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One-pot Synthesis of β-Azido and β-Amino Alcohols Using $Fe_3O_4@SiO_2@CS@POCl_{2-x}$ as a Heterogenous and Magnetic Nanocatalyst

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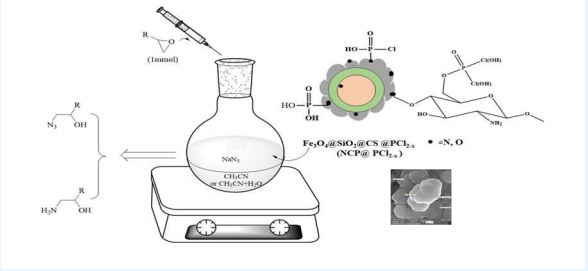
Abstract: This study introduces $Fe_3O_4@SiO_2@CS@POCl_{2-x}$ (NCP@ $POCl_{2-x}$), as an innovative and eco-friendly nanocatalyst for the regioselective azidolysis of epoxides using NaN_3. This process efficiently yields *β*-azido alcohols or *β*-amino alcohols, depending on the reaction conditions. In the presence of the catalyst, rapid azidation occurs, and the subsequent addition of water and application of heat efficiently produce β-amino alcohols. The catalyst is notable for its high yield and precise targeting of specific chemical sites (regioselectivity). Incorporating chitosan into the catalyst enhances its environmental friendliness, while surface hydroxyl and amine groups promote the smooth conversion of *β*-azido to *β*-amino alcohols via water incorporation in the mechanism and proton transfer during heating. This catalyst's performance acts under mild conditions and excellent yield potential. Its ability to be easily recovered magnetically, its diverse composition, and efficient, clean reactions make it a vital tool for transforming epoxides.



Keywords: NCP@$POCl_{2-x}$, Azidolysis, Epoxides, magnetic nanocatalyst, *β*-azido alcohols, *β*-amino alcohols.

1. Introduction

β-Azido alcohols are widely recognized as important precursors for synthesizing a diverse range of compounds, including β-amino alcohols,[1] amino acids,[2] 1,2-vicinal diamines,[3] aziridines,[4] vicinal azidoacetamides,[5] and triazole derivatives.[6] These compounds serve as crucial building blocks in the preparation of various natural products and bioactive compounds, especially in the pharmaceutical field.[3e] Additionally, *β*-amino alcohols have played significant roles in the chemistry of carbohydrates and nucleosides.[7] The incorporation of β-amino alcohols into carbohydrate and nucleoside frameworks enhances the scope of chemical diversity, enabling the design and development of novel molecules with potential applications in medicinal chemistry, drug discovery, and other biologically relevant areas.[8]

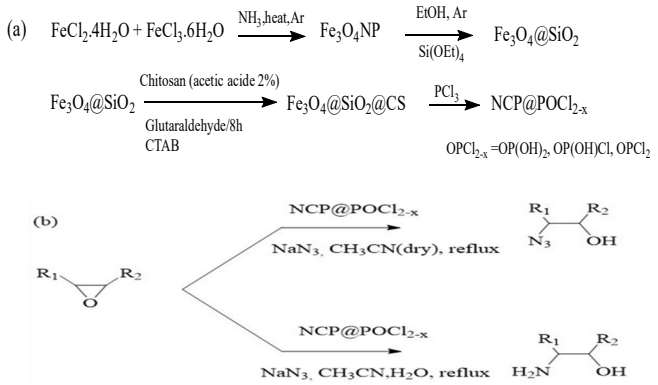
The preferred method for synthesizing *β*-azido alcohols involves the ring-opening of epoxides.[9] The azidolysis of epoxides can be promoted by either homogeneous or heterogeneous systems. In the homogeneous category, metal chlorides, salts, and alkyl metal azides have been utilized. Various metal salts, including $Zr(OTf)_4$,[10] $In(OTf)_3$,[11] $Nd(OTf)_3$,[12] and $Gd(OTf)_3$,[12] $Hf(OTf)_4$,[13] $Yb(OTf)_3$,[12] $LiClO_4$[14] and ammonium-12-molybdophosphate[15] and Bronsted acidic ionic liquid (BAIL),[16] have been reported for this transformation.

