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Aluminum tris (dodecyl sulfate) trihydrate $Al(DS)_3 \cdot 3H_2O$ as an efficient Lewis acid–surfactant-combined catalyst for organic reactions in water Efficient conversion of epoxides to thiiranes and to amino alcohols at room temperature

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Abstract

High yielding preparation of structurally different thiiranes, β-amino alcohols and bis(β-amino) alcohols from the reaction of epoxides with thiourea and amines in the presence of catalytic amounts of aluminum tris (dodecyl sulfate) trihydrate $Al(DS)_3 \cdot 3H_2O$ as a Lewis acid–surfactant-combined catalyst at room temperature in water has been described. Ring opening of epoxides with amines in the presence of this catalyst proceeded with high chemo- and regioselectivity.

Keywords: Aluminum tris (dodecyl sulfate) trihydrate; Water; Epoxide; Thiirane; Thiourea; Amio alcohol; Chemoselective reaction; Regioselective reaction

1. Introduction

Nowadays, environmental consciousness encourages replacement of hazardous organic solvents with water which is a relatively green and a cheap media for organic reactions [1–11]. Along this line, we have studied Michael addition of amines and thiols to α,β-unsaturated ketones [9], regioselective iodination of aromatic compounds with NaI/$Ce(OH)_3O_2H$ [12] and ring opening of epoxides with different nucleophiles in water catalyzed by micellar solution of sodium dodecyl sulfate (SDS) [13]. We have recently reported oxidation of sulfides to their sulfoxides by H_2O_2 in the presence of the *in situ* generated dodecyl hydrogen sulfate [14]. We have also reported aluminum tris (dodecyl sulfate) trihydrate [$Al(DS)_3 \cdot 3H_2O$] as an efficient Lewis acid–surfactant-combined catalyst for Michael addition of indoles and pyrrole to α,β-unsaturated electron-deficient compounds in water [15].

Epoxides are small molecules with vast synthetic applications. They are able to react with various nucleophiles and their potential to undergo regioselective ring opening reactions adds to their importance as highly useful precursors for the synthesis of organic compounds. Transformation of epoxides to their corresponding thiiranes is a useful reaction [16,17]. Thiirane is the simplest heterocyclic ring system which carrying a sulfur atom in the ring and is found in naturally occurring compounds, herbicides, pesticides, polymeric compounds, pharmaceuticals and in many other useful man-made chemicals [18]. For the preparation of thiiranes from epoxides, variety of methods have been employed and reported in the literature [19–43]. Preparation of thiiranes from epoxides in water as the reaction media using an ionic liquid with KSCN [44], β-cyclodextrin as a catalyst with KSCN or thiourea in water [45,46] and also synthesis of thiirane in micellar solution of sodium dodecyl sulfate (SDS) in the presence of catalytic amounts of $Ce(OTf)_4$ has been recently reported [13].

β-Amino alcohols are important intermediates for the synthesis of a vast range of biologically active natural and synthetic products, for the synthesis of amino acids, and also used as chiral auxiliaries/ligands for asymmetric synthesis [47–56]. One of the most accessible synthetic procedures for the preparation of

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β-amino alcohols involves the ring opening of epoxides with amines [57–80].

Recently, ring opening of epoxides with aromatic amines in water catalyzed by β-cyclodextrin [81], 1,4-diazabicycl[2.2.2]octane (DABCO) [82], bismuth triflate [83], asymmetric ring opening of meso-epoxides with aromatic and aliphatic amines catalyzed by scandium tris (dodecyl sulfate) in the presence of a chiral bipyridine ligand [84] and using heteropoly acid [85] has been reported. Catalyst-free ring opening of epoxides with amines in water has been also introduced [86]. However, this method observes limitations such as for example, addition of aniline and 4-nitroaniline to alkyl epoxides that is discouraging and gives very low yields of the desired β-amino alcohols in elongated reaction times. By this method, styrene oxide also reacts with aniline in a rather long reaction time (14 h).

Now we report the use of aluminum tris (dodecyl sulfate) trihydrate as an effective Lewis acid–surfactant-combined catalyst for efficient conversion of structurally diverse epoxides to their corresponding thiiranes using thiourea as sulfurating agent and also high yielding regio- and chemoselective ring opening of different epoxides with structurally different amines at room temperature in water.

