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Highly efficient synthesis of amine-substituted diazoacetoacetates from aldimines and vinyldiazoacetate catalysed by MgI_2 etherate

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A mild and efficient procedure is described for the synthesis of amine-substituted diazoacetoacetates by the addition of ethyl 3-(*tert*-butyldimethylsilanoxy)-2-diazobut-3-enoate to aldimines catalysed by MgI_2 etherate (MgI_2•$(OEt_2)_n$) in MeCN. The reaction produces good to excellent yield at room temperature.

Keywords: aldimine, silyl enol ether, diazoacetoacetates, MgI_2 etherate

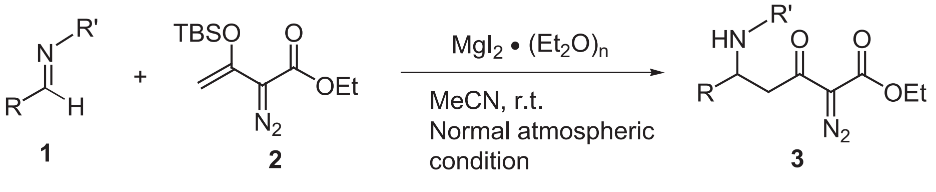
The chemistry of α-diazocarbonyl compounds has received considerable attention because of the diverse range of synthetically useful transformations that these compounds can undergo.[1–2] Diazoacetoacetates are widely used reactants for metal carbene transformations in the synthesis of natural products and compounds of pharmaceutical interest.[3–6] Catalytic reactions of diazoacetoacetate involving addition or insertion that produce cyclopropanes, cycloalkanones and β-lactams are well known because of their higher thermal stability and unique reactivity compared to other classes of diazo compounds.[7–11] A Mukaiyama–Mannich type reaction of methyl 3-(*tert*-butyldimethylsilanoxy)-2-diazobut-3-enoate with imines catalysed by scandium(III) triflate was reported by Doyle's research group.[12] In addition, Mukaiyama–Mannich type reaction of a silyl enol of α-diazoacetate and an *N*-(benzylidene) derivative was investigated in the presence of Lewis acids, such as BF_3•Et_2O, $TiCl_4$, $Cu(OTf)_2$, $La(OTf)_3$ and TBSOTf. However, only TBSOTf showed reaction and gave a low yield of the desired product. Typically, the nucleophilic addition product was obtained by reactions of the diazoacetate enolate with the imine in the presence of a strong base LiHMDS [lithium bis(trimethylsilyl)-amide] in good yield.[13] Although the anionic species which was generated can efficiently add to carbonyl compounds, some of these methods clearly require strongly basic reagents and anhydrous conditions. To the best of our knowledge, there are few reports of Mukaiyama–Mannich addition reactions of the silyl enol ethers of diazo compounds with imines.

We have recently reported an efficient and facile method for the synthesis of α-diazo-β- hydroxy esters by coupling aldehydes with ethyl diazoacetate (EDA) and promoted by MgI_2 etherate (MgI_2•$(OEt_2)_n$) in the presence of Et_3N at room temperature under normal atmospheric conditions.[14] In continuation of our interest on the catalytic applications of MgI_2 etherate for various organic transformations, we wish to describe a simple and efficient protocol for the Mukaiyama–Mannich addition of ethyl 3-(tertbutyldimethylsilanoxy)-2-diazobut-3-enoate **2** with imines catalysed by MgI_2 etherate under mild and neutral reaction conditions in good to excellent yields (Scheme 1).

Results and discussion

Initially, we prepared a variety of imines from aromatic aldehydes and aromatic amines. To optimise the reaction (Scheme 1), we chose *N*-(4-nitrobenzylidene)aniline as a model substrate and we determined the yield of product **3b** using various solvents (Table 1). Amongst the various solvents that were screened, an excellent yield was obtained in MeCN as the reaction media in a short time. A good yield was also obtained in CH_2Cl_2, $ClCH_2CH_2Cl$ and $CHCl_3$. Moderate yields were produced in toluene. The reaction was very sluggish in the coordinative polar solvents, such as Et_2O and THF. The reaction was not observed in more polar solvents, such as DMF, dioxane, DMSO and MeOH. In order to examine the halide anion effect, halogen analogs of MgI_2 etherate, $MgBr_2$ etherate and $MgCl_2$ etherate, were compared under parallel reaction conditions in MeCN (5 mol% of promoters). $MgCl_2$ etherate was almost inactive. $MgBr_2$ etherate is less effective in terms of substrate conversion and yield.

