RESEARCH LETTER

Protection and deprotection chemistry catalyzed by zirconium oxychloride octahydrate (ZrOCl$_2$·8H$_2$O)

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(Received 14 September 2010; final version received 14 March 2012)

An efficient, chemoselective, convenient, and straightforward methodology has been developed for the protection of C=O group of aldehydes/ketones as C=N moiety of hydrazones catalyzed by ZrOCl$_2$·8H$_2$O (10 mol%) in acetonitrile and the same catalyst in methanol oxidatively cleaves C=N moiety of hydrazones to provide parent carbonyl compounds in high yields. The reactions have been performed in aerobic condition. The catalyst is inexpensive, readily available, easy to handle, insensitive to air and moisture, easily recoverable and can be reused and importantly less toxic.

Keywords: protection; deprotection; catalyst; hydrazone; zirconium oxychloride octahydrate

Introduction

As far as the synthesis of complex target molecules is concerned, protection–deprotection is essential (1) and hence serves as a central theme in organic chemistry (2). Hydrazine is regarded as one of the important protective moieties for carbonyl compound in multi-step synthesis of many target molecules (3). Apart from carbonyl protection they also serve as carbanion equivalents in C–C bond forming reactions (4,5). Accordingly, lots of efforts have been exercised for the development of a mild and efficient methodology for masking carbonyl moiety of aldehydes/ketones in the form of hydrazone and the subsequent oxidative cleavage of hydrazones to regenerate the parent carbonyl compounds. But, some of the reported methods (6–21) have one or the other limitations such as requirement of strongly oxidizing or reducing acidic or basic reagents, use of reagents in stoichiometric amounts or more, tedious work up procedure, low yield, longer reaction time, harsh reaction condition, lack of selectivity, associated with environmental hazard and importantly, tolerability to other functional groups. It is also likely that by product formed in the reaction may block the active sites of the catalyst thereby reducing its activity. Importantly, presence of sensitive structural features in molecules restricts the choice of reagents. Hence, there has been considerable interest in this area and still scope is there for further development.

The application of ZrOCl$_2$·8H$_2$O as a catalyst in organic synthesis has attracted our attention as it is relatively cheaper, readily available, easy to handle, insensitive to air and moisture (22) and importantly less toxic (23). This octahydrate of Zirconium oxychloride is a mild Lewis acid having some distinct differences (24,25) from other metal hydrates. An account by Zhang et al. recently reviewed effectiveness of Zirconium-based compounds in many organic transformations, for example addition reaction, rearrangement reaction, protection of common functional groups such as carbonyl, carboxylic acid, amino and hydroxy groups (26) and their subsequent deprotection. Zolfigol et al. described the application of zirconium compounds in deprotection, oxidation, C–C, C–N, and C–O bond forming reactions (27).

Catalytic activity of ZrOCl$_2$·8H$_2$O has been described for the oxidation of alcohols (28); acylation of alcohols, phenols, amines, and thiols (29); esterification of long chain carboxylic acids and alcohols (30); enaminones and enaminoo ester synthesis (31); chemoselective synthesis of 2-aryloxazolines and bis-oxazolines (32); and synthesis of benzoazoles, benzothiazoles, benzimidazoles, and oxazolo[4,5-b]-pyridines (33).

Results and discussion

In continuation to our interest in protection and deprotection (34) and zirconium chemistry (35), we want to divulge herein a new and convenient protocol

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ISSN 1751-8253 print / ISSN 1751-7192 online
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http://dx.doi.org/10.1080/17518253.2012.677480
http://www.tandfonline.com
for the protection of aldehydes/ketones as corresponding hydrazones in refluxing acetonitrile and subsequent deprotection of hydrazones back to their parent carbonyl compounds in refluxing methanol using ZrOCl$_2$·8H$_2$O as catalyst (10 mol%) (Scheme 1).

In order to search for the optimum experimental condition, the reaction of benzaldehyde (1a) and hydrazine hydrate (1b) was chosen as the model reaction (Scheme 2). The need for a catalyst was realized by the observation that very poor yields (trace-19%) were obtained when the reactions were carried out in the absence of a catalyst at room temperature or heating at 80 °C under neat condition or refluxing in Dimethyl formamide/Dimethyl sulfoxide (DMF/DMSO) for 24 h. Various compounds were screened for their effectiveness as catalysts (Table 1) in the model reaction. In addition, various solvents were also screened.

