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Formation of Benzodiazepines and Pyrazinylquinoxalines from Aromatic and Heteroaromatic Ketones *via* Deoximation

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The report stated that the treatment of *o*-phenylenediamine with acetone dicarboxylic acid, acetone and acetophenone afforded 2,4,4-trimethyl-3*H*-5-hydro-1,5-benzodiazepine. However, direct reactions of *o*-phenylenediamine with oximes (acetone oxime, acetophenone oxime, and benzophenone oxime) as ketone equivalents did not occur. In the course of present investigations, it is found that dichloroamine-T can be an efficient reagent for the conversion of oximes into the corresponding carbonyl compounds. As a part of a research program related to the synthetic study of pharmacologically interesting benzodiazepine compounds, herein the synthesis of 1*H*-1,5-benzodiazepine derivatives from heteroaromatic ketones and acetone equivalents obtained using dichloroamine-T. On the other hand, when diamine (1,2-phenylene diamine or 1,2-naphthalene diamine) with heterocyclic ketone (acetyl pyridine or acetyl pyrazines) in the presenece of conc. HCl and SiO_2 was refluxed, quinoxaline derivatives as yellow crystalline solids were isolated in high yields.

Keywords: Deoximation, Dichloroamine-T, 1,5-Benzodiazepines, Quinoxalines, Benzoquinoxalines.

INTRODUCTION

Benzodiazepines are a type of drugs known as tranquilizers [1,2]. Benzodiazepines act on the central nervous system, produce sedation and muscle relaxation, and lower anxiety levels. Doctors may prescribe benzodiazepines for amnesia induction for uncomfortable procedures, muscle relaxation, alcohol withdrawal, premedication before anaesthesia (such as before surgery), insomnia, anxiety and seizure control. 1,4-Benzodiazepines and 1,5-benzodiazepines, being in the spotlight recently possess similar activity spectra [3]. We studied the synthesis of 1,5-benzodiazepine derivatives using various reagents [4]. The report stated that the treatment of *o*-phenylenediamine with acetone dicarboxylic acid, acetophenone and acetone, afforded 2,4,4-trimethyl-3*H*-5-hydro-1,5-benzodiazepine. However, direct reactions of *o*-phenylenediamine with oximes (acetone oxime, acetophenone oxime and benzophenone oxime) as ketone equivalents did not occur. The synthesis of ketone compounds from oximes represents a potential route for the syntheses of carbonyl compounds and aldehydes.

Hence, the preparation of ketone compounds from the corresponding oximes is a radical new development in organic chemistry. The general approach used for deoximation involves oxidation using various oxidants, such as pyridinium fluorochromate/Al_2O_3 [5], permanganate/Al_2O_3 [6], *N*-chloro-3-methyl-2,6-diphenylpiperidin-4-one [7], bismuth compounds [8], *N*,*N*-dibromo-*N*,*N*-1,2-ethanediyl*bis*-(*p*-toluenesulfonamide) [9], ammonium persulfate/SiO_2 [10], *N*-haloamides [11], dimethyl dioxirane [12], 2-iodylbenzoic acid [13], Dess-Martin periodinane [14] or manganese triacetate [15]. Most of the reagents used in these deoximation methods suffer from different disadvantages such as long reaction times, severe environmental pollution, generation of over oxidation products and difficult product isolation. Hence, inexpensive and clean oxidants for deoximation reactions are extremely important and significant from environmental and economical viewpoints in green chemistry.

In this article, synthesis of 1,5-benzodiazepine derivatives through the conversion of oximes into the corresponding carbonyl compounds in the presence of dichloramine-T (*N*,*N*-

dichloro-4-toluenesulfonamide) is reported. In the course of present investigations, it is found that dichloramine-T can be an efficient reagent for the conversion of oximes into the corresponding carbonyl compounds. According to the reaction mechanism, the reaction of dichloramine-T with an oxime proceeds through an initial electrophilic attack of a positive chlorine (A) atom provided by dichloramine-T on the oxime nitrogen. Subsequent nucleophilic addition of (B) water followed by elimination of chlorohydroxylamine gives the target compound [16].

EXPERIMENTAL

Synthesis of acetone from acetone oxime using dichloramine-T: A mixture of acetone oxime (7.3 g, 0.1 mol) and dichloramine-T (24.0 g, 0.1 mol) in 5 mL of H_2O and 50 mL of acetonitrile was stirred at room temperature for 15 min. Afterwards, acetone was obtained in high yield through simple distillation (5.22 g, 90%).

