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An Effective Process for Conversion of Diphenylurea to CC-2, a Potential Decontaminant of Sulfur Mustard

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Abstract:

N,N′-Dichlorobis(2,4,6-trichlorodiphenyl) urea, also known as 2-chlorocarbinol (CC-2) is a potential decontaminant of Sulfur Mustard (SM), which is also known as bis(2-chloroethyl) sulfide (HD), a well-known warfare agent. A new process has been developed for the synthesis of CC-2 from diphenylurea (DPU) through an intermediate, known as hexachlorocarbanilide (HCC). The conversion of DPU to HCC was studied in the temperature range 35−100 °C in the presence of several homogeneous scavengers of HCl, like pyridine, triethylamine, ethylenediamine, hexamethylenetetramine, 4,4′-bipyridine, diethylamine, dicyclohexylamine, etc. Experiments were carried out to study the activity of these homogeneous scavengers to get maximum conversion, yield, and purity of the product. Reaction temperature, reaction time, substrate-to-scavenger ratio, and solvent requirements were studied to optimize the reaction conditions. Both pyridine (product purity ~80% and yield ~89%) and ethylenediamine (product purity ~85% and yield ~70%) were found to be effective in improving the performance of the reaction.

Introduction

Sulfur Mustard (SM), also known as bis(2-chloroethyl) sulfide (HD) is a chemical warfare agent with serious toxic effects. SM causes serious blisters upon contact with human skin, and due to this reason it has been used as a chemical warfare agent.[1−9] There also exists the potential threat of use of SM by a terrorist group. So, protection of the soldiers and the civilians against HD remains one of the main concerns of the scientific community. However, there is no specific antidote available against HD.

Decontamination of chemical warfare agents (CWAs) is required in the case of chemical attack by adversaries or terrorists. Decontamination is one of the important combating activities (detection, protection, and decontamination) against CWAs. Decontamination of CWAs is achieved either by physically removing the toxic substances from contaminated surfaces (materials in the field like vehicles, buildings, equipment, and living objects) or by chemically converting them into relatively less or nontoxic substances.[10] For physical removal of CWAs from a contaminated site, adsorbents such as Fuller's earth (native aluminium silicate) and detergent/soap solutions are used. Since these decontaminants do not detoxify the toxic agents, they are not considered as reliable means. The second major problem of physical decontaminants is their safe disposal. Physical decontaminants themselves get contaminated during decontamination operation; their subsequent safe disposal requires further treatment to neutralise CWAs. Another problem of physical decontaminants is secondary contamination, which is caused by the ensuing desorption of adsorbed CWAs. Yet another problem of physical decontaminants such as washing solutions is spreading of the contaminated area, since during washing operation the solution is spread over the contaminated surface.

Keeping in view the hazards of SM, its decontamination is of paramount importance. It is required on the battlefield, laboratories, production and storage plants, destruction sites, and more importantly in case of sabotage and usage of CWAs by terrorists. In all cases the chemical decontamination of the HD is still the best method of protection against HD.[11] Currently used reactive decontaminants include nucleophile/base-amine mixtures and bleach formulations.[12−14] Although these formulations are effective in decontaminating the CWAs, they cannot be used on persons because they are highly corrosive and toxic and hence best suited for material decontamination only. Consequently, there was a need to develop a safe and effective decontamination formulation against CWAs for human application.

A requirement for a topical skin protectant (TSP) to protect skin from toxic CWAs was immediately recognised

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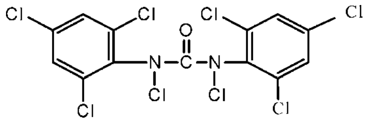
following the introduction of these agents in World War I. Although several creams and ointments were made, none of them was effective in deactivating the CWAs. Just prior to and during World War II, a concentrated effort to develop ointments for protection against sulphur mustard was made at the chemical warfare service in Edgewood Arsenal, Maryland USA, which resulted in the development of M-5 ointment, but it left room for improvement. For several years, research concentrated on introducing active ingredients such as alkalies, chelators, sorbents, etc. into polyethylene-based formulations. However, none of them provided the required protection.[15]