The best result was obtained when we used 10 mol% of ZrOCl$_2$·8H$_2$O in refluxing acetonitrile for 2 h (Table 1, entry 17, yield 93%). In solvents such as Dichloromethane (DCM), DMSO, DMF and Tetrahydrofuran (THF), the reaction required longer time to afford either very poor yield (trace-15%) or no reaction at all.

In addition, to determine the effective catalyst loading, we employed varying amounts (1, 2, and 5 mol%, respectively) of ZrOCl$_2$·8H$_2$O in the model reaction, but the reaction did not undergo. However, using 7 mol% of ZrOCl$_2$·8H$_2$O under the same reaction condition, 52% yield was obtained. When the catalyst loading was increased to 10 mol%, to our delight the reaction underwent efficiently resulting excellent yield (Table 2, entry 5, 93%) of the product. Further increasing the catalyst to 15 and 20 mol% resulted lower yield (60 and 57%, respectively) requiring longer reaction times.

Encouraged by these results, next, the reaction of 1a with several hydrazine sources were carried out in the presence of ZrOCl$_2$·8H$_2$O (10 mol%) to investigate the influence of various hydrazine sources (Table 3). The reaction was found to be faster with hydrazine hydrate than with hydrazine sulfate and hydrazine chloride. Notably, with hydrazine nitrate no reaction occurred. The reaction of 1a with hydrazine hydrate

![Scheme 1. Protection of carbonyl compounds 1 as hydrazones 2 and deprotection of hydrazones 2 to carbonyl compounds 1 by ZrOCl$_2$·8H$_2$O.](image)

![Scheme 2. Screening of catalysts for the reaction between benzaldehyde and hydrazine hydrate.](image)

**Table 1. Reaction of 1a and 1b in the presence of various catalysts.**

| Entry | Catalyst | Solvent | Temp (°C) | Time (h) | Yield (%)$^a$ |
|-------|----------|---------|-----------|---------|--------------|
| 1     | FeCl$_3$ | Neat    | RT        | 48      |  b           |
| 2     | FeCl$_3$ | Neat    | 80        | 7       |  b           |
| 3     | FeCl$_3$ | H$_2$O  | RT        | 48      |  b           |
| 4     | FeCl$_3$ | MeCN    | RT        | 48      |  b           |
| 5     | FeCl$_3$ | EtOH    | Reflux    | 48      |  b           |
| 6     | BiOCl$_2$ | Neat    | RT        | 48      |  b           |
| 7     | BiOCl$_2$ | Neat    | 80        | 6       |  b           |
| 8     | BiOCl$_2$ | MeOH    | RT        | 15      |              |
| 9     | BiOCl$_2$ | MeOH    | Reflux    | 38      | Trace        |
| 10    | BiOCl$_2$ | EtOH    | Reflux    | 38      | Trace        |
| 11    | BiOCl$_2$ | MeCN    | Reflux    | 38      | 10           |
| 12    | BiOCl$_2$ | DMSO    | RT        | 38      |  b           |
| 13    | BiOCl$_2$ | DCM     | RT        | 24      |  b           |
| 14    | ZrOCl$_2$·8H$_2$O | Neat    | RT        | 24      |  b           |
| 15    | ZrOCl$_2$·8H$_2$O | Neat    | 80        | 7       |  b           |
| 16    | ZrOCl$_2$·8H$_2$O | MeCN    | RT        | 12      | 48           |
| 17    | ZrOCl$_2$·8H$_2$O | MeCN    | Reflux    | 2       | 93           |
| 18    | ZrOCl$_2$·8H$_2$O | Toluene | Reflux    | 12      | Trace        |
| 19    | ZrOCl$_2$·8H$_2$O | THF     | Reflux    | 12      | 10           |
| 20    | ZrOCl$_2$·8H$_2$O | DCM     | Reflux    | 12      | 15           |
| 21    | ZrOCl$_2$·8H$_2$O | DMF     | Reflux    | 12      | Trace        |
| 22    | None      | Neat    | 80        | 24      | Trace        |
| 23    | None      | Neat    | RT        | 24      | Trace        |
| 24    | None      | MeCN    | RT        | 24      | Trace        |
| 25    | None      | MeCN    | Reflux    | 24      | 19           |
| 26    | None      | DMSO    | Reflux    | 24      | Trace        |

Note: 1a (1 mmol) was treated with 1b (1 mmol) in the presence of the catalyst (10 mol%) except for the entries 22–27 where no catalyst was used.

