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ORIGINAL PAPER

Selective conversion of C=N bonds to their corresponding carbonyl compounds by the tribromoisocyanuric acid/wet SiO_2 system as a novel reagent

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Abstract Tribromoisocyanuric acid/wet SiO_2 was used for the conversion of C=N bonds to their corresponding carbonyl compounds in oximes, semicarbazones, azines, and Schiff bases. The interesting feature of this system is that in those oximes, semicarbazones, azines, and Schiff bases which have conjugated or unconjugated C=C bonds, the C=N bond will selectively change to the relevant C=O bond while the conjugated or unconjugated C=C bond will remain intact.

Keywords Tribromoisocyanuric acid · Wet SiO_2 · Heterogeneous catalyst · C=N bond · C=O bond

Introduction

Oximes, azines, semicarbazones, and Schiff bases have been employed as ketone or aldehyde equivalent functional groups in organic synthesis. Protection and subsequent deprotection of a functional group is almost inevitable in multistep organic synthesis [1]. Protection of carbonyl compounds as C=N derivatives is of great interest to organic chemists, as they are highly stable and readily prepared compounds [2]. Therefore, regeneration of carbonyl compounds from their derivatives provides an attractive method for the regeneration of aldehydes and ketones [3]. Oximes can be prepared from noncarbonyl compounds [4], and regeneration of the carbonyl compounds from oximes therefore provides an alternative method for the preparation of aldehydes and ketones [5–32]. Some methods of carbonyl compound generation from hydrazones, oximes, semicarbazones, and hydrazo compounds [33–44] involve reagents which are often hazardous or very toxic, expensive, or not readily available. Moreover, many need to be freshly prepared or the reactions require drastic conditions, long reaction times, and tedious workup. Thus milder, selective, nonhazardous, and inexpensive reagents are still in demand.

Although many methods have been developed for the oxidative cleavage of oximes, hydrazones, and semicarbazones to the corresponding carbonyl compounds [45, 46], little attention has been paid to the cleavage of the carbon–nitrogen double bonds of azines [47] and Schiff bases.

Recently a new methodology was reported for the preparation of tribromoisocyanuric acid (TBCA) by the reaction of an aqueous solution of Oxone$^{®}$ with sodium cyanurate in the presence of KBr [48]. This methodology is simpler and considerably less expensive than the older method [49]. There are some reports on the application of TBCA in organic transformations such as regioselective bromination of deactivated and activated aromatic rings [50, 51], oxidative coupling of thiols [52], nitration of phenols [53], and regioselective halogenation of *β*-dicarbonyl compounds [54].

In continuation of our studies on the applications of *N*-halo reagents in organic functional group transformations [55] and cleavage of the C=N bond to its corresponding carbonyl functional group [56], we found that TBCA is a suitable reagent for the regeneration of carbonyl compounds in the presence of wet SiO_2 under mild conditions (Scheme 1).

Results and discussion

We have studied several substrates to show that TBCA/wet SiO_2 can serve as an efficient heterogeneous catalytic system for the cleavage of their C=N bonds to the

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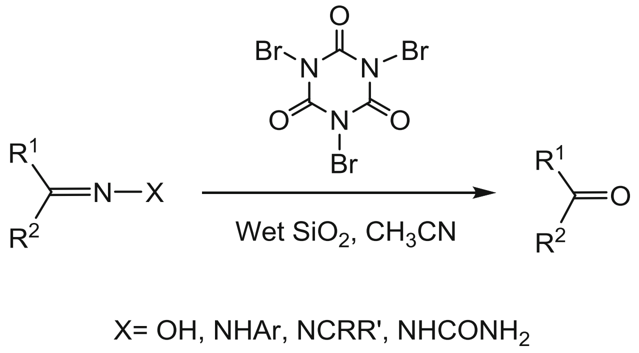


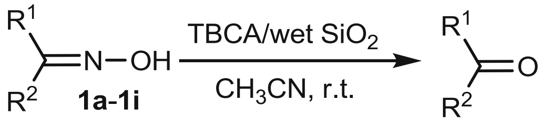
Scheme 1

corresponding carbonyl compounds without any overoxidation. The results are summarized in Tables 1, 2, 3, 4. The comparison between different reagents and conditions (screening data) is depicted in Table 5.

The applied method has advantages in terms of yields, heterogeneous nature, cheapness and availability of the reagents, short reaction time, and easy work-up. Products were isolated by filtering the mixture and evaporating the solvent. Yields are good to excellent and the procedure is simple and convenient. We believe that TBCA will be a suitable reagent for various functional group transformations [55].

