CHEMISTRY & CHEMICAL TECHNOLOGY

*Chem. Chem. Technol., 2022,*
*Vol. 16, No. 1, pp. 126–132*

**Chemical Technology**

# A GREEN AND EFFICIENT SOLVENT- AND CATALYST-FREE ULTRASONIC DIBENZYLATION PROCEDURE

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https://doi.org/10.23939/chcht16.01.126

**Abstract.** A greener improvement was achieved for the synthesis of diverse *N,N*-dibenzylated compounds from the reaction of various aromatic amines with benzyl bromide using the ultrasound irradiation in solvent- and catalyst-free conditions. The dibenzylation reactions were carried out in different solvents and solvent-free conditions under ultrasound irradiation at various temperatures. The yields were very low in all applied solvents, while in the solvent-free condition and at room temperature, the yields were excellent. Due to obtaining the high reaction yields, the catalyst was not used.

**Keywords:** *N,N*-dibenzylation, aromatic amines, ultrasound irradiation, solvent and catalyst-free reactions, room temperature.

## 1. Introduction

Benzylation of aromatic compounds has been studied extensively using various catalysts. For example, Anand *et al.*[1] investigated the Friedel-Crafts benzylation of benzene and various substituted benzenes employing benzyl chloride as an alkylating agent over a series of three-dimensional cage type mesoporous ferrosilicate catalysts. Benzylation of phenol was successfully achieved by Yu *et al.*[2] in water under room temperature mediated by tetrabutylammonium bromide for only two hours affording the corresponding benzyl phenyl ether with good to excellent yields. Hoa and coworkers report the synthesis of nanoporous $\alpha$-$Fe_2O_3$ nanoparticles *via* a hydrothermal method and their application in the catalytic benzylation of benzene and benzyl chloride in the fabrication of diphenylmethane.[3] The benzylation of benzene was carried out by Viswanadham *et al.*[4] with benzyl alcohol in the presence of mesoporous mordenite. A novel Brønsted-Lewis acidic catalyst, $Hf_{0.5}[TEAPS]PW_{12}O_{40}$, was prepared by Xie and coworkers *via* the replacement of protons in neat phosphotungstic acid with both organic and metal cations to promote the conversion of dibenzyl ether to the benzylation products.[5] The benzylation of phenol with benzyl alcohol was carried out by Yadav *et al.*[6] using 20% (w/w) $Cs_{2.5}H_{0.5}PW_{12}O_{40}$ supported on mesocellular foam silica. The silica-immobilized $FeCl_3$ nano-catalysts were dispersed by Su *et al.*[7] in benzyl chloride to form a nanofluid, and then were applied for the benzylation of toluene with benzyl chloride to produce monobenzyl toluene and dibenzyl toluene. Kurian and coworkers investigated the liquid phase benzylation of benzene with benzyl chloride over different compositions of cobalt zinc ferrite ($Co_xZn_{1-x}Fe_2O_4$, x: 0.0, 0.25, 0.5, 0.75, 1.0) nano composites.[8] Lee *et al.*[9] fabricated the Ru nanodots uniformly dispersed on graphene oxide nanosheets *via* a hydrothermal process to investigate the synergistic interactions of the bifunctional reduced graphene oxide nanosheets and Ru composites, for the hydrogenative-reductive benzylation of *N*-heteroaromatics. Dar and coworkers described a one-pot procedure for the direct *N,N*-dibenzylation of anilines using a catalytic amount of the Cu modified montmorillonite-KSF and showed that the applied catalyst is a simple, efficient, mild, convenient, and effective catalyst for the selective benzylation of anilines with benzyl bromide and the Cu loading plays significant role in product yield and the selectivity of the reactions were controlled by solvents.[10]

Now, we would like to report the synthesis of diverse *N,N*-dibenzylated compounds (**2a-k**) by the reaction of different aromatic amines (**1a-k**) with benzyl bromide under ultrasound irradiation in solvent- and catalyst-free conditions at room temperature (Scheme 1).

## 2. Experimental

### 2.1. Materials

All chemicals (starting materials, reagents, solvents, *etc.*) were purchased from the Aldrich and Merck chemical companies with high-grade quality and used

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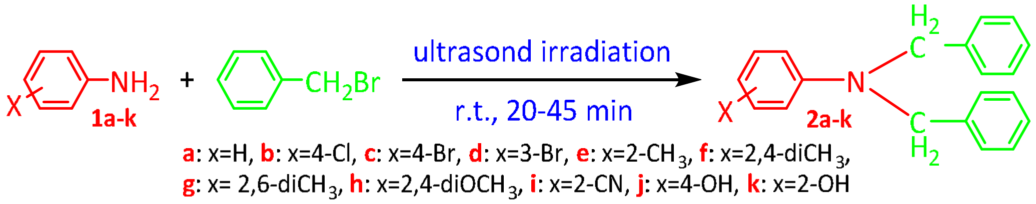
without further purification. Melting points were taken in open capillary tubes with a BUCHI 510 melting point apparatus and were uncorrected. NMR spectra were recorded in DMSO-$d_6$ on a JEOL FX-NMR 90 MHZ spectrometer. FT-IR (KBr) spectra were recorded on a Perkin-Elmer 781 spectrophotometer.