2. Results and discussion

2.1. Preparation of thiiranes from epoxides

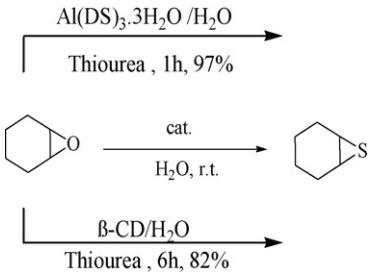
In this study facile and efficient conversion of epoxides to thiiranes in the presence of a catalytic amount of aluminum tris (dodecyl sulfate) trihydrate [$Al(DS)_3{\cdot}3H_2O$] in water is presented (Scheme 1).

For optimization of the reaction conditions, we studied the reaction of phenylglycidyl ether with thiourea in the presence of this catalyst in water at room temperature. We found that the optimized molar ratio of phenylglycidyl ether/thiourea/catalyst was 1.0 mmol/1.5 mmol/0.1 mmol in water (5 mL). The reaction was proceeded well at room temperature and the desired thiirane was isolated in 96% yield after 3 h. Then we applied similar reaction conditions for the preparation of structurally diverse thiiranes from different epoxides in order to show the general applicability of the method. The reactions proceeded smoothly and efficiently in this optimized reaction condition with excellent isolated yields usually in short reaction times (Table 1).

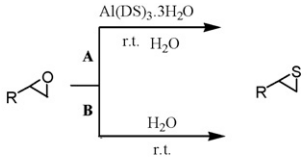
In order to show the advantage of the presented catalyst we have compared the results obtained using $Al(DS)_3{\cdot}3H_2O$ and β-cyclodextrin as catalysts [45] for the conversion of cyclohexene oxide to its corresponding thiirane using thiourea in water as a model reaction. As it is evident from Scheme 2, $Al(DS)_3{\cdot}3H_2O$ is a more efficient catalyst than β-cyclodextrin for this reaction.

In order to show the importance of the catalyst we have also conducted several reactions in the absence of $Al(DS)_3{\cdot}3H_2O$ in

Scheme 1.



Scheme 2.



Entry	Epoxide	Thiirane	A (Time/isolated Yield%)	B (Time/isolated Yield%)[a]
1			3h/96	no reaction
2			1h/97	no reaction
3			1h/97	no reaction

[a] our observations

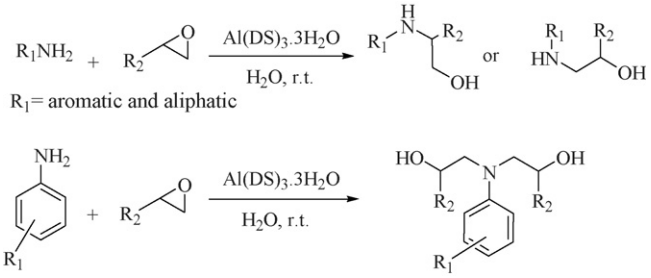
Scheme 3.

water and compared the results obtained using $Al(DS)_3{\cdot}3H_2O$ as a catalyst. These results are presented in Scheme 3.

2.2. Preparation of β-amino alcohols or bis(β-amino) alcohols

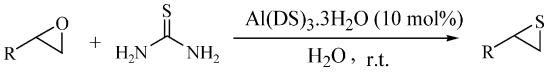
In this study, we report facile and efficient cleavage of epoxides with aromatic and aliphatic amines in the presence of a catalytic amount of aluminum tris (dodecyl sulfate) trihydrate [$Al(DS)_3{\cdot}3H_2O$] as a Lewis acid–surfactant-combined catalyst in aqueous media to produce the corresponding β-amino alcohols or bis(β-amino) alcohols in excellent yields at room temperature (Scheme 4).