With the optimal conditions in hand, a variety of aldimines were subjected to this reaction. There was no need to exclude moisture and oxygen from the reaction system. The experimental results are summarised in Table 2. As shown in Table 2, the reaction proceeded smoothly at room temperature and gave good to excellent yields. The Mukaiyama–Mannich addition of the aldimines **1a**–**1d**, derived from benzaldehyde, *p*-nitrobenzaldehyde, *p*-anisaldehyde and *p*-methylbenzaldehyde with aniline, proceeded very smoothly and provided the Mannich adduct **3a**–**3d** in excellent yield, (Table 2, entries 1–4). The use of *p*-nitroaniline, *p*-methoxyaniline and *p*-methylaniline as the imine component was examined and the Mannich reaction products **3e**–**3g** was obtained in good yields (Table 2, entries 5–7). Significantly, the reaction of aldimine, prepared from *p*-nitrobenzaldehyde and *p*-methoxyaniline, also gave the desired product **3h** in nearly quantitative yield (Table 2, entry 8). The Mannich addition of the imine **1i** derived from cinnamaldehyde and aniline produced the adduct **3i** in 90% yield without any decomposition or polymerisation under the present reaction conditions (Table 2, entry 9). Moreover, the reaction of heteroaromatic imine **1j** and **1k** derived from 2-thiophenealdehyde and furfural also proceeded in good



Scheme 1 MgI_2 etherate-promoted Mukaiyama–Mannich type addition of aldimines with vinyldiazoacetate **2**.

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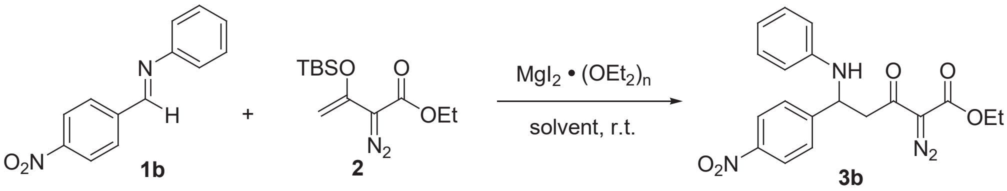
Table 1 The effect of solvents on the addition of the aldimines of vinyldiazoacetate 2 catalysed by $MgI_2{\bullet}(Et_2O)_n$[a]



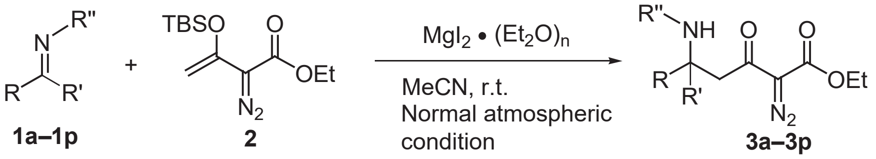
Entry	Solvent	Time/h	Yield/%[b]
1	CH_2Cl_2	3	85
2	$ClCH_2CH_2Cl$	3	86
3	$CHCl_3$	3	89
4	CH_3CN	0.5	98
5	$PhCH_3$	5	77
6	THF	5	62
7	Et_2O	5	58
9	DMF	12	NR[c]
10	MeOH	12	NR
11	Dioxane	12	NR
12	DMSO	12	NR

[a] Reaction conditions: A stirred solution of aldimine **1b** (1.0 mmol) and vinyldiazoacetate **2** (1.2 mmol) in the various solvents was treated with 5 mol % $MgI_2{\cdot}(Et_2O)_n$ and the reaction mixture was stirred at room temperature for various lengths of time.

[b] Yields after silica gel column chromatography.

[c] NR denotes no reaction.

Table 2 The Mukaiyama–Mannich addition to aldimines of vinyldiazoacetate 2 catalysed by $MgI_2{\bullet}(OEt_2)_n$[a]



