

Green synthesis of quinoxaline derivatives using phthalic acid as difunctional Brønsted acid at room temperature

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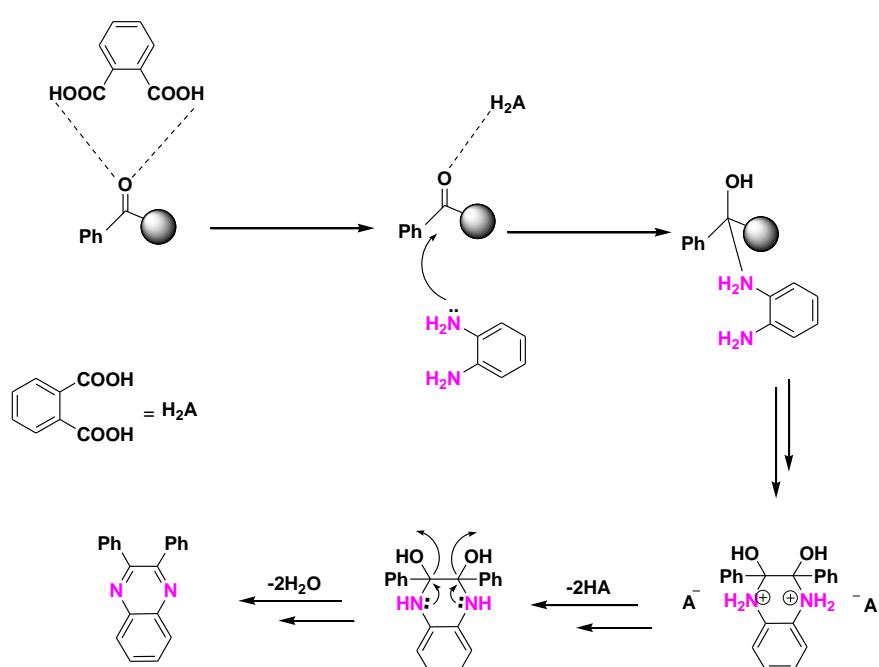
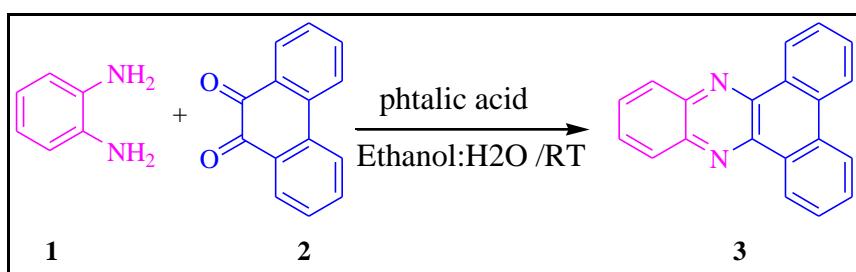
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Abstract: A simple, highly efficient and green procedure for the condensation of *o*-phenylenediamine with α -diketones in the presence of catalytic quantity of phthalic acid(0.0083 g, 5mol%) at room temperature was done. Using this method, quinoxaline derivatives as biological fascinating compound are produced in good to excellent yields and short reaction times.

Keywords: Quinoxaline, *o*-Phenylenediamine, α -Diketone, Phthalic acid, Green chemistry.

