

KF/Nano-clinoptilolite Catalyzed Aldol-Type Reaction of Aldehydes with Ethyl Diazoacetate

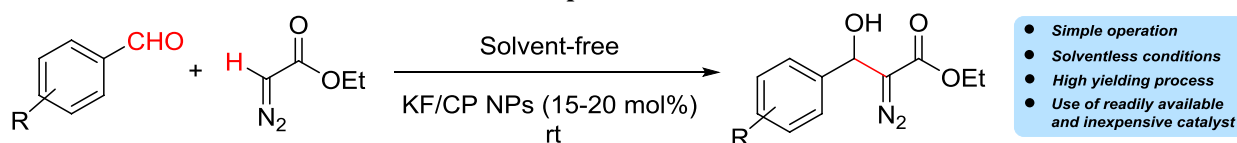
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Abstract Potassium fluoride supported on nano-clinoptilolite (KF/CP NPs) was used as an excellent catalytic system for direct aldol-type condensation of aldehydes with

ethyl diazoacetate under solvent-less conditions. A variety of α -diazo carbonyl derivatives were prepared in good to excellent yields in short reaction times.

Graphical Abstract



Keywords KF/nano-clinoptilolite · Aldol-type reaction · Ethyl diazoacetate · α -Diazo carbonyl derivatives

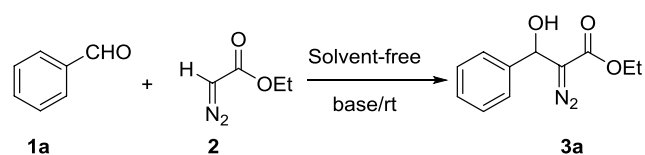
1 Introduction

Diazo compounds have received considerable attention in recent years due to their wide applications in synthetic organic chemistry [1], among which α -diazo carbonyl derivatives are the most fundamental chemicals as they are usually the starting materials for the synthesis of other useful organic chemicals [2, 3]. These compounds are useful intermediates in the synthesis of amino alcohols and amino acids. α -Diazo carbonyl compounds are readily obtained from the corresponding aldehyde derivatives upon treatment with acyldiazomethanes, generally by deprotonation of the acyldiazomethane in the presence of a strong base catalyst. A large number of base catalysts have been reported to effect the condensation of acyldiazomethanes with aldehydes including lithium diisopropylamide (LDA) [4, 5], sodium hydride [6], potassium hydroxide [7, 8] and quaternary ammonium hydroxide [9]. Although the generated anionic species can efficiently react with aldehydes to afford α -diazo- β -hydroxy carbonyl compounds, the strong

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Table 1 Effect of various bases for the addition of EDA to benzaldehyde

Entry	Base (mol%)	Time (h)	Yield (%) ^a
1	NaOH (20)	8	50
2	KOH (20)	5	51
3	K ₂ CO ₃ (20%)	10	45
4	Na ₂ CO ₃ (20%)	12	38
5	KF (20%)	8	42
6	CP ^b	10	<5
7	KF/CP (20%)	5	56
8	CaH ₂ (20%)	12	30
9	–	24	0
10	K ₃ PO ₄ (20%)	12	trace
11	KF/CP NPs (20%)	5	86
12	KF/CP NPs (15%)	5	85 ^c
13	KF/CP NPs (10%)	5	67