Synthesis of acetophenone from acetophenone oxime using dichloramine-T: A solution of acetophenone oxime (1.36 g, 10^{-2} mol) and dichloramine-T (2.4 g, 10^{-2} mol) in 2 mL of H_2O and 10 mL of acetonitrile was refluxed under stirring for 20 min. After it had cooled to room temperature, the mixture was diluted with 20 mL of water and 50 mL of CH_2Cl_2. The organic layer was separated from residual water, dried ($MgSO_4$) and distilled. The remaining sticky oil was purified using flash column chromatography on silica gel (*n*-hexane:ethyl acetate = 10:1 v/v) to give 1.08 g of acetophenone (yield: 90%).

Synthesis of benzophenone from benzophenone oxime using dichloramine-T: Benzophenone oxime (0.20 g, 1 mmol) and dichloramine-T (0.24 g, 1 mmol) were added to a mixture of 2 mL of H_2O and 10 mL of acetonitrile. The reaction mixture was refluxed under stirring for 20 min. After it had cooled to room temperature, the mixture was diluted with 20 mL of water and neutralized with aqueous NaOH. The aqueous solution was extracted with dichloromethane (50 mL). The dichloromethane extract was washed with water and dried ($MgSO_4$). The CH_2Cl_2 was removed under aspirator pressure and the remaining sticky oil was purified using flash column chromatography on silica gel(*n*-hexane:ethyl acetate = 5:1 v/v) to yield benzophenone (0.17 g, 93%).

Synthesis of 2,4,4-trimethyl-3*H*-5-hydro-1,5-benzodiazepine from acetone oxime using dichloramine-T: A mixture of acetone oxime (7.3 g, 0.1 mol) and dichloramine-T (24.0 g, 0.1 mol) in 5 mL of H_2O and 50 mL of acetonitrile was stirred at room temperature for 15 min. After 15 min, *o*-phenylenediamine (5.4 g, 5×10^{-2} mol) and polyphosphoric acid (0.16 g) was added in mixture solution. The reaction mixture was stirred at room temperature for 3 h. The reaction mixture adding water 10 mL was stirred and neutralized with 5% $NaHCO_3$ (50 mL). Then, it was extracted with chloroform (3 × 100 mL), and the extract was washed with water and dried ($MgSO_4$). The organic layer was concentrated. Removal of chloroform gave 2,4,4-trimethyl-3*H*-5-hydro-1,5-benzodiazepine as a yellow solid (7.67 g, 85%), which crystallized from *n*-hexane.

Synthesis of 2,4-diphenyl-4-methyl-3*H*-5-hydro-1,5-benzodiazepine from acetophenone oxime using dichloramine-T: A solution of acetophenone oxime (1.36 g, 10^{-2} mol) and dichloramine-T (2.4 g, 10^{-2} mol) in 2 mL of H_2O and 10 mL of acetonitrile was refluxed under stirring for 20 min. After 20 min, *o*-phenylenediamine (1.08 g, 10^{-2} mol) and polyphosphoric acid (0.63 g) was added and stirred at 40-50 ºC for 5 h. The reaction mixture was diluted with water and neutralized with 5% $NaHCO_3$ (50 mL). Then, it was extracted with $CHCl_3$ (3 × 100 mL) and the extract was washed with water and dried ($MgSO_4$). The chloroform was removed under aspirator pressure and the remaining sticky oil was purified using flash column chromatography on silica gel (*n*-hexane:ethyl acetate = 8:1 v/v) to yield 2,4-diphenyl-4-methyl-3*H*-5-hydro-1,5-benzodiazepine (4.35 g, 56%) as a yellow crystalline solid.

Synthesis of 1,5-benzodiazepine derivative from benzophenone oxime using dichloramine-T: Benzophenone oxime (5.0 g, 2.5×10^{-2} mol) and dichloramine-T (6.0 g, 2.5×10^{-2} mol) were added to a mixture of 2 mL of H_2O and 10 mL of acetonitrile. The reaction mixture was refluxed under stirring for 20 min. After 20 min, *o*-phenylenediamine (2.7 g, 2.5×10^{-2} mol) and polyphosphoric acid (0.63 g) was added and stirred at 40-50 ºC for 5 h. The reaction mixture was diluted with water and neutralized with 5% $NaHCO_3$ (50 mL). Then, it was extracted with chloroform (3 × 100 mL), and the extract was washed with water and dried over $MgSO_4$. This reaction identified by TLC and did not occur.

