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To cite this article: Zakaria Benzekri, Houda Serrar, Sara Sibous, Said Boukhris, Ali Ouasri, Ali Rhandour & Abdelaziz Souizi (2016) Hybrid crystal $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ as an efficient catalyst for the synthesis of benzoxazoles, benzimidazoles and benzothiazoles under solvent-free conditions, Green Chemistry Letters and Reviews, 9:4, 223-228, DOI: [10.1080/17518253.2016.1242662](https://doi.org/10.1080/17518253.2016.1242662)

To link to this article: <https://doi.org/10.1080/17518253.2016.1242662>



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Published online: 21 Oct 2016.



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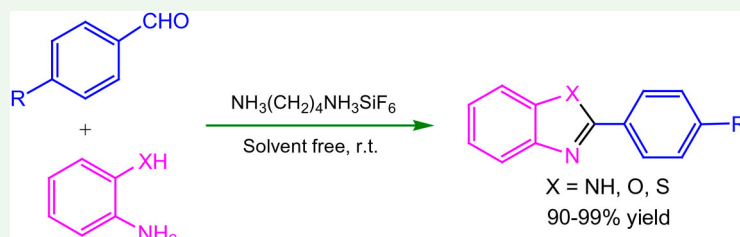
Hybrid crystal $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ as an efficient catalyst for the synthesis of benzoxazoles, benzimidazoles and benzothiazoles under solvent-free conditions

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ABSTRACT

An easy synthetic protocol for the synthesis of biologically active benzimidazole, benzothiazole and benzoxazole derivatives has been demonstrated using a hybrid crystal $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ as a mild and efficient heterogeneous catalyst. Short reaction times, solvent-free conditions, good to excellent yields, easy reusability and use of an eco-friendly catalyst are some of the significant attributes of the present method.



ARTICLE HISTORY

Received 21 February 2016

Accepted 26 September 2016

KEYWORDS

Heterogeneous catalyst; benzimidazole; benzothiazole; benzoxazole; butylene diammonium hexafluorosilicate; solvent free


Introduction

The benzimidazole, benzoxazole and benzothiazole skeletons may be found in numerous pharmaceutical agents with a diverse spectrum of biological properties (1–4). They are considered as privileged structures in the medicinal chemistry field (5) and are found in a large variety of natural products. They have been used as antiviral (6), antimicrobial (7), anti-tumor (8), antibiotic, antifungal (9), anti-oncuvulsant (10), anti-inflammatory (11), antiulcer (12a), antihelminthic (12a), anti-hypertensive (12b) and anti-analgesic (12c) agents. Moreover, their application in the field of advanced materials is worthy of note (13). The protocols usually followed for their synthesis involve condensation of ortho-esters (14–15), nitriles (16), aldehydes (17–18), carboxylic acids (19), amides (20) and esters (21) with ortho-substituted aminoaromatics, in the presence of different acids or catalysts such as Pd-catalyzed oxidative cyclization (22), base-assisted reaction of 1,1-dibromoethanes (23), different hetero-polyacid catalysts (24), In (OTf)₃ (25), Cu-np/SiO₂ (26), iron phthalocyanine (27), nanoCeO₂ (28), nanosolid acids (29), CuFe₂O₄ (30), Fe (NO₃)₃/Tempo (31), RHA-SO₃H (32) and Fe(III)-Schiff base/

SBA-15 (33). Therefore, there is a strong demand for a more effective, simple and environmentally friendly process for the synthesis of these heterocycles.

In order to develop such processes, it was used in the present synthetic protocols, the butane-1,4-diammonium hexafluorosilicate $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ as a solid catalyst; that is a novel hybrid compound (34) that belongs to the family of alkylendiammonium halogenometallate salts of the general formula $\text{NH}_3(\text{CH}_2)_n\text{NH}_3\text{MX}_6$ (IV) (M: Sn, Si, Te; X: Cl, Br, I and F) that have recently attracted considerable attention due to their important chemical and physical properties (35–37). Generally, the hexafluorosilicate $\text{NH}_3(\text{CH}_2)_n\text{NH}_3\text{SiF}_6$ salts were less studied in comparison with other hexahalogenometallate $\text{NH}_3(\text{CH}_2)_n\text{NH}_3\text{MX}_6$ salts (M: Sn, Te; X: Cl, Br, I) (38–42). But, the monoalkylammonium hexafluorosilicate compounds have extensively been studied and some of them have been found to be of interest for the study of crystal dynamic and phase transition involving hydrogen bonds as well as the reorientation motions of alkylammoniums cations (39–42). The synthesis of complex $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ is a practical way