TBCA behaves as a noble reagent which selectively cleaves the C=N bond without interfering with other sensitive compounds and functional groups such as phenol (Table 3, entry **3e**; Table 4, entries **4c**, **4d**, and **4e**), conjugated C=C bonds (Table 1, entry **1g**), and unconjugated C=C bonds (Table 2, entry **2k**).

Table 1 Conversions of oximes to their corresponding carbonyl compounds by the TBCA/wet SiO_2 system

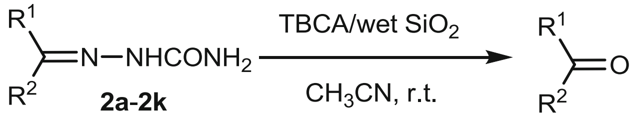


No.	R^1	R^2	Time/h	Yield/%[a]
1a	CH_3	C_6H_5	3	96
1b	H	4-Cl-C_6H_4	1	98
1c	H	4-OCH_3-C_6H_4	1.5	96
1d	CH_3	4-CH_3-C_6H_4	3	94
1e	H	4-CH_3-C_6H_4	1.33	92
1f	CH_3CH_2	C_6H_5	2.33	92
1g	H	C_6H_5CH=CH	1.5	92
1h	H	2-OCH_3-C_6H_4	1.5	95
1i	CH_3	CH_3CH_2	2.5	90

TBCA/substrate 0.33:1 mmol + 0.5 g wet SiO_2

[a] Isolated yields

Table 2 Conversions of semicarbazones to their corresponding carbonyl compounds by the TBCA/wet SiO_2 system

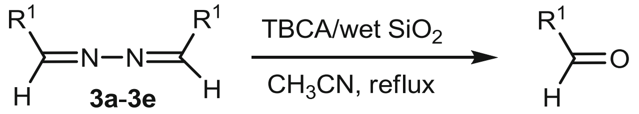


No.	R^1	R^2	Time/h	Yield/%[a]
2a	CH_3	C_6H_5	4	95
2b	CH_3	2-Thienyl	4.5	98
2c	H	4-Cl-C_6H_4	5.5	92
2d	H	4-OCH_3-C_6H_4	2.45	90
2e	H	4-NO_2-C_6H_4	4	80
2f	H	C_6H_5	3	92
2g	C_6H_5	C_6H_5	10	60
2h	H	2,4-Di-OCH_3-C_6H_3	4	78
2i	CH_3	4-Br-C_6H_4	6	94
2j	H	2-Naphthyl	3.5	92
2k	CH_3	$(CH_3)_2CHCH$=$CHCH_2$	3	83

TBCA/substrate 0.33:1 mmol + 0.5 g wet SiO_2

[a] Isolated yields

Table 3 Conversions of azines to their corresponding carbonyl compounds by the TBCA/wet SiO_2 system



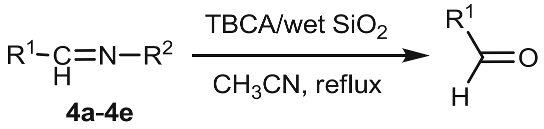
No.	R^1	Time/h	Yield/%[a]
3a	C_6H_5	0.75	95
3b	2-OCH_3-C_6H_4	1.5	90
3c	4-OCH_3-C_6H_4	1	95
3d	4-Cl-C_6H_4	1	85
3e	3-HO-C_6H_4	1.5	90

TBCA/substrate 0.33:1 mmol + 0.5 g wet SiO_2

[a] Isolated yields

Wet SiO_2 or the TBCA/dry SiO_2 system alone does not carry out any reactions [60]. In order to prove the role of the TBCA/wet SiO_2 system, the results of the conversion of some suitable C=N containing compounds into the corresponding carbonyl compounds were compared by application of the following two different conditions: (i) TBCA/wet SiO_2, CH_3CN, and (ii) TBCA/aqueous CH_3CN. Method (i) is more efficient because the yields were about 90% whereas in method (ii) the yields were about 60%, which shows the interesting role of wet SiO_2 (these yields refer to the compound bearing the 4-Cl-C_6H_4 group: Table 1, entry **1b**, 98%; Table 2, entry **2c**, 92%; Table 3, entry **3d**,

Table 4 Conversions of Schiff bases to their corresponding carbonyl compounds by the TBCA/wet SiO_2 system



No.	R^1	R^2	Time/h	Yield/%[a]
4a	4-$(CH_3)_2N$-C_6H_4	4-CH_3-C_6H_4	1	90
4b	4-Cl-C_6H_4	4-Cl-C_6H_4	0.75	95
4c	4-$(CH_3)_2N$-C_6H_4	2-HO-C_6H_4	2.5	80
4d	2-HO-C_6H_4	2-OH-5-CH_3-C_6H_3	5	48
4e	2-HO-C_6H_4	4-OH-C_6H_4	1	50