Benzaldehyde (1 mmol), EDA (1.2 mmol), at room temperature

^aYield by NMR^b0.1 g of CP was used^cAverage of two runs

basic reaction medium makes these reactions less attractive for synthetic organic chemists. The use of Lewis bases like 1,8-diazobicyclo-[5.4.0]undec-7-ene (DBU) enables the same transformation under milder conditions [10, 11]. In addition, various efficient catalytic systems in water including pyrrolidine [12], DBU [13], KO^tBu [14], ionic liquids [15, 16], KO^tBu under solvent-free conditions [17, 18] and DBU/flow conditions [19] have been developed as a green alternative to the traditional organic solvents. Heterogeneous base catalysts have also shown great potential as catalysts for this transformation. Kantam et al. reported that ethyl diazoacetate (EDA) can be condensed with a range of aromatic aldehydes in the presence of magnesium/lanthanum mixed oxide to afford the desired products in reasonable yields [20, 21]. The same authors later utilized silica-supported tetramethylguanidine catalyst to the reaction of EDA with divers aldehydes, leading to the improvement of yields with shorter reaction times [22]. The use of a solid catalyst allows replacement of the soluble homogeneous catalysts, contributing to a reduction of waste to conform to the concept of green technology. Recently, we have developed a simple catalytic system using potassium fluoride impregnated on clinoptilolite (KF/CP) in various

organic transformations [23–28]. KF/CP exhibits a greatly enhanced basicity compared to the potassium fluoride. This property can be boosted when CP nanoparticles were used, as the large surface areas of such particles provides enhanced catalytic activities [29].

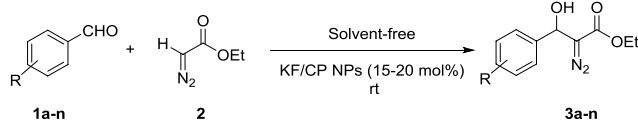
In the past decade, green chemistry has attracted much attention, and it currently encompasses major areas of the chemical sciences. The recent increase of environmental consciousness towards clean technology emphasizes the point that the best solvent is no solvent. Solvent-free reactions have many benefits that are reduced pollution, lower costs and the simplicity of the processes. In continuation of our ongoing program to develop green synthetic protocols, we here report a simple, green, and selective protocol for the synthesis of α -diazo carbonyl compounds using catalytic amounts of KF/CP NPs as an efficient solid base catalyst [29, 30].

2 Results and Discussion

To confirm the catalytic activity of KF/CP NPs, various solid bases were studied for the preparation of ethyl-2-diazo-3-hydroxy-3-phenylpropionate (**3a**) by the reaction of benzaldehyde (**1a**) with ethyl diazoacetate (**2**) at room temperature under solvent-free conditions, and the results are summarized in Table 1.

Potassium hydroxide shows better catalytic performance at shorter reaction time than other bases such as NaOH, K₂CO₃ and Na₂CO₃ (Table 1, entries 1–4). Potassium fluoride alone afforded only low yield of product **3a** (Table 1, entry 5). The reaction mediated clinoptilolite afforded less than 5% of desired product (Table 1, entry 6). Potassium fluoride supported on clinoptilolite afforded moderate yield of desired product (Table 1, entry 7). Only 30% of product **3a** was formed when CaH₂ was used as catalyst (Table 1, entry 8). No addition product was obtained when the reaction carried out without catalyst (Table 1, entry 9), even after prolonged reaction time. Only trace of product was formed when K₃PO₄ was used as catalyst (Table 1, entry 10). However, the outcome of reaction was significantly enhanced when 20% of KF/CP NPs was used as catalyst (Table 1, entry 11). Reducing the amounts of catalyst to 15 mol% did not affect the outcome of reaction and product **3a** was obtained in 85% yield after 5 h (Table 1, entry 12). However, decreasing the catalyst loading to 10 mol% led to a substantial decrease in the yield of **3a** (Table 1, entry 13). Thus 15 mol% of KF/CP NPs was chosen for subsequent substrates.

The efficiency of the condensation involving the KF/CP NPs was examined with a series of aldehydes **1** and ethyl diazoacetate **2** under solvent-free conditions at room temperature (Table 2).

