



The application of *N,N'*-dibromo-*N,N'*-1,2-ethanediylbis (*p*-toluenesulphonamide) as a powerful reagent for deoxygenation of various oximes

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Abstract—*N,N'*-Dibromo-*N,N'*-1,2-ethanediylbis (*p*-toluenesulphonamide) [BNBTS] **2** was reacted with oximes and converts them to their corresponding carbonyl compounds in good yields under mild conditions. © 2001 Elsevier Science Ltd. All rights reserved.

Protection of carbonyl compounds as oximes is of great interest to synthetic chemists, as they are readily prepared and highly stable compounds.¹ Oximes are extensively used for characterization of carbonyl compounds and in the preparation of amides via the Beckman rearrangement.² Since oximes can be prepared from non-carbonyl compounds,^{3–7} the regeneration of carbonyl compounds from oximes provides an alternative method for preparation of aldehydes and ketones. Some of the reagents reported for deoxygenation^{8–10} are often hazardous or very toxic, expensive, or not readily available, they need to be freshly prepared or the reactions require drastic conditions, long reaction times and tedious work-up. Thus, a milder, selective, non-hazardous and inexpensive reagent is still in demand.

We now report a convenient method for the deoxygenation of ketone and aldehyde oximes to their corresponding carbonyl compounds using a new reagent (BNBTS) **2** that was prepared from *N,N'*-1,2-ethanediylbis (*p*-toluenesulphonamide)[**1**] (Fig. 1).¹¹

The reaction of oximes (**3**) with BNBTS in CCl₄ afforded carbonyl compounds (**4**) without side products (Scheme 1).

The results of the conversion of various ketoximes and aldoximes to ketones and aldehydes are presented in Table 1. The products of the reaction with BNBTS were isolated simply by filtering off (**1**) and evaporating

the solvent from the filtrate. The method has advantage in terms of yields, simplicity of the reaction conditions, shorter reaction times and no side products. The recovered starting material (**1**), was rebrominated and used many times without reducing the yield.

The oximes were prepared by a standard procedure. The purity of the compounds was checked by TLC. A mixture of oxime (5 mmol), carbon tetrachloride (15 ml) and BNBTS (5 mmol) was stirred at room temperature for the specified time (Table 1). The reaction was monitored by TLC. After completion of the reaction,

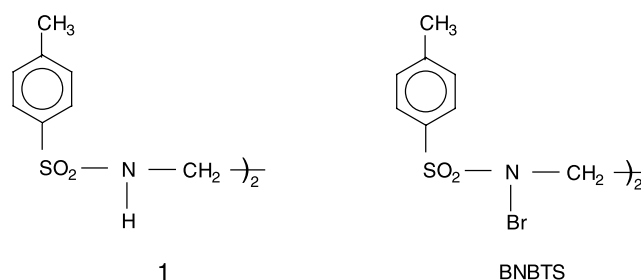
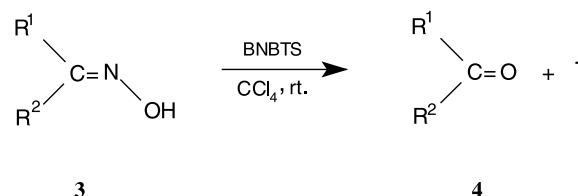


Figure 1.



Scheme 1.

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Table 1. Deoximation with BNBTS

Entry	Yield (%)	R ¹	R ²	Product ^a	Reaction times (h)
1	95	CH ₃	C ₆ H ₅	Acetophenone	2
2	94	CH ₃	<i>p</i> -ClC ₆ H ₅	<i>p</i> -Chloroacetophenone	4
3	97	CH ₃	<i>p</i> -MeOC ₆ H ₅	<i>p</i> -Methoxyacetophenone	2
4	96	CH ₃	<i>p</i> -BrC ₆ H ₅	<i>p</i> -Bromoacetophenone	2
5	94	C ₆ H ₅	C ₆ H ₅	Benzophenone	4
6	94	C ₆ H ₅	<i>p</i> -ClC ₆ H ₅	<i>p</i> -Chlorobenzophenone	0.5
7	85	H	C ₆ H ₅	Benzaldehyde	2
8	91	H	<i>p</i> -ClC ₆ H ₅	<i>p</i> -Chlorobenzaldehyde	1
9	88	H	<i>p</i> -MeC ₆ H ₅	<i>p</i> -Methylbenzaldehyde	2
10	95	H	<i>o</i> -MeOC ₆ H ₅	<i>o</i> -Methoxybenzaldehyde	1
11	90	C ₆ H ₅	C ₆ H ₅ CH(OH)	Benzoin	2

^a Products were characterized by their physical constants, comparison with authentic samples and IR spectra.

water was added to hydrolyze the intermediate, and the insoluble sulfonamide (**1**) was removed by filtration and washed with cold carbon tetrachloride (10 ml). Removal of the solvent under reduced pressure gave the crude product. Solid products were recrystallized from diethyl ether, oily products being dissolved in ether and the ether solution washed, dried and concentrated.

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