Entry	R	R’	R”	Time/h	Product	Yield/%[b]
1	C_6H_5	H	C_6H_5	3	**3a**	92
2	4-$NO_2C_6H_4$	H	C_6H_5	0.5	**3b**	96
3	4-$CH_3OC_6H_4$	H	C_6H_5	1	**3c**	97
4	4-$CH_3C_6H_4$	H	C_6H_5	3	**3d**	95
5	C_6H_5	H	4-$NO_2C_6H_4$	2	**3e**	93
6	C_6H_5	H	4-$CH_3OC_6H_4$	2	**3f**	91
7	C_6H_5	H	4-$CH_3C_6H_4$	3	**3g**	90
8	4-$NO_2C_6H_4$	H	4-$CH_3OC_6H_4$	1	**3h**	98
9	(*E*)-$C_6H_5CH{=}CH$	H	C_6H_5	3	**3i**	90
10	2-thienyl	H	C_6H_5	3	**3j**	89
11	2-furfuryl	H	C_6H_5	2	**3k**	90
12	C_6H_5	H	$C_6H_5CH_2$	12	NA[c]	NR[d]
13	C_6H_5	H	(*S*)-$C_6H_5CH(CH_3)$	12	NA	NR
14	C_6H_5	CH_3	C_6H_5	8	NA	NR
15	4-$NO_2C_6H_4$	CH_3	4-$CH_3OC_6H_4$	8	NA	NR
16	2-thienyl	CH_3	4-FC_6H_4	8	NA	NR

[a] Reaction conditions: A solution of aldimine **1** (1.0 mmol) and vinyldiazoacetate **2** (1.2 mmol) in MeCN was treated with 5 mol% of $MgI_2{\cdot}(Et_2O)_n$ and the reaction mixture was stirred at room temperature for various lengths of time.

[b] Yields after silica gel column chromatography.

[c] NA denotes not formed.

[d] NR denotes no reaction.

yields (Table 2, entries 10 and 11). Apparently, the aldimines derived from aromatic aldehydes or anilines bearing electron-donating and electron-withdrawing groups in the aromatic ring reacted smoothly to afford the desired Mannich adducts in good to excellent yields. Furthermore, we have observed that the use of aromatic amine as the imine component gave, in general, good results whereas aliphatic amine did not yield any product even after prolonging the reaction time in the presence of MgI_2 etherate (Table 2, entries 12 and 13). In addition, various ketoimine substrates were also unreactive toward to vinyldiazoacetate **2** in the presence of MgI_2 etherate (Table 2, entries 14–16).

In conclusion, we have found that a facile and efficient Mukaiyama–Mannich addition to aldimines of TBSO-vinyldiazoacetate derivatives occurs in the presence of a MgI_2 etherate system. This catalytic reaction permits the Mukaiyama–

Mannich reaction to be carried out under essentially neutral conditions at room temperature in good to excellent yields. We are examining other carbon–carbon bond forming reactions based on TBSO-vinyldiazoacetate derivatives catalysed by MgI_2 etherate. Enantioselective variants of this reaction will be our next target in this area.

Experimental

The purification of the products by flash column chromatography, utilised silica gel (200 ~ 300 mesh) and light petroleum ether (PE, b.p. 60 ~ 90 °C) were used. ^1H NMR spectra were obtained on a Bruker AM-500 spectrometer with TMS as the internal standard and $CDCl_3$ as the solvent. The reactions were monitored by TLC on silica-gel polygram SILG/UV 254 plates. Melting points were measured on a BUCHI B-540 and uncorrected. FTIR were recorded on a Bruker Tensor 27 spectrometer. HRMS were determined on a Waters **GCT** Premier spectrometer. All compounds were identified by ^1H NMR and the data are in good agreement with those reported.

The typical procedure for the synthesis of ethyl 2-diazo-5-phenyl-3-oxo-5-(phenylamino)pentanoate: A stirred solution of *N*-benzylideneaniline **1a** (1.0 mmol) in MeCN (5 mL) was treated with freshly prepared MgI_2 etherate (0.05 mmol) [15] at room temperature, followed by the addition of the vinyldiazoacetate **2** (1.2 mmol). The resulting homogeneous reaction mixture was stirred at room temperature for 3 h and quenched by saturated $NaHCO_3$ aqueous solution. The product was recovered with ethyl acetate. Chromatographic purification of the crude product on silica gel gave *ethyl 2-diazo-5-phenyl-3-oxo-5-(phenylamino) pentanoate* **3a** in 92% yield.

Spectroscopic data for products **3a**–**3k** *(Table 2, entries 1–11)*

Ethyl 2-diazo-5-phenyl-3-oxo-5-(phenylamino)pentanoate (**3a**)[16]: white solid. m.p. 108–109 °C. R_f = 0.11 (PE:EA = 5:1). δ_H 1.53 (t, 3H, J = 7.1 Hz), 3.28 (dd, 1H, J = 4.6, 14.4 Hz), 3.65 (dd, 1H, J = 9.6, 14.4 Hz), 4.49 (dd, 1H, J = 4.6, 9.4 Hz), 4.59 (q, 2H, J = 7.1 Hz), 5.03 (s, 1H), 6.33 (d, 2H, J = 8.0 Hz), 6.61 (t, 1H, J = 7.3 Hz), 6.90 (d, 2H, J = 8.3 Hz), 7.23 (t, 1H, J = 7.5 Hz), 7.33 (t, 2H, J = 7.6 Hz), 7.45 (d, 2H, J = 7.5 Hz).

