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### Rapid Regeneration of Carbonyl Compounds from Tosylhydrazones with Thallium Trinitrate(TTN)

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## RAPID REGENERATION OF CARBONYL COMPOUNDS FROM TOSYLHYDRAZONES WITH THALLIUM TRINITRATE(TTN)

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**Abstract:** Tosylhy,drazones can be rapidly converted into corresponding carbonyl compounds in good to excellent yields with thallium trinitrate (TTN) under mild conditions.

Tosylhydrazones, which are a class of highly crystalline compounds, can be used for the purification and characterization of aldehydes and ketones. The conversion of tosylhydrazones into corresponding carbonyl compounds is a useful transformation. As a result, a number of methods have been developed for this purpose. Among these, many were based on the oxidative cleavage of

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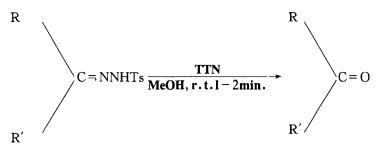
tosylhydrazones with various oxidizing agents such as  $(\mathbf{Bu}_4\mathbf{N})_2\mathbf{S}_2\mathbf{O}_8^{[1]}$ ,

$$Cu(NO_3)_2/SiO_2^{[2]}$$
, PhI(OAC)<sub>2</sub><sup>[3]</sup>, Ts-1/H<sub>2</sub>O<sub>2</sub><sup>[4]</sup>, Na<sub>2</sub>CO<sub>3</sub>· $\frac{3}{2}$ H<sub>2</sub>O<sub>2</sub><sup>[5]</sup>

etc.. Recently, a novel method has been reported for the hydrolysis of tosylhydrazones to their corresponding carbonyl compounds.<sup>[6]</sup>

Thallium trinitrate(TTN) is a versatile oxidizing agent.<sup>[7]</sup> It has been extensively applied to organic synthesis over the last two decades. A · Mckillop etal have reported the oxidative cleavage of oximes, semicarbazones and phenyl hydrazones with TTN. Herein we wish to report the conversion of tosylhydrazones into corresponding carbonyl compounds with TTN. In our experimental work, tosylhydrazones can be rapidly converted into parent aldehydes and ketones in good to excellent yields in MeOH under mild conditions. The carbon-carbon double bonds in  $\alpha$ ,  $\beta$ -unsaturated carbonyl compounds were not affected under reaction conditions. The reactions were completed in one to two minutes. Some results were summarized in Table.





Entry	Product	Yield * (%)
1	(2)— СНО	90
2	сі – (Д)– сно	86
3	0 <sub>z</sub> N −⊘− CHO	87
4	⊚– сн=сн-сно	75
5	о — сн=сн-с-@	81
6	о () () () С-СН,	84
7	0 0 	89
8	()=0	93 <sup>b</sup>
9	O ∐ CH₃CH₂C CH₃	91 <sup><i>b</i></sup>

Table. Oxidative cleavage of tosylhydrazones with TTN.

a. Yields of isolated products.

b. Yields determined by Gas Chromatography.

c.All tosylhydrazones were prepared by known method. [8]

d.Products were characterized by comparison of their melting points, IR and <sup>1</sup> H-NMR spectra with authentic samples.

# General procedure for the oxidative cleavage of tosylhydrazones with TTN:

To a magnetically stirred solution of TTN (2mmol) in 5ml CH<sub>3</sub> OH in a 50ml round-bottom flask was added tosylhydrazone(1mmol)in one portion. The mixture was stirred for 1 - 2 mintues. The evolution of gas(N<sub>2</sub>) and white

precipitate can be seen. Ether (30ml) was added to the reaction mixture. Organic layer was separated and then was washed with water( $20ml \times 3$ ). After being dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, the solvent was removed under reduced pressure. the residue was then purified by preparative TLC on silica gel (hexane/acetate as eluent)

In view of good yield, short reaction time, simple operation and mild reaction conditions, the present procedure provides a useful method for the regeneration of aldehydes and ketones from tosylhydrazones.

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