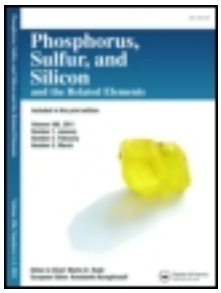

Phosphorus, Sulfur, and Silicon and the Related Elements

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An efficient One-pot synthesis of new 2- thioxo and 2-oxo-pyrimidine-5-carbonitriles in Ball-Milling under Solvent-Free and Catalyst-Free conditions

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An efficient One-pot synthesis of new 2- thioxo and 2-oxo-pyrimidine-5-carbonitriles in Ball-Milling under Solvent-Free and Catalyst-Free conditions

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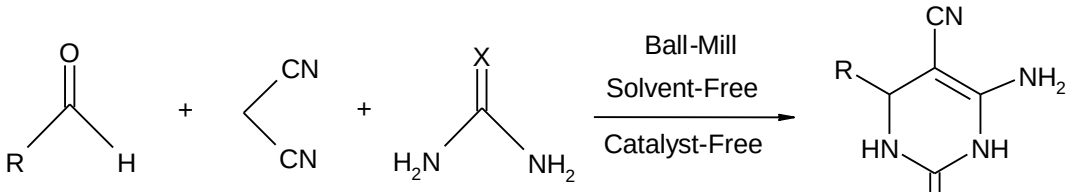
Abstract:

Various 2-thioxo and 2-oxo-1,2,3,4-tetrahydro-pyrimidine-5-carbonitriles were synthesized in very high yields in 40 min through a direct condensation of an equimolar amount of an aldehyde, malononitrile and thiourea/urea under ball mill solvent-free and catalyst-free conditions.



R = p-CH_3-O-C_6H_4, p-OH-C_6H_4, p-NO_2-C_6H_4, p-Cl-C_6H_4, Ph

X = S, O

1a-j

Keywords:

Ball milling solvent-free reaction, catalyst-free, thioxo-pyrimidine, oxo-pyrimidine.

1. Introduction

Ball milling is a mechanical technique that is widely used to grind minerals into extremely fine particles, and the preparation or modification of inorganic solids [1-4]. The applications of ball milling solvent-free in organic synthesis are relatively rare. However, in the last decade, it is gaining noticeable attention due to its simplicity, low cost and friendly impact on the environment, as well as its capability to achieve very high yields.

The applications of ball milling in organic synthesis [5-13], has inspired other researchers to synthesis different types of organic materials including functionalized indan-1,3-diones [14], coordination polymers [15], nitrones [16], the reductive benzylation of malononitrile [17], preparation of phosphorus ylides [18], Knoevenagel condensations [19], the protection of diols diamines [20], functionalization of fullerenes [21], Heck-type reactions [22-23], aldol reaction [24,25], and Suzoku-type reaction [26-29].

Pyrimidine derivatives have a great importance due to their different biological properties such as anticancer [30,31], antitumor [32], analgesic [33], antibacterial activity [34] and fungicides [35] anti-inflammatory [36,37] and cardioprotective effects [38]. The commonly method used for the synthesis of pyrimidines is the Biginelli reaction, which is a direct condensation of an aldehyde, keto ester and thiourea / urea. This condensation is usually done by using heat in different solvents and in the presence of a catalyst [39-43].

2. Results and Discussion

Mashkouri et al. [44] used ball milling for the synthesis of pyrano [2,3-d] pyrimidines via direct condensation of an aldehyde, malononitrile and barbituric acid. The authors failed to have results with an ordinary ball milling system. An excellent yield is obtained only by using a specific setup for the ball milling, by circulating warm water in order to heat the reaction. Despite this excellent yield, the disadvantage of this method is the use of a specific milling machine allowing the circulation of warm water and that makes the milling system very complicated and difficult to setup.

In this paper, we present a simple ball milling procedure without any specific setup for the synthesis of new 2-thioxo and 2-oxo-pyrimidine-5-carbonitriles by direct condensation of an aldehyde, malononitrile and the thiourea / urea using ball milling solvent-free, catalyst-free (Scheme 1). In this one pot synthesis process, we need to change only the weight of the balls used in the ball milling process in order to obtain high yield. This easy method can be used for the synthesis of several others organic materials.