1. Introduction

Quinoxaline and their derivatives are an important class of bioactive molecules and are broadused as anticancer and antihelmintic agents[1], antiviral, antibacterial[2], anti-inflammatory, and askinase inhibitors[3]. They have been reported for their application in dye[4], pharmaceuticals[5], used as builds blocks for the synthesis of organic semiconductors[6], and served as benefitstiff subunits in macrocyclicreceptor molecular diagnose[7] and chemically controllable shifts[8]. However, the most common method used for the synthesis of quinoxaline is based on the condensation of an *o*-phenylenediamine with a α -diketone compound in refluxing ethanol or acetic acid for 2–11 h giving 34%–85% yields[9]. However, most of these methods endure unsatisfactory product isolation procedure, costly detrimental metal precursors, and tough reaction condition. Nevertheless, many enhanced methods have reported the synthesis of quinoxalines using catalytic quantity of variety of metal precursors, acids, zeolites, and molecular iodine[10–12]. It is clear that green chemistry requires the use of environmentally benign reagent and solvent, and it is very important to retrieve and reuse the catalyst. Solid acids have many progresses such as being simple in manipulation; to reduce reactor and plant corrosion problem, and more environmentally safe disposal in different chemical processes. Moreover, squander and by-product can be minimalized or avoided by using solid acids in expanding the cleanest synthesis routes[13–15]. These methods have their own value and drawbacks. Very previously, we have developed convenient and efficient procedures for the synthesis of quinoxaline derivatives using cupric sulfate pentahydrate, IBX, Zn(l-proline) [16], Microwave[17] / I₂[18], Ultrasound Itradition[19], Citric acid[20], Ionic liquid[21], Phenol[22], SBSSA[23], SnCl₂/SiO₂[24], NH₄Cl[25], (NH₄)₆Mo₇O₂₄.4H₂O[26], Bentonit Clay K-10[27], AcOH[28] and BSA[29]. Nevertheless, many of them endure drawbacks such as unsatisfactory yields, or require high temperatures and a lot of time. All these facts clearly demonstrate the importance of expanding new, efficient and versatile procedure for the preparation of this class of compounds. Our research toward the expansion of efficient and environmentally benign synthetics methodologies using eco-friendly conditions[30], we report here the synthesis of quinoxalines from *o*-phenylenediamine and various α -diketones in the presence of phthalic acidin (EtOH:H₂O) at room temperature (Scheme1).



2. Experimental

2.1. General

IR spectra of the compounds were obtained on a Shimadzu IR-435 spectrometer using a KBr disk. The ¹H nuclear magnetic resonance (¹H NMR) spectra were recorded on a Bruker AQS 300 Avance instrument at 300 MHz in dimethyl sulfoxide (DMSO-d₆) using tetramethylsilane as an internal standard. The progress of reaction is followed with thin-layer chromatography (TLC) using silica gel SILG/UV 254 and 365 plates. All the products are known compounds and characterized by comparing the IR, ¹H NMR, and ¹³C NMR spectroscopic data and their melting points with the literature values.

2.2. General procedure for synthesis of quinoxaline

A mixture of *o*-phenylenediamine(1 mmol) and α -diketones compound(1 mmol) in Ethanol:H₂O(7:3, 10 mL) was stirred at room temperature in the presence of catalytic amount of phthalic acid(0.0083g, 5mol%). The progress of the reaction was monitored by TLC(*n*-hexan:ethylacetate, 10:1). After completion of the reaction, H₂O(20 mL) was added to the reaction mixture and was allowed to stand at room temperature for 30 min. The reaction mixture was collected by filtration, washed with H₂O and dried.

2.3. Selected spectral data of quinoxalines

2.3.1. 2,3-Diphenylquinoxaline(1P)

White solid; m.p 125-127[lit. 128-129], FT-IR (KBr): 1556 cm⁻¹; ¹H NMR (FT-300 MHz, CDCl₃/TMS): δ ppm 7.33977(bs, 6H, Ar-H) 7.54183(bs, 4H, Ar-H) 7.74584(bs, 2H, Ar-H) 8.20007(bs, 2H, Ar-

H); ^{13}C NMR (300 MHz, CDCl_3): 128.290, 128.896, 129.121, 129.913, 130.066, 138.921, 141.115, 153.384; MS: $m/z = 282$ (M^+).

2.3.2. 6-Nitro-2,3-diphenylquinoxaline(**2P**)

Red solid; m.p 185-187 [lit. 185-187], FT-IR (KBr): 1656 cm^{-1} (stretching C=N); ^1H NMR (FT-300 MHz, CDCl_3/TMS): dppm 7.38(bs, 6H, Ar-H) 7.56(bs, 4H, Ar-H) 8.28(bs, 1H, Ar-H) 8.45(bs, 1H, Ar-H) 9.02(bs, 1H, Ar-H); ^{13}C NMR (300 MHz, CDCl_3): MS: 123.269, 125.512, 128.450, 129.667, 129.854, 129.953, 130.666, 137..950, 139.870, 143.390, 147.801, 155.621, 156.176; MS: $m/z = 327$ (M^+).

