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Microwave-induced calcium(II) chloride-catalyzed Paal–Knorr pyrrole synthesis: a safe, expeditious, and sustainable protocol

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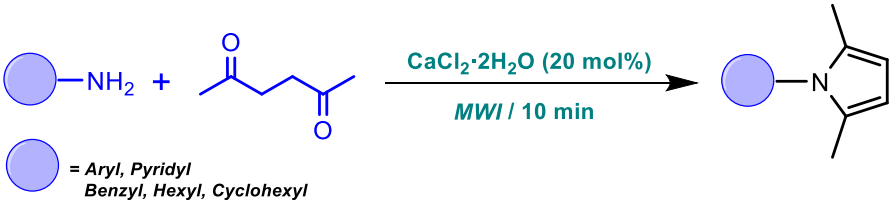
Abstract Among various alkali (Na, K) and alkaline-earth (Ca, Mg, Sr, Ba) chlorides, calcium(II) chloride was found to be a cost-effective Lewis acid catalyst for solvent-free synthesis of pyrroles from primary aromatic and aliphatic amines under open-vessel focused microwave irradiation. The salient features of this environmentally benign method are high to quantitative conversion, short reaction time, safe and clean reaction profile, possibility of scale-up to multigram quantities, and use of a low-cost, widely available, nontoxic catalyst.

Graphical abstract



Keywords Calcium(II) chloride · Ecofriendly method · Microwave heating · Paal–Knorr reaction · 2,5-Dimethylpyrrole

Electronic supplementary material The online version of this article (https://doi.org/10.1007/s11164-018-3355-7) contains supplementary material, which is available to authorized users.

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Introduction

Microwave-induced organic reaction enhancement (MORE) is now a well-established tool in development of clean organic transformations. This scientific tool has the merits of user-friendliness, operational simplicity, uniform heating, and greater reproducibility of chemical reactions. The ability to adjust various parameters (microwave power, reaction time, and temperature), accelerate the reaction rate, minimize byproduct generation, and significantly reduce energy consumption and reaction time (compared with conventional thermal heating) makes it a valuable asset for organic chemists aiming to implement green chemical processes [1].

Pyrrole moieties are one of the most important classes of heterocyclic compounds, being present in a large number of naturally occurring, biological, and drug molecules [2–9]. The growing importance of these heterocycles as intermediates in pharmaceutical and chemical processes has led to considerable effort to develop a variety of methodologies for their synthesis. Among the various methods developed for straightforward synthesis of pyrrole ring, Paal–Knorr cyclization and the Clauson-Kaas reaction have received considerable attention due to their use of readily available starting materials [10, 11]. Among catalysts reported for the Paal–Knorr reaction, Lewis acid catalysts are the most widely adopted, e.g., $Bi(NO_3)_3{\cdot}5H_2O$ [12], $Sc(OTf)_3$ [13], $SnCl_2{\cdot}2H_2O$ [14], $RuCl_3$ [15], $CoCl_2$ [16], $InCl_3$, $InBr_3$ or $In(OTf)_3$ [17], $ZrCl_4$ [18], $Ga(OTf)_3$ [19], $AlCl_3$/PS [20], $ZrOCl_2{\cdot}8H_2O$ [21], $UO_2(NO_3)_2{\cdot}6H_2O$ [22], CuI/C [23], $SbCl_3/SiO_2$ [24], $BiCl_3/SiO_2$ [25], $GaCl_3$/PS [26], $Al(DS)_3$ [27], $MgI_2{\cdot}(OEt_2)_n$ [28], $FePO_4$ [29], and MgI_2 [30]. Besides, some ecofriendly protocols have recently been reported for this purpose, e.g., mechanical activation [31], use of nanomagnetically modified sulfuric acid [32], and a deep eutectic solvent under ultrasonic activation [33]. However, the search for inexpensive, abundant, and greener Lewis acid catalysts is still being actively pursued.