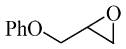
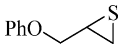
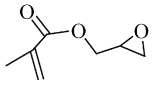
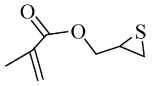
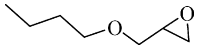
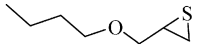
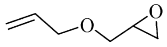
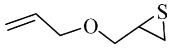
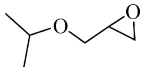
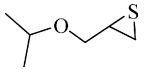
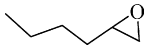
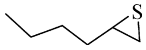
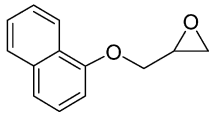
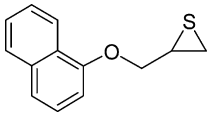
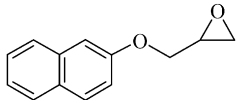
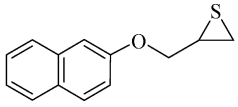
In order to optimize the reaction conditions, first the reaction of phenylglycidyl ether with 3-nitrtoaniline in the presence of the catalyst as a model reaction at room temperature was studied



Scheme 4.

Table 1
Reaction of thiourea with epoxides catalyzed with $Al(DS)_3{\cdot}3H_2O$ in water at room temperature

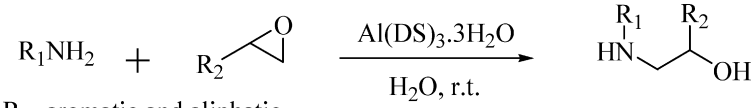


Entry	Epoxide	Product[a]	Time (h)	Isolated yield (%)
1			3	96
2			1.45	90
3			1	95
4			1.45	95
5			1	87
6			1	97
7			1	97
8			1	90
9			2.5	83
10			3	87

[a] All the products were known compounds and were characterized by their ^{1}H NMR and ^{13}C NMR spectra.

in water. It was found that the optimized molar ratio of phenylglycidyl ether with respect to 3-nitrtoaniline and $Al(DS)_3{\cdot}3H_2O$ was 1 mmol/1.5 mmol/0.1 mmol in 5 mL of water. The reaction proceeded smoothly with high regioselectivity and the desired β-amino alcohol, as a crystalline compound, was isolated by a simple filtration in an excellent yield (97%) after 5 h (Table 2, entry 1). Then, we applied similar reaction conditions for the ring opening of structurally diverse epoxides with aromatic and aliphatic amines. All the reactions proceeded well in excellent yields with high regioselectivity and most of the epoxide rings

Table 2
Reaction of amines which attack various epoxides from less hindered cite catalyzed by $Al(DS)_3{\cdot}3H_2O$ in water at room temperature

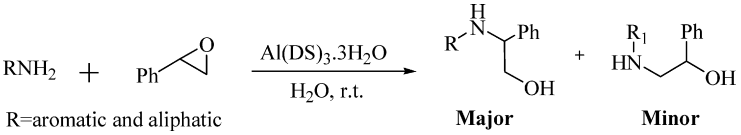


R_1=aromatic and aliphatic

Entry	Aromatic amines	Epoxide	Time (h)	Isolated yield (%)[a]
1	3-Nitroaniline	Phenylglycidyl ether	5	97
2	3-Nitroaniline	2,3-Epoxypropylmethacrylate	3	94
3	3-Cyanoaniline	Phenylglycidyl ether	5	94
4	3-Cyanoaniline	2,3-Epoxypropylmethacrylate	3	96
5	4-Nitroaniline	Phenylglycidyl ether	10	93
6	4-Cyanoaniline	2,3-Epoxypropylmethacrylate	6	95
7	Aniline	Phenylglycidyl ether	1.45	95
8	Aniline	Cyclohexeneoxide	1.5	96
9	α-Naphtyl amine	2,3-Epoxypropylmethacrylate	4	97
10	α-Naphtyl amine	Cyclohexeneoxide	2	98
11	2-Phenylaminoethanol	Phenylglycidyl ether	1	90
12	Piperidine	Phenylglycidyl ether	45 min	91

[a] Products were characterized by comparison of their ^{1}H NMR and ^{13}C NMR spectra with those of known compounds.

Table 3
Highly selective reaction of amines which attack styrene oxide from more hindered side catalyzed by $Al(DS)_3{\cdot}3H_2O$



R=aromatic and aliphatic **Major** **Minor**

Entry	Anilines	Time	Major product isolated yield (%)[a]
1	Aniline	45 min	88
2	3-Nitroaniline	1 h	90
3	3-Cynoaniline	1 h	90
4	Piperidine	30 min	80

[a] Products were isolated and purified by column chromatography and their structures were identified by ^{1}H NMR spectroscopy and compared with those reported for authentic samples.