2.3.3. 6-Methyl-2,3-diphenylquinoxaline(**3P**)

Brown solid; m.p 113-115 [lit. 116-117], FT-IR (KBr): 1619 cm^{-1} (stretching C=N); ^1H NMR (FT-300 MHz, CDCl_3/TMS): dppm 2.61(s, 3H, Ar- CH_3) 7.35(s, 6H, Ar-H) 7.55(d, $J=6.48$, 4H, Ar-H) 7.60(s, 1H, Ar-H) 7.98(s, 1H, Ar-H) 8.09(d, $J=8.4$, 1H, Ar-H); ^{13}C NMR (300 MHz, CDCl_3): 21.948, 128.040, 128.244, 128.663, 128.731, 129.903, 129.915, 132.321, 139.246, 139.728, 140.486, 141.289, 152.552, 153.289; MS: $m/z = 296$ (M^+).

2.3.4. Dibenzo[a,c]Phenazine(**8P**)

Yellow solid; m.p 224-226 [lit. 223-225], FT-IR (KBr): 1604 cm^{-1} (stretching C=N); ^1H NMR (FT-300 MHz, CDCl_3/TMS): dppm 7.71(s, 4H, Ar-H) 7.85(s, 2H, Ar-H) 8.35(s, 2H, Ar-H) 8.43(s, 2H, Ar-H) 9.33(s, 2H, Ar-H); ^{13}C NMR (300 MHz, CDCl_3): 122.875, 126.448, 128.023, 128.925, 129.356, 130.205, 130.661, 132.019, 141.323, 141.876; MS: $m/z = 280$ (M^+).

2.3.5. 11-Methyl-dibenzo[a,c]Phenazine(**9P**)

Brown solid; m.p 219-221 [lit. 208-210], FT-IR (KBr): 1624 cm^{-1} (stretching C=N); ^1H NMR (FT-300 MHz, CDCl_3/TMS): dppm 2.65(s, 3H, CH_3) 7.72(bs, 5H, Ar-H) 8.04(s, 1H, Ar-H) 8.15(s, 1H, Ar-H) 8.47(s, 2H, Ar-H) 9.32(bs, 2H, Ar-H); ^{13}C NMR (300 MHz, CDCl_3): 20.058, 122.795, 126.032, 126.178, 127.793, 128.797, 129.978, 130.127, 131.727, 131.922, 132.368, 140.393, 140.573, 141.508, 141.971; MS: $m/z = 294$ (M^+).

2.3.6. 2-Bromopyrido-[2,3-b]dibenzo[5,6-7,8]quinoxaline(**10P**)

Yellow solid; m.p 216-218 °C, FT-IR (KBr): 1603 cm^{-1} (stretching C=N); ^1H NMR (FT-300 MHz, CDCl_3/TMS): dppm 7.72(bs, 4H, Ar-H) 8.41(bs, 2H, Ar-H) 8.66(s, 1H, Ar-H) 9.06(s, 1H, Ar-H) 9.12(s, 1H, Ar-H) 9.30(s, 1H, Ar-H); ^{13}C NMR (300 MHz, CDCl_3): 122.858, 126.519, 127.292, 128.048, 129.026, 131.393, 139.507, 143.815, 155.170; MS: $m/z = 360$ (M^+).

2.3.7. 11-Benzoyl-dibenzo[a,c]Phenazine(**12P**)

Yellow solid; m.p 245-247, FT-IR (KBr): 1653, 1606 cm^{-1} (stretching C=N); ^1H NMR (FT-300 MHz, CDCl_3/TMS): dppm 7.27(s, 1H, Ar-H) 7.60(s, 2H, Ar-H) 7.71(s, 2H, Ar-H) 7.79(s, 2H, Ar-H) 7.98(s, 2H, Ar-H) 8.35(s, 1H, Ar-H) 8.52(s, 3H, Ar-H) 8.69(s, 1H, Ar-H) 9.31(s, 1H, Ar-H) 9.44(s, 1H, Ar-H); ^{13}C NMR (300 MHz, CDCl_3): 123.048, 128.254, 128.601, 129.450, 130.220, 130.754, 131.056, 132, 916, 140.540, 140.980, 141.323, 141.876, 154.046; MS: $m/z = 384$ (M^+).