Recently, two compounds have been used as active ingredients in the formulations made to protect human skin from sulphur mustard. A substance coded as S-330 (1,3,4,6-tetrachloro-7,8-diphenyl-2,5-diiminoglycoluril) was mixed with perfluorinated ethers and evaluated as a reactive decontaminant against sulphur mustard.[16,17] But the main disadvantages of this system are the use of an uneconomic and nonecofriendly perfluorinated compound as base material, and S-330 decomposes within 2−3 weeks at room temperature in the presence of even a little moisture. Another disadvantage of this system is that it is not very fast acting, which is the prime requirement of any decontaminant.[18,19] The second compound which was found to be very efficient, fast acting, and effective at subzero temperatures also in decontaminating the sulphur mustard is *N*,*N′*-dichlorobis-(2,4,6-trichlorophenyl) urea (**1**).[20] It is a solid at room-temperature having a melting point of 178−180 °C. Theoretically **1** contains 14.54% active chlorine, which is responsible for the instantaneous reaction with HD. Therefore **1** can be used for personal protection against SM on the battlefield. Formulations made by incorporating **1** in different bases such as Gum acacia and hydroxypropyl cellulose were found to be very effective, stable, and safe and provided the best protection against topically applied sulphur mustard in mice and rats. Since SM is highly lipophilic and gets absorbed very quickly after contact with skin, the best way of protection from SM is to decontaminate it instantaneously after contact with skin without causing any damage to delicate human skin. This stringent requirement of decontaminant was successfully met by developing a formulation based on **1** that deactivated the topically applied SM instantaneously without causing any irritation or harm to the skin. A formulation based on **1** has been approved by competent authority for introduction into services after passing through necessary toxicological and pharmacological tests and trials.[21,22] Keeping in view the requirement of formulation based on **1** in large quantities, there is a need to develop the technology for preparation of **1** itself. The methods reported in the literature for preparation of **1** are inadequate in meeting the requirement of an industrially viable process. In one method, **1** is prepared by reacting diphenylurea (**10**) with chlorine for 48 h in two steps.[23] The major disadvantages of the process are that complete chlorination of aromatic rings did not take place in this method, the yield of the product is less (<40%), the rate of reaction is very slow, and it takes 7 days (with 40% yield) for aromatic chlorination and 48 h for N-chlorination (second step) (with ≈ 30−40%), and also several impurities are formed (partial chlorinated products). The second method makes use of phosgene, which is reacted with 2,4,6-trichloroaniline to produce HCC-2 (**11**), the precursor of **1**. In a second step, **11** is converted into **1** by reacting it with chlorine.[24] Both methods are not suitable for production of **1** in large amounts, as they are time-consuming and multistep and involve highly toxic phosgene as a starting material.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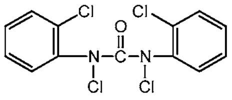
Keeping in view limitations of existing synthetic procedures of **1**, there was a dire need to develop an industrially viable process for the production of **1**. More importantly, there was a need for development of a one-pot process (without any requirement of isolation of intermediates or precursors) to produce **1** by making use of readily available chemicals.

A new laboratory method for the synthesis of **1** has been reported.[20] According to the process, **1** can be synthesized from **10** through a two-stage chlorination process. But the process has some bottlenecks. First, the product contains a lot of impurities such as **2**, **3**, **4**, **5**, **6**, **7**, **8**, and **9** that are produced through an incomplete reaction. Once these impurities are formed it is very difficult (almost impossible) to separate them by any separation process, in an economical manner. Second, even if all the impurities are separated, then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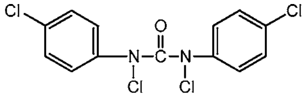
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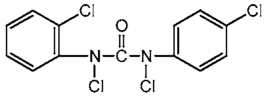
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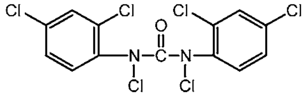
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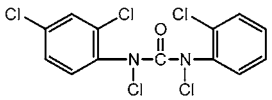
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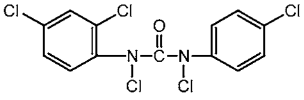
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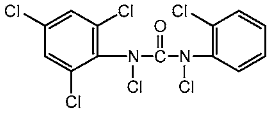
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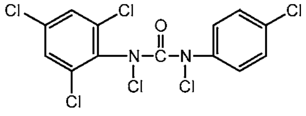
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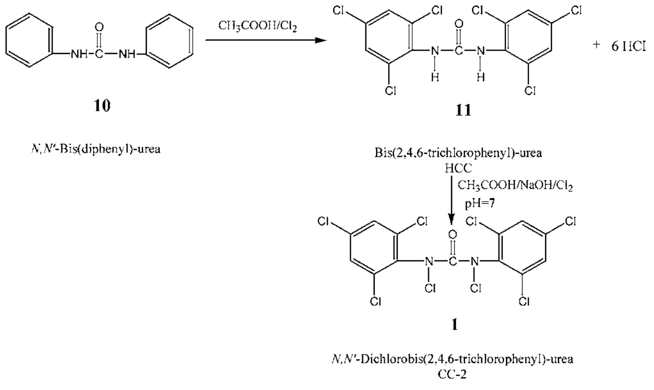
Scheme 1. **Conversion of 10 to 1 through aromatic and N-chlorination**