Table 2 KF/CP NPs-catalyzed synthesis of α -diazo carbonyl compounds

Entry	Aldehyde	Time (h)	Product	Yield (%) ^a
1		4	3a	85
2		3	3b	77
3		3	3c	80
4		3	3d	87
5		4	3e	78
6		5	3f	60
7		4	3g	72
8		4	3h	70
9		4	3i	78
10		5	3j	50 ^b
11		5	3k	55 ^b
12		6	3l	52 ^b
13		3	3m	70 ^b
14		4	3n	72

Reaction conditions benzaldehyde (1 mmol), EDA (1.2 mmol), KF/CP NPs (15–20 mol%), at room temperature

^aIsolated yield

^bReactions performed using 20 mol% of KF/CP NPs

The reaction is efficient for variety of aliphatic and aromatic aldehydes, giving α -diazo carbonyl compounds in moderate to high yields. Both electron-rich and electron-deficient aldehydes worked well, affording the desired products in good to high yields. Electron-deficient aldehydes gave relatively higher yields than their electron-rich counterparts (Table 2). Heterocyclic aldehydes, such as furfural and 2-pyridinecarboxaldehyde produced corresponding products **3h** and **3i** in good yields. However, aliphatic aldehydes gave comparatively lower yields than aromatic aldehydes. α,β -Unsaturated aldehydes such as cinnamaldehyde also gave good yields of product. The effect of temperature on the outcome of the reaction has been investigated. Interestingly, heating the reaction to 80 °C led to the formation of substantial amounts of ethyl 2-benzoylacetate **4** through the intermediate **3a** (Scheme 1).

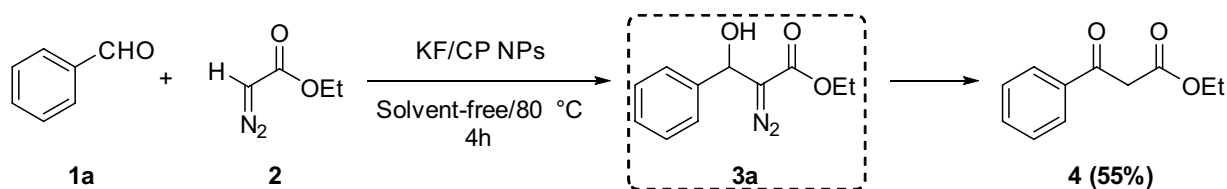
Clinoptilolite nanoparticles have been easily prepared by grinding in a planetary ball mill using a zirconia vial set under dry conditions with a time period of about 20 min. The KF/CP NPs catalyst was then prepared according to previously reported procedure. The particle size and morphology of CP NPs and KF@CP NPs samples have been analyzed by SEM and TEM. The SEM images showed polydisperse of morphology and wide range of particle size distributions (Fig. 1). These features are influenced by milling process and thus can be varied. Although, the TEM images showed some particles with micro-dimension but the major particles are formed in smaller sizes with spherical shapes in about 35–65 nm size range. The TEM images of the KF@CP NPs samples show the KF@CP NPs are rather dispersed relative to CP NPs. This could be explained by adsorption of potassium fluoride onto the CP NPs that increase the negative charges resulted from free fluoride anions on the surface of zeolite leading the electrostatic repulsion between the layers.

A plausible reaction mechanism of the present fluoride-catalyzed aldol-type condensation has shown in Scheme 2. The reaction involves the abstraction of the proton from EDA to generate a carbanion stabilized by potassium captured in the nano-CP. The EDA carbanion would react with aldehydes to release the product upon protonation with HF formed due to the abstraction of protons.

In order to show the merit of this method, the yield of the reaction of ethyl diazoacetate and benzaldehyde has been compared with some other protocols using different catalysts and conditions (Table 3).

It is clear that KF/CP nano particles can efficiently catalyze the synthesis of ethyl 2-diazo-3-hydroxy-3-phenylpropanoate (**3a**) and it is comparable to previous reported methods, however, the present method benefits from cheapness and catalyst recovery.