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 Supplemental data for this article can be accessed at [10.1080/17518253.2016.1242662](http://dx.doi.org/10.1080/17518253.2016.1242662).

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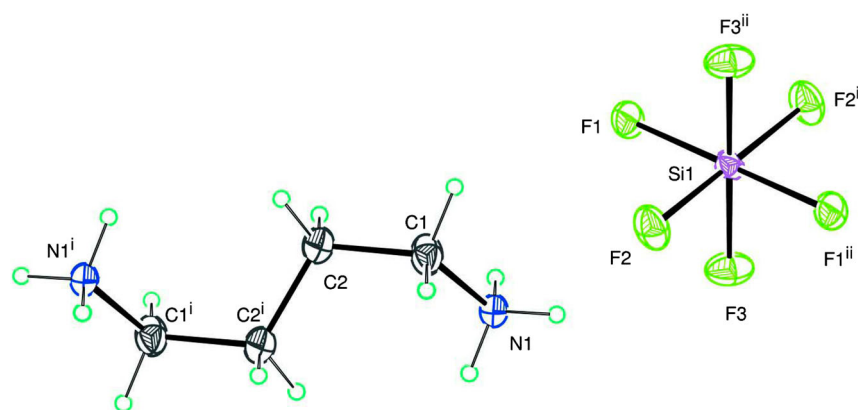


Figure 1. The molecular structure of the $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ compound with the atom-labeling scheme as has been determined. The symmetry codes are: (i) $-x-1, -y+1, -z+1$; (ii) $-x+1, -y, -z$.

to produce heterogeneous catalyst with the associated advantages such as easy catalyst separation, possible recycling, high activity and selectivity.

In the present study, we report the synthesis of benzimidazole, benzoxazole and benzothiazole derivatives by condensation of 1,2-phenylenediamine, ortho-aminophenol and ortho-aminothiophenol with various aromatic aldehydes using $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ as an efficient catalyst in free solvent conditions.

Results and discussion

The $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ crystal has been recently synthesized and characterized by X-ray diffraction (34). The crystal structure of this compound has been determined in the triclinic system with $P\bar{1}$ ($Z=1$) as space group and $a=5.796$ (1) Å, $b=5.889$ (1) Å, $c=7.774$ (2) Å and

$\alpha=87.02$ (1)°, $\beta=82.15$ (1)° and $\gamma=61.87$ (1)°, $V=231.79$ (8) Å³ as parameters of the crystal unit cell.

The molecular structure of the $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ compound is given in Figure 1. The crystal structure of this little compound has been found to be built up from inorganic anions SiF_6^{2-} linked to the organic cations $^+\text{NH}_3(\text{CH}_2)_4\text{NH}_3^+$ through N–H...F hydrogen bonds producing an infinite two-dimensional layer parallel to (0 1 1) (Figure 2).

The intermolecular hydrogen bonding contacts N–H...F types provide a linkage between cationic entities $^+\text{NH}_3(\text{CH}_2)_4\text{NH}_3^+$ and SiF_6^{2-} anionic complexes. The hydrogen bonds involved (donors and acceptors) ensure the three-dimensional cohesion of the atomic arrangement. The unit cell of the $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ compound contains only one organic cation $^+\text{NH}_3(\text{CH}_2)_4\text{NH}_3^+$ and one slightly distorted SiF_6^{2-} anion. In this structure, all