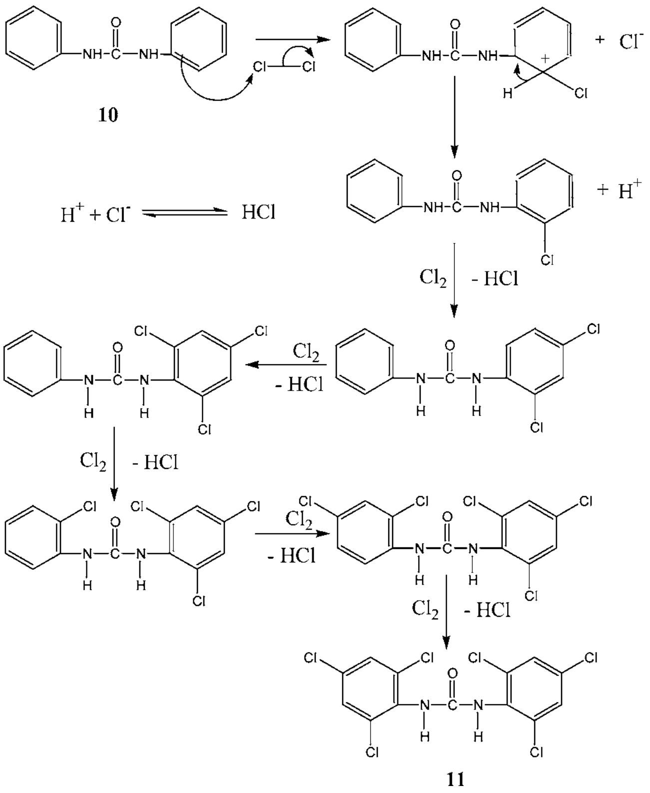


the overall yield of the pure product is very less. Hence, new reactions were tried in the presence of a scavenger to enhance the conversion towards the desired product and thereby increase the yield of product by restricting conversions towards impurities. In this paper all the activities of different scavengers to remove the impurities were explained. The effect of each scavenger on the yield and the conversion has also been described in this paper.

Results and Discussion

The conversion of **10** to **1** takes place in two steps, where in the first step aromatic chlorination takes place and the second step is the N-chlorination. Chlorine is absorbed by the reaction mixture, where chlorination of phenyl rings takes place at 2,4,6 positions as shown in Scheme 1 first and then N-chlorination takes place in an acetic acid and sodium hydroxide mixture at a pH of 7.0. As the reaction proceeds and aromatic chlorination was achieved, the precipitation of **11** occurs and absorption of chlorine gas gradually ceases. This initial aromatic chlorination is complete within 4−6 h, which was indicated by the appearance of a fluffy precipitate of **11** and the IR and NMR analyses. In this reaction one of the major disadvantages is that a lot of impurities as described in the previous section were formed, and the formation of these impurities is explained as incomplete chlorination of the aromatic ring (Scheme 2). The mechanism of conversion of **11** to **1** is explained in the Scheme 3. The N-chlorination takes place via the formation of hypochlorous acid, in which chlorine has a slight positive charge. This positive charge on chlorine is responsible for the attack of the lone pair on the nitrogen of **11**.

Scheme 2. **Mechanism of formation of mono- and disubstituted impurities of 11**



Scheme 3. **Mechanism of N-chlorination of 11**

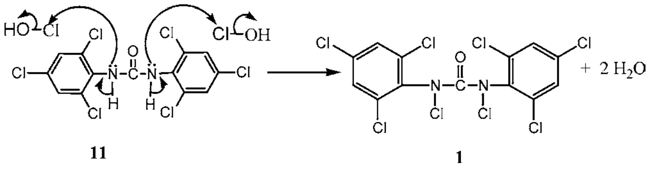
$$2\,NaOH + Cl_2 \rightleftharpoons NaOCl + NaCl + H_2O$$

$$NaOCl + H_2O \rightleftharpoons NaOH + HOCl$$

Over all Reaction:

$$NaOH + Cl_2 \rightleftharpoons NaCl + HOCl$$

HOCl formed react with HCC in following maner:



Reaction without a Scavenger. By performing the reaction without a scavenger it was observed that incomplete aromatic ring chlorination products (mono- and bisubstituted) are formed as impurities. These impurities are formed through a series reaction due to the incomplete chlorination of the aromatic ring. It was observed that once partial chlorinated products were formed as impurities, it was not possible to separate them by any economical separation technique. Therefore, the second step (N-chlorination) was continued in the same reactor, and all the partial aromatic chlorinated intermediates had undergone N-chlorination in the second step (in the same reactor) and became impurities with the desired **1**. Since, in the second step, all the intermediates, whether partially or completely chlorinated in the phenyl group, undergo N-chlorination and contribute to the lower conversion towards the desired product, the purity (<50%) and the yield (only 80%) are also less. In this case the product was yellowish due to the presence of impurities. Also, it is not possible to separate these impurities from **1** by any separation process economically. Even several recrystallizations of **1** could not lead to a product of more than 90% pure. It was observed that in the presence of impurities the product becomes unstable and the colour changes to reddish yellow with time.