2.3.8. 2,3-bis(4-Methoxyphenyl)quinoxaline(**15P**)

Yellow solid; m.p 134-136 [lit. 148-150], FT-IR (KBr): 1615 cm^{-1} (stretching C=N); ^1H NMR (FT-300 MHz, CDCl_3/TMS): dppm 3.85(s, 6H, $2\times\text{CH}_3$) 6.87(d, $J=7.77$, 1H, Ar-H) 6.94 (d, $J=7.77$, 4H, Ar-H) 7.50 (d, $J=7.14$, 1H, Ar-H) 7.71(s, 1H, Ar-H) 7.93(d, $J=7.62$, 4H, Ar-H) 8.13(s, 1H, Ar-H); ^{13}C NMR (300 MHz, CDCl_3): 55.291, 55.615, 113.786, 114.300, 126.271, 128.875, 129.645, 131.307, 131.449, 132.315, 140.867, 152.926, 160.267, 164.867, 193.506; MS: $m/z = 342$ (M^+).

2.3.9. Acenaphtho[1,2-b]quinoxaline(**22P**)

Yellow solid; m.p 241-242 [lit. 238-240], FT-IR (KBr): 1614 cm^{-1} (stretching C=N); ^1H NMR (FT-300 MHz, CDCl_3/TMS): dppm 7.74-7.81(m, 4H, Ar-H) 8.05(d, $J=8.16$, 2H, Ar-H) 8.20-8.23(m, 2H, Ar-H) 8.4(d,

$J=6.87$, 2H, Ar-H); ^{13}C NMR (300 MHz, CDCl_3): 122.328, 128.679, 129.285, 129.442, 129.694, 129.900, 131.248, 136.479, 140.707, 153.595; MS: $m/z = 254$ (M^+).

2.3.10. 9-Methylacenaphtho[1,2-b]quinoxaline(23P)

Brown solid; m.p 231-242 [lit. >300 °C], FT-IR (KBr): 1626 cm^{-1} (stretching C=N); ^1H NMR (FT-300 MHz, CDCl_3 /TMS): d ppm 2.56(s, 3H, CH_3) 7.49(d, $J=8.31$, 1H, Ar-H) 7.70(t, $J=7.5$, 2H, Ar-H) 7.88(s, 1H, Ar-H) 7.94(d, $J=8.07$, 2H, Ar-H) 7.99(d, $J=8.61$, 1H, Ar-H) 8.27(t, $J=6.40$, 2H, Ar-H); ^{13}C NMR (300 MHz, CDCl_3): 21.735, 121.583, 121.785, 128.484, 128.554, 128.938, 129.113, 129.284, 129.799, 131.250, 131.645, 136.097, 139.359, 139.666, 140.915, 153.035, 153.661; MS: $m/z = 268$ (M^+).

3. Results and Discussion

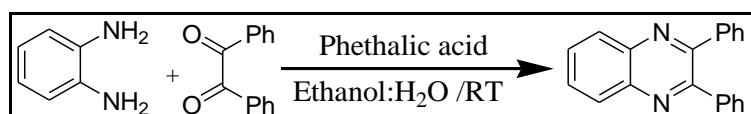
The effect of catalyst loading on the condensation reaction between α -diketone and *o*-phenylenediamine was also studied and the results were summarized in Table 1. On the optimized amount of catalyst, we realize that (0.0083g, 5mol%) of phthalic acid can effectively catalyze the reaction for the synthesis of the desired product.

Table 1. Optimization of catalyst in synthesis of 2,3-diphenylquinoxaline.

| Entry | Cat(%) | Time (min) | Yield(%) |
|----------|----------------------|---------------|------------|
| 1 | 1 ^a | 52 | 92.22 |
| 2 | 3 ^a | 43 | 98.43 |
| 3 | 5^a | 2 | 100 |
| 4 | 10 ^a | 60 | 92 |
| 5 | 20 ^a | 5 | 97 |