$^a$Isolated yield of benzaldehyde hydrazone.

$^b$No reaction.
in the presence of 10 mol% ZrOCl$_2$·8H$_2$O under near condition provided low yield (Table 3, entry 7, 40%), whereas in case of hydrazine sulfate and hydrazine chloride the reaction was very slow providing only trace conversion to the product.

In an attempt to broaden the scope of the methodology and to establish the generality of the reaction, a range of aldehydes/ketones were reacted with hydrazine hydrate in the presence of 10 mol% ZrOCl$_2$·8H$_2$O in refluxing acetonitrile and the results are summarized in Table 4 (entries 1–24). Entries 1–22 provided corresponding hydrazones in relatively good yields, except the entries 19 and 21. Groups such as −Cl, −NO$_2$, −OH, −OMe, and −COOEt

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**Table 2. Optimization of the catalyst ZrOCl$_2$·8H$_2$O for the protection of carbonyl functionality.**

| Entry | Mol% of catalyst | Time (h) | Yield (%)$^a$ |
|-------|------------------|----------|---------------|
| 1     | 1                | 9        | b             |
| 2     | 2                | 9        | Trace         |
| 3     | 5                | 9        | Trace         |
| 4     | 7                | 9        | 52            |
| 5     | 10               | 2        | 93            |
| 6     | 15               | 6        | 60            |
| 7     | 20               | 6        | 57            |

Note: Reaction condition: 10 mmol of 1a, 10 mmol of 1b, acetonitrile (15 mL), reflux.
$^a$Isolated pure product.
$^b$No reaction.

**Table 3. Different hydrazine sources tested for the formation of hydrazones.**

| Entry | Hydrazines sources | Solvent | Time (h) | Yield (%)$^a$ |
|-------|--------------------|---------|----------|---------------|
| 1     | NH$_2$NH$_2$, H$_2$SO$_4$ | MeCN    | 24       | 58            |
| 2     | NH$_2$NH$_2$, HNO$_3$ | MeCN    | 24       | b             |
| 3     | NH$_2$NH$_2$, HCl  | MeCN    | 18       | Trace         |
| 4     | NH$_2$NH$_2$, H$_2$O | MeCN    | 2        | 93            |
| 5$^c$| NH$_2$NH$_2$, H$_2$SO$_4$ | Neat    | 24       | Trace         |
| 6$^c$| NH$_2$NH$_2$, HCl  | Neat    | 24       | Trace         |
| 7$^c$| NH$_2$NH$_2$, H$_2$O | Neat    | 9        | 40            |

Note: Reaction condition: 1 mmol of 1a and 1 mmol of hydrazine source, acetonitrile (5 mL), reflux.
$^a$Isolated yield.
$^b$No reaction.
$^c$The reaction was performed at 80°C.

**Table 4. Protection of carbonyl compounds 1 by ZrOCl$_2$·8H$_2$O vide Scheme 1.**

| Entry | Substrate (aldehydes/ketones 1) | Products (hydrazones 2) | Time for protection (h) | Yield (%)$^{a,b}$ |
|-------|---------------------------------|-------------------------|-------------------------|-------------------|
| 1     | ![Aldehyde 1](image)             | ![Hydrazone 1](image)   | 1                       | 93                |
| 2     | ![Aldehyde 2](image)             | ![Hydrazone 2](image)   | 2                       | 90                |
| 3     | ![Aldehyde 3](image)             | ![Hydrazone 3](image)   | 3                       | 92                |
| 4     | ![Aldehyde 4](image)             | ![Hydrazone 4](image)   | 3                       | 90                |
| 5     | ![Aldehyde 5](image)             | ![Hydrazone 5](image)   | 1                       | 90                |
| 6     | ![Aldehyde 6](image)             | ![Hydrazone 6](image)   | 2                       | 88                |
| 7     | ![Aldehyde 7](image)             | ![Hydrazone 7](image)   | 3                       | 82                |
Table 4 (Continued)

