolled rate of reaction,  
temperature overshoot)

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## I CLAIM:

1. An improved process for the manufacture of cyclopropyl nitrile which comprises mixing 4-halo-butyronitrile, at least 0.25 mole of an inorganic salt and a catalytic amount of water in the presence of an aprotic polar solvent at an elevated temperature of about 50°-100°C and reacting the mixture with an alkali metal base at the elevated temperature; wherein the inorganic salt is a metal halide, a metal sulfate or a metal carbonate and the metal is Na, K or Li.
2. The process according to claim 1 wherein the alkali metal base is MOR wherein M is Na, K or Li and R is hydrogen or C<sub>1</sub>-C<sub>6</sub> alkyl.
3. The process according to claim 1 wherein about 0.5 to 1.0 mole of the inorganic salt is added.
4. The process according to claim 1 wherein the aprotic polar solvent is a sulfoxide or a carboxylic acid amide.
5. The process according to claim 4 wherein the solvent is dimethylsulfoxide.
6. The process according to claim 1 wherein the alkali metal base is sodium hydroxide, the aprotic polar solvent is dimethylsulfoxide, the inorganic salt is sodium chloride and the temperature is about 60°-90°C.

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7. The process according to claim 1 which further comprises (a)cooling the reaction temperature to room temperature when the reaction is complete, (b)neutralizing the cooled completed reaction, (c)diluting the neutralized reaction with water and (d)isolating the reaction product cyclopropyl nitrile by azeotropic distillation.

8. The process according to claim 1 wherein the neutralized diluted reaction has a pH of about 4-9.

9. The process according to claim 8 wherein the pH is about 7-8.

10. The process according to claim 7 wherein the product cyclopropyl nitrile is azeotropically distilled to an essentially anhydrous condition.

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**IMPROVED PROCESS FOR THE MANUFACTURE OF CYCLO-  
PROPYLNITRILE**

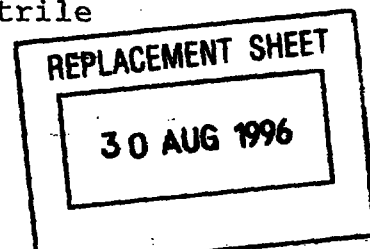
Cyclopropyl nitrile is a vital raw material in the manufacture of herbicidal agents. It is particularly useful in the manufacture of herbicidally potent, yet environmentally benign 1-(o-cyclopropylcarbonyl)-phenylsulfamoyl urea derivatives. The preparation of cyclopropyl nitrile from 4-chlorobutyronitrile is described in U. S. 3,843,709. However, when this preparation is applied to a manufacturing scale, an unstirrable mass forms causing decreased reaction yield, broken or damaged equipment and problematic product isolation.

Therefore it is an object of this invention to provide an improved process for the manufacture of cyclopropyl nitrile which is suitable for large scale production with increased isolated product yield.

It is another object of this invention to provide an efficient and effective method of isolation of cyclopropyl nitrile in high purity and with essentially no water content.

It is a further object of this invention to provide a convenient and economic source of essential starting material in the production of herbicidal sulfamoyl urea derivatives.

Herbicidal agents and in particular 1-(o-cyclopropylcarbonyl)phenylsulfamoyl urea derivatives are prepared using cyclopropyl nitrile



as the basic starting material. It has now been found that cyclopropyl nitrile can be prepared on a commercial manufacturing scale without the formation of an unstirrable mass and accompanying equipment damage or breakage and decreased product yield by reacting 4-halobutyronitrile with an alkali metal base in the presence of an aprotic polar solvent, an inorganic salt and a catalytic amount of water at a temperature of about 50°-100°C. The preparation is shown in flow diagram I wherein X is halogen, M is an alkali metal and R is hydrogen or C<sub>1</sub>-C<sub>6</sub>alkyl.