TBCA/substrate 0.33:1 mmol + 0.5 g wet SiO_2

[a] Isolated yields

85%; and Table 4, entry **4b**, 95%). In method (i) the wet SiO_2 acts as a useful heterogeneous catalyst that releases water gradually into the reaction mixture to prevent it from becoming a two-phase system, and consequently the C=N containing compounds will remain in the reaction mixture [57, 59, 60]. In the absence of wet SiO_2 and presence of aqueous CH_3CN, the substrate will be substituted by water and will go out from the reaction mixture, so the yield will be dramatically decreased.

So far, hydrolysis of the C=N compounds has generally been carried out in aqueous acidic media. Because the acidic conditions will hydrolyze some sensitive functional groups such as nitrile, in our applied conditions an acidic medium was avoided. The HOBr species causes hydrolysis of these substrates more efficiently than the usual acids and the bromonium ion is a more powerful Lewis acid to be coordinated to the nitrogen atom, which is an interesting feature of this method. The proposed mechanism is depicted in Scheme 2 [55, 57].

To support the proposed mechanism, an extra reaction was carried out with 2,3-dimethylbut-2-ene as a suitable trapping agent and the bromohydrin compound was produced. In C=N containing compounds possessing conjugated or an unconjugated C=C bond, the bromohydrin formation does not occur (the C=N bond is probably more reactive than the C=C bond). These observations indicate that the applied catalytic system selectively converts the C=N bonds into their carbonyl compounds while a conjugated or unconjugated C=C bond will remain intact.

This method is also suitable for compounds containing acid-sensitive functional groups such as –CN, which will not participate in the reaction. In addition, the original aldehydes were obtained with excellent yields and their oxidation to the corresponding acids was not observed, which is another advantage of the proposed system.

The following three factors probably influence the reactivity of compounds **3e**, **4d**, and **4e**:

1. The α effect between the nitrogen atoms in **3e** (=N–N=) causes the nitrogen atom to abstract the Br^+ better rather than the nitrogen atom in Schiff bases **4d** and **4c** (–CH=N–) as a result of the lone pair repulsion effect between the two nitrogen atoms.
2. The OH group in the *meta* position of **3e** exerts only an inductive effect, so the electron density of the carbon atom in the C=N bond will decrease and consequently the nucleophilic attack of a water molecule will be favored, whereas in **4d** and **4e** the resonance effect of the OH group in the *ortho* position increases the electron density of the carbon atom in the C=N bond, so the nucleophilic attack of a water molecule will be disfavored.
3. The hydrogen bonding between the OH and the nitrogen atom in **4d** and **4e** will decrease the tendency of the nitrogen atom to abstract the Br^+. Consequently the yield of **3e** is much greater than that of **4d** and **4e**, and the reaction time is lower. The above explanations are roughly applicable for **4c** as well.

Experimental

General procedure

The substrate (1.00 mmol of oximes, semicarbazones, azines, or Schiff bases, which were prepared by standard procedures [61]), wet SiO_2 (0.5 g, SiO_2 powder with 60 mesh/H_2O, 50% w/w), and TBCA (0.33 mmol, the molar ratio of TBCA and wet SiO_2 to the substrate was optimized, see footnotes of the tables) were added to 5 cm^3 acetonitrile, and the mixture was stirred for the specific time at room temperature (for oximes, semicarbazones), or reflux conditions (for azines and Schiff bases). After completion of the reaction which was monitored by TLC (eluent, acetone/*n*-hexane 1:8), 5–15 cm^3 CH_2Cl_2 was added and the mixture was filtered. Aqueous $NaHCO_3$ (10 cm^3, 10% w/w) was then added to the filtrate, the organic layer separated, anhydrous $MgSO_4$ was added, and the mixture filtered. Evaporation of the solvent gave the corresponding carbonyl compounds in good to excellent yields.

All products are known and characterized by comparison of their spectra (IR and NMR), melting points, and physical data with authentic samples [62–64].

Note: Isocyanuric acid will not dissolve in the organic solvents and will be absorbed by water existing at the surface of wet SiO_2, so it will be removed by simple filtration.