Calcium(II) chloride is an abundant moisture-tolerant salt having several industrial applications, e.g., deicing of roads, dust control, and imparting stability to roads and buildings. It is also used as a desiccant for dehydrating gases and liquids, and as a calcium source in liquid feed supplements for dairy cattle [34]. It is used as catalyst for preparation of tetrahydropyranyl ethers [35], 3,4-dihydropyrimidin-2(1*H*)-ones [36, 37], α-aminophosphonic esters [38], carbamoyl and thiocarbamoyl phosphonates [39], β-aminoketones [40], 4*H*-pyrans [41], 9-aryl-1,8-dioxooctahydroxanthene [42], 3-(aryliminomethyl) chromones [43], allylic oxidation of alkenes [44], esterification of aromatic aldehydes [45], and C–F functionalization of fluoroarenes [46]. Its conjunction with chiral ligands has also proved to be effective for asymmetric reactions [47–49].

Microwave-assisted organic synthesis (MAOS) has had a tremendous impact in generation of *N*-heterocycle libraries [50–55]. Its contribution to sustainable development of the pharmaceutical industry and drug discovery is now undeniable [56–58]. As part of our continuing efforts to develop sustainable processes for pyrrole synthesis [24, 25, 59–61], we introduce herein calcium(II) chloride as an

inexpensive, environmentally benign, commercially available catalyst for rapid Paal–Knorr pyrrole cyclocondensation under microwave irradiation.

Results and discussion

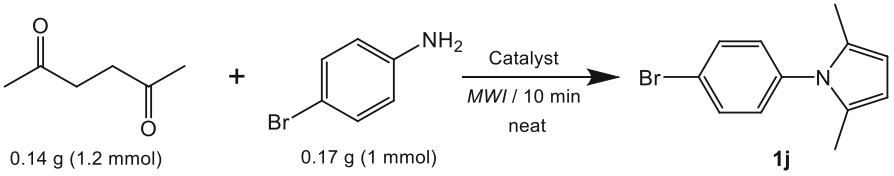
Initially, condensation of hexane-2,5-dione (1.2 mmol) with 4-bromoaniline (1 mmol) was examined in presence of catalytic amount (20 mol%) of various alkali and alkaline-earth chlorides (Scheme 1). The reaction was screened under microwave-induced solvent-free condition within 10 min. Among these Lewis acid salts, NaCl, KCl, and $BaCl_2$ showed poor catalytic activity (Table 1, entries 2, 3, 7) whereas $MgCl_2{\cdot}6H_2O$, $CaCl_2{\cdot}2H_2O$, and $SrCl_2{\cdot}6H_2O$ (Table 1, entries 4–6) catalyzed the reaction to afford the desired product in excellent yield.

Synthesis of **1j** was also performed in presence of various calcium salts as readily available catalysts (Table 2). $CaSO_4$, $CaCO_3$, $Ca(OH)_2$, and CaO exhibited poor catalytic activity (Table 2, entries 4–7). While $CaHPO_4$ gave good yield of product (Table 2, entry 3), calcium chloride catalyzed the reaction to afford the desired product in excellent yield (Table 2, entries 1, 2). Consequently, $CaCl_2{\cdot}2H_2O$ was chosen as catalyst of choice due to its easy availability, nontoxicity, ecofriendliness, and quantitative yield of **1j** (Table 2, entry 1). The classical reaction was also studied in presence of $CaCl_2{\cdot}2H_2O$ at 25 and 70 °C for 1 h (Table 2, entries 12, 13). The same results were obtained under microwave irradiation and classical heating at 70 °C (Table 2, entries 1, 12) to highlight the role of the catalyst.

The effect of the microwave power in presence of $CaCl_2{\cdot}2H_2O$ was also investigated. The results revealed that decrease of the microwave power led to lower conversion (Fig. 1).

To evaluate the performance and generality of $CaCl_2{\cdot}2H_2O$ applied as catalyst, various primary amines were tested with hexane-2,5-dione under the optimized conditions; the results are presented in Table 3. In most cases, the reaction proceeded expeditiously, although the yield was highly dependent on the substrate used (Table 3).