The reusability of the catalyst was also investigated. The used KF/CP NPs, obtained after filtration from the reaction



Scheme 1 Direct formation of ethyl 2-benzoylacetate in the presence of KF/CP NPs at 80 °C

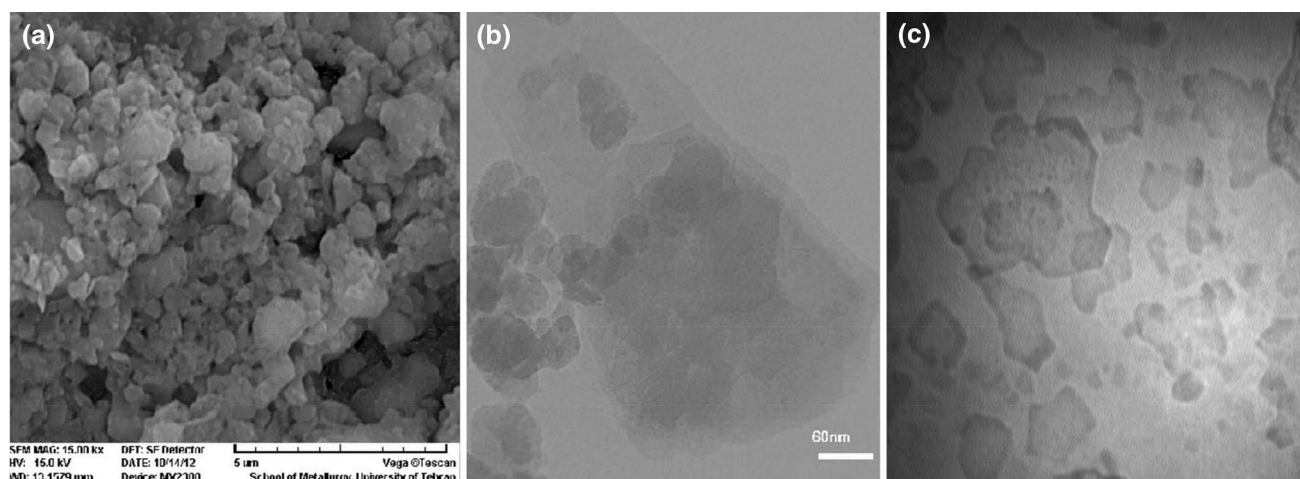


Fig. 1 SEM (a) and TEM (b) images of CP NPs. TEM image of KF/CP NPs (c)

Scheme 2 A plausible mechanism for aldol-type condensation of aldehydes with ethyl diazoacetate in the presence of KF/CP NPs

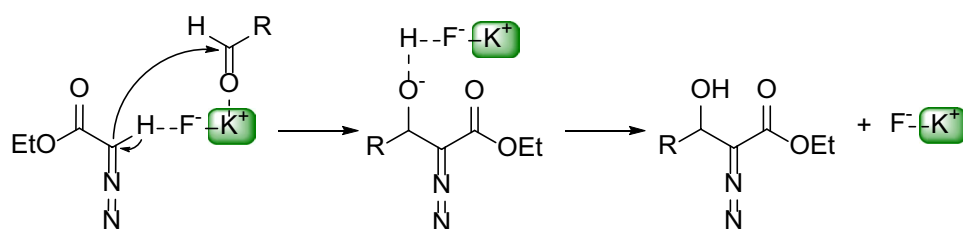


Table 3 Comparison among synthesis of ethyl 2-diazo-3-hydroxy-3-phenylpropanoate (**3a**) from the reaction of benzaldehyde with ethyl diazoacetate using various catalysts and conditions

Entry	Conditions	Time (h)	Catalyst	Yield%
1 [20]	Water/rt	7	Mg/La mixed oxide	85
2 [15]	Water/rt	6	MR-IMZ-OH	90
3 [17]	Solvent-free/rt	2	<i>tert</i> -BuOK	87
4 [10]	MeCN/rt	8	DBU	78
5 [13]	water/rt	24	DBU	74
6 [12]	Water/rt	2	pyrrolidine	65
7 [22]	DMSO/rt	2	SiO ₂ -TMG	90
8 [21]	DMSO/rt	2	NAP-MgO	78
9	Solvent-free/rt	4	KF/CP NPs	85