Table 5 Screening data (conversion of C=N to C=O bonds by different reagents and conditions)

Reagent	R^1	R^2	Time/h	Yield/%	Substrate	Ref.
TBCA, wet SiO_2, CH_3CN, rt	CH_3	C_6H_5	3	96	Oxime	This work
PVCBSA[a], CCl_4, reflux			3	92		[22]
CDBACN[b], H_2SO_4, dioxane, reflux			4	65		[11]
CTACN[c], H_2SO_4, dioxane, reflux			3	75		[11]
BNBTS[d], CCl_4, rt			2	95		[32]
$CuCl_2 \cdot 2H_2O$, CH_3CN/H_2O (4:1), reflux			3	92		[6]
MnTPPCl[e], benzaldehyde, toluene, 50 °C			2	99		[42]
NBS, a few drops of water, rt, solvent-free			2*	86		[25]
HMTAB[f], a few drops of water, rt, solvent-free			2*	82		[25]
Amberlyst-15, $NaNO_2$, O_2, CH_2Cl_2/H_2O (5:1)			9	85		[18]
DMDBH[g], wet SiO_2, CH_2Cl_2, rt			2	91		[57]
$Al(NO_3)_3 \cdot 9H_2O$, NaBr, CH_2Cl_2, rt			1.83	98		[58]
PTA[h], CH_3CN, mercury lamp			6	93		[44]
TBCA, wet SiO_2, CH_3CN, rt	CH_3	4-CH_3-C_6H_4	3	94		This work
BNBTS, CCl_4, rt			2	88		[32]
DMDBH, wet SiO_2, CH_2Cl_2, rt			2.5	90		[57]
TBCA, wet SiO_2, CH_3CN, rt	H	4-CH_3-C_6H_4	1.33	92		This work
PVCBSA, CCl_4, reflux			3	90		[22]
$Al(NO_3)_3 \cdot 9H_2O$, NaBr, CH_2Cl_2, rt			3.5	90		[58]
$Al(NO_3)_3 \cdot 9H_2O$, CH_2Cl_2, rt			22.5	88		[58]
TBCA, wet SiO_2, CH_3CN, rt	H	$C_6H_5CH{=}CH$	1.5	92		This work
PVCBSA, CCl_4, reflux			3	93		[22]
$CuCl_2 \cdot 2H_2O$, $CH_3CN:H_2O$ (4:1), reflux			1.5	92		[6]
NBS, a few drops of water, rt, solvent-free			2*	63		[25]
HMTAB, a few drops of water, rt, solvent-free			2*	72		[25]
PTA, CH_3CN, mercury lamp			6	10		[44]
TBCA/wet SiO_2, CH_3CN, rt	CH_3CH_2	C_6H_5	2.33	92		This work
Amberlyst-15, $NaNO_2$, O_2, CH_2Cl_2/H_2O (5:1)			9	82		[18]
TBCA, wet SiO_2, CH_3CN, rt	H	4-Cl-C_6H_4	1	98		This work
PVCBSA, CCl_4, reflux			2	90		[22]
BNBTS, CCl_4, rt			1	91		[32]
DMDBH, wet SiO_2, CH_2Cl_2, rt			2	90		[57]
NBS, a few drops of water, rt, solvent-free			4*	95		[25]
HMTAB, a few drops of water, rt, solvent-free			10*	96		[25]
Amberlyst-15, $NaNO_2$, O_2, CH_3CN/H_2O (5:1)			1	76		[18]
$Zr(HSO_4)_4$, wet SiO_2, hexane, reflux			1	80		[59]
Amberlyst-15, $NaNO_2$, O_2, CH_2Cl_2/H_2O (5:1)			5	95		[18]
TBCA, wet SiO_2, CH_3CN, rt	H	4-OCH_3-C_6H_4	1.5	96		This work
$CuCl_2.2H_2O$, CH_3CN/H_2O (4:1), reflux			0.5	97		[6]
$Al(NO_3)_3 \cdot 9H_2O$, NaBr, CH_2Cl_2, rt			3.25	94		[58]
TBCA, wet SiO_2, CH_3CN, rt	H	2-OCH_3-C_6H_4	1.5	95		This work
PVCBSA, CCl_4, reflux			2	96		[22]
BNBTS, CCl_4, rt			1	95		[32]