Effect of Reaction Temperature. For studying the effect of temperature on the yield, conversion, and purity of

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Table 1. **Results of reaction carried out in the presence of different scavengers**[a]

scavenger no.	scavenger	moles of scavenger	molar ratio (scavenger/DPU)	yield (%)	% purity of **1**
1	pyridine	0.0030	0.0667	54.2	62.8
		0.0120	0.2667	55.0	65.6
		0.0310	0.6889	59.4	69.2
		0.0620	1.3778	68.2	75.6
		0.0920	2.0444	74.3	78.2
		0.1220	2.7111	82.0	80.3
		0.1470	3.2667	79.0	77.3
		0.1840	4.0888	78.2	74.3
		0.2810	6.2444	68.9	58.3
2	ethylenediamine	0.0070	0.1555	64.0	62.0
		0.0374	0.8311	65.0	75.0
		0.0750	1.6667	77.0	85.0
		0.1220	2.7111	64.2	84.2
		0.1500	3.3333	45.7	80.2
		0.1800	4.0000	41.0	55.0
3	hexamethylenetetramine	0.00043	0.0096	32.0	31.0
		0.0009	0.0200	59.5	35.0
		0.0018	0.0400	54.8	33.0
		0.0036	0.0800	48.0	34.0
		0.0047	0.1044	43.0	32.2
		0.0357	0.7933	27.4	00.0
		0.0710	1.5778	00.0	00.0
4	bypyridine	0.0064	0.1422	32.0	88.4
		0.0160	0.3556	54.9	75.6
		0.0320	0.7111	57.1	33.4
5	dicyclohexylamine	0.0025	0.0556	36.0	
		0.0050	0.1111	36.6	
		0.0125	0.2778	27.4	
		0.0250	0.5556	27.4	
		0.0500	1.1111	25.2	
		0.1004	2.2311	12.0	
6	triethylamine	0.0247	0.5488	45.7	29.6
		0.0494	1.0978	44.0	34.8
		0.0988	2.1956	54.8	43.2
		0.148	3.2889	68.6	53.2

[a] Conditions: substrate (DPU), 0.045 mol; reaction temperature in first step, 80−82 °C; reaction temperature in second step, 10−15 °C; reaction time, 8 h (4 h in first step and 4 h in second step).

product, experiments were carried out at different temperatures. The aromatic chlorination and the N-chlorination were carried out in the temperature range 30 to 80 °C and 5 to 40 °C, respectively. The reactions were allowed to complete by bubbling chlorine for 4 h in both reaction steps. To see the effect of temperature on each step separately, the product mixture was filtered and **11**, formed in the reaction, was separated. With an increase in the temperature the solubility of chlorine decreases, but the rate of reaction increases. The experimental results suggest that the optimum temperature in aromatic chlorination (first step reaction) is 80−82 °C. It was observed that below this temperature the yield of HCC was less due to less reactivity at lower temperature, whereas at higher temperature the HCC formed in the reaction may undergo backward reaction due to dissociation at higher temperature. Similarly it was found that for the N-chlorination the optimum temperature is 10−15 °C.

Effect of Amount of Solvent. In both reactions acetic acid was used as solvent. It was observed that the amount of solvent has an influence in the purity of **11** and **1**. If too little of an amount of acetic acid is used, the product was found to be less pure. This may be attributed to improper mixing of the reactants. On the other hand larger amounts of acetic acid lead to an increased reactor size and thereby increased capital cost and operating cost. Therefore, the amount of solvent was experimentally optimized, and it was found that 500 mL of acetic acid for 0.047 mol of **10** were optimum. At this ratio, the mixing was good and the optimum yield and purity were obtained.

Effect of pH. In the formation of **11** from **10**, as it is depicted in Schemes 1 and 2, hydrochloric acid is produced in the first step. Therefore the pH of the reaction decreases with time. It was experimentally observed that in the beginning of the reaction the pH was 1.7 and as reaction progresses towards the forward direction it reduced to 0.41.