Ethyl 2-diazo-5-(4-nitrophenyl)-3-oxo-5-(phenylamino)pentanoate (**3b**): yellowish oil. R_f = 0.21 (PE:EA = 5:1). IR (film) ν (cm^{-1}) 3397 (N–H), 3054, 2141 (C=N=N), 1713 (C=O), 1650 (C=O), 1603, 1519, 1313, 1016, 856, 751, 695. δ_H 1.37 (t, 3H, J = 7.2 Hz), 3.26 (dd, 1H, J = 9.4, 14.45 Hz), 3.67 (dd, 1H, J = 4.2, 14.5 Hz), 4.35 (q, 2H, J = 7.1 Hz), 4.94(s, 1H), 4.99 (dd, 1H, J = 4.2, 9.3 Hz), 6.49 (d, 2H, J = 7.8 Hz), 6.69 (t, 1H, J = 7.3 Hz), 7.10 (dd, 2H, J = 7.6, 8.3 Hz), 7.66 (d, 2H, J = 8.7 Hz), 8.18 (dd, 2H, J = 1.7, 7.1 Hz). δ_C 14.21, 46.92, 54.46, 61.75, 113.49, 118.07, 123.89, 127.26, 129.10, 146.09, 147.13, 150.42, 161.27, 189.79. *m/z* (ESI): 383.14 [M + H]$^+$; HRMS (ESI) calcd. for $C_{19}H_{19}N_4O_5$: 383.1355; found for [M + H]$^+$: 383.1365.

Ethyl 2-diazo-5-(4-methoxyphenyl)-3-oxo-5-(phenylamino)pentanoate (**3c**)[16]: white solid. m.p. 103–104 °C. R_f = 0.26 (PE:EA = 10:1). δ_H 1.37 (t, 3H, J = 7.1 Hz), 3.28 (dd, 1H, J = 9.6, 14.2 Hz), 3.39 (dd, 1H, J = 4.5, 14.3 Hz), 3.79 (s, 1H), 4.35 (q, 2H, J = 7.1 Hz), 4.72 (s, 1H), 4.88 (q, 1H, J = 4.5 Hz), 6.55 (d, 2H, J = 7.9 Hz), 6.66 (t, 1H, J = 7.3 Hz), 6.87 (d, 2H, J = 8.7 Hz), 7.10 (t, 2H, J = 8.0 Hz), 7.38 (d, 2H, J = 8.6 Hz).

Ethyl 2-diazo-5-(p-tolyl)-3-oxo-5-(phenylamino)pentanoate (**3d**)[16]: white solid. m.p. 97–99 °C. R_f = 0.4 (PE:EA = 10:1). δ_H 1.40 (t, J = 7.1 Hz, 3H), 2.38 (s, 1H), 3.34 (dd, J = 9.3, 14.3 Hz, 1H), 3.44 (dd, J = 4.6, 14.3 Hz, 1H), 4.37 (q, J = 7.2 Hz, 2H), 4.8 (s, 1H), 4.95 (q, J = 4.5, 1H), 6.59 (d, J = 7.9 Hz, 2H), 6.7 (t, J = 7.3 Hz, 1H), 7.14 (d, J = 7.6Hz, 2H), 7.17 (q, J = 7.9Hz, 2H), 7.27 (s, 2H), 7.40 (d, J = 8.0 Hz, 2H).

Ethyl 2-diazo-5-phenyl-3-oxo-5-((4-nitrophenyl)amino)pentanoate (**3e**): yellowish solid. m.p. 139–140 °C. R_f = 0.28 (PE:EA = 3:1). IR (film) ν (cm^{-1}) 3973 (N–H), 2138 (C=N=N), 1714 (C=O), 1650 (C=O), 1596, 1305, 1112, 706. δ_H 1.35 (t, 3H, J = 7.2 Hz), 3.25 (dd, 1H, J = 4.6, 14.6 Hz), 3.42 (dd, 1H, J = 9.0, 14.5 Hz), 4.35 (q, 2H, J = 7.1 Hz), 4.95–4.99 (m, 1H), 5.76 (d, 1H, J = 6.2 Hz), 6.46–6.49 (m, 2H), 7.27–7.28 (m, 1H), 7.34–7.37 (m, 2H), 7.40 (d, 2H, J = 7.3 Hz), 7.99 (dd, 2H, J = 3.0, 12.2 Hz). δ_C 14.32, 29.69, 46.82, 54.85, 61.94, 112.12, 126.09, 125.14, 127.87, 129.02, 138.41, 140.81, 152.06, 161.50, 190.29. *m/z* (ESI): 383.14 [M + H]$^+$; HRMS (ESI) calcd. for $C_{19}H_{19}N_4O_5$: 383.1355; found for [M + H]$^+$: 383.1336.