An equimolar amount (0.02 mol) of paramethoxybenzaldehyde, malononitrile and thiourea (with a total mass of 5.56 g) were introduced in a stainless steel bowl. In order to optimize the reaction conditions, several milling times and balls weights were used (Table 1). The progress of the reaction was followed by determining the conversion rate calculated relative to the reagents. The analysis of these results shows that when the ratio (balls weight / reagents weight) is equal to 1, the reaction does not change even after 4 h of grinding. With increasing value of the balls weight, the conversion increases until the optimal value for the ratio: balls weight to reagents is equal to 5.

3. Conclusion

In conclusion, we have developed a simple and efficient method for the synthesis of new functionalized pyrimidines (Table **2**). This method allowed us to obtain the products synthesized in pure form without further purification and in quantitative yields.

Experimental

Materials and techniques

The ball-mill was a SPEX 8000 mixer with 10 cm^3 stainless steel vials. Melting points were determined in open capillaries and were uncorrected. IR spectra were taken on a FT-IR-Tensor 27 spectrometer in KBr pellets. ^1H and ^{13}C NMR spectra were measured on a Jeol-400 NMR spectrometer in DMSO-d_6 using TMS as an internal standard and chemical shifts are expressed as δ ppm units. Elemental analysis was carried out on an Elementar Vario EL analyzer. The progress of all the reactions was monitored by thin layer chromatography on silica gel 60 for TLC (Merck) using chloroform-ethanol.

General procedure for the synthesis of pyrimidine compound 1a

An equimolar amount (0.02 mol) of paramethoxybenzaldehyde, malononitrile and thiourea, were introduced into stainless steel vials with 27.80 gram of stainless steel balls (12 mm in diameter)

in a SPEX 8000 mixer. We obtained the product 1a after 40 min of milling in pure form without further purification.

4.3. 6-amino-4-(4-methoxy-phenyl)- 2-thioxo 1,2,3,4- tetrahydro-pyrimidine-5-carbonitrile (1a).

Yield: 98%; Mp 199-200 ^{0}C; IR(KBr): 3440, 3350, 3230, 2220, 1250; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.30-7.28 (m, Ar, 2H), 7.11-7.08 (m, Ar, 2H), 6.31 (br, s, 2NH, NH_2, 4H), 5.49 (s, CH, 1H), 3.81 (s, 3H, OCH_3); ^{13}C NMR (100 MHz. DMSO-d6) δ 167.65, 163.27, 160.39, 137.33, 128.08, 119.19, 117.80, 81.90, 56.89, 52.99; Anal. Calcd. for $C_{12}H_{12}N_4OS$ (260.316): C (55.37%), H (4.65%), N (21.52%), O (6.15%), S(12.32%). Found: C (55.01%), H (4.60%), N (21.11%), O (5.98%), S (12.35%).

4.4. 6-Amino-4-(4-hydroxy-phenyl)-2-thioxo-1,2,3,4-tetrahydro-pyrimidine-5-carbonitrile (1b).

Yield: 96%; Mp 220-221 ^{0}C; IR(KBr): 4510, 3430, 3360, 3220, 2221, 1260; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.26-7.24 (m, Ar, 2H), 6.80-6.78 (m, Ar, 2H), 6.60 (br, s, 2NH, NH_2,OH, 5H), 5.51 (s, CH, 1H); ^{13}C NMR (100 MHz. DMSO-**d_6**) δ 167.65, 163.27, 159.39, 136.83, 127.68, 118.59, 117.89, 81.76, 52.16; Anal. Calcd. For $C_{11}H_{10}N_4O$**S** (246.29): C (53.64%), H (4.09%), N (22.75%), O (6.50%), S (13.02%). Found: C (53.24%), H (4.11%), N (22.80%), O (6.13%), S (12.96%).

4.5. 6-Amino-4-(4-nitro-phenyl)--2-thioxo-1,2,3,4-tetrahydro-pyrimidine-5-carbonitrile (1c).

Yield: 97%; Mp 215-216 ^{0}C; IR(KBr): 3430, 3340, 3230, 2222, 1215; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.30-8.24 (m, Ar, 2H), 7.81-7.78 (m, Ar, 2H), 6.30 (br, s, 2NH, NH_2, 4H), 5.47 (s, CH, 1H); ^{13}C NMR (100 MHz. DMSO-**d_6**) δ 167.97, 162.87, 151.39, 148.30, 132.14, 129.16, 118.91, 82.10, 53.29; Anal. Calcd. For $C_{11}H_9N_5O_2S$ (275.29): C (47.99%), H (3.30%), N (25.44%), O (11.62%), S (11.65%). found: C (47.19%); H (3.29%); N (25.36%); O (11.54%); S (11.37%).