| Entry | Substrate (aldehydes/ketones) | Products (hydrazones) | Time for protection (h) | Yield (%)<sup>a,b</sup> |
|-------|-------------------------------|------------------------|-------------------------|--------------------------|
| 8     | ![substrate](image1)           | ![product](image2)     | 4                       | 80                       |
| 9     | ![substrate](image3)           | ![product](image4)     | 3                       | 85                       |
| 10    | ![substrate](image5)           | ![product](image6)     | 2                       | 87                       |
| 11    | ![substrate](image7)           | ![product](image8)     | 3                       | 82                       |
| 12    | ![substrate](image9)           | ![product](image10)    | 4                       | 80                       |
| 13    | ![substrate](image11)          | ![product](image12)    | 3                       | 80                       |
| 14    | ![substrate](image13)          | ![product](image14)    | 2                       | 84                       |
| 15    | ![substrate](image15)          | ![product](image16)    | 3                       | 81                       |
| 16    | ![substrate](image17)          | ![product](image18)    | 4                       | 70                       |
| 17    | ![substrate](image19)          | ![product](image20)    | 5                       | 68                       |
| 18    | ![substrate](image21)          | ![product](image22)    | 5                       | 65                       |
| 19    | ![substrate](image23)          | ![product](image24)    | 7                       | 30                       |
| 20    | ![substrate](image25)          | ![product](image26)    | 7                       | 55                       |
| 21    | ![substrate](image27)          | ![product](image28)    | 7                       | 40                       |
Among the aliphatic carbonyl compounds, those with shorter alkyl chains showed a higher yield (Table 4, entry 16) as compared to those with longer alkyl chain (Table 4, entries 17 and 18, respectively). Good chemoselectivity was observed for the substrate containing \(-\text{COOEt}\) group that did not experience any competition with the existing carbonyl group in the substrate (Table 4, entry 19), although yield was low (30%). In case of dicarbonyl compounds, when one equivalent of hydrazine hydrate was used, the reaction underwent producing corresponding mono-hydrazone in moderate yield (Table 4, entries 20 and 21, respectively). When two equivalents of hydrazine hydrate were used for masking both the carbonyl moieties, we did not observe any reaction occurring at both the carbonyl groups, that is selectively only mono-hydrazones were obtained. We believe that the active species, zirconium cation cluster \([\text{Zr}_4(\text{OH})_8(\text{H}_2\text{O})_{16}]^{8+}\) is too bulky to coordinate with the two carbonyl groups at the same time. Hence, complexation with only one carbonyl group is possible and thus showing the chemoselectivity. The acyclic ketone, cyclohexanone (Table 4, entry 22) also provided good yield under the present reaction condition. Furthermore, the reaction was also found to proceed for the heteroatom containing aldehydes (Table 4, entries 12 and 13, respectively) with high yields. However, both benzil and benzoin (Table 4, entries 23 and 24, respectively) remained inert under the reaction condition. In general, aromatic compounds showed better yields than the aliphatic or acyclic ones.

In order to find the selectivity of the reaction, we investigated the competitive reaction: protection of 4-chlorobenzaldehyde in the presence of acetophenone (in equimolar amounts) using \(\text{ZrOCl}_2\cdot 8\text{H}_2\text{O}\) (10 mol\%) in acetonitrile under refluxing condition. We have found that 4-chlorobenzaldehyde was converted to the corresponding hydrazone selectively and faster than acetophenone. Alternatively, in the competitive reaction using equimolar amounts of hydrazones of 4-nitrobenzaldehyde and acetophenone, under the same reaction condition, 4-nitrobenzaldehyde hydrazone was cleanly and selectively cleaved in the presence of acetophenone hydrazone (Scheme 3).

Alternatively, to extend our study, we also investigated oxidative deprotection of hydrazones to yield the corresponding carbonyl compounds catalyzed by \(\text{ZrOCl}_2\cdot 8\text{H}_2\text{O}\) (10 mol\%) in refluxing methanol (Scheme 1). The cleavage of hydrazones was equally effective for both aldehydes and ketones as summarized in Table 5 (entries 1–22). Interestingly, deprotection of hydrazones was much faster than the protection of carbonyl compounds. Both hydroxyl and nitro groups present in the compounds did not have any observable effect on the yields of the products (Table 5, entries 6, 7, 10 and 11, respectively). The deprotection was also found to proceed efficiently for the hetero atom containing hydrazones (Table 5, entries 12 and 13, respectively). It was also

Table 4 (Continued)

| Entry | Substrate (aldehydes/ketones) | Products (hydrazones) | Time for protection (h) | Yield (%)\(^{a,b}\) |
|-------|------------------------------|-----------------------|------------------------|------------------|
| 22    | ![Image](image1.png)          | ![Image](image2.png)  |                        | 4                | 72               |
| 23    | ![Image](image3.png)          | ![Image](image4.png)  |                        | c                |                  |
| 24    | ![Image](image5.png)          | ![Image](image6.png)  |                        | c                |                  |

\(^{a}\) Isolated yield.