It was found that aniline derivatives bearing either electron-donating (such as methoxy, methyl, and dimethyl) or electron-withdrawing (such as nitro, bromo, chloro, dichloro, and cyano) substituents reacted smoothly to give corresponding *N*-substituted pyrroles (Table 3, entries **1a**–**1o**) in high to excellent yield. An exception to this rule was observed for 2-nitroaniline as substrate, for which strong



Scheme 1 Catalyst screening of alkali and alkaline-earth salts as potential Lewis acids for synthesis of **1j**

Table 1 Catalyst effect on synthesis of **1j** under microwave heating

Entry	Catalyst (mol%)	Conversion (%)[a]
1	–	24
2	NaCl (20)	28
3	KCl (20)	15
4	$MgCl_2{\cdot}6H_2O$ (20)	100
5	$CaCl_2{\cdot}2H_2O$ (20)	100
6	$SrCl_2{\cdot}6H_2O$ (20)	96
7	$BaCl_2$ (20)	31

Microwave irradiation: 420 W

[a]Gas chromatography assay (%)

Table 2 Screening of various Ca salts for synthesis of **1j**

Entry	Catalyst (mol%)	Conditions	Time (min)	Conversion (%)
1	$CaCl_2{\cdot}2H_2O$ (20)	MWI	10	100
2	$CaCl_2$ anhydrous (20)	MWI	10	100
3	$CaHPO_4$ (20)	MWI	10	82
4	$CaSO_4$ (20)	MWI	10	39
5	$CaCO_3$ (20)	MWI	10	18
6	$Ca(OH)_2$ (20)	MWI	10	29
7	CaO (20)	MWI	10	13
8	$CaCl_2{\cdot}2H_2O$ (15)	MWI	10	99
9	$CaCl_2{\cdot}2H_2O$ (10)	MWI	10	98.5
10	$CaCl_2{\cdot}2H_2O$ (20)	MWI	8	96
11	$CaCl_2{\cdot}2H_2O$ (20)	MWI	5	55
12	$CaCl_2{\cdot}2H_2O$ (20)	70 °C	60	100
13	$CaCl_2{\cdot}2H_2O$ (20)	25 °C	60	75

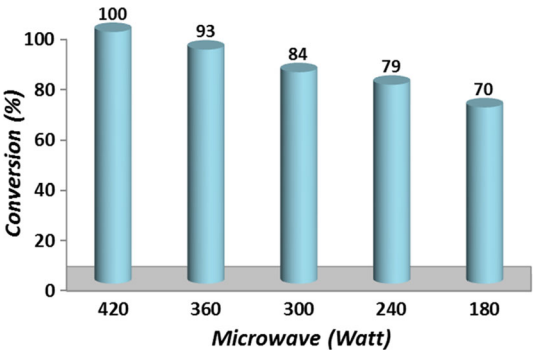


Fig. 1 Effect of microwave power in presence of $CaCl_2{\cdot}2H_2O$ (20 mol%) as catalyst in synthesis of **1j** within 10 min

Table 3 Microwave-induced synthesis of *N*-substituted pyrroles catalyzed by $CaCl_2 \cdot 2H_2O$ (20 mol%)