^aIn presence of phthalic acid

Table 2. Solvent optimization in synthesis of 2,3-diphenyl quinoxaline

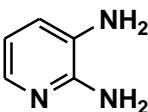
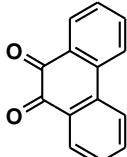
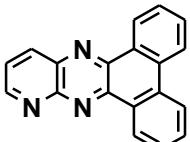
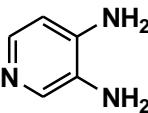
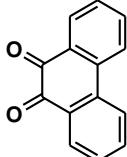
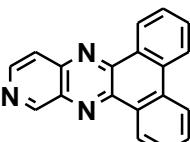
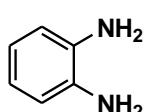
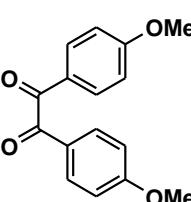
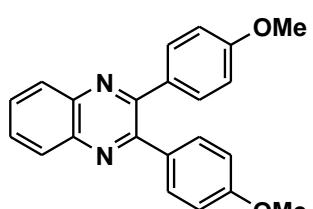
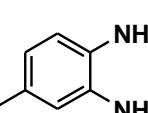
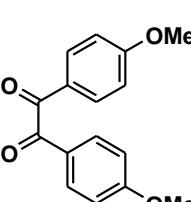
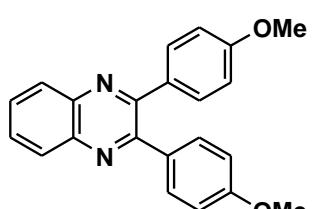
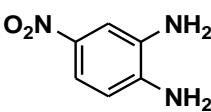
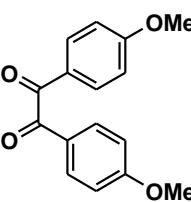
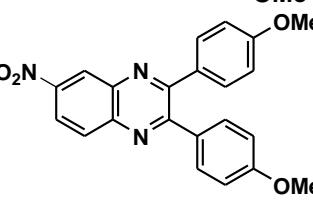
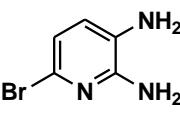
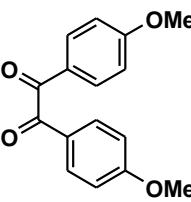
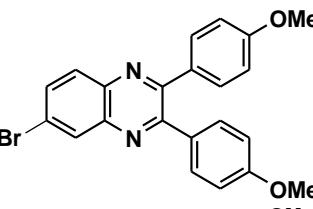
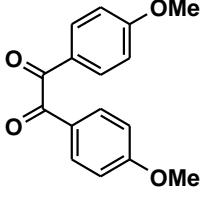
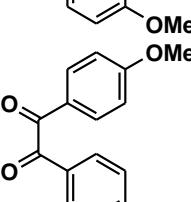
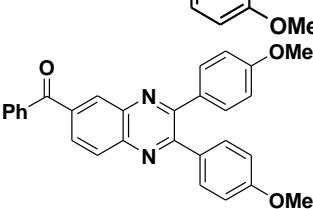
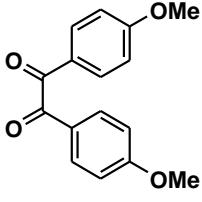
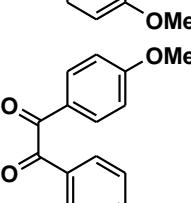
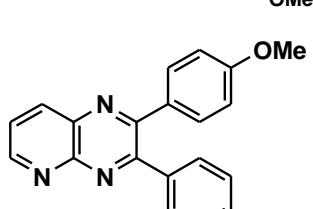
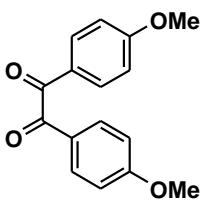
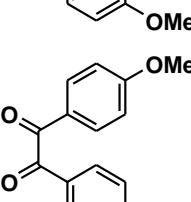
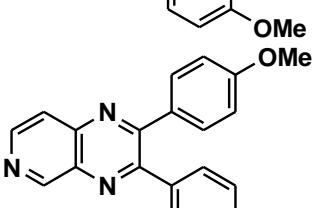


| Number | Solvent | Time (min) | Yield(%) |
|----------|--|---------------|------------|
| 1 | $\text{H}_2\text{O}(10 \text{ mL})$ | 60 | 92 |
| 2 | $\text{EtOH:H}_2\text{O (1:1, 10 mL)}$ | 5 | 97 |
| 3 | $\text{EtOH:H}_2\text{O (4:1, 10 mL)}$ | 6 | 98.13 |
| 4 | EtOH:H₂O(7:3, 10 mL) | 2 | 100 |
| 5 | $\text{EtOH:H}_2\text{O(3:7, 10 mL)}$ | 42 | 91.65 |
| 6 | $\text{EtOH:H}_2\text{O (9:1, 10 mL)}$ | 11 | 95 |
| 7 | $\text{EtOH}(10 \text{ mL})$ | 11 | 91 |