Moving on to the heterogeneous category, a range of catalysts have been explored. These include amberlite IRA-400 supported azide,[17] sodium azide supported on Zeolite CaY,[18] Ni^{2+} supported on hydroxyapatite-core-shell γ-Fe_2O_3,[19] $Fe_3O_4@SiO_2$/bipyridinium dichloride,[20] γ-Fe_2O_3@HAp@β-CD[21] and Montmorillonite K10[22] as well as functionalized polymeric catalysts such as quaternate amino functionalized cross-linked polyacrylamide,[23] poly(*N*-bromo acrylamide),[24] polyethylene glycol immobilized on silica gel,[25] poly(vinylamine) and poly(allylamine),[26] and PEG-300[27] have also been investigated for their potential to promote the azidolysis of epoxides. Each of the methods and procedures mentioned above has its own set of advantages. However, it is important to note that many of these methodologies also come with certain disadvantages. Some of the common drawbacks include unsatisfactory yields, strong acidic conditions, hygroscopic nature of the reagents, lack of regioselectivity, high cost, and reagent stability issues.

The regio- and stereoselective azidolysis and the ring-opening reaction of epoxides are seriously influenced by various factors, including the nature of the substrate, the nucleophilicity of the nitrogen, and the reaction conditions, encompassing the solvent, concentration, temperature, additive, catalyst, and procedural parameters.[28] Over the past two decades, water has played an important role not only as an ideal solvent but also as a remarkable medium and co-

catalyst for a variety of catalyzed and uncatalyzed organic reactions.[29]

We have recently demonstrated the remarkable versatility of Fe_3O_4@SiO_2@CS@$POCl_{2-x}$ (NCP@ $POCl_{2-x}$) in various organic transformations, including the conversion of alcohols to alkyl halides[30] and amines.[31] The steps for the preparation of the catalyst are briefly shown in Scheme 1a. Expanding on these achievements, our focus is to emphasize the distinctive capability of this complex in activating epoxides for subsequent reactions with sodium azide (NaN_3) as a nucleophilic compound. The objective of this study is to explore the intriguing reactivity of the epoxides with NaN_3 in dry acetonitrile and aqueous acetonitrile in producing β-azido alcohols and subsequently advancing *β*-amino alcohols synthesis in one pot reaction to overcome the challenges associated with traditional methods by presenting for achieving regioselective conversions (Scheme 1b).



Scheme 1. (a) Steps in the preparation of the NCP@$POCl_{2-x}$ catalyst (b) Schematic for the synthesis of *β*-azido and *β*-amino alcohols using NCP@$POCl_{2-x}$.

2. Results and Discussion

As mentioned earlier, the catalyst has been extensively studied in existing literature [30]. In this research, we provide a detailed comparative analysis, exploring both the morphology observed through field emission scanning electron microscopy (FESEM) and the magnetic strength using vibrating sample magnetometry (VSM) analysis.

It's important to note that while the magnetic activity of NCP@PCl_{2-x} particles with an average size of 157 nm, is lower compared to Fe_3O_4@SiO_2 particles, they still possess enough magnetic strength to respond to external magnetic fields. This decrease in magnetic activity is attributed to their larger size and the increased organic layer surrounding the Fe_3O_4 core. The NCP@PCl_{2-x} particles can be easily separated using a magnet with a magnetic field strength of 0.5 T, making them well-suited for applications requiring efficient magnetic separation.

Figure 1 illustrates the comparison in magnetic strength between the catalyst and both nano Fe_3O_4@SiO_2 and nano Fe_3O_4.

A series of experiments aimed to optimize the reaction conditions and study the influence of catalyst amount, solvent, and temperature. The focus was on the azidolysis of 2-(phenoxymethyl) oxirane as a representative reaction, detailed in Table 1. In these experiments, 2 mmol of sodium azide (NaN_3) reacted with 0.2-2 mmol of catalyst (NCP@$POCl_{2-x}$) under various conditions, either at room temperature or under reflux. Different solvents, such as $CHCl_3$, CH_2Cl_2, THF, EtOAc, H_2O, CH_3CN, and a mix of CH_3CN-H_2O, were systematically examined to understand their effects on the reaction's performance and selectivity.