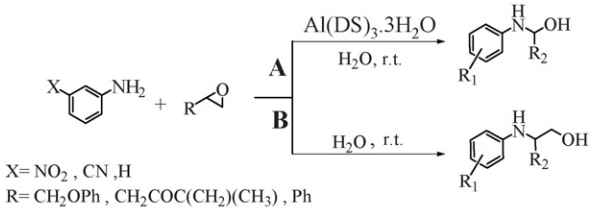
were opened from the less hindered cites in the appropriate reaction times. This type of the ring opening shows that steric hindrance plays a major role in the ring opening reactions. The results of this investigation are summarized in Table 2.

In the case of the reaction of styrene oxide with piperidine and different aromatic amines, the reaction proceeded well and the epoxide ring was opened from the more hindered cite by amines with high regioselectivity in excellent yields in the appropriate reaction times. The ring opening reaction from the more hindered cite of styrene oxide indicates the formation of a carbocationic character during the progress of the reaction is the most probable. The results of this study are tabulated in Table 3.

The important catalytic role of $Al(DS)_3{\cdot}3H_2O$ for the ring opening of epoxides with aromatic amines in water, has been also shown by the reaction of several aromatic amines with epoxides in presence and in the absence of the catalyst. Our results show that in the presence of the catalyst, the drastic rate enhancement and also the yield improvements are quite noticeable. The results of this investigation are tabulated in Scheme 5.

Preparation of bis(β-amino) alcohols which are potential compounds for polymerization and also could be used as mutidentate ligands was achieved by the reaction of epoxides (2.5 M equiv.) with aromatic or aliphatic amines (1 M equiv.) in the presence of $Al(DS)_3{\cdot}3H_2O$ (0.1 mmol) in water. The reactions proceeded well and smoothly at room temperature and the desired bis(β-amino) alcohols were produced in excellent yields (Table 4).

Chemoselectivity of the method is of practical importance. Chemoselectivity of the method was also studied by the reaction of phenylglycidyl ether with 2-phenylamino ethanol which carries both –NH and –OH functional groups in a single molecule and also with different binary mixtures as indicated in Table 5. In the presence of this catalyst in aqueous media, excellent chemoselectivity for the ring opening of phenylglycidyl ether

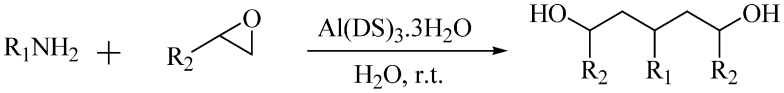


Entry	Aromatic Amine	Epoxide	**A** (Time/Yield %)[a]	**B** (Time/Yield %)[a]
1	3-Nitroaniline	phenylglycidyl ether	5/97	72/10
2	3-Nitroaniline	2,3-Epoxypropylmethacrylate	3/94	48/90
3	3-Cyanoaniline	phenylglycidyl ether	5/94	72/20
4	3-Cyanoaniline	2,3-Epoxypropylmethacrylate	3/96	48/92
5	3-Nitroaniline	styrene oxide	1/90	24/98
6	aniline	styrene oxide	45 min/88	14/97

[a]our observations

Scheme 5.

Table 4
Conversion of epoxides to bis(β-amino) alcohols using 2.5 molar ratios of epoxides with 1 molar ratio of aromatic amines

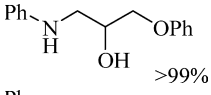
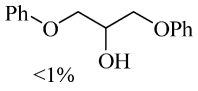
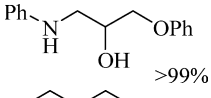
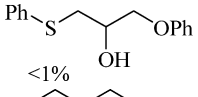
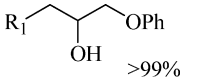
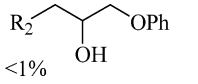
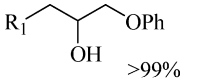
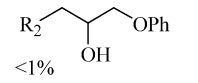
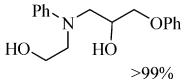
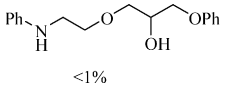


R_1=aromatic and aliphatic

Entry	Aromatic amines	Epoxide	Time (h)	Isolated yield (%)[a]
1	4-Chloroaniline	Phenylglycidyl ether	5	97
2	4-Methylaniline	Phenylglycidyl ether	3	94
3	Aniline	Phenylglycidyl ether	1	91
4	Aniline	2,3-Epoxypropylmethacrylate	5	94
5	Isopropyl amine	Phenylglycidyl ether	1	>85

[a] Products were characterized by ^{1}H NMR, ^{13}C NMR spectroscopy.