\(^{b}\) All the compounds were characterized by IR, \(^1\)H, \(^13\)C-NMR spectroscopy, mass spectrometry and physical data, which were found to be consistent with that reported in literature, finally, compared with authentic compounds.

\(^{c}\) No reaction.
observed that the substrate having either electron donating or electron withdrawing groups on the aromatic ring reacted with varying reaction times (Table 5, entries 2, 4, and 6–11, respectively). The hydrazones of aliphatic aldehydes were also cleanly cleaved by ZrOCl₂·8H₂O under the present reaction condition (Table 5, entries 16–18, respectively). The deprotection of hydrazone of acyclic ketone took place with good yield (Table 5, entry 22). However, regeneration of corresponding carbonyl compounds from the hydrazones of α,β-unsaturated aldehyde, p-alkoxybenzaldehyde and alkylacetoacetate (Table 5, entries 14, 15, and 19, respectively) failed under the reaction condition. The actual mechanism of the reaction is not clear to us at this stage. Notably the aldehydes/ketones regenerated from their hydrazones did not undergo further oxidation under the present reaction condition.

A range of protic solvents was screened in order to evaluate the scope and limitation as far as the

![Scheme 3. Competitive reaction between p-chlorobenzaldehyde and acetophenone.](image)

| Entry | Hydrazones 2 | Carbonyl compounds 1 | Time for deprotection (h) | Yield (%)<sup>a,b</sup> |
|-------|--------------|----------------------|--------------------------|------------------------|
| 1     | ![Image](image) | ![Image](image) | 1                        | 95                     |
| 2     | ![Image](image) | ![Image](image) | 1                        | 94                     |
| 3     | ![Image](image) | ![Image](image) | 2                        | 95                     |
| 4     | ![Image](image) | ![Image](image) | 2                        | 93                     |
| 5     | ![Image](image) | ![Image](image) | 1                        | 90                     |

Table 5. Deprotection of hydrazones 2 by ZrOCl₂·8H₂O.
| Entry | Hydrazones 2 | Carbonyl compounds 1 | Time for deprotection (h) | Yield (%)<sup>a,b</sup> |
|-------|--------------|----------------------|--------------------------|------------------|
| 6     | ![Structure](image1) | ![Structure](image2) | 1                        | 90               |
| 7     | ![Structure](image3) | ![Structure](image4) | 1                        | 90               |
| 8     | ![Structure](image5) | ![Structure](image6) | 2                        | 88               |
| 9     | ![Structure](image7) | ![Structure](image8) | 1                        | 90               |
| 10    | ![Structure](image9) | ![Structure](image10) | 2                        | 84               |
| 11    | ![Structure](image11) | ![Structure](image12) | 2                        | 80               |
| 12    | ![Structure](image13) | ![Structure](image14) | 3                        | 80               |
| 13    | ![Structure](image15) | ![Structure](image16) | 3                        | 90               |
| 14    | ![Structure](image17) | ![Structure](image18) | c                        |                   |
| 15    | ![Structure](image19) | ![Structure](image20) | c                        |                   |
| 16    | ![Structure](image21) | ![Structure](image22) | 2                        | 90               |
| 17    | ![Structure](image23) | ![Structure](image24) | 3                        | 70               |
| 18    | ![Structure](image25) | ![Structure](image26) | 3                        | 70               |
deprotection of hydrazones into their parent carbonyl compounds using ZrOCl$_2$·8H$_2$O (10 mol%) (Table 6) is concerned. For this purpose, 4-chlorobenzaldehyde hydrazone was chosen as the model reactant with 10 mol% ZrOCl$_2$·8H$_2$O in various solvents. Methanol was found to be the choice of solvent among all the solvents with faster conversion and quantitative yield.

It is also noteworthy to mention that the catalyst is recyclable, an important aspect from green chemistry and industrial point of view. After the aqueous work-up, ZrOCl$_2$·8H$_2$O was recovered from the aqueous solution by concentrating under reduced pressure. Alternatively, after filtration, the catalyst was washed with CH$_2$Cl$_2$ or CHCl$_3$, dried at 60°C and subjected to reaction again. Even after five runs for protection and deprotection reaction, the catalytic activity of ZrOCl$_2$·8H$_2$O was found to be almost same as that of freshly used catalyst (Figure 1), without significant loss of its activity.