The N-chlorination is highly pH sensitive. It is clear from the mechanism that it takes place through the formation of hypochlorous acid and hypochloride ion, where the chlorine bears a slightly positive charge, which must be generated for the N-chlorination. But at low pH (as in acetic acid) HOCl dissociates to HCl and nascent oxygen. Positive chlorine will not be available, and therefore the N-chlorination cannot take place at a lower pH. It was observed that it takes place at neutral pH (≈ 7.0).

Effect of Scavenger of Hydrochloric Acid. The formation of partial chlorinated products is a problem when the

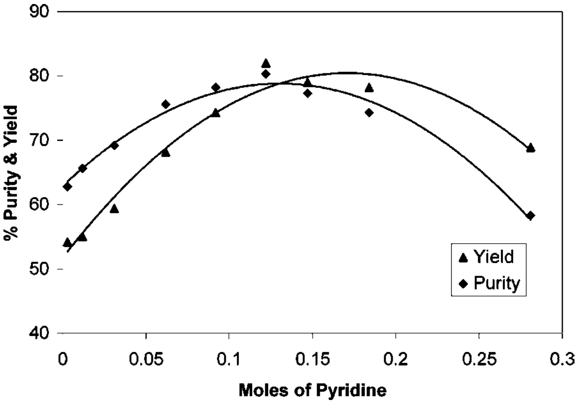


Figure 1. **Effect of moles of pyridine on purity and yield of CC-2 obtained from reaction in the presence of pyridine as catalyst. Conditions: substrate (DPU), 0.045 mol; reaction temperature in first step, 80−82 °C; reaction temperature in second step, 10−15 °C; reaction time, 8 h (4 h in first step and 4 h in second step).**

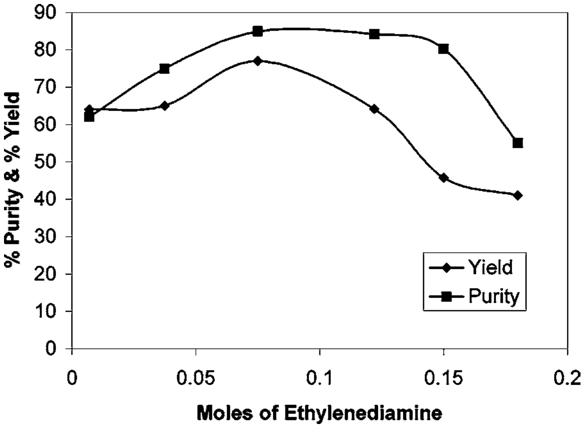
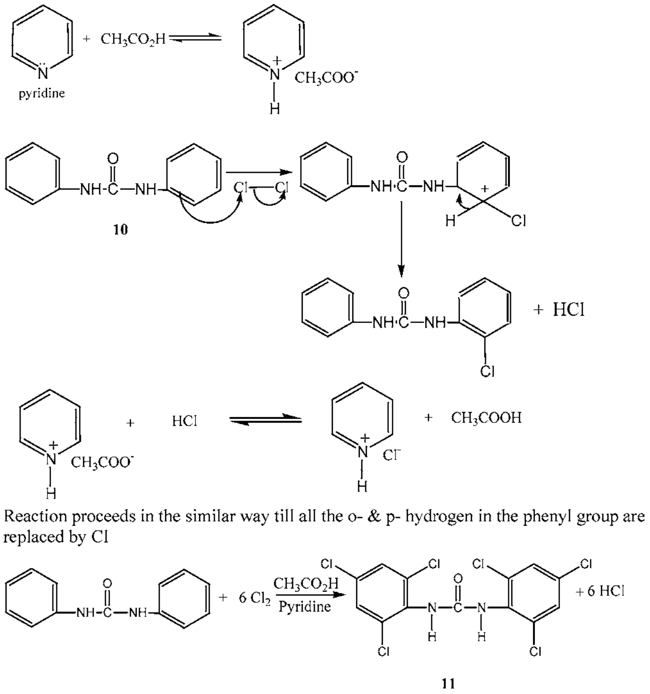


Figure 2. **Effect of moles of ethylenediamine on purity and yield of CC-2 obtained from reaction in the presence of ethylenediamine as catalyst. Conditions: substrate (DPU), 0.045 mol; reaction temperature in first step, 80−82 °C; reaction temperature in second step, 10−15 °C; reaction time, 8 h (4 h in first step and 4 h in second step).**