Ethyl 2-diazo-5-phenyl-3-oxo-5-((4-methoxyphenyl)amino)pentanoate (**3f**)[16]: white solid. m.p. 103–104 °C. R_f = 0.19 (PE:EA = 7:1). δ_H 1.37 (t, 3H, J = 7.1 Hz), 3.28 (dd, 1H, J = 9.3, 14.2 Hz), 3.39 (dd, 1H, J = 4.5, 14.3 Hz), 3.79 (s, 1H), 4.35 (q, 2H, J = 7.1 Hz), 4.72 (s, 1H), 4.88 (q, 1H, J = 4.5 Hz), 6.55 (d, 2H, J = 7.9 Hz), 6.66 (t, 1H, J = 7.3 Hz), 6.87 (d, 2H, J = 8.7 Hz), 7.10 (t, 2H, J = 7.8 Hz), 7.38 (d, 2H, J = 8.6 Hz).

Ethyl 2-diazo-5-phenyl-3-oxo-5-(p-tolylamino)pentanoate (**3g**): yellowish solid. m.p. 101–102 °C. R_f = 0.33 (PE:EA = 7:1). IR (film) ν (cm^{-1}) 3364 (N–H), 2997, 2140 (C=N=N), 1716 (C=O), 1645 (C=O), 1522, 1454, 1317, 1280, 702. δ_H 1.35 (t, 3H, J = 7.2 Hz), 2.19 (s, 3H), 3.26 (dd, 1H, J = 9.6, 14.2 Hz), 3.38–3.41 (m, 1H), 4.33 (q, 2H, J = 7.2 Hz), 4.88 (dd, 1H, J = 4.4, 9.6 Hz), 6.45 (d, 2H, J = 8.5 Hz), 6.90 (d, 2H, J = 8.3 Hz), 7.23 (t, 1H, J = 7.4 Hz), 7.33 (t, 2H, J = 7.5 Hz), 7.45 (d, 2H, J = 7.4 Hz). δ_C 14.35, 20.32, 47.57, 55.33, 61.61, 113.75, 126.31, 126.73, 127.17, 128.66, 129.56, 142.81, 144.57, 161.44, 190.83. *m/z* (ESI): 352.16 [M + H]$^+$; HRMS (ESI) calcd. for $C_{20}H_{22}N_3O_3$: 352.1656; found for [M + H]$^+$: 352.1645.

Ethyl 2-diazo-5-(4-nitrophenyl)-3-oxo-5-((4-methoxyphenyl)amino) pentanoate (**3h**): yellowish solid. m.p. 126–127 °C. R_f = 0.26 (PE:EA = 10:1). IR (film) ν (cm^{-1}) 3400 (N–H), 2952, 2907, 2145 (C=N=N), 1713 (C=O), 1655 (C=O), 1515, 1343, 1242, 1032, 743. δ_H 1.37 (t, 3H, J = 7.2 Hz), 3.28 (dd, 1H, J = 9.4, 14.5 Hz), 3.41 (dd, 1H, J = 4.2, 14.6 Hz), 3.69 (s, 1H), 4.35 (q, 2H, J = 7.2 Hz), 4.56 (s, 1H), 4.92 (dd, 2H, J = 4.1, 9.3 Hz), 6.44 (d, 2H, J = 8.9 Hz), 6.68 (d, 2H, J = 8.7 Hz), 7.64 (d, 2H, J = 8.7 Hz), 8.18 (d, 2H, J = 8.7 Hz). δ_C 14.31, 47.02, 55.44, 55.66, 61.81, 77.29, 114.83, 114.98, 123.98, 127.39, 140.26, 147.24, 150.71, 152.57, 161.35, 190.01 ppm. *m/z* (ESI): 413.15 [M + H]$^+$; HRMS (ESI) calcd. for $C_{20}H_{21}N_4O_6$: 413.1456; found for [M + H]$^+$: 413.1464.