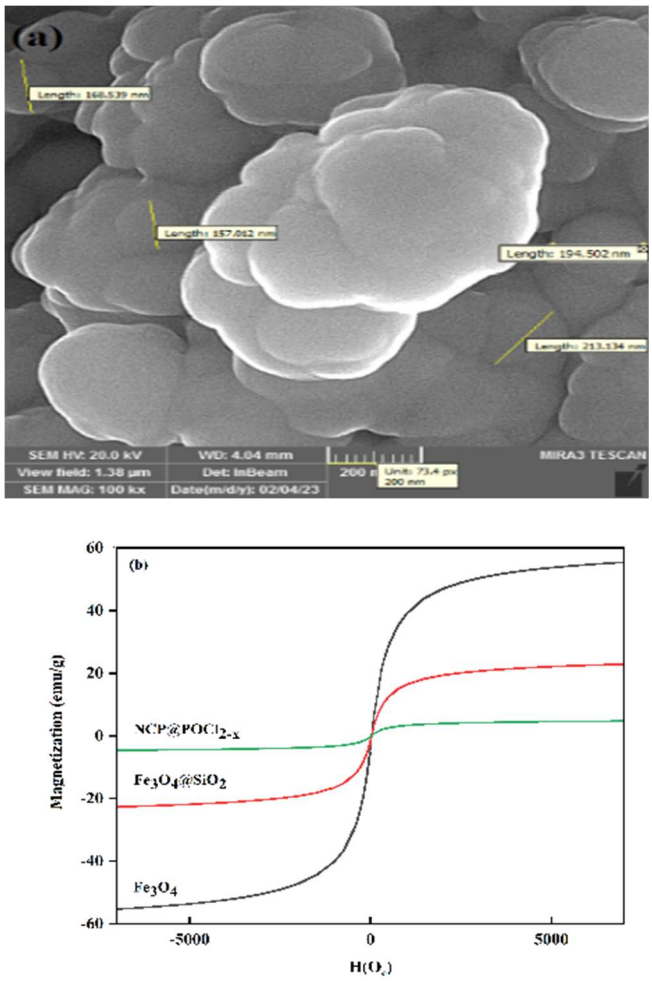


Figure 1. (a) FESEM image of NCP@$POCl_{2-x}$ at a magnification of 200 nm, featuring an average size of 157 nm (b) Comparing magnetic behaviors through VSM curves: Fe_3O_4, Fe_3O_4@SiO_2 and NCP@$POCl_{2-x}$.

Out of the explored solvents, CH_3CN was found to be suitable for the azidolysis of 2-(phenoxymethyl)oxirane. Introducing water into CH_3CN led to a transformation of the product into *β*-amino alcohol. Optimal results were obtained using 0.5 mmol of the catalyst under reflux conditions (see Table 1, Entry 10, 13). In these specific conditions, the catalyst exhibited high effectiveness, yielding the maximum product.

Several factors, including the specific structure of NCP@PCl_{2-X}, the chosen epoxide, reaction conditions (temperature, concentration), and the existence of additives or co-catalysts like H_2O, may impact the success and efficiency of this reaction. To evaluate the extensive versatility of the procedure, we treated various substituted epoxides with NaN_3 under optimized conditions. The detailed results from these experiments are provided in Table 2, offering a comprehensive overview of the findings. The data in Table 2 indicates that excellent yields and high regioselectivity were achieved in the formation of β-azido alcohols in terminal epoxides using NCP@PCl_{2-X} in CH_3CN under reflux conditions.

Table 1. Optimization of azidolysis for 1 mmol of 2-(phenoxymethyl)oxirane with 2 mmol sodium azide

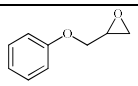
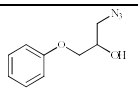
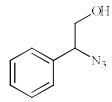
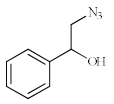
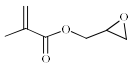
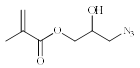
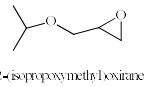
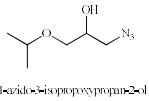
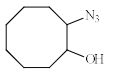
Entry	Solvent	Catalyst (mmol)	Temp. (ºC)	Time (h)	Conversion (%)[a]
1	$CHCl_3$	2	rt	24	10
2	CH_2Cl_2	2	rt	24	21
3	THF	2	rt	24	20
4	THF	2	reflux	5	30
5	EtOAc	2	rt	24	53
6	EtOAc	2	reflux	24	70
7	CH_3CN(dry)	2	rt	5	85
8	CH_3CN(dry)	2	reflux	3	100
9	CH_3CN(dry)	1	reflux	3	100
10	CH_3CN(dry)	0.5	reflux	3	100
11	CH_3CN(dry)	0.2	reflux	24	70
12	H_2O	0.5	reflux	24	80[b]
13	CH_3CN-H_2O	0.5	reflux	5	90[b]