Scheme 4. **Mechanism of conversion of 10 to 11 in the presence of pyridine**



Reaction proceeds in the similar way till all the o- & p- hydrogen in the phenyl group are replaced by Cl

reaction is carried out without any scavenger of hydrochloric acid. On the other hand by performing the reaction in the presence of a scavenger, complete chlorination takes place and the conversion towards the desired product is enhanced and thereby increases the yield of product by restricting formation of impurities. But, use of Lewis acid catalysts such as $AlCl_3$ and BF_3 offered no advantage in this case. It is assumed (Scheme 2) that hydrochloric acid is produced in the reaction, and any compound that acts as a scavenger for the HCl can shift the reaction towards the completion of substitution of the aromatic hydrogen. That means the reaction is shifted in the forward direction. Therefore, the reaction was studied with the use of pyridine, triethylamine, ethylenediamine, hexamethylenetetramine, 4,4′-bipyridine, diethylamine, and dicyclohexylamine, as these are possible scavengers of HCl. So, these amines are capable of accelerating the aromatic chlorination.

The result of reactions carried out in the presence of different scavengers is tabulated in Table 1. The effect of different scavengers on purity and yield is shown in Table 1. The same has been shown in graphical form in order to find out the optimum amount of each scavenger. The result of reactions carried out with pyridine is shown in Figure 1. Here the pyridine takes part in the reaction as depicted through Scheme 4. It is clear that the aromatic chlorination is facile in this case, since pyridine reacts with the HCl produced and forms pyridiniumhydrochloride salt, which ensures the reaction shifts towards the forward direction. This is also clear from the observation that the pH initially decreases (due to the formation of HCl) but, after a certain time, again increases because HCl is consumed for the formation of pyridiniumhydrochloride salt. From Figure 1 it is clear that as the scavenger-to-substrate ratio increases, both the yield and purity of the product obtained increase, attain maxima, and decrease again. It was observed that the optimum yield and purity of **1** obtained were 80% and 78%, respectively, and 0.13 mol of pyridine is optimum for 0.045 mol of **10**.

The result of reactions that were carried out with ethylenediamine as a scavenger is shown in Figure 2. It was observed that, for 0.045 mol of **10**, the purity of **1** gradually increases up to 0.15 mol of ethylenediamine, but the yield improvement is not appreciable. Similarly the results of the reactions carried out in the presence of hexamethylenetetramine, bipyridine, dicyclohexylamine, and triethylamine are shown in Figures 3−6, respectively. In all these cases the optimum conditions and the yield and purity were obtained.

In the case of selection of a proper scavenger for a particular process, cost is one of the major criteria along with its activity or efficiency. Among all the scavengers tried, hexamethylenetetramine is the cheapest and is available in solid form. Obviously there is an inclination to use this cheap material as a scavenger of HCl for conversion of **10** to **11** in the production of **1**. But the main problem is that when the reaction was carried out in the presence of hexamethylene-

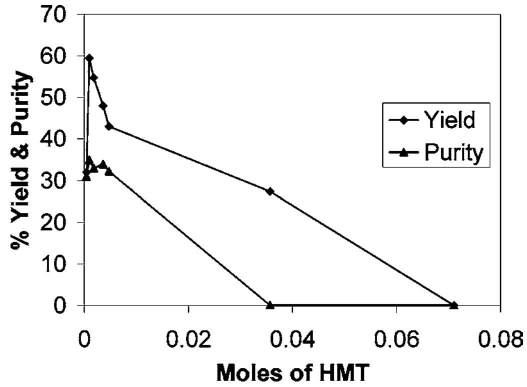


Figure 3. **Effect of moles of hexamethylenetetramine on purity and yield of CC-2 obtained from reaction in the presence of hexamethylenetetramine as catalyst. Conditions: substrate (DPU), 0.045 mol; reaction temperature in first step, 80−82 °C; reaction temperature in second step, 10−15 °C; reaction time, 8 h (4 h in first step and 4 h in second step).**

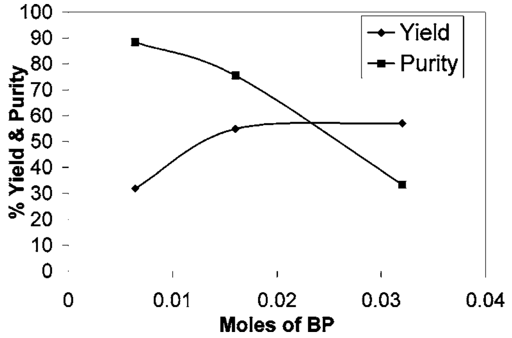


Figure 4. **Effect of moles of bipyridine on purity and yield of CC-2 obtained from reaction in the presence of bipyridine as catalyst. Conditions: substrate (DPU), 0.045 mol; reaction temperature in first step, 80−82 °C; reaction temperature in second step, 10−15 °C; reaction time, 8 h (4 h in first step and 4 h in second step).**