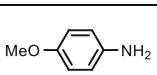
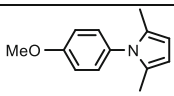
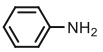
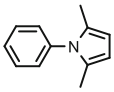
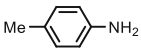
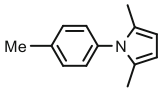
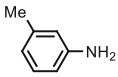
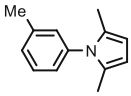
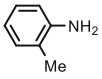
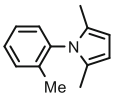
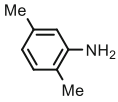
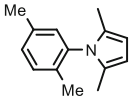
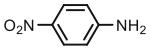
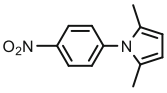
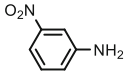
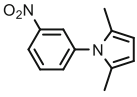
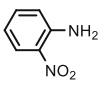
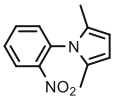
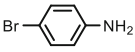
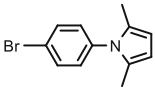
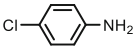
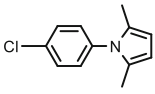
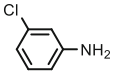
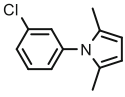
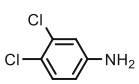
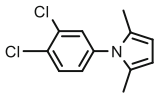
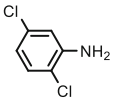
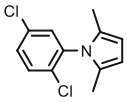
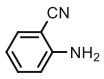
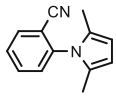
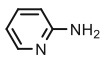
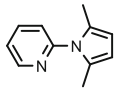
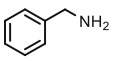
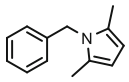
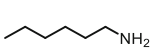
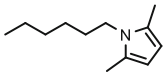
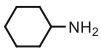
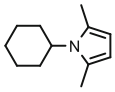
Entry	Amine	Product	Time (min)	Conversion (%)[a]	Yield (%)[b]	m.p. (°C)
1a			10	100	96	58–59
1b			10	100	95	50–51
1c			10	100	94	46–47
1d			10	100	94	51–52
1e			10	100	94	Oil
1f			10	95	90	Oil
1g			10	91	86	145–146
1h			10	100	93	85–86
1i			10 / 20	10 / 14	5	87–88
1j			10	100	95	74–75
1k			10	100	95	56–57
1l			10	100	94	48–49

Table 3 continued

1m			10	100	94	102–103
1n			10	100	95	138–139
1o			10	89	80	84–85
1p			10	92	84	Oil
1q			10	100	97	44–45
1r			10	100	94	Oil
1s			10	89	74	Oil

[a]Gas chromatography assay (%)

[b]Yields refer to pure isolated product

electron-withdrawing nature and steric hindrance led to poor conversion [24, 25, 59–61], even after extended irradiation time (Table 3, entry **1i**).

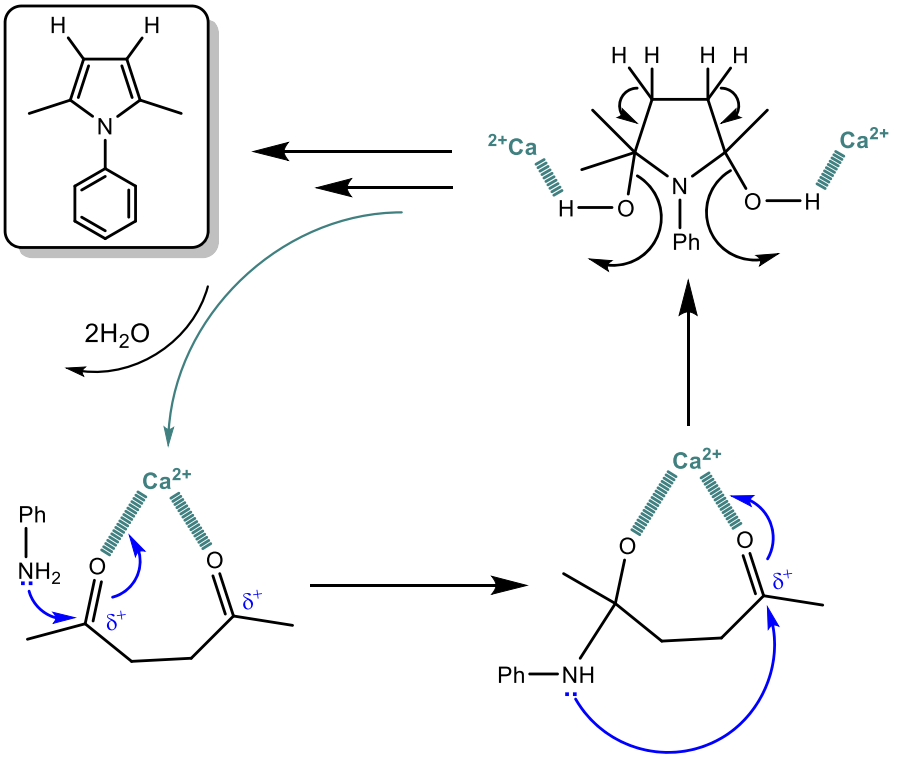
The reaction was also carried out with 2-aminopyridine and aliphatic amines such as benzylamine, *n*-hexylamine, and cyclohexylamine, giving high to excellent conversion (Table 3, entries **1p**–**1s**).