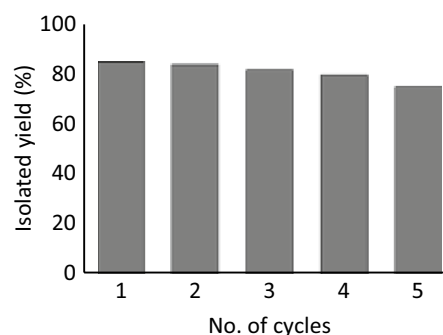


Fig. 2 Reusability of KF/CP NPs for the condensation of EDA with benzaldehyde

mixture was thoroughly washed with dry ethyl acetate, dried under vacuum and reused again for the condensation of EDA with benzaldehyde. The recyclability of the

catalyst was confirmed when it was found to exhibit good activity even after the five run without significant decrease in the yield (Fig. 2).

In conclusion, we have developed a convenient and efficient method for the preparation of β -hydroxy- α -diazo carbonyl compounds by the condensation of aldehydes with ethyl diazoacetate using KF/CP NPs as a heterogeneous catalyst under solvent-less conditions at room temperature. The catalyst can be easily recovered and reused for next run without significant loss of catalytic activity. This methodology offers significant improvements for the synthesis of β -hydroxy- α -diazo carbonyls with regard to yield of products, cost efficiency, simplicity in operation, and green aspects by avoiding toxic catalysts and solvents.

3 Experimental Section

All chemicals used in this work were purchased from Merck and Sigma and were used without further purification. Clinoptilolite was obtained from Afrandtooska Company in the region of Semnan. The morphology of nanoparticles of KF/cclinoptilolite was characterized by scanning electron microscopy (SEM) using a Holland Philips XL30 microscope. Transmission electron microscopy (TEM) was carried out using a Zeiss EM10C microscope at 80 KV. The ^1H NMR (400 MHz) and ^{13}C NMR (100 MHz) were measured with a Bruker AVANCE-DRX. FT-NMR spectrometer (δ in ppm). ^1H and ^{13}C spectra were obtained for solutions in CDCl_3 using TMS as internal standard.

3.1 Preparation of KF/CP NPs

Nano-sized natural clinoptilolite zeolite was prepared by grinding in a planetary ball mill using a zirconia vial set in dry conditions with a time period of about 20 min. The KF/CP NPs catalyst was prepared according to our previously reported procedure [28]. Thus, 1 g of KF was dissolved in distilled water (10 ml) and nano-clinoptilolite (9 g). The mixture was stirred for an hour and then, the water was evaporated at 60–70 °C under reduced pressure and further dried at 70–80 °C in a vacuum drying oven for 30 h. The dried material was powdered using a mortar and pestle and stored in a desiccator until use.

3.2 General Procedure for the Synthesis of Compounds 3a–n

A mixture of aldehyde (1 mmol), ethyl diazoacetate (1.2 mmol), and KF/CP NPs (15–20 mol%) was stirred at room temperature for the time indicated in Table 2. The progress of reaction was monitored by TLC. Upon completion, dry ethyl acetate was added and the catalyst was

separated by filtration from the product solution and the filtrate washed further with dry ethyl acetate. The solvent of residue was removed under reduced pressure. The crude product was purified by column chromatography on silica gel. The products were characterized by the use of spectral data and comparison of their physical data with the literature.

3.2.1 Ethyl-2-diazo-3-hydroxy-3-phenylpropanoate (3a) [31]

^1H NMR (400 MHz, CDCl_3) δ : 1.28 (t, $J=7.1$, 3H), 3.48 (bs, 1H), 4.24 (q, $J=7.1$ Hz, 2H), 5.83 (s, 1H), 7.28–7.43 (m, 5H); ^{13}C NMR (100 MHz, CDCl_3) δ : 14.5, 61.2, 68.7, 125.7, 128.3, 128.7, 139.0, 166.6.