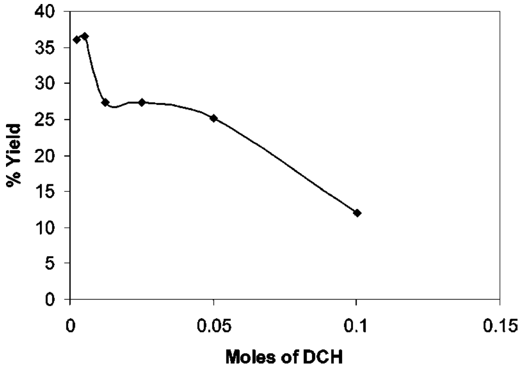


Figure 5. **Effect of moles of dicylohexylamine on purity and yield of CC-2 obtained from reaction in the presence of dicylohexylamine as catalyst. Conditions: substrate (DPU), 0.045 mol; reaction temperature in first step, 80−82 °C; reaction temperature in second step, 10−15 °C; reaction time, 8 h (4 h in first step and 4 h in second step).**

tetramine, it was observed that both the yield and the purity were very poor. In this case the product mixture in the reactor also became dark yellow, making the isolation difficult. Excellent purity product was obtained when bipyridine was used as a scavenger, but the yield was very poor. When the reaction was carried out with dicyclohexylamine, the purity of the product was very poor and in fact less than that when the reaction was carried out without a scavenger of HCl. Good purity product was obtained when the reaction was carried out in the presence of triethylamine, though the yield was not appreciable. But 0.045 mol of **10** requires a large amount of triethylamine (20 mL), which means the reactor volume requirement will be higher in the case of large scale production.

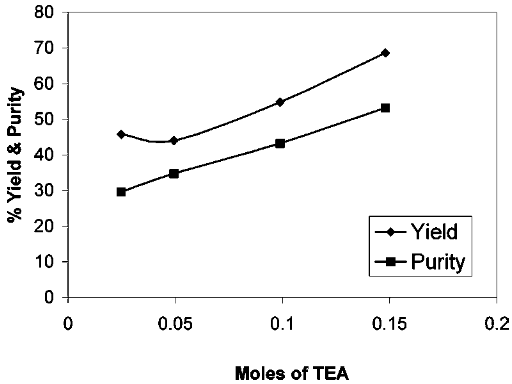


Figure 6. **Effect of moles of triethylamine on purity and yield of CC-2 obtained from reaction in the presence of triethylamine as catalyst. Conditions: substrate (DPU), 0.045 mol; reaction temperature in first step, 80−82 °C; reaction temperature in second step, 10−15 °C; reaction time, 8 h (4 h in first step and 4 h in second step).**

Conclusion

The conversion of **10** to **1** was studied in the presence of different homogeneous scavengers of HCl, and a much higher conversion was obtained in the best case. One of the coproducts was removed in situ from the reaction mixture, which forced the forward reaction to go to completion, and the problem of simultaneous generation of impurities with the desired product was solved.

It was observed that the presence of pyridine and ethylenediamine are almost equally effective in the completion of aromatic chlorination of **10**, thereby improving the yield and purity of the final product.

The effects of pH, temperature, and the amount of solvent were studied. The optimum conditions for maximum yield and purity of **1** for different scavengers were obtained.

The result of this study will be of immense help for further study in this reaction, and it will also help in the scale-up of the process. The study will also be of value to the design engineers for designing a plant for the manufacture of **1**.

Experimental Section

Materials and General Methods. **10** is the main precursor material in the synthesis of **1**. **10**, synthesis grade, is obtained from M/s Sigma-Alrich. Acetic acid, sodium hydroxide, dichloromethane, and hexane (all synthesis grades) were purchased form M/S Qualigens India Ltd. Pyridine, triethylamine, ethylenediamine, hexamethylenetetramine, 4,4′-bipyridine, diethylamine, diethylamine, and dicyclohexylamine (all analytical grade) were purchased from M/s S.D. Fine Chemicals, India. Acetone and dichlo-