Reaction of *o*-phenylenediamine with acetone prepared through deoximation in the presence of polyphosphoric acid: A solution of *o*-phenylenediamine (5.4 g, 5×10^{-2} mol), acetone (20 g, 3×10^{-2} mol) and polyphosphoric acid (0.16 g) was stired at room temperature for 3 h. The reaction mixture was stirred with water and neutralized with 5% $NaHCO_3$ (50 mL). Then, it was extracted with $CHCl_3$ (3 × 100 mL) and the extract was washed with water and dried over $MgSO_4$. The organic layer was concentrated. Removal of chloroform gave 2,4,4-trimethyl-3*H*-5-hydro-1,5-benzodiazepine 4 as a yellow solid (7.67 g, 85%), which crystallized from *n*-hexane.

Reaction of *o*-phenylenediamine with acetophenone prepared through deoximation in the presence of SiO_2. To a solution of *o*-phenylenediamine (2.70 g, 2.5×10^{-2} mol) and acetophenone (8.67 g, 7×10^{-2} mol), a catalytic quantity of SiO_2 (0.08 g) was added, and the reaction mixture was stirred at 40-50 ºC for 5 h. The mixture was extracted with 10% aqueous $NaHCO_3$. The organic layer was dried over $MgSO_4$, filtered, and concentrated. The residue was chromatographed on silica gel (*n*-hexane:ethyl acetate = 8:1 v/v) to yield 2,4-diphenyl-4-methyl-3*H*-5-hydro-1,5-benzodiazepine (4.06 g, 52%) as a yellow crystalline solid.

General procedure for synthesis of benzodiazepine derivatives using polyphosphoric acid with heteroaromatic ketones prepared through deoximation. Polyphosphoric acid (0.16 g) was added to a solution of 1,2-phenylenediamine (2.70 g, 2.5×10^{-2} mol) and a heteroaromatic ketone (5×10^{-2} mol) in chloroform (15 mL), and the resulting solution was stirred at 40-45 ºC for 5 h. After it was stirred for 6-8 h, the reaction mixture was diluted with water and neutralized with 5% $NaHCO_3$ (50 mL). Then it was removed with chloroform (3 × 100 mL), and the extract was washed with water and dried over $MgSO_4$. The chloroform was then removed under reduced pressure to

give a sticky oil that was purified using flash column chromatography on silica gel (*n*-hexane:ethyl acetate).

General procedure for quinoxaline: Formation in methanol (20 mL) solution of 1,2-phenylenediamine (2.70 g, 2.5. × 10^{-2} mol) and acetylpyridine (2.5 × 10^{-2} mol) catalytic amount of SiO_2 (0.08g) and 10% HCl (2 mL) were added and stirred. The reaction mixture was refluxed for 5 h. After refluxing for 5 h, the reaction mixture was diluted with water and neutralized with 5% $NaHCO_3$ (50 mL). It was extracted with chloroform (3 × 100 mL). The extract was washed with water and dried ($MgSO_4$). The chloroform was then removed under aspirator pressure and the remaining sticky oil was separated by flash column chromatography on silica gel (*n*-hexane:EtOAc).

2-Pyrazin-2-yl-quinoxaline (4c): m.p.: 133-134 ºC; ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.91 (s, 1H, quinoxalinyl-H), 9.82 (s, 1H, pyridinyl-H), 8.72 (m, 1H, pyridinyl-H), 8.19 (d, 2H, pyridinyl-H), 7.84 (d, 1H, phenyl H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 146.7, 145.8, 144.4, 144.3, 142.3, 142.2, 129.6 129.3; GC/MS: M+ = 208.

2-(Pyrazin-2-yl)benzo[*g*]quinoxaline (4d): m.p.: 236-237 ºC; ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.98 (s, 1H), 8.77 (d, 1H), 8.60 (t, 1H), 8.16 (m, 2H), 7.89 (t, 1H), 7.78 (m, 2H), 7.41 (t, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 146.6, 144.7 144.4, 144.3, 142.3, 142.2, 132.7, 127.3, 127.2,126.4; GC/MS: M+ = 258.

RESULTS AND DISCUSSION

Dichloramine-T ($TsNCl_2$), an *N*-chlorosulfonyl amide derivative, was found to be selective for the conversion of oximes to carbonyl compounds and no product formation was observed. Acetoxime, a ketoxime with two electron-donating alkyl groups, was easily converted into acetone (Table-1, entry 1). After the reaction was finished, the acetone was obtained through simple distillation to react with a diamine. The usefulness of this reagent was further demonstrated by the good to excellent yields of the products obtained from the deoximations of acetophenone oxime (Table-1, entry 2) and benzophenone oxime (Table-1, entry 3) in shorter reaction times. It is found that among all deoximation reagents, dichloramine-T was the fastest and most efficient.