3.2.2 Ethyl-2-diazo-3-hydroxy-3-(4-chlorophenyl)-propanoate (3b) [31]

^1H NMR (400 MHz, CDCl_3) δ : 1.27 (t, $J=7.1$, 3H), 4.19–4.27 (m, 3H), 5.76 (d, $J=4.3$ Hz, 1H), 7.36–7.44 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ : 14.4, 61.5, 67.9, 127.2, 128.9, 134.1, 137.6, 166.4.

3.2.3 Ethyl-3-hydroxy-2-diazo-3-(2-chlorophenyl)-propanoate (3c) [31]

^1H NMR (400 MHz, CDCl_3) δ : 7.71 (m, 1H), 7.25–7.39 (m, 3H), 6.12 (s, 1H), 4.27 (q, $J=7.0$ Hz, 2H), 3.48 (bs, 1H), 1.29 (t, $J=7.0$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 14.3, 61.3, 66.2, 127.1, 127.3, 129.2, 129.4, 131.6, 136.5, 166.4.

3.2.4 Ethyl 2-diazo-3-(4-fluorophenyl)-3-hydroxypropanoate (3d) [31]

^1H NMR (400 MHz, CDCl_3) δ : 1.26 (t, $J=7.12$ Hz, 3H), 3.60 (s, 1H), 4.21 (q, $J=7.12$ Hz, 2H), 5.88 (s, 1H), 7.05–7.08 (m, 2H), 7.40–7.43 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 14.5, 61.3, 68.1, 115.6, 127.5, 134.9, 161.3, 163.7, 166.4.

3.2.5 Ethyl 2-diazo-3-(4-methylphenyl)-3-hydroxypropanoate (3e) [31]

^1H NMR (400 MHz, CDCl_3) δ : 1.28 (t, $J=7.1$ Hz, 3H), 2.35 (s, 3H), 3.42 (s, 1H), 4.25 (q, $J=7.1$ Hz, 2H), 5.88 (s, 1H), 7.18 (d, $J=8.0$ Hz, 2H), 7.30 (d, $J=8.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 14.5, 21.1, 61.2, 68.5, 125.7, 129.4, 135.5, 138.2, 166.5.

3.2.6 Ethyl-2-diazo-3-hydroxy-3-(4-methoxyphenyl)propanoate (**3f**) [31]

¹H NMR (400 MHz, CDCl₃) δ: 1.29 (t, *J* = 7.2, 3H), 3.18 (s, 1H), 3.79 (s, 3H), 4.24 (q, *J* = 7.2 Hz, 2H), 5.86 (s, 1H), 6.90 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 8.3 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ: 14.5, 55.3, 61.2, 68.3, 114.1, 127.0, 131.1, 159.4, 166.5.

3.2.7 Ethyl-2-diazo-3-hydroxy-3-(3-methoxyphenyl)propanoate (**3g**) [32]

¹H NMR (400 MHz, CDCl₃): δ 1.29 (t, *J* = 7.3 Hz, 3H), 3.82 (s, 3H), 3.05 (s, 1H), 4.27 (q, *J* = 7.3 Hz, 2H), 5.89 (s, 1H), 6.83 (m, 1H), 7.05–6.97 (m, 2H), 7.28–7.36 (m, 1H); ¹³C NMR (100 MHz, CDCl₃) δ: 14.4, 55.2, 61.2, 68.6, 111.1, 113.8, 117.9, 129.9, 140.5, 159.9, 166.5.

3.2.8 Ethyl-2-diazo-3-(furan-2-yl)-3-hydroxypropanoate (**3h**) [20]

¹H NMR (400 MHz, CDCl₃) δ: 1.27 (t, *J* = 7.12 Hz, 3H), 3.40 (s, 1H), 4.24 (q, *J* = 7.12 Hz, 2H), 5.80 (s, 1H), 6.33–6.38 (m, 2H), 7.38–7.35 (m, 1H); ¹³C NMR (100 MHz, CDCl₃) δ: 14.4, 61.3, 63.4, 107.5, 110.4, 142.8, 152.1, 166.1.