Table 5
Chemoselective ring opening reaction of phenylglycidyl ether with different binary mixtures and also with 2-phenylamino-ethanol

Entry	Epoxide	Binary mixture	Product[a]	
1	Phenylglycidyl ether	Aniline + phenol	 >99%	 <1%
2	Phenylglycidyl ether	Aniline + thiophenol	 >99%	 <1%
3	Phenylglycidyl ether	4-Nitro aniline + 4-methoxy phenol	 >99% R_1= 4-nitro aniline	 <1% R_2= 4-methoxy phenol
4	Phenylglycidyl ether	4-Nitro aniline + 4-methyl thiophenol	 >99% R_1= 4-nitro aniline	 <1% R_2= 4-methyl thiophenol
5	Phenylglycidyl ether	2-Phenylamino-ethanol	 >99%	 <1%

[a] GC yields.

with different nucleophiles has been observed. The results of this study are shown in Table 5.

However, in order to explain the unique catalytic activity of aluminum tris (dodecyl sulfate) trihydrate in these reactions, we may suggest that $Al(DS)_3{\cdot}3H_2O$ acts as a Lewis acid–surfactant-combined catalyst in water and enforces its catalytic activity by its dual action. One is to activate the epoxide ring by the interaction of oxygen atom with aluminum cation core of the catalyst (Lewis acid part) and the second role of the catalyst is to wrap around the reacting molecules by its non-polar hydrocarbon tails and bring them closely together to facilitate their reaction [14].

3. Conclusion

In this study, we have reported facile and efficient conversion of epoxides to thiiranes in the presence of a catalytic amount of aluminum tris (dodecyl sulfate) trihydrate [$Al(DS)_3{\cdot}3H_2O$] in water at room temperature in excellent yields. We have also extensively studied the ring opening of structurally diverse epoxides with aromatic and aliphatic amines using water as a media in the presence of aluminum tris (dodecyl sulfate) trihydrate at room temperature. The yields of β-amino alcohols and bis(β-amino) alcohols by this catalytic method is excellent and proceeded with excellent regio- and chemoselectivity. The simplicity, efficiency, mild reaction conditions and using simply prepared water stable and a cheap Lewis acid–surfactant-combined catalyst are worthy of mention for the presented method which is a useful addition to the available methodologies for the preparation of amino alcohols and thiiranes from epoxides in aqueous media.

4. Experimental

4.1. General remarks

The chemicals were obtained from Fluka and Merck Chemical Companies. $Al(DS)_3{\cdot}3H_2O$ was prepared according to our reported procedure by the addition of hydrated aluminium chloride to an aqueous solution of sodium dodecyl sulfate in water [15]. Progress of the reactions were monitored by TLC using silica gel SILG/UV 254 plates and Shimadzu GC MS-QP 1000 EX. The ^{1}H NMR and ^{13}C NMR spectra were recorded on a

Bruker Avance DPX-250, FT-NMR Spectrometer (δ in ppm). All yields refer to the isolated products.

4.2. General procedure for conversion of epoxides to their thiiranes with thiourea catalyzed by $Al(DS)_3{\cdot}3H_2O$ in water

To a solution of epoxide (1 mmol) in water (5 mL) was added $Al(DS)_3{\cdot}3H_2O$ (0.082 g, 0.1 mmol) and the resulting solution was stirred for 5 min. Then, thiourea (0.114 g, 1.5 mmol) was added to the mixture and the resulting mixture was magnetically stirred vigorously at room temperature for the appropriate reaction time (Table 1). The progress of the reaction was monitored by TLC or GC. Then the resulting mixture was extracted by continuous extraction with Et_2O (5 mL). The organic phase was separated and dried over anhydrous Na_2SO_4 which after filtration and evaporation of the solvent gave the crude desired product. Purification of the crude product was performed by column chromatography on silica gel eluted with petroleum ether (40–60 °C) to afford the pure product in excellent yields (Table 1).