[a]GC yield (%). [b]β-amino alcohol was synthesized.

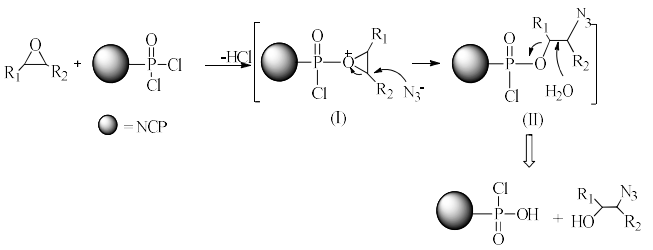
Significantly, nucleophilic attacks from a specific side are less hindered in this reaction, leading to regioselectivity in the product. The preference for attacking a particular site is influenced by electronic and steric factors in the reaction environment and the catalytic species' structure. In these reactions, the primary target is the carbon atom of the epoxide ring, influenced by both steric and electronic considerations. A larger catalyst activates the substrate, making the N_3^- ion attack easier at the site. This regioselective behavior is seen in the results, except for the reaction involving styrene oxide, which produced an alternative regio isomer as a side product (Table 2, Entry 2). Despite hindrance at the phenyl position, it prefers attack due to its greater stability concerning cationic and electronic effects. Consequently, two distinct products are formed. Despite this exception, the reactions with other epoxides showed high regioselectivity, resulting in the exclusive formation of a single isomer in each case.

Understanding this mechanistic insight is crucial for optimizing reaction conditions, predicting regioselectivity, and designing efficient synthetic pathways in the development of this catalytic process. NCP@$POCl_{2-x}$ has proven to be an exceptionally active catalyst in facilitating this transformation. In this process, when an epoxide is present, the oxygen atom in the epoxide tends to interact with the phosphorus group in NCP@PCl_{2-X}, leading to the formation of an intermediate known as (I) (Scheme 2).

Table 2. Reaction of epoxides with NaN_3 in CH_3CN catalyzed by NCP@PCl_{2-x}

Entry	Substrate	Product	Time (h)	Isolated yield (%)
1	 2-(phenoxymethyl)oxirane	 1-azido-3-phenoxypropan-2-ol	1	93
2	 2-phenyloxirane	 2-azido-2-phenylethanol  2-azido-1-phenylethanol	1	80 (20)
3	 oxiran-2-ylmethyl methacrylate	 3-azido-2-hydroxypropyl methacrylate	2	88
4	 2-(isopropoxymethyl)oxirane	 1-azido-3-isopropoxypropan-2-ol	3	85
5	 9-oxabicyclo[6.1.0]nonane	 2-azidocyclooctanol	5	91

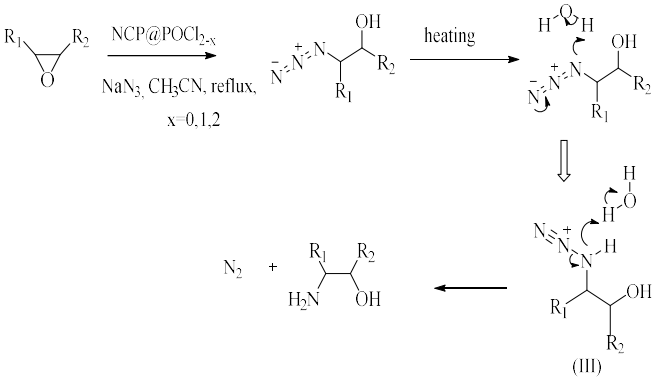
The intermediate formed from the epoxide displays increased reactivity as a consequence of the inherent instability arising from oxygen's possession of three bonds. Furthermore, the nucleophilic attack of NaN_3 leads to the conversion of the intermediate (I) into a different form depicted as (II) in Scheme 2. Acting as a catalyst, NCP@PCl_{2-X} offers an active site that facilitates the nucleophilic attack of the N_3^- on the epoxide, particularly at less hindered positions. The highly regioselective azidation of terminal epoxides, resulting in quantitative yields, is attributed to the predominant attack of the N_3^- on the less hindered carbon of the epoxide. Consequently, the presence of NaN_3 as a nucleophile in the solution facilitates the ring-opening process, ultimately resulting in the synthesis of the desired product. This specific regioselectivity is a noteworthy outcome, showcasing the efficiency of the NCP@$POCl_{2-x}$ catalyzed reaction. The general mechanism of the reaction can be illustrated as follows:



Scheme 2. Proposed mechanism for preparation of *β*-azido alcohols

upon prolonged heating with the addition of water to the mixture, the *β*-azido alcohol undergoes a transformation into

a β-amino acid. The core-shell layer on NCP@$POCl_{2-x}$ plays a crucial role in forming an intermediate with the β-azido alcohol via hydrogen bond, activating the substrate for further reactions. In the presence of water, electron transfer occurs within the azide compound. The presence of an amine group, serving as a base within the chitosan layer of the catalyst, plays a pivotal role in facilitating the transfer of hydrogen from the oxygen in water to the nitrogen in the azide group, forming intermediate III. This process simultaneously results in the release of nitrogen gas (N_2), ultimately leading to the synthesis of β-amino alcohol. Scheme 3 illustrates the mechanistic details of this process, providing a comprehensive understanding of the reaction pathway and revealing how the reaction proceeds.



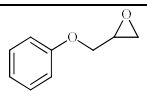
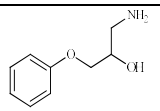
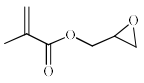
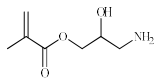
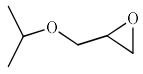
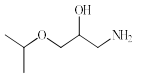
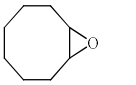
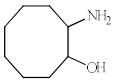
Scheme 3. Proposed mechanism for preparation of β-amino alcohols

The absence of NCP@$POCl_{2-x}$ has a notable effect on the conversion, leading to a less favorable yield. This observation suggests that NCP@$POCl_{2-x}$ plays a crucial role as a catalyst in facilitating the formation of β-amino alcohols from epoxides. The catalytic activity of NCP@$POCl_{2-x}$ is essential for promoting the desired transformation, resulting in improved yields and the specific formation of β-amino alcohols.

The results for the ring opening of various substituted epoxides to β-amino alcohol with NCP@$POCl_{2-x}$ are detailed in Tables 3, highlighting the regioselective nature of the reaction across different substrate structures.

It is worth noting that the functional groups, such as simple esters, alkyl groups, and alkenes, remain stable during the reaction. No transesterification reactions occur, which is beneficial. This stability allows the reaction to proceed smoothly without any unintended side reactions. Furthermore, the magnetic catalyst's easy separation simplifies the workup process, enhancing the practicality of the synthetic procedure. Table 4 provides a comprehensive comparison between the method described in this study and other catalysts employed in similar reactions. This comparative analysis aids in understanding the unique benefits and potential drawbacks of the magnetic catalyst utilized in this study, ultimately contributing to the advancement of catalyst design and selection in synthetic processes.

Table 3. Reaction of epoxides with N_3^- in CH_3CN: H_2O (70:30) catalyzed by NCP@$POCl_{2-x}$ during 24 h

Entry	Substrate	Product	Isolated yield (%)
1	 2-(phenoxymethyl)oxirane	 1-amino-3-phenoxypropan-2-ol	90
2	 oxiran-2-ylmethyl methacrylate	 3-amino-2-hydroxypropyl methacrylate	80
3	 2-(isopropoxymethyl)oxirane	 1-amino-3-isopropoxypropan-2-ol	79
4	 9-oxabicyclo[6.1.0]nonane	 2-aminocyclooctanol	84

It is worth noting that the functional groups, such as simple reaction. No transesterification reactions occur, which is esters, alkyl groups, and alkenes, remain stable during the beneficial. This stability allows the reaction to proceed smoothly without any unintended side reactions. Furthermore, the magnetic catalyst's easy separation simplifies the workup process, enhancing the practicality of the synthetic procedure. Table 4 provides a comprehensive comparison between the method described in this study and other catalysts employed in similar reactions. This comparative analysis aids in understanding the unique benefits and potential drawbacks of the magnetic catalyst utilized in this study, ultimately contributing to the advancement of catalyst design and selection in synthetic processes.