4.6. 6-Amino-4-(4-chloro-phenyl)-2-thioxo-1,2,3,4-tetrahydro-pyrimidine-5-carbonitrile (1d).

Yield: 95%; Mp 202-203 ^{0}C; IR(KBr): 3420, 3350, 3220, 2221, 1260; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.46-7.42 (m, Ar, 2H), 7.33-7.29 (m, Ar, 2H), 6.37 (br, s, 2NH, NH_2, 4H), 5.45 (s, CH, 1H); ^{13}C NMR (100 MHz. DMSO-**d_6**) δ 167.86, 163.11, 142.17, 133.16, 132.60, 128.77, 119.25, 81.10, 52.12; Anal. Calcd. For $C_{11}H_9ClN_4S$ (264.74): C (49.91%), H (3.43%), Cl (13.39%), N (21.16%), S (12.11%). found: C (49.88%), H (3.38%), Cl (13.47%), N (21.11%), S (12.03%).

4.7. 6-Amino-4-phenyl-2-thioxo-1,2,3,4-tetrahydro-pyrimidine-5-carbonitrile (1e).

Yield: 96%; Mp 196-197 ^{0}C; IR(KBr): 3440, 3350, 3210, 2214, 1250; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.63-7.28 (m, Ar, 5H), 6.41 (br, s, 2NH, NH_2, 4H), 5.47 (s, CH, 1H); ^{13}C NMR

(100 MHz. DMSO-**d_6**) δ 167.30, 162.05, 141.12, 133.47, 129.23, 127.81, 119.20, 81.66, 52.73; Anal. Calcd. For $C_{11}H_{10}N_4S$ (230.29): C (57.37%), H (4.38%), N (24.33%), S (13.92%). found: C (57.50%), H (4.21%), N (24.19%), S (14.01%).

4.8. 6-Amino-4-(4-methoxy-phenyl)-2-oxo-1,2,3,4-tetrahydro-pyrimidine-5-carbonitrile (1f).

Yield: 96%; Mp 142-143 ^{0}C; IR(KBr): 3415, 3340, 3230, 2222, 1678; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.89-(s, NH. 1H), 7.24-7.04 (m, Ar, 4H), 6.35 (br, s, NH_2, 2H), 6.12 (s, NH. 1H), 5.18 (s, CH, 1H), 3.84 (s, 3H, OCH_3); ^{13}C NMR (100 MHz. DMSO-**d_6**) δ 162.65, 160.17, 152.63, 132.17, 127.05, 116.44, 115.43, 72.91, 56.19, 48.29; Anal. Calcd. For $C_{12}H_{12}N_4O_2$ (244.26): C (59.01%), H (4.95%), N (22.94%), O (13.10%). Found: C (58.92%), H (4.91%), N (22.88%), O (13.26%).

4.9. 6-Amino-4-(4-hydroxy-phenyl)-2-oxo-1,2,3,4-tetrahydro-pyrimidine-5-carbonitrile (1g).

Yield: 94%; Mp 189-190 ^{0}C; IR(KBr): 3460, 3410, 3350, 3230, 2220, 1672; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.49 (s, NH, 1H),7.16-7.14 (m, Ar, 2H), 6.78-6.12 (m, Ar, NH_2, OH, 5H), 6.21 (br, s, NH, 1H), 5.29 (s, CH, 1H); ^{13}C NMR (100 MHz. DMSO-**d_6**) δ 162.35, 160.57, 153.12, 133.23, 128.18, 118.67, 116.13, 72.97, 48.49; Anal. Calcd. For $C_{11}H_{10}N_4O_2$ (230.23): C (57.39%), H (4.38%), N (24.34%), O (13.90%). Found: C (57.31%), H (4.32%), N (24.28%), O (13.95%).

4.10. 6-Amino-4-(4-nitro-phenyl)-2-oxo-1,2,3,4-tetrahydro-pyrimidine-5-carbonitrile (1h).

Yield: 93%; Mp 198-199 ^{0}C; IR(KBr): 3420, 3350, 3230, 2220, 1660; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.28-8.22 (m, Ar, 2H), 7.70-7.68 (m, Ar, 2H), 7.52 (s, NH, 1H), 6.56 (br, s, NH_2, 2H), 6.22 (s, NH, 1H), 5.39 (s, CH, 1H); ^{13}C NMR (100 MHz. DMSO-**d_6**) δ 162.85, 153.67, 148.82, 147.63, 128.22, 125.21, 116.20, 71.97, 47.79; Anal. Calcd. For $C_{11}H_9N_5O_3$ (259.23): C (50.97%), H (3.50%), N (27.02%), O (18.52%). Found: C (51.12%), H (3.17%), N (27.00%), O (18.48%).