Ultrasonication was performed in a PARSONIC 2600s ultrasound cleaner with the frequency of 28 kHz and an output power of 50 W (Builtin heating, 293–343 K thermo-statically adjustable). The reaction flask was located at the maximum energy area in the ultrasound cleaner and the surface of the reactants was placed slightly lower than the level of water. The reaction temperature was controlled by addition or removal of water from the ultrasonic bath. The temperature of the water bath was controlled at about 298–303 K.



**Scheme 1.** Synthesis of diverse *N,N*-dibenzylated compounds under ultrasound irradiation

## 2.2. General Procedure for the Preparation of Different Dibenzylated Products

An aromatic amine (1.0 mmol) and benzyl bromide (2.0 mmol) were mixed in a test tube and put in an ultrasonic bath for the appropriate time at room temperature. After completion of the reaction, which was monitored by TLC, the reaction mixture was dissolved in ethanol (5 mL), and distilled water was gradually added until the solid separated. The solid was then filtered and washed several times with water to give the corresponding dibenzylated product which was characterized by different spectroscopic methods. Since the dibenzylated products were washed several times with water, the elemental analyses did not show any bromine in the products.

All obtained products were known compounds and characterized by comparing their physical or spectral data and melting points with those of authentic samples. In the IR spectra of the product, disappearance of two N-H peaks at about 33300–3500 cm$^{-1}$, confirms the formation of the dibenzylated products.

## 2.3. Selected Spectral Data

***N,N*-Dibenzylaniline (2a):** m.p. 339–341 K; IR (KBr, cm$^{-1}$): 3030, 2981, 2832, 2582, 1615, 1593, 1497, 1392, 1261, 750; $^1$H NMR (90 MHz, DMSO-$d_6$) $\delta_H$ (ppm): 7.25–6.63 (m, 15H, aromatic), 4.66 (s, 2-CH$_2$, 4H).

***N,N*-Dibenzyl-4-chloroaniline (2b):** m.p. 378 K; IR (KBr, cm$^{-1}$): 3086, 3026, 2904, 1652, 1597, 1499, 1354, 1200, 804, 730; $^1$H NMR (90 MHz, DMSO-$d_6$) $\delta_H$ (ppm): 7.26–6.58 (m, 14H, aromatic), 4.678 (s, 2-CH$_2$, 4H).

***N,N*-Dibenzyl-3-bromoaniline (2d):** m.p. 375–377 K; IR (KBr, cm$^{-1}$): 3062, 3028, 2867, 1592, 1452, 1360, 1232, 1075, 814, 759; $^1$H NMR (90 MHz, DMSO-$d_6$) $\delta_H$ (ppm): 7.24–6.64 (m, 14H, aromatic), 4.67 (s, 2-CH$_2$, 4H).

***N,N*-Dibenzyl-2,4-dimethylaniline (2f):** m.p. 418 K; IR (KBr, cm$^{-1}$): 3033, 2985, 2830, 2705, 2580, 1618, 1495, 1390, 1263, 1084, 752, 698, 615; $^1$H NMR (90 MHz, DMSO-$d_6$) $\delta_H$ (ppm): 7.23 (m, 13H, aromatic), 4.67 (s, 2-CH$_2$, 4H).

***N,N*-Dibenzyl-2,6-dimethylaniline (2g):** m.p. 421–423 K; IR (KBr, cm$^{-1}$): 3025, 2933, 1605, 1506, 1410, 750, 731, 692, 503; $^1$H NMR (90 MHz, DMSO-$d_6$) $\delta_H$ (ppm): 6.9–7.23 (m, 13H, aromatic), 5 (s, 2-CH$_2$, 4H), 2.1 (s, 2-CH$_3$, 6H).