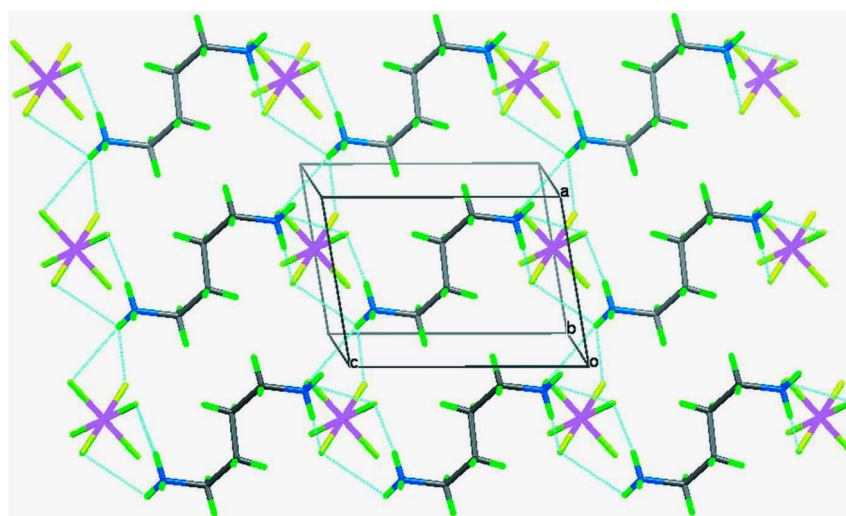
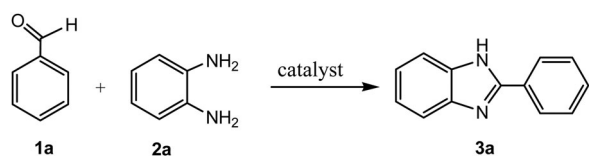


Figure 2. The three-dimensional plot of the $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ compound, showing inorganic sheets linked through N–H...F hydrogen bonds to the organic layers (dashed lines) (34).



Scheme 1. Model reaction for the synthesis of 2-phenylbenzimidazole.

atoms have been found to be in general positions, except the silicon atom Si which has been located at a crystallographic center of inversion (1/2, 0, 0) of the $P1$ space group. The silicon atom was surrounded by six fluorine atoms building a slightly distorted SiF_6^{2-} octahedron. In addition, the center of the bond C2–C2 (i) [with (i) being a symmetry code: $-x-1, -y+1, -z+1$] was also situated on another crystallographic center of inversion.

Working on a series of new alkylendiammonium halogenometallate compounds, we found it interesting to study the efficiency of the hybrid compound $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ as heterogeneous solid catalysts for the preparation of benzoxazoles, benzothiazoles and benzimidazoles. At first, 1,2-phenylenediamine **1a** and benzaldehyde **2a** were selected as the model substrates to investigate the best reaction conditions (Scheme 1). Then several reaction conditions were tried to accomplish this reaction and the efficiency of the reaction was found to be influenced by the quantity of the catalyst and solvent at room temperature. In the absence of a catalyst, only 12% yield of the desired product was obtained even after longer reaction time (Table 1, entry 1). However, in the presence of the catalyst the product **3a** was obtained in 93% yield and only after 14 min (Table 1, entry 2). Next, the optimization of reaction conditions was undertaken to increase the yield of the product using various solvents, which showed a prominent influence on reaction time and yields to obtain the desired product (Table 1, entries

Table 1. Screening of the reaction conditions for the reaction of 1,2-phenylenediamine **1a** and benzaldehyde **2a**^a.

Entry	Catalyst (mol%)	Solvent (1 mL)	Time ^b (min)	Yield (%) ^c
1	–	EtOH	240	12
2	5	EtOH	14	93
3	5	MeOH	10	93
4	5	Butanol	23	80
5	5	Isopropanol	30	77
6	5	CH_3CN	40	67
7	5	THF	25	69
8	5	ACOEt	15	79
9	5	Solvent free	4	94
10	4	Solvent free	4	94
11	3	Solvent free	4	95
12	2	Solvent free	4	95
13	1	Solvent free	4	98

^aReaction conditions: benzaldehyde (**1a**, 1 mmol), o-phenylenediamine (**2a**, 1 mmol), room temperature.

^bTime reported in min monitored by thin layer chromatography (TLC).