Table 5 continued

Reagent	R^1	R^2	Time/h	Yield/%	Substrate	Ref.
TBCA, wet SiO_2, CH_3CN, rt	C_6H_5	C_6H_5	10	60	Semicarbazone	This work
DMDBH, wet SiO_2, CH_2Cl_2, rt			2.5	94		[57]
TBCA, wet SiO_2, CH_3CN, rt	H	4-NO_2-C_6H_4	4	80		This work
DMDBH, wet SiO_2, CH_2Cl_2, rt			2	92		[57]
TBCA, wet SiO_2, CH_3CN, rt	H	4-OCH_3-C_6H_4	2.45	90		This work
$Zr(HSO_4)_4$, wet SiO_2, hexane, reflux			0.03	90		[59]
TBCA, wet SiO_2, CH_3CN, rt	CH_3	C_6H_5	4	95		This work
$Zr(HSO_4)_4$, wet SiO_2, hexane, reflux			0.25	90		[59]
TBCA, wet SiO_2, CH_3CN, rt	CH_3	4-Br-C_6H_4	6	94		This work
$Zr(HSO_4)_4$, wet SiO_2, hexane, reflux			0.12	82		[59]
TBCA, wet SiO_2, CH_3CN, reflux	C_6H_5		0.75	95	Azine	This work
$Zr(HSO_4)_4$, wet SiO_2, hexane, reflux			3.75	89		[59]
TBCA, wet SiO_2, CH_3CN, reflux	2-OCH_3-C_6H_4		1.5	90		This work
$Zr(HSO_4)_4$, wet SiO_2, hexane, reflux			2.8	84		[59]

[a] Poly[4-vinyl-*N*,*N*-dichlorobenzenesulfonamide]

[b] Cetyldimethylammonium cerium nitrate

[c] Cetyltrimethylammonium cerium nitrate

[d] *N*,*N*-Dibromo-*N*,*N*-1,2-ethanediylbis(*p*-toluenesulfonamide)

[e] Manganese *meso*-tetraphenylporphyrin chloride

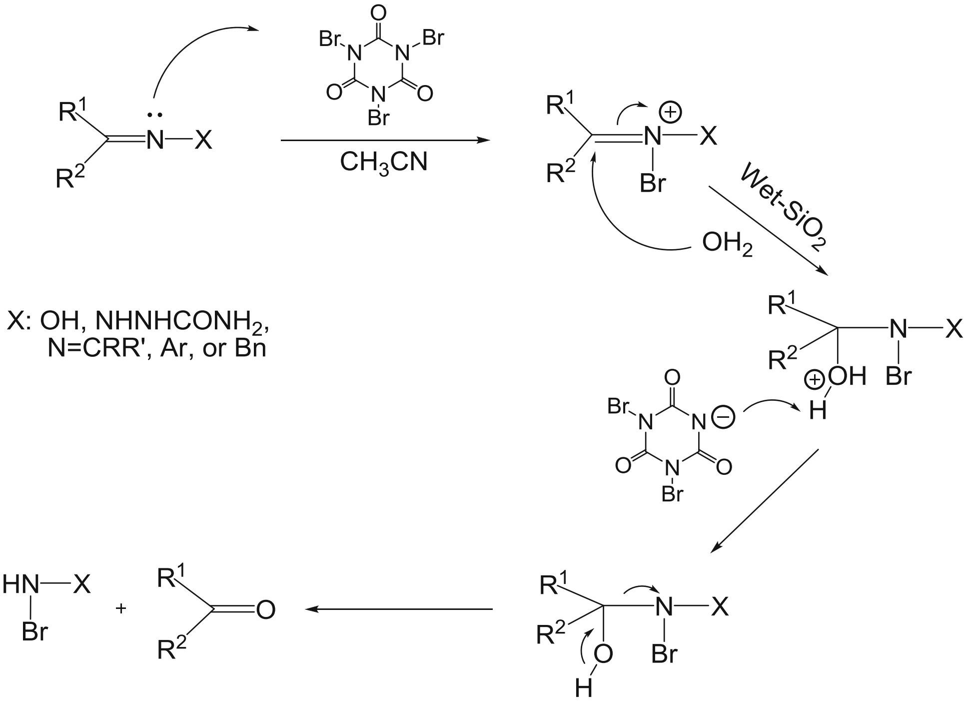
[f] Hexamethylenetetramine/bromine

[g] 1,3-Dibromo-5,5-dimethylhydantoin

[h] Platinum(II) terpyridyl acetylide

* Time in minutes

Scheme 2



*Procedure for HOBr trapping using 2,3-dimethylbut-2-ene (**1**) with TBCA/wet SiO_2*

The substrates (1.00 mmol of **1**), wet SiO_2 (0.5 g, SiO_2 powder/H_2O, 50% w/w), and TBCA (0.33 mmol) were added to 5 cm^3 acetonitrile at room temperature or reflux (see general procedure) and the mixture was stirred for 3 h. Monitoring the reaction by TLC showed that **1** had participated in the reaction [48]. Similar results were obtained when the reaction was conducted by HOBr itself.

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