3.2.9 Ethyl-2-diazo-3-hydroxy-3-(2-pyridinyl)-propanoate (**3i**) [20]

¹H NMR (400 MHz, CDCl₃) δ: 1.29 (t, *J* = 7.1 Hz, 3H), 4.24 (q, *J* = 7.1 Hz, 2H), 5.80 (s, 1H), 7.24–7.27 (m, 1H), 7.43 (d, *J* = 8.1 Hz, 1H), 7.73 (t, *J* = 8.0 Hz, 1H), 8.53 (d, *J* = 8.1 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ: 14.6, 61.1, 67.3, 121.1, 123.3, 137.1, 148.1, 158.1, 166.1.

3.2.10 Ethyl-2-diazo-3-hydroxy-pentanoate (**3j**) [33]

¹H NMR (400 MHz, CDCl₃) δ: 1.02 (t, *J* = 7.8 Hz, 3H), 1.32 (t, *J* = 7.0 Hz, 3H), 1.69–1.55 (m, 2H), 1.83–1.57 (m, 3H), 2.83 (bs, 1H), 4.24 (q, *J* = 7.8 Hz, 2H), 4.67–4.58 (m, 1H); ¹³C NMR (100 MHz, CDCl₃) δ: 9.9, 14.5, 27.1, 27.8, 60.9, 68.1.

3.2.11 Ethyl-2-diazo-3-hydroxyheptanoate (**3k**) [15]

¹H NMR (400 MHz, CDCl₃) δ: 0.89 (t, *J* = 7.1 Hz, 3H), 1.29 (t, *J* = 7.1 Hz, 3H), 1.30–1.77 (m, 6H), 2.62 (bs, 1H),

4.25 (q, 7.2 Hz, 2H), 4.63–4.69 (m, 1H); ¹³C (100 MHz, CDCl₃) δ: 13.9, 14.4, 22.3, 27.7, 33.7, 61.0, 66.6, 166.7.

3.2.12 Ethyl-2-diazo-3-hydroxy-4-methylpentanoate (**3l**) [15]

¹H NMR (400 MHz, CDCl₃) δ: 0.95 (d, 3H, *J* = 6.8 Hz), 1.07 (d, *J* = 6.9 Hz, 3H), 1.29 (t, *J* = 7.8 Hz), 1.84–1.94 (m, 1H), 2.57 (bs, 1H), 4.20–4.28 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 14.4, 18.7, 18.8, 32.7, 60.9, 72.2, 166.7.

3.2.13 Ethyl-3-cyclohexyl-2-diazo-3-hydroxypropanoate (**3m**) [15]

¹H NMR (400 MHz, CDCl₃) δ: 0.93–1.27 (m, 9H), 1.51–1.76 (m, 4H), 1.99–2.01 (m, 1H), 3.02 (s, 1H), 4.17 (q, *J* = 7.11 Hz, 2H), 4.25 (d, *J* = 8.57 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ: 14.5, 25.7, 26.3, 29.2, 42.1, 61.0, 71.3, 166.9.

3.2.14 Ethyl-2-diazo-3-hydroxy-5-phenylpent-4-enoate (**3n**) [31]

¹H NMR (400 MHz, CDCl₃) δ: 1.28 (t, *J* = 7.1, 3H), 2.83 (bs, 1H), 4.26 (q, *J* = 7.1 Hz, 2H), 5.43–5.55 (m, 1H), 6.21 (dd, *J* = 15.8, 5.4 Hz, 1H), 6.79 (d, *J* = 15.8 Hz, 1H), 7.22–7.40 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ: 14.4, 61.1, 66.8, 114.2, 126.1, 126.7, 128.5, 131.8, 135.1, 166.2.

3.2.15 Ethyl-2-benzoylacetate (**4**) [34]

¹H NMR (400 MHz, CDCl₃) δ: 1.25 (t, *J* = 7.1 Hz, 3H), 3.98 (s, 2H), 4.20 (q, *J* = 7.1 Hz, 2H), 7.43–7.95 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ: 14.1, 46.0, 61.5, 128.4, 128.6, 133.7, 136.0, 167.6, 192.7.

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