To investigate the viability of this protocol in pharmaceutical industry, gram-scale synthesis of **1j** (using 20 mmol 4-bromoaniline) was also carried out under the same optimized conditions. The synthesis was successfully scaled up to produce *N*-(4′-bromophenyl)-2,5-dimethylpyrrole on gram scale with almost the same isolated yield (96 %) of **1j** as in the small-scale reaction (Table 3, entry **1j**, yield 95 %). Moreover, the catalyst could be readily recovered. After reaction completion, ethyl acetate was added and the catalyst together with water produced from the reaction was separated. The aqueous solution was evaporated under reduced pressure or put in the microwave oven (5 min, 420 W), and the catalyst was reused in the subsequent run with little loss of activity.

A plausible mechanism for calcium(II) chloride-catalyzed synthesis of pyrroles is depicted in Scheme 2. Ca^{2+} ion interacts with hexane-2,5-dione and activates the oxygen of carbonyl groups to promote the reaction via nucleophilic attack of primary amines. Eventually, after completion of cyclocondensation, detachment of $CaCl_2$ occurs with subsequent loss of water, leading to formation of the pyrrole ring.

Conclusions

Catalyst screening of various alkali (Na, K) and alkaline-earth (Ca, Mg, Sr, Ba) salts was performed for microwave-induced Paal–Knorr pyrrole cyclocondensation of 4-bromoaniline and hexane-2,5-dione under solvent-free condition. Among these potential catalysts, $CaCl_2{\cdot}2H_2O$ led to quantitative conversion and was chosen as catalyst of choice due to its inexpensiveness and innocuous nature. The scope and generality of this protocol were explored with respect to various primary aromatic and aliphatic amines, revealing high to excellent yields. The combination of microwave irradiation with a nontoxic and abundant catalyst under solvent-free condition, together with the possibility of scale-up to multigram quantities of



Scheme 2 Plausible mechanism for calcium(II) chloride-catalyzed Paal–Knorr pyrrole cyclocondensation

desired pyrroles, makes this protocol a useful asset for generation of pyrrole libraries.

Experimental

Materials and methods

^{1}H and ^{13}C nuclear magnetic resonance (NMR) spectra were recorded on a Bruker 300 and 500 MHz spectrometer. All NMR samples were run in $CDCl_3$, and chemical shifts are expressed as ppm relative to internal Me_4Si. Mass spectra were obtained on a Fisons instrument. Substrates are commercially available and used without further purification. A laboratory microwave oven (MW 3100, Landgraf Laborsysteme HLL GmbH, Langenhagen, Germany) equipped with magnetic stirrer operating at 2450 MHz was used for synthesis of 2,5-dimethylpyrroles.