Flow Diagram I



Methods available in the art to prepare cyclopropyl nitrile, upon scale-up, give the formation of an unstirrable mass. The inability to stir or agitate a reaction mixture results in incomplete reaction, hazardous reaction conditions and damaged or broken equipment. Surprisingly, it has been found that the addition of at least 0.25 mole, preferably about 0.4 to 1.5 mole and more preferably about 0.5 to 1.0 mole of an inorganic salt eliminates the formation of an unstirrable mass thus preventing agitation problems, poor yields and a broken or damaged agitator. Advantageously, it has been found that the addition of a catalytic amount of water to the reaction mixture is critical to the initiation of the reaction. Predictable and reproducible reaction initiation prevents the potential hazard of large scale run-

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30 AUG 1996

away reaction rates and sudden uncontrollable exotherms.

In general, the reaction rate increases as the temperature increases, however it has now  
5 been found that under large scale conditions, reaction temperatures greater than about 110°C give mainly amide side-product formation and little or no cyclopropyl nitrile product. Elevated  
10 temperatures of about 50°- 100°C, preferably about 60°- 90°C are suitable in the inventive process.

Aprotic polar solvents suitable for use in the inventive process are sulfoxides, sulfones, carboxylic acid amides, pyrrolidones and the like. Sulfoxides and carboxylic acid amides are  
15 preferred, dimethylsulfoxide and dimethyl formamide are more preferred and dimethylsulfoxide is most preferred.

Alkali metal bases useful in the process of the invention are any alkali metal hydroxides or  
20 alkoxides or mixtures thereof. Preferable alkali metal bases are monovalent bases such as NaOR, KOR or LiOR, more preferred are the hydroxides of sodium or potassium and most preferred is NaOH. Stoichiometric amounts of the alkali metal base may  
25 be used in the process of the invention. In a preferred embodiment of the invention, the alkali metal base is added to the reaction mixture incrementally over a period of time.

Inorganic salts suitable for use in the  
30 present invention are metal halides, metal sulfates or metal carbonates, preferably metal halides such as sodium halide or potassium halide, more preferably sodium halide and most preferably sodium chloride.

35 The product cyclopropyl nitrile, in order to be suitable for use in manufacturing 1-(o-cyclopropyl-carbonyl)phenylsulfamoyl urea

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30 AUG 1996

derivatives, must be essentially free of water and minor components. It has now been found that highly pure and essentially anhydrous cyclopropyl nitrile product can be isolated directly from the crude reaction mixture by direct azeotropic distillation thus avoiding the use of large amounts of extraction solvents and tedious and costly fraction distillation procedures. Upon completion of the reaction, the reaction mixture is cooled to about room temperature, neutralized to a pH range of about 4.0 - 9.0, preferably about 6-8, to avoid amide by-product formation, diluted with water to aid the azeotropic removal of the cyclopropyl nitrile, and azeotropically distilled using a Dean Stark trap to give high purity, essentially anhydrous product cyclopropyl nitrile. Advantageously, this procedure allows the isolation of pure cyclopropyl nitrile on a manufacturing scale without the use and handling of extraction solvents and without the need for costly fraction distillation columns.

For a more clear understanding of the invention, specific examples thereof are set forth below. These examples are merely illustrative and are not to be understood as limiting the scope and underlying principles of the invention in any way.

The term GLC designates gas liquid chromatography.