Table 4. compares various catalysts used in the azidation of 2-(phenoxymethyl) oxirane to 1-azido-3-phenoxypropan-2-ol

Entry	Catalyst	Conditions and isolated yield	Reference
1	NCP@$POCl_{2-x}$	1h, 93%	This work
2	Montmorillonite-K10	8h, 90%	6a
3	1-(1-Alkylsulfonic)-3-methylimidazolium chloride	2h, 95%	32
4	γ-Fe_2O_3@HAp@β-CD	1h, 89%	21
5	Fe_3O_4@SiO_2/bipyridinium	1h, 90%	20
6	Poly (*N*-bromoacrylamide)	1h, 92%	24b
7	Poly (vinylamine)	5h, 89%	26
8	Poly(allylamine)	4h, 92%	26
9	[pyridine-SO_3H]Cl	4h, 95%	16
10	PEG-300	1h, 90%	27

3. Experimental

All Chemical material from Merck or Fluka were used in reactions and the rection monitored by thin-layer chromatography (TLC) on silica gel SILG-UV 254 plates. Column chromatography with silica gel 60 was employed for more purification. Product identification relied on comparing physical and spectral characteristics with literature data.

Nuclear magnetic resonance (NMR) spectra were recorded with a Bruker Advance DPX 250MHz instrument.

General procedure for transformation epoxide to *β*-azido alcohol

To a stirred suspension, 1 mmol of oxirane was combined with 2 mmol of sodium azide (NaN_3) in 5 ml of CH_3CN under reflux conditions. NCP@$POCl_{2-x}$ (0.5 mmol, 0.8 g) was introduced, and the reaction proceeded for a specified duration as detailed in Table 2. After the reaction was complete, the catalyst was separated by filtration using external magnetic force. The filtrate was treated with CH_2Cl_2, followed by washing steps with brine (10 mL) and water (2 × 10 mL). Drying of the filtrate was achieved using anhydrous Na_2SO_4, and solvent evaporation was carried out with a rotary evaporator. The resulting pure product was collected for further analysis. Characterization of the obtained products included confirming their structural identity by comparing their ^{1}H-NMR spectra with those of authentic samples.

General procedure for transformation epoxide to *β*-amino alcohol

A stirred mixture of oxirane (1 mmol) in 7 ml of CH_3CN and sodium azide (2 mmol) underwent the addition of NCP@$POCl_{2-x}$, followed by 1h of stirring under reflux conditions. Subsequently, H_2O (3 ml) was added to the reaction mixture, which was stirred for 24h under reflux conditions. After completing the reaction, the mixture was filtered to remove the catalyst using an external magnetic force. CH_2Cl_2 was added, and the organic phase underwent washing with brine (10 mL) and water (2 × 10 mL). The filtrate was then dried using anhydrous Na_2SO_4. After evaporating the solvent with a rotary evaporator, the resulting pure product was collected. The obtained products were characterized by comparing their ^{1}H-NMR spectra with those of authentic samples.

4. Conclusions

The nanocatalyst NCP@$POCl_{2-x}$ holds great potential for synthesizing *β*-azido and *β*-amino alcohols. In the presence of the catalyst, azidation occurs rapidly, but upon adding water and applying sufficient heat, *β*-amino alcohols are synthesized with high yield and regioselectivity. This catalyst possesses unique qualities, such as easy separation from mixtures and magnetic properties. Incorporating chitosan into the nano-catalyst structure enhances its environmental friendliness. The presence of hydroxyl and amine groups on the catalyst's surface attracts β-azido alcohols, and through the formation of a five-atom ring intermediate, the transformation into *β*-amino alcohols occurs easily. It works well in mild conditions, producing excellent yield and proving its practical use in organic synthesis.

Declaration of Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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