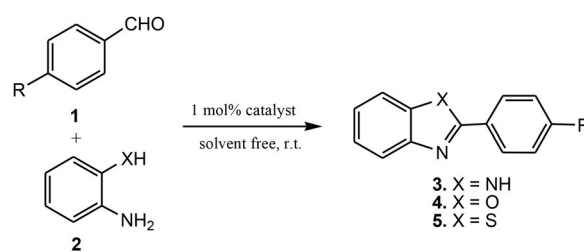
^cIsolated yield.

Table 2. Substrate scope for synthesis of desired heterocycles derivatives.

Product	R	X	Time min ^a	Yield % ^b	Melting point (°C)		Ref.
					Found	Reported	
3a	H	NH	4	98	>266	292–294	33
3b	Cl	NH	2	97	>266	288–290	33
3c	Me	NH	6	95	>266	275–276	33
3d	NO_2	NH	2	99	>266	318–320	33
4a	H	S	4	97	112–113	110–114	33
4b	Cl	S	6	94	114–116	114–116	33
4c	Me	S	3	90	85–87	86–89	43
4d	NO_2	S	5	99	226–227	227–229	33
5a	H	O	5	98	100–102	100–102	43
5b	Cl	O	3	97	144–145	144–146	43
5c	Me	O	6	96	88–90	88–89	43
5d	NO_2	O	5	99	261–262	262–264	43
5e	OH	O	2	98	220–221	220–223	43

^aIsolated yields.

^bTime reported in min monitored by TLC.



Scheme 2. Synthesis of compounds **3**, **4** and **5** derivatives catalyzed by $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$.

2–8). It was also found that the reaction proceeded efficiently without solvent and resulted in high yields of the desired product (Table 1, entries 9–13). Furthermore, the catalytic efficiency of $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ was examined using different amounts of this catalyst. Interestingly, the yield of **3a** was significantly increased to 98% by employing 1 mol% of the catalyst, without solvent, at room temperature (Table 1, entry 13). The opportunity of applying solvent-free conditions is an environmentally significant benefit of the procedure.

The generality of the procedure was evaluated for the synthesis of benzimidazole **3**, benzoxazole **4** and benzothiazole **5** derivatives by condensation of different 1,2-phenylene diamine, 1,2-aminophenol and 1,2-aminothiophenol with substituted aldehydes (Scheme 2). As shown in Table 2, good to excellent yields were obtained for these reactions. The reactivity of **1** with

Table 3. Reusability of the catalyst in the synthesis of 2-phenylbenzimidazole **3a**.

Run	Time (min)	Yield (%) ^a
1	4	98
2	4	97
3	4	97
4	4	96
5	4	95
6	4	95

^aIsolated yields.

Table 4. Comparative synthesis of desired heterocycles using the reported catalysts versus $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$.

Catalyst	Amount of catalyst/reaction conditions	Time	Yield %
$\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$	1 mol% solvent free, RT	2–6 min	90–99
Fe(III)–Schiff base/SBA-15 (33)	0.01 g, 0.0014 mmol (0.01 based on metal ion), water, reflux	3 h	79–92
ZnBr ₂ /ABM (44a)	100 mg, 5 mL, toluene, 111°C	10–25 min	83–96
Indion 190 resin (44b)	10%/weight, 5 mL EtOH, 70°C	4 h	78–92
Nano-ZnO (29)	10 mol%, 10 mL EtOH, reflux	80–100 min	64–88
CuO-np/SiO ₂ (26)	10 mol %, 10 mL, MeOH, RT	4–14 h	68–93
ZnO PNs (43)	5 mg; solvent free, or in EtOH, RT	2–8 min	90–99
t-Butyl hypochloride (44c)	2 mmol, 10 mL CH ₃ CN, reflux	1–4 h	65–92
ABMs-ZnBr ₂ (44d)	Toluene; air, 110°C	10–25 min	81–96
PIFA (44e)	1.05 equiv, 3 mL EtOH, 800C, μW	15 min	59–90
Cu NPs/SiO ₂ (44f)	10 mol%, methanol, RT	4–8 h	72–86
CeO ₂ NPs (44g)	10 mol%, H ₂ O, RT	20–40 min	58–97
PTSA (44h)	10 mol%, water, 70°C	1–5 h	60–97
FeCl ₃ /montmorillonite K-10 (44i)	10%w/w, CH ₃ OH, condensation, ultrasonic irradiation	0.7–5 h	33–95