30

#### EXAMPLE 1

##### Preparation of cyclopropyl nitrile (2 molar scale)

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A stirred mixture of 4-chloro-butyronitrile (213.4g, 2.0 mole), sodium chloride (58.0g, 0.5 mole) and 2.0g of water in 200g of dimethylsulfoxide is heated to 80°C, treated with  
 5 ground solid sodium hydroxide (88g, 2.2 mole) over a 3 hour period and held at 80°C for about 1 additional hour. The reaction mixture is cooled to room temperature neutralized to about pH6.8 with concentrated HCl (37% aqueous solution), diluted  
 10 with 200ml water (readjusted pH to 6.8 with 37% HCl) and azeotropically distilled with a Dean Stark trap to remove the cyclopropyl nitrile/water azeotrope. The aqueous layer is continuously returned to the distillation pot to give the  
 15 initial product as the distillate cyclopropyl nitrile, 133.5g. A 121.5g sample of this cyclopropyl nitrile is azeotropically distilled using a Dean Stark trap (to remove water) and continuous return of the organic layer to give the  
 20 final product cyclopropyl nitrile, 106.6g, 87.8% yield, 98% pure by GLC analysis and 0.04% H<sub>2</sub>O by Karl Fischer titration.

#### EXAMPLE 2

25

#### Preparation of cyclopropyl nitrile (30 molar scale)



30

A stirred mixture of 4-chloro-butyronitrile (3.17kg, 30 mole), sodium chloride (0.87kg, 15 mole) and .03kg water in 3.0kg of dimethylsulfoxide is heated to 80°C, treated with  
 35 NaOH pellets (1.24kg, 30.9 mole) over a 2 hour period and held at 80°C for about 1 additional

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30 AUG 1996

hour. (Another 0.012kg of NaOH was added during the hold period.) The reaction mixture is cooled to room temperature, neutralized to about pH 6.8 - 7.0, diluted with 3.0 L of water (readjusted pH to about 7.0) and azeotropically distilled with a Dean Stark trap to remove the cyclopropyl nitrile/water azeotrope. The aqueous layer is continuously returned to the distillation pot to give the initial product cyclopropyl nitrile distillate, 1.811kg. A 1.72kg sample of this cyclopropyl nitrile is azeotropically distilled using a Dean Stark trap (to remove water) and continuous return of the organic layer to give the final product cyclopropyl nitrile, 1.63kg, 85% yield, 97.1% pure by GLC analysis and 0.28% H<sub>2</sub>O by Karl Fischer titration.

### EXAMPLE 3

#### 20 Preparation of cyclopropyl nitrile (Plant scale)



25 A)

Using essentially the same procedure described in Examples 1 and 2, cyclopropyl nitrile is prepared on a 111kg scale (1.071 kmole) in a pilot plant using 100 gallon and 500 gallon glass-lined reactors to give the initial cyclopropyl nitrile product distillate, 59.3kg, 82.48% yield, 95.3% purity and 4.4% water content. This cyclopropyl nitrile distillate is combined with another pilot plant batch and azeotropically dried as described in Examples 1 and 2 to give the final

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cyclopropyl nitrile product, 95.1% recovery, 98.3% pure and 0.4% water content.

B)

5           Using the essentially the same procedure described above, cyclopropyl nitrile is prepared on a plant scale of 1,021kg (9.86 kmole), using a 2,000 gallon reactor to give the initial  
10 cyclopropyl nitrile distillate, 592kg, 89.5% yield, 95.2% pure and 4.4% water content. This product is combined with another plant run and azeotropically distilled to give the final cyclopropyl nitrile product, 95.5% recovery, 96.4% pure and 0.4% water content.

15

#### EXAMPLES 4-8

#### Comparative preparation of cyclopropyl nitrile

##### General Procedure

20

A stirred mixture of 4-chlorobutyronitrile and NaOH in dimethylsulfoxide, with and without NaCl and with and without a catalytic amount of water, is heated to 80°C and stirring  
25 observations are made and recorded in Table I.

As can be seen from Table I, the absence of the addition of NaCl to the reaction mixture leads to an unstirrable mass and the absence of a catalytic amount of water may induce hazardous  
30 reaction conditions wherein the reaction rate is uncontrollable and large exotherms occur. When the stirring is poor or completely stopped, the reaction remains incomplete and product yields and purity are diminished.