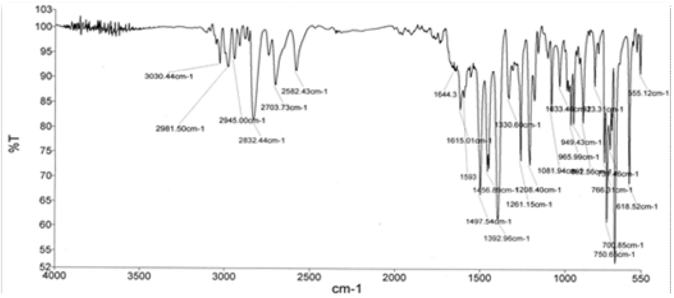


**Fig. 1.** The IR spectrum of *N,N*-dibenzylaniline (**2a**)

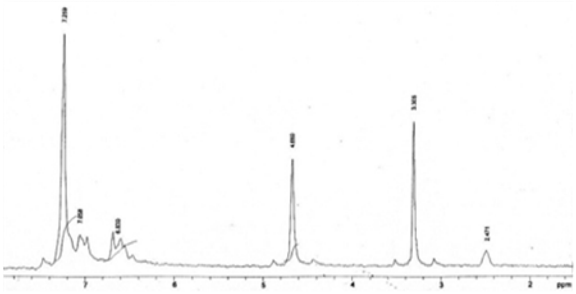


**Fig. 2.** The NMR spectrum of *N,N*-dibenzylaniline (**2a**)

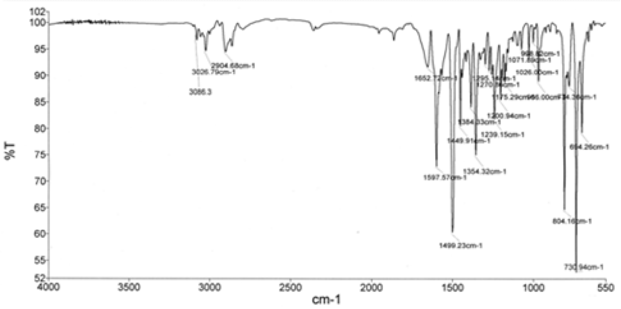


Fig. 3. The IR spectrum of *N,N*-dibenzyl-4-chlorolaniline (**2b**)

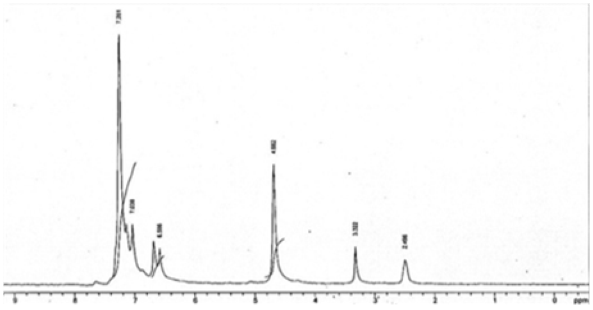


Fig. 4. The NMR spectrum of *N,N*-dibenzyl-4-chlorolaniline (**2b**)

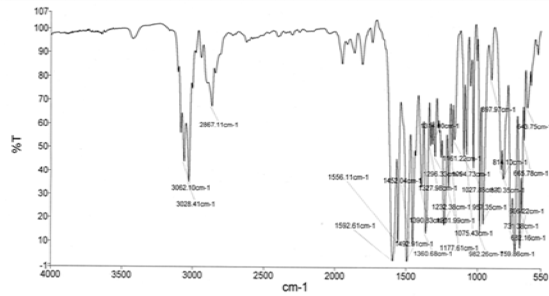


Fig. 5. The IR spectrum of *N,N*-dibenzyl-3-bromolaniline (**2d**)

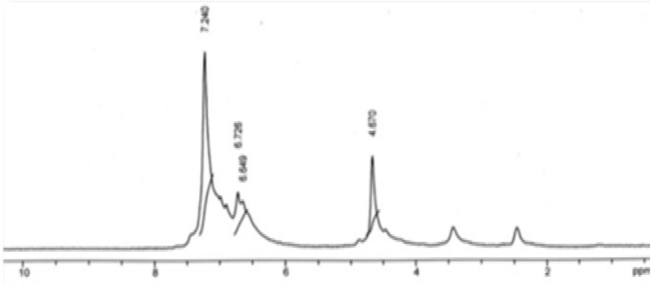


Fig. 6. The NMR spectrum of *N,N*-dibenzyl-3-bromolaniline (**2d**)

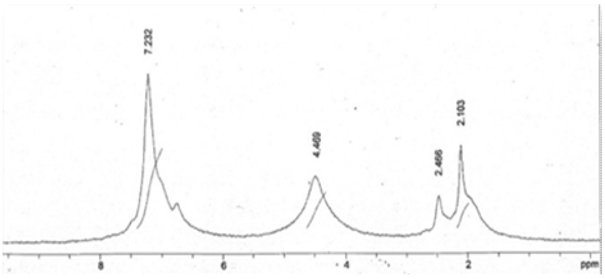


Fig. 7. The NMR spectrum of *N,N*-dibenzyl-2,4-dimethylaniline (**2f**)