aldehyde derivatives **2** appear to be not controlled by electronic effects. Indeed, the reaction with both electron-rich and electron-deficient aldehydes is good and affording benzimidazole **3**, benzothiazole **4** and benzoxazole **5** in high yields (Scheme 2). The prepared products are known compounds and were confirmed by comparing the ¹H NMR and ¹³C NMR spectral data with authentic samples reported in the literature (33, 43).

The recycling of the catalyst was also studied. For this, the catalyst was filtered, separated, washed with methanol and dried at 80°C for 45 min and then the residual catalyst as such was reused without loss of any significant catalytic activity. The structure and aspect of the catalyst remains unchanged after recovery and reuse. In the case of the model reaction, the catalyst was recovered and reused six times without any significant changes in the yield and the reaction time (Table 3).

In order to compare the capability and efficiency of our catalyst with respect to the previously reported catalysts in the literature (26, 29, 33, 43–44), the results for the synthesis of benzimidazoles, benzothiazoles and benzoxazoles employing these catalysts are tabulated in Table 4. As it is clear from the Table, $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ is more efficient than the others.

Conclusion

In conclusion, an efficient and mild one-pot protocol for the synthesis of benzimidazoles, benzothiazoles and benzoxazoles using a crystal $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ as a catalyst in free solvent has been developed. The use of the crystal as reusable heterogeneous catalyst, solvent-free conditions, excellent product yields and shorter reaction time make this protocol practical and environment-friendly. In addition, the preliminary toxicity evaluation has showed that the catalyst is non-toxic at low concentration levels. The study on the exact mechanism of $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ applicable to the preparation of these heterocycle derivatives is underway in our laboratory.

Experimental

Chemicals and apparatus

All the chemicals used were purchased from Sigma-Aldrich and were used as such. All products are known, and were identified by comparison of spectral and physical data with the literature. Melting points were taken on a KOFER hot stage apparatus and are uncorrected. ¹H NMR and ¹³C NMR spectra were recorded on a Bruker 300-MHz spectrometer in DMSO-d₆.

General procedure for the synthesis of catalyst

The catalyst single crystal has been recently synthesized by slow evaporation of aqueous solution containing $\text{NH}_2(\text{CH}_2)_4\text{NH}_2$ and H_2SiF_6 by the following method: the solid butane-1,4-diamine $\text{NH}_2(\text{CH}_2)_4\text{NH}_2$ (percentage $\geq 99\%$) was primarily dissolved in the necessary distilled water and then was mixed with the stoichiometric amounts (1:1) to H_2SiF_6 (percentage = 34% in weight). The obtained solution was taken under room temperature for evaporation. The colorless single crystals were so obtained

General procedure for the preparation of benzimidazoles, benzothiazoles and benzoxazoles

Aldehyde (1 mmol), o-phenylenediamine, o-amino thiophenol or o-amino phenol (1 mmol) and 1 mol % (2.32 mg) of $\text{NH}_3(\text{CH}_2)_4\text{NH}_3\text{SiF}_6$ were stirred, in free solvent conditions, at room temperature for the appropriate time (Table 2). The progress of the reaction was monitored by TLC hexane/ethyl acetate (70:30) as eluent. After completion of the reaction, the crude reaction mixture was dissolved in EtOH, and the catalyst was separated out by simple filtration. The product was recrystallized from ethanol to give respectively

benzimidazole **3**, benzothiazole **4** and benzoxazole **5** in high yields (90–99%, Table 2).

Disclosure statement

No potential conflict of interest was reported by the authors.

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Zakaria Benzekri received a master's degree in Organic Chemistry from Faculty of Sciences, Ibn Tofail University, Morocco, in 2013 and is now a Ph.D. student in organic chemistry at Ibn Tofail University. His research has focused on the organic synthesis, catalysis and application of natural and synthetic compounds as catalyst in the organic transformations.

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