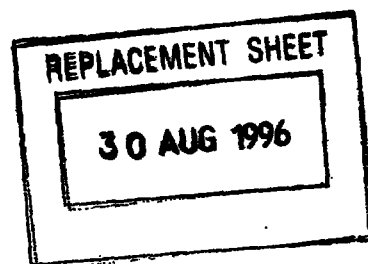


Table I

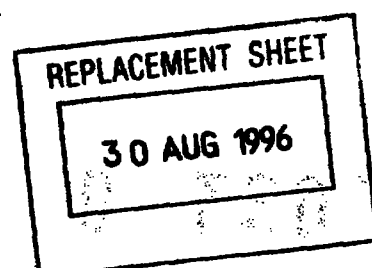
|    | 4-CBN <sup>1</sup> | NaOH         | Water        | NaCl         | DMSO         |                         |
|----|--------------------|--------------|--------------|--------------|--------------|-------------------------|
| 5  | <u>Example</u>     | <u>moles</u> | <u>moles</u> | <u>moles</u> | <u>grams</u> | <u>Stirring</u>         |
|    | 4                  | 1.0          | 1.1          | 0.0          | 0.0          | 200.0 Poor <sup>2</sup> |
|    | 5                  | 1.0          | 1.1          | 0.0          | 1.0          | 200.0 Good              |
|    | 6                  | 2.0          | 2.0          | 0.1          | 1.0          | 200.0 Good              |
|    | 7                  | 6.0          | 6.6          | 0.05         | 3.0          | 600.0 Good              |
| 10 | 8                  | 1.0          | 1.1          | 0.0          | 0.5          | 200.0 Good <sup>3</sup> |

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<sup>1</sup>Chlorobutyronitrile

<sup>2</sup>Stirring completely impeded by reaction mass

<sup>3</sup>Hang-fire conditions (delayed reaction initiation  
15 followed by uncontrolled rate of reaction,  
temperature overshoot)



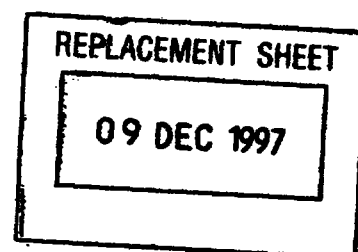


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## I CLAIM:

| REPLACEMENT SHEETS                             |                                   | SG |
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1. A process for the manufacture of cyclopropyl nitrile which comprises mixing 4-halo-butyronitrile, at least 0.25 mole of an inorganic salt and a catalytic amount of water in the presence of an aprotic polar solvent at an elevated temperature of about 50°-100°C and reacting the mixture with an alkali metal base at the elevated temperature; wherein the inorganic salt is a metal halide, a metal sulfate or a metal carbonate and the metal is Na, K or Li.
2. The process according to claim 1 wherein the alkali metal base is MOR wherein M is Na, K or Li and R is hydrogen or C<sub>1</sub>-C<sub>6</sub> alkyl.
3. The process according to claim 1 wherein about 0.5 to 1.0 mole of the inorganic salt is added.
4. The process according to claim 1 wherein the aprotic polar solvent is a sulfoxide or a carboxylic acid amide.
5. The process according to claim 4 wherein the solvent is dimethylsulfoxide.
6. The process according to claim 1 wherein the alkali metal base is sodium hydroxide, the aprotic polar solvent is dimethylsulfoxide, the inorganic salt is sodium chloride and the temperature is about 60°-90°C.





7. The process according to claim 1 which further comprises (a)cooling the reaction temperature to room temperature when the reaction is complete, (b)neutralizing the cooled completed reaction, (c)diluting the neutralized reaction with water and (d)isolating the reaction product cyclopropyl nitrile by azeotropic distillation.

8. The process according to claim 1 wherein the neutralized diluted reaction has a pH of about 4-9.

9. The process according to claim 8 wherein the pH is about 7-8.

10. The process according to claim 7 wherein the product cyclopropyl nitrile is azeotropically distilled to an essentially anhydrous condition.

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