(E)-ethyl 2-diazo-7-phenyl-3-oxo-5-(phenylamino)hept-6-enoate (**3i**): yellowish oil. R_f = 0.29 (PE:EA = 7:1). IR (film) ν (cm^{-1}) 3393 (N–H), 3025, 2137 (C=N=N), 1713 (C=O), 1650 (C=O), 1602, 1504, 1374, 1312, 1179, 967. δ_H 1.37 (t, 3H, J = 7.2 Hz), 3.28 (dd, 1H, J = 5.4, 15 Hz), 3.39 (dd, 1H, J = 7.5, 15.0 Hz), 4.35 (q, 2H, J = 7.1 Hz), 4.62–4.65 (m, 1H), 6.30 (dd, 1H, J = 6.0, 16.0 Hz), 6.67–6.77 (m, 4H), 7.20–7.23 (m, 2H), 7.27 (d, 1H, J = 5.8 Hz), 7.32–7.35 (m, 2H), 7.38 (d, 2H, J = 7.2 Hz). δ_C 14.21, 44.93, 52.65, 61.48, 77.28, 113.64, 117.61, 126.35, 127.40, 128.37, 129.08, 130.28, 130.50, 136.59, 146.86, 161.31, 190.63. *m/z* (ESI): 364.19 [M + H]$^+$; HRMS (ESI) calcd. for $C_{21}H_{22}N_3O_3$: 364.1656; found for [M + H]$^+$: 364.1638.

Ethyl 2-diazo-5-(thiophen-2-yl)-3-oxo-5-(phenylamino)pentanoate (**3j**): yellowish solid. m.p. 80–82 °C. R_f = 0.33 (PE:EA = 5:1). IR (film) ν (cm^{-1}) 3373 (N–H), 2985, 2134 (C=N=N), 1713 (C=O), 1653 (C=O), 1602, 1507, 1377, 1222, 747. δ_H 1.35 (t, 3H, J = 7.1 Hz), 3.48 (dd, 1H, J = 5.2, 15.3 Hz), 3.56 (dd, 1H, J = 8.4, 15.3 Hz), 4.33 (q, 2H, J = 7.1 Hz), 5.25 (dd, 1H, J = 5.2, 8.0 Hz), 6.66 (d, 2H, J = 7.8 Hz), 6.74 (t, 1H, J = 7.4 Hz), 6.94 (dd, 1H, J = 3.6, 5.0 Hz), 7.04 (d, 1H, J = 3.5 Hz), 7.13 (t, 1H, J = 1.7 Hz), 7.15–7.18 (m, 3H). δ_C 14.31, 47.19, 51.01, 61.63, 77.28, 113.83, 118.17, 123.83, 124.08, 126.81, 129.13, 146.54, 147.48, 161.34, 190.17. *m/z* (ESI): 344.11 [M + H]$^+$; HRMS (ESI) calcd. for $C_{17}H_{18}N_3O_3S$: 344.1063; found for [M + H]$^+$: 344.1069.

Ethyl 2-diazo-5-(furan-2-yl)-3-oxo-5-(phenylamino)pentanoate (**3k**): yellowish oil. R_f = 0.27 (PE:EA = 7:1). IR (film) ν (cm^{-1}) 3366 (N–H), 3120, 2131 (N=N), 1720 (C=O), 1656 (C=O), 1507, 1379, 1305, 1024, 740. δ_H 1.35 (m, 3H), 3.41 (dd, 1H, J = 5.5, 15.7 Hz), 3.56 (dd, 1H, J = 7.8, 15.6 Hz), 4.33 (q, 2H, J = 7.1 Hz), 5.14 (dd, 1H, J = 5.6, 7.6 Hz), 6.22 (d, 1H, J = 3.2 Hz), 6.28 (dd, 1H, J = 1.8, 3.2 Hz), 6.67 (m, 2H), 6.73 (t, 1H, J = 7.4 Hz), 7.14–7.34 (m, 2H), 7.34 (s, 1H). δ_C 14.32, 43.83, 48.91. 61.61, 106.22, 110.23, 113.87, 118.23, 129.18, 141.71, 146.60, 154.88, 161.38, 190.27. *m/z* (ESI): 328.13 [M + H]$^+$; HRMS (ESI) calcd. for $C_{17}H_{18}N_3O_4$: 328.1292; found for [M + H]$^+$: 328.1283.

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