TABLE-1
CONVERSION OF OXIMES INTO THE CORRESPONDING CARBONYL COMPOUNDS USING DCT

Entry	R1	R2	Temp.	Time (min)	Yield[a] (%)
1	Me	Me	r.t.	10	99
2	Ph	Me	Reflux	10	99
3	Ph	Ph	Reflux	10	98

[a]All products were characterized using GC/MS and compared with authentic samples; r.t. = room temperature.

When *o*-phenylenediamine was treated acetone obtained using dichloramine-T at room temperature for 3 h, a yellow crystalline solid (**1a=2a**) was produced (Table-2). Its structure was assigned on the basis of NMR, which appeared singlets at δ 1.33 and δ 2.36 ppm, representing two methyl protons and one methyl proton, respectively. The C3-methylene protons gave at δ 2.22 ppm and the N5 proton appeared at δ 2.97 ppm. The MS spectrum displayed a protonated molecular ion peak at *m/z* 188 corresponding to the molecular formula $C_{12}H_{16}N_2$. Similar results were obtained when polyphosphoric acid (isolated yield: 85%) and SiO_2 (isolated yield: 96%) were added to the reaction mixture. In the case of acetophenone obtained using dichloramine-T, a yellow crystalline solid (**1e=2e**) was produced after 5-10 h (Table-3). Its structure was assigned on the basis

TABLE-2
CONVERSION OF OXIMES INTO THE CORRESPONDING CARBONYL COMPOUNDS USING DCT

Amine	Ketone	Catalyst	Time (h)	Product	Yield (%)[a]
1,2-Phenylenediamine	Acetone	PPA	3	**1a, 2a**	83
		SiO_2	3		95
4-Chloro-1,2-phenylenediamine	Acetone	PPA	3	**1b, 2b**	83
		SiO_2	3		87
3,4-Diaminotoluene	Acetone	PPA	3	**1c, 2c**	82
		SiO_2	3		81
4-Nitro-1,2-phenylenediamine	Acetone	PPA	3	**1d, 2d**	92
		SiO_2	3		75

[a]Isolated yield; PPA = polyphosphoric acid

TABLE-3
YIELD OF SYNTHESIZED 1*H*-1,5-BENZODIAZEPINES (**1e-h**, **2e-h**) FROM ACETOPHENONE OBTAINED USING DCT

Amine	Ketone	Catalyst	Time (h)	Product	Yield (%)[a]
1,2-Phenylenediamine	Acetophenone	PPA	5	**1e, 2e**	46
		SiO_2	5		42
4-Chloro-1,2-phenylenediamine	Acetophenone	PPA	10	**1f, 2f**	74
		SiO_2	10		72
3,4-Diaminotoluene	Acetophenone	PPA	10	**1g, 2g**	83
		SiO_2	10		81
4-Nitro-1,2-phenylenediamine	Acetophenone	PPA	10	**1h, 2h**	81
		SiO_2	10		90

[a]Isolated yield; PPA = polyphosphoric acid

of nuclear magnetic resonance (NMR) spectroscopy, which gave a singlet at δ 1.75 ppm and two doublets at δ 3.03 ppm, representing one methyl proton and two methylene protons, respectively, while the 5N proton appeared at δ 3.51 ppm. Mass spectrometry (MS) gave a protonated molecular ion peak at *m/z* 312 corresponding to the molecular formula $C_{22}H_{20}N_2$ (Table-3). A possible mechanism for the formation of benzodiazepines is that to begin with, that each amine of *o*-phenylenediamine attacks the carbonyl group of a ketone, giving diimine intermediate A (Fig. 1). A 1,3-shift of a methyl hydrogen then occurs to afford isomeric enamine D, which cyclizes to produce a seven-membered ring (E) (Fig. 2). Treatment of substituted *o*-phenylenedi-amines (4-chloro-1,2-phenylenediamine, 3,4-diaminotoluene, 4-nitro-1,2-phenylenediamine) with acetone obtained using dichloramine-T in the presence of polyphosphoric acid (or SiO_2) afforded **1b** and **2b**, **1c** and **2c**, and **1d** and **2d** in excellent yields (Table-2). When 1,2-diaminobenzene was treated with 2-acetylpyridine in the presence of polyphosphoric acid at 40-45 ºC for 6 h, a yellow crystalline solid (**3a**) was produced (67%). Its structure was assigned on the basis of ^1H & ^{13}C NMR and GC/MS (Table-4). Specially, we report synthesis of quinoxaline derivation with diamine (1,2-phenylene diamine or 1,2-naphthalene diamine) and heterocyclic ketone (acetylpyridines or acetylpyrazines) in aqueous 10% conc. HCl solution and SiO_2. It is found that treatment of 1,2-phenylene diamine with heterocyclic ketone in the presence of conc. HCl at room temperature afforded only benzodiazepine derivative in good yield. On the other hand, when diamine (1,2-phenylene diamine or 1,2-naphthalene diamine) with heterocyclic ketone (acetylpyridine or acetylpyrazines) in the presence of conc. HCl and SiO_2 was refluxed, quinoxaline derivatives yellow crystalline solids were isolated in high yields. This result indicates that not 2 equiv. of diamine but 1 equiv. of diamine in reacted. Seeing the plausible formation mechanism of quinoxaline, first of all, amino group of diamine attaches carbonyl group of ketone to give the imine. Then a 1,3-shift of hydrogen attached methyl group then occur to provide isomeric enamine F. Enamine F changed into intermediate G by the movement of the lone-paired electron of nitrogen. Then, proton shift, ring formation and proton elimination occur to afford six-membered ring intermediate H (Fig. 3). In order to form quinoxaline, the aromatization subsequent to the formation of the intermediate H occurred. Yields of synthesized quinoxalines with diamine (1,2-phenylene diamine or 1,2-naphthalene diamine) and heterocyclic ketone (acetylpyridines or acetylpyrazines) are shown in Table-5.