General procedure for preparation of 2,5-dimethylpyrroles

In a typical reaction, primary amine (1.0 mmol), hexane-2,5-dione (1.2 mmol), and $CaCl_2{\cdot}2H_2O$ (0.03 g, 0.2 mmol) were mixed thoroughly for 5 min. The mixture was taken in an open vessel and irradiated at 420 W (70 % of maximum power) for 10 min. Ethyl acetate (5 mL) and water (2 × 10 mL) were added to the reaction mixture. The organic layer was separated and dried over Na_2SO_4. After filtration, the recovered organic medium was subjected to gas chromatography (GC) analysis to determine the conversion. The solvent was then evaporated under reduced pressure to obtain the corresponding product. In cases where the reaction did not proceed to completeness, crude product was passed through a short column of neutral alumina [eluted with ethyl acetate/petroleum ether (3:7)] to give pure pyrrole **1**.

Selected spectroscopic data

N-(4′-Methoxyphenyl)-2,5-dimethylpyrrole (**1a**) [62] *[CAS registry no. 5044-27-9]* ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 7.13$ (d, $J = 8.7$ Hz, 2H), 6.97 (d, $J = 8.7$ Hz, 2H), 5.89 (s, 2H), 3.87 (s, 3H), 2.02 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 158.9, 131.7, 129.2, 129.0, 114.2, 105.3, 60.4, 14.2$. MS (EI): *m/z* (rel. intensity %) = 201 (M^+, 100), 200 (48), 186 (23), 154 (7), 145 (10).

N-Phenyl-2,5-dimethylpyrrole (**1b**) [63] *[CAS registry no. 83-24-9]* ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 7.52–7.48$ (m, 2H), 7.45–7.42 (m, 1H), 7.30–7.25 (d, 2H), 5.95 (s, 2H), 2.08 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$): $\delta = 138.9, 128.9, 128.8, 128.2, 127.6, 105.6, 12.9$. MS (EI): *m/z* (rel. intensity %) = 171 (M^+, 100), 154 (13).

N-(2′,5′-Dichlorophenyl)-2,5-dimethylpyrrole (**1n**) [64] ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 7.49$ (d, $J = 8.4$ Hz, 1H), 7.41–7.34 (m, 2H), 5.95 (s, 2H), 1.99 (s,

6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 138.0, 132.9, 132.3, 131.0, 130.7, 130.2, 128.8 (2C), 106.2 (2C), 12.5 (2C). MS (EI): *m/z* (rel. intensity %) 241 (45), 240 (73), 239 (M^+, 68), 238 (100), 224 (7), 203 (7), 168 (18), 109 (7), 83 (20).

N-(2′-Cyanophenyl)-2,5-dimethylpyrrole (**1o**) [65] *[CAS registry no. 124678-40-6]* ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.81 (d, J = 7.8 Hz, 1H), 7.74 (t, J = 7.8 Hz, 1H), 7.56 (t, J = 7.5 Hz, 1H), 7.38 (d, J = 8.1 Hz, 1H), 5.98 (s, 2H), 2.04 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 142.0, 133.6, 130.1, 128.8, 128.6, 115.9, 113.6, 107.1, 106.8, 12.5. MS (EI): *m/z* (rel. intensity %) = 196 (M^+, 80), 195(100), 181 (35), 154 (10), 102 (15), 92 (10), 75 (5).

N-Benzyl-2,5-dimethylpyrrole (**1q**) [66] *[CAS registry no. 5044-20-2]* ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.44–7.34 (m, 3H), 7.02 (d, J = 9.0 Hz, 2H), 6.00 (s, 2H), 5.14 (s, 2H), 2.27 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 139.1, 129.2, 128.5, 127.4, 126.1, 105.9, 47.2, 12.7; MS (EI): *m/z* (rel. intensity %) = 185 (M^+, 70), 91 (100).

N-Cyclohexyl-2,5-dimethylpyrrole (**1s**) [67] *[CAS registry no. 24836-02-0]* ^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.76 (s, 2H), 3.94 (m, 1H), 2.33 (s, 6H), 1.98–1.23 (m, 10H). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 128.3 (2C), 106.5 (2C), 56.9, 32.9 (2C), 27.1 (2C), 26.1 (2C), 14.9. MS (EI): *m/z* (rel. intensity %) = 178 (40), 177 (M^+, 100), 94 (10).

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