The satisfactory results were procured when a mixture of EtOH and H_2O was used as solvent. The best ratio of EtOH: H_2O (v:v) was realized to be (7:1). To establish the most and scope of our method, various *o*-phenylenediamine were reacted with some α -diketones. The results are manifested in Table 3. However, the reactions were preceded efficiently and the respective quinoxaline was procured in good to excellent yields and short reaction times.

Table 3: Quinoxalines synthesis from *o*-phenylenediamine and α -diketones using phthalic acid

| Entr y | Diamine (DA) | Diketone (DK) | Product (Q) | Time (h:min) Yield (%) | m.p(Found) m.p [lit.] |
|-----------|-----------------|------------------|----------------|---------------------------------|------------------------------------|
| 1 | | | | 00:02 100 | 124-126 [128-129] ³² |
| 2 | | | | 03:53 96.24 | 187-189 [193-194] ³² |
| 3 | | | | 00:02 93.92 | 112-113 [117-118] ³² |
| 4 | | | | 02:28 99.14 | 137-139 [141-143] ³² |
| 5 | | | | 04:43 89.36 | 143-145 |
| 6 | | | | 03:29 91.23 | 133-134 |
| 7 | | | | 08:13 93.17 | 127-128 |
| 8 | | | | 00:02 98.92 | 225-226 [223-225] ³⁴ |
| 9 | | | | 00:13 95.27 | 211-213 [208-210] ³⁴ |
| 10 | | | | 00:46 85.61 | 215-216 |
| 11 | | | | 00:20 92.86 | 259-260 [262-264] ³⁴ |
| 12 | | | | 03:11 98.33 | 243-245 [246-247] ³⁴ |

| | | | | | |
|----|---|---|--|----------------|------------------------------------|
| 13 |  |  |  | 02:08 90.82 | 218-219 |
| 14 |  |  |  | 03:02 94.31 | 159-162 |
| 15 |  |  |  | 03:22 91.72 | 145-147 [151-152] ³¹ |
| 16 |  |  |  | 00:05 89.75 | 122-123 [125-127] ³¹ |
| 17 |  |  |  | 03:00 91.38 | 188-189 [184-186] |
| 18 |  |  |  | 05:45 90.03 | 120-121 |
| 19 |  |  |  | 00:25 74.04 | 145-147 |
| 20 |  |  |  | 02:38 76.48 | 131-134 |
| 21 |  |  |  | 07:16 92.10 | 120-122 |

| | | | | | |
|----|--|--|--|----------------|------------------------------------|
| 22 | | | | 00:28 91.14 | 235-236 [238-240] ³⁴ |
| 23 | | | | 00:02 97.24 | 228-229 [>300] ³⁴ |
| 24 | | | | 04:37 92.67 | 318-319 [>300] ³³ |
| 25 | | | | 04:18 93.96 | 221-223 |
| 26 | | | | 05:11 84.93 | 245-246 |
| 27 | | | | 03:05 92.38 | 229-231 |

To begin this work, the condensation reaction between α -diketones and *o*-phenylenediamine was employed as the model reaction to screen the best condition (Table 3). As shown in the Table, The condensation of *o*-phenylenediamine (1 mmol) with α -diketones (1 mmol) in the different presence of catalyst phthalic acid (0.0083g, 5 mol%) in EtOH:H₂O(7:3, 10 mL) give the best results in terms of time and yield, so we chose this solvent system for environmental acceptability.

4. Conclusions

A gentle, efficient and environmentally benign method has been developed for the synthesis of quinoxalines that is established as more important in every respect in comparison to the previously reported method. The method is tested appropriately for aliphatic, aromatic and heterocyclic α -diketones. The advantages of this method are efficiency, completeness, excellent yield, short reaction time, cleanest reaction profile, simplicity, easy work up.

5. Acknowledgment

The authors gratefully acknowledge partial support of this work by Payame Noor University of Ilam, I.R. of Iran.

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