4.11. 6-Amino-4-(4-chloro-phenyl)-2-oxo-1,2,3,4-tetrahydro-pyrimidine-5-carbonitrile (1i).

Yield: 92%; Mp 178-179 ^{0}C; IR(KBr): 3386, 3360, 3230, 2220, 1650; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.53 (s, NH, 1H), 7.39-7.36 (m, Ar, 2H), 7.22-7.18 (m, Ar, 2H), 6.55 (br, s, NH_2, 2H), 6.13 (s, NH, 1H), 5.27 (s, CH, 1H); ^{13}C NMR (100 MHz. DMSO-**d_6**) δ 162.63, 152.33, 137.83, 134.52, 131.27, 126.29, 115.19, 71.24, 47.39; Anal. Calcd. For $C_{11}H_9ClN_4$**O** (248.67): C (53.13%), H (3.65%), Cl (14.26%), N (22.53%), O (6.43%). Found: C (52.79%), H (3.51%), Cl (14.86%), N (22.21%), O (6.22%).

4.12. 6-Amino-2-oxo-4-phenyl-1,2,3,4-tetrahydro-pyrimidine-5-carbonitrile (1j).

Yield: 92%; Mp 198-199 ^{0}C; IR(KBr): 3340, 3327, 3137, 2220, 1654; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.66-7.42 (m, Ar, NH, 4H), 7.21-7.15 (m, Ar, 2H), 6.39 (br, s, NH_2, 2H), 6.29 (s, NH, 1H), 5.41 (s, CH, 1H); ^{13}C NMR (100 MHz. DMSO-**d_6**) δ 161.95, 153.62, 140.31, 130.25, 128.68, 125.17, 115.49, 71.24, 46.31; Anal. Calcd. For $C_{11}H_{10}N_4O$ (214.23): C (61.67%), H (4.71%), N (26.15%), O (7.47%). found: C (61.36%), H (4.62%), N (26.11%), O (7.51%).

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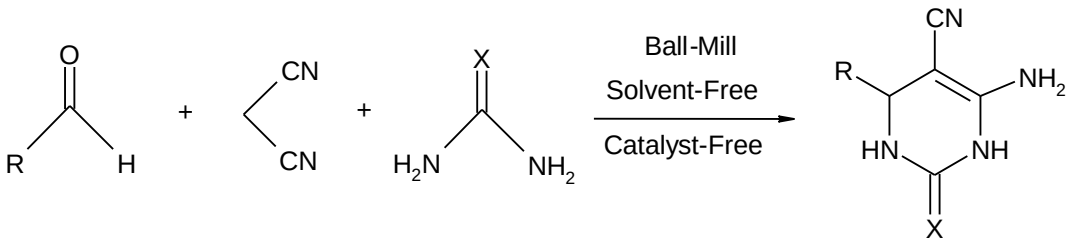

R = p-CH_3-O-C_6H_4, p-OH-C_6H_4, p-NO_2-C_6H_4, p-Cl-C_6H_4, Ph

X = S, O

1a-j

Schem1: Synthesis of 2-thioxo **and** 2-oxo-pyrimidine-5-carbonitriles

Table 1: Milling parameters and the conversion rate for the synthesis of 2-thioxo and 2-oxo pyrimidine-5-carbonitriles

Entry*	Balls(g)	Tim**e** (min)	**Conversion** (%)
1	5.56	240	0
2	11.12	240	20
3	16.68	180	40
4	22.24	60	70
5	27.8**0**	40	100

*0.02 mol of paramet**h**oxybenzaldhyde, malonitrile and thiourea (5.56 g)

Table 2: Synthesis of 2-thioxo **and** 2-oxo-1,2,3,4-tetrahydro-pyrimidine-5-carbonitriles using ball-mill

Entry	R	X	Product
1	*p*-CH_3-O-C_6H_4	S	1a
2	*p*-HO-C_6H_4	S	1b
3	*p*-NO_2-C_6H_4	S	1c
4	*p*-Cl-C_6H_4	S	1d
5	Ph	S	1e
6	*p*-CH_3-O-C_6H_4	O	1f
7	*p*-HO-C_6H_4	O	1g
8	*p*-NO_2-C_6H_4	O	1h
9	*p*-Cl-C_6H_4	O	1i
10	Ph	O	1j