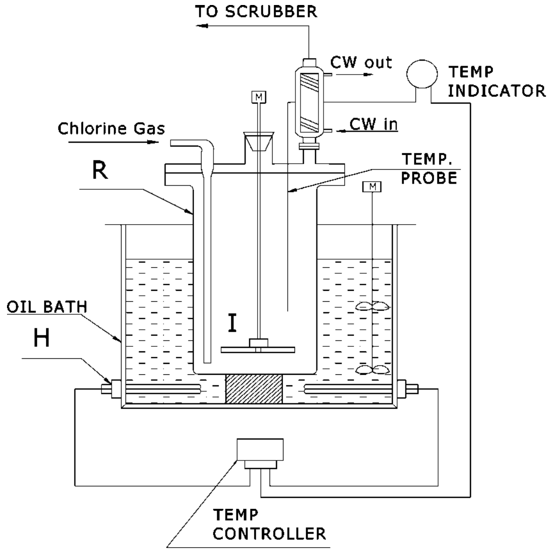


Figure 7. **Experimental setup for chlorination of diphenylurea in the presence of different scavengers of hydrochloric acid: CW, cooling water; H, heating coil; R, reactor; M, motor; I, impeller.**

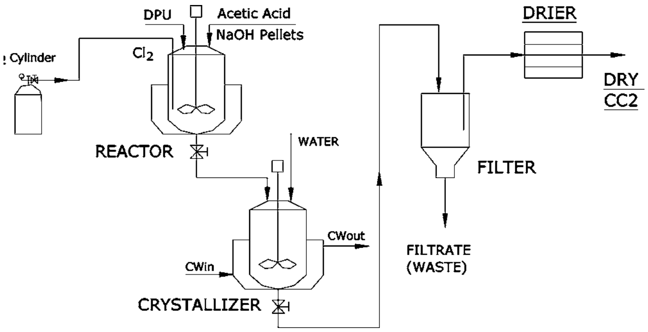


Figure 8. **Flow diagram of the process as used for the synthesis of CC-2.**

romethane, used as reagents for analysis on high performance liquid chromatography (HPLC), were of HPLC grade and obtained from M/S E. MERCK, India.

The conversion of **10** to **11** and then **1** by chlorination was studied in a mechanically agitated glass reactor with a 1000 mL capacity. The glass reactor was equipped with a four-bladed turbine impeller, a thermowell for measuring the temperature, and a gas inlet tube. A reflux condenser with a provision for chilled water as a coolant was also mounted on the reactor to condense and reflux solvent vapors. The entire setup was immersed in a constant temperature oil bath, maintained by using a temperature indicator and controller (TIC). The schematic diagram of the experimental setup is given in Figure 7.

A measured amount of solvent, acetic acid (500 mL), was put in the reactor. A weighed quantity of precursor **10** (10 g, 0.045 mol) was added into the reactor. Then the content of the reactor was heated with constant stirring. When the temperature of the reaction mixture reached the desired value, a measured quantity of homogeneous scavenger was added and passing (bubbling) of chlorine through the liquid was started. This was considered as zero time. Samples were taken out at various time intervals during the experiment. The samples were filtered, and the solid product was separated from the substrate, scavenger, and the solvent. These samples were analyzed by NMR and IR.

At this stage, the passing of chlorine was stopped and reaction mixture was cooled to 5−15 °C in an ice−water bath. When the temperature of the mixture reached 10 °C, sodium hydroxide was added and then bubbling of chlorine gas was again started with constant stirring. Addition of NaOH was continued in portions at 10−15 min intervals until the pH of the mixture became almost neutral (checked by pH paper and a pH meter), and chlorine was passed with continuous stirring. N-chlorination takes place during this step, which is evident by redissolution of fluffy precipitate that was formed in the first step. This step requires approximately 3−4 h. After complete dissolution of the fluffy precipitate, the passing of chlorine was stopped and the reaction mixture was poured into ice-cold water. In cold water, precipitation of CC-2 occurs, which was filtered on a sintered glass funnel and dried. The pyridiniumhydrochloride is soluble in water and hence will go with the filtrate making the separation of the product from the mixture very easy. The product was analyzed by HPLC and NMR. The complete flow diagram of the process is shown in Figure 8.

The samples were analyzed using high performance liquid chromatography (HPLC), (Shimadzu, Japan, model LC-6A) fitted with a CTO-2A column oven. A UV detector (SPD-6AV) was used for scanning. μ Porasil (300 × 3.9 mm^2) supplied by M/S Waters, India was used as the column, and the column temperature was kept at 30 °C. The mobile phase was a mixture of hexane and dichloromethane (65 to 35), and the flow rate of the mobile phase was 2 mL/min. The samples were dissolved and diluted in dichloromethane, and 10 μL were injected into the column through a Reheodyne injector. Chromatopac (C-R3A) was used as the integrator and recorder.

Future Scope of Work

The kinetics of the reaction will be studied, and the activation energy, reaction order, and other kinetic parameters will be found out. The overall rate expression needs to be found out. The effect of hydrodynamic parameters in the reaction needs to be studied, and the scale-up parameters will be found out. The reaction and the process will be studied in different types of reactors. The complete system will be modeled, and simulated results will be verified experimentally.

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