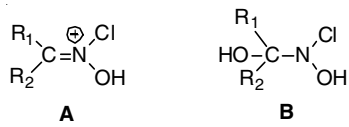

Fig. 1. Product A, B

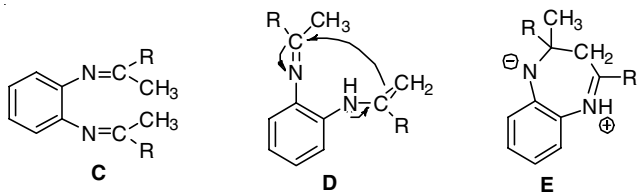

Fig. 2. Product C, D, E

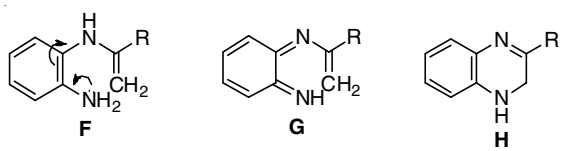

Fig. 3. Product F, G, H

Conclusion

In conclusion, herein we synthesized new benzodiazepine derivatives with heteroaromatic ketones by using polyphosphoric acid, conc. HCl or SiO_2. Dichloramine-T was found to be an efficient reagent for the conversion of oximes into their corresponding carbonyl compounds. The conversion was very efficient and fast and gave better product yields in comparison to reactions that use previously reported deoximation reagents. The reagent is very mild, and byproducts and overoxidation products were not formed. The peculiarity of dichloramine-T

TABLE-4
YIELD OF SYNTHESIZED 1*H*-1,5-BENZODIAZEPINES (**3a-e**) FROM ACETOPHE OBTAINED USING DCT

Amine	Ketone	Catalyst	Time (h)	Product	Yield (%)[a]
1,2-Phenylenediamine	2-Acetyl pyridine	PPA	6	**3a**	67
	3-Acetyl pyridine	PPA	5	**3b**	73
	4-Acetyl pyridine	PPA	6	**3c**	78
	2-Acetyl pyrrole	PPA	8	**3d**	81
	2-Acetyl thiophene	PPA	7	**3e**	63

[a]Isolated yield; PPA = polyphosphoric acid

TABLE-5
YIELD OF SYNTHESIZED QUINOXALINES (**4a-d**) FROM HETEROAROMATIC KETONES

Amine	Ketone	Catalyst	Time (h)	Product	Yield (%)[a]
1,2-Phenylenediamine	2-Acetyl pyridine	Conc. HCl, SiO_2	5	2-Pyridylquinoxaline	84
	3-Acetyl pyridine		8	3-Pyridylquinoxaline	79
1,2-Naphtalenediamine	2-Acetyl pyrazine		8	2-Pyrazylquinoxaline	75
	2-Acetyl pyrazine		5	2,2-Pyrazylbenzo[g]quinoxaline	64

[a]Isolated yield

as a deoximation reagent lies in the fact that sensitive functional groups survive the reaction. In this article, we have reported the synthesis of benzodiazepine derivatives though the conversion of oximes into the corresponding carbonyl compounds in the presence of dichloramine-T. And also we obtained novel quinoxaline derivatives with 1,2-diamine (1,2-phenylene diamine or 1,2-naphthalene diamine) and heterocyclic ketone (acetylpyridines or acetylpyrazines) in aqueous 10% conc. HCl and SiO_2.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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