The catalyst recyclability chart is shown below (Figure 2).

| Entry | Hydrazones | Carbonyl compounds | Time for deprotection (h) | Yield (%)$^{a,b}$ |
|-------|------------|--------------------|---------------------------|-----------------|
| 19    | ![Image](image1.png) | ![Image](image2.png) | c                         |                 |
| 20    | ![Image](image3.png) | ![Image](image4.png) | 4                         | 52              |
| 21    | ![Image](image5.png) | ![Image](image6.png) | 4                         | 50              |
| 22    | ![Image](image7.png) | ![Image](image8.png) | 2                         | 90              |

$^a$Isolated yield.

$^b$All the compounds were characterized by IR, $^1$H, $^{13}$C-NMR spectroscopy, mass spectrometry and physical data, which were found to be consistent with that reported in literature, finally, compared with authentic compounds.

$^c$No reaction.

In conclusion, we have developed a simple methodology for the protection of aldehydes/ketones into hydrazones and deprotection of hydrazones back to corresponding aldehydes/ketones under non-aqueous and protic condition catalyzed by ZrOCl$_2$·8H$_2$O (10 mol%). Moreover, this is a good development as far as chemistry of Zirconium is concerned.

**Experimental**

**General**

$^1$H-NMR and $^{13}$C-NMR spectra were recorded on a JNM ECS 400 MHz (JEOL) spectrophotometer in CDCl$_3$ with Tetramethylsilane (TMS) as the internal standard. IR spectra were recorded on a Nicolet...
(Impact 410) FT-IR spectrophotometer using KBr disks. Mass spectra were recorded on a Waters Q-TOF Premier & Aquity UPLC spectrometer. Melting points/boiling points were checked on a Büchi 504 apparatus and are uncorrected. All the chemicals were used as received. ZrOCl$_2$·8H$_2$O was purchased from Fluka. All reactions were monitored by TLC on silica gel 60 F$_{254}$ (0.25 mm), visualization was effected with UV and/or by developing in iodine. Purification of the reaction products was carried out by column chromatography using 60–120 mesh silica gel.

**Typical procedure for the protection of carbonyl compounds 1 into hydrazones 2 using ZrOCl$_2$·8H$_2$O vide Scheme 1**

In an oven-dried round-bottomed flask (50 mL), benzaldehyde (1 mmol), hydrazine hydrate (1 mmol), and ZrOCl$_2$·8H$_2$O (10 mol%) were taken and acetonitrile (5 mL) was added to it. The reaction mixture was refluxed for required time under aerobic condition. After the completion of the reaction (monitored by TLC), the reaction system was allowed to cool to room temperature, acetonitrile was removed in a rotary evaporator and then extracted with ethyl acetate (3 × 10 mL), washed with water and brine, dried over Na$_2$SO$_4$, concentrated in a rotary evaporator and subjected to column chromatography to furnish the pure product (Yield 93%). Finally, the product was recrystallized from ethanol to afford the purest product, which was identified by IR, $^1$H, $^{13}$C-NMR, mass, mp, and compared with authentic sample.

**Typical procedure for the deprotection of hydrazones 2 into their parent aldehydes/ketones 1 using ZrOCl$_2$·8H$_2$O vide Scheme 1**

The benzaldehyde hydrazone (1 mmol) and ZrOCl$_2$·8H$_2$O (10 mol%) were taken in an oven-dried round-bottomed flask (50 mL) and methanol (5 mL) was added to it. It was then refluxed for the appropriate time under aerobic condition. After the completion of the reaction (vide TLC), the reaction mixture was allowed to cool to room temperature, methanol was removed in a rotary evaporator and then extracted with ethyl acetate (3 × 10 mL), washed with water and brine, dried over Na$_2$SO$_4$, concentrated in a rotary evaporator and finally performed column chromatography to afford the pure product (Yield 95%). The product was identified by IR, $^1$H, $^{13}$C-NMR, mass, bp and compared with authentic samples.

When the product was solid, recrystallization from ethanol provided the pure compound.

**Acknowledgements**

AJT thanks Council of Scientific and Industrial Research, New Delhi, India for financial support. VKD and SD thank University Grants Commission, New Delhi, India for Rajiv Gandhi fellowship.

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