4.3. Typical spectral data

Table 1, entry 1: ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 2.14$ (dd, $J = 5.2$ Hz, 1.2 Hz, 1H), 2.41 (dd, $J = 6.0$ Hz, 1.2 Hz, 1H), 3.08 (m, 1H), 3.73 (dd, $J = 10.2$ Hz, 6.8 Hz, 1H), 3.98 (dd, $J = 10.2$ Hz, 5.2 Hz, 1H), 6.77 (m, 3H), 7.14 (m, 2H); ^{13}C NMR (62.5 MHz, $CDCl_3$): $\delta = 158.51, 129.69, 121.33, 114.78, 72.98, 31.60, 24.07$.

4.4. General procedure for the reaction of aromatic amines with epoxides catalyzed by $Al(DS)_3{\cdot}3H_2O$ to produce β-amino alcohols in water

To a solution of an epoxide (1 mmol) in water (5 mL) was added $Al(DS)_3{\cdot}3H_2O$ (0.082 g, 0.1 mmol), and the resulting solution was stirred for 5 min. Then, an amine (1.5 mmol) was added to the mixture and was magnetically stirred at room temperature for the appropriate reaction time (Tables 2 and 3). The progress of the reaction was monitored by TLC and GC. Then the resulting mixture was extracted by continuous extraction with Et_2O (5 mL). The organic phase was dried over anhydrous Na_2SO_4 which after filtration and evaporation of the solvent gave the crude desired product. Purification of the crude product was performed by column chromatography on silica gel eluted with EtOAc/petroleum ether (40–60 °C) to afford the pure product in excellent yields (Tables 2 and 3). For the preparation of bis(β-amino) alcohols the molar ratio of epoxide/amine/catalyst was 2.5 mmol/1 mmol/0.1 mmol (Table 4).

4.5. Typical spectral data

Table 2, entry 9: ^{1}H NMR (250 MHz, $CDCl_3$): 1.90 (s, 3H), 3.05–3.36 (complex, 2H), 3.90–4.10 (broad, exchangeable with D_2O, 1H), 4.10–4.19 (m, 2H), 4.60–4.80 (broad, exchangeable with D_2O, 1H), 5.53 (s, 1H), 6.11 (s, 1H), 6.51–6.54 (m, 1H), 7.22–7.38 (complex, 4H), 7.70–7.80 (complex, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$): $\delta = 18.36, 49.54, 66.97, 68.44, 104.82, 118.05, 120.22, 125.64, 126.54, 126.68, 128.67, 129.37, 134.38, 135.89, 148.02, 167.73$.

Table 2, entry 11: ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 3.22$–3.36 (m, 2H), 3.63–3.93 (complex, 6H), 4.29–4.31 (m, 1H), 4.70 (broad, exchangeable with D_2O, 1H), 5.30 (broad, exchangeable with D_2O, 1H), 6.74–7.31 (complex, 10H); ^{13}C NMR (62.5 MHz, $CDCl_3$): $\delta = 55.53, 56.73, 60.46, 68.66, 69.68, 112.68, 115.17, 117.05, 121.32, 129.52, 129.73, 147.96, 158.64$.

Table 4, entry 1: ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 3.22$–3.26 (complex, 1H), 3.51–3.55 (complex, 2H), 3.87–3.96 (complex, 6H), 4.23–4.29 (complex, 2H), 4.23–4.29 (broad, 1H, exchangeable with D_2O), 6.58–7.56 (complex, 14H); ^{13}C NMR (62.5 MHz, $CDCl_3$): $\delta = 55.49, 68.41, 69.47, 114.57, 114.77, 121.36, 121.95, 129.21, 129.64, 146.21, 158.34$.

Table 4, entry 4: ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 1.90$–1.97 (partially by long range coupling, 6H), 2.70 (broad, exchangeable with D_2O, 1H), 3.38–4.25 (complex, 10H), 5.30 (broad, exchangeable with D_2O, 1H), 5.57–5.62 (m, 2H), 6.07–6.16 (m, 2H), 6.77–7.27 (complex, 5H). ^{13}C NMR (62.5 MHz, $CDCl_3$): $\delta = 17.98, 44.25, 49.19, 64.99, 112.05, 116.74, 125.99, 129.13, 135.87, 147.37, 167.00$.

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