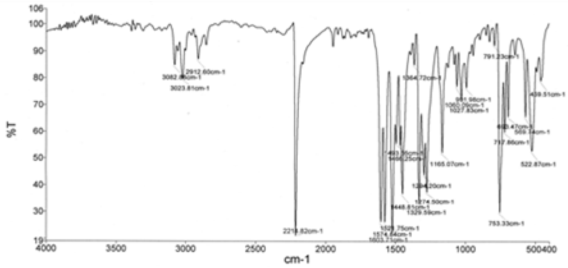


Fig. 8. The IR spectrum of 2-(dibenzylamino)benzonitrile (**2i**)

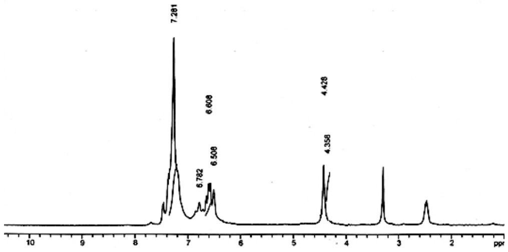


Fig. 9. The NMR spectrum of 2-(dibenzylamino)benzonitrile (**2i**)

***N,N*-Dibenzyl-2,6-dimethoxylaniline (2h):** m.p. 365–367 K; IR (KBr, $cm^{-1}$): 3032, 2937, 1600, 1510, 1403, 1062, 755, 733, 691, 506; $^1H$ NMR (90 MHz, DMSO-$d_6$) $\delta_H$ (ppm): 6.5–7.3 (m, 13H, aromatic), 5 (s, 2-$CH_2$, 4H), 3.8 (s, 2-$CH_3$, 6H).

**2-(Dibenzylamino)benzonitrile (2i):** m.p. 377–380 K; IR (KBr, $cm^{-1}$): 3082, 3023, 2912, 2216, 1603, 1448, 1329, 753, 522; $^1H$ NMR (90 MHz, DMSO-$d_6$) $\delta_H$ (ppm): 7.28–6.50 (m, 14H, aromatic), 4.42 (s, -2$CH_2$, 4H).

**4-(Dibenzylamino)phenol (2j):** m.p. 385 K; IR (KBr, $cm^{-1}$): 3165, 3000, 2905, 1610, 1502, 1490, 1223, 1081, 1065, 710, 653, 580, 405; $^1H$ NMR (90 MHz, DMSO-$d_6$) $\delta_H$ (ppm): 9.5 (s, 1H, phenolic H), 6.6-7.3 (m, 14H, aromatic), 5 (s, 2-$CH_2$, 4H).

**2-(Dibenzylamino)phenol (2k):** m.p. 353–355 K; IR (KBr, $cm^{-1}$): 3156, 2995, 2910, 1606, 1495, 1225, 1082, 705, 652, 583, 402; $^1$H NMR (90 MHz, DMSO-$d_6$) $\delta_H$ (ppm): 10 (s, 1H, phenolic H), 6.7-7.3 (m, 14H, aromatic), 5 (s, 2-$CH_2$, 4H).

## 3. Results and Discussion

As was stated before, the benzylation of nitrogen atoms is an important and frequently utilized protection strategy in organic synthesis. The most commonly used benzylation methodology has some disadvantages such as the use of toxic and carcinogenic reagents and working in harsh conditions with tedious work-up. So, we decided to carry out the dibenzylation of aromatic compounds by ultrasound irradiation. This method is cheap, and the reaction was carried out in solvent- and catalyst-free conditions. This simple, fast, and highly efficient green procedure provided the high pure products and quantitative yield within minutes at room temperature. In addition, this protocol does not require any water quenches, solvent separations, and purification steps such as column chromatography or recrystallization.

### 3.1. Optimization

To optimize the reaction conditions, dibenzylation reaction of aniline with benzyl bromide was carried out in different solvents such as water, ethanol, ethyl acetate, acetonitrile, hexane, and dichloromethane under ultrasound irradiations at room temperature and reflux conditions. The yields were very low in all applied solvents, so we decided to realize the dibenzylation reaction in solvent-free condition at room temperature and the yield was excellent.

Despite the guess about the best molar ratio of aniline to benzyl bromide (1:2, according to the starting materials molar ratio equivalence), reaction of aniline with three different molar ratios of benzyl bromide were carried out in solvent-free condition at room temperature under ultrasound irradiations to find out the best reaction conditions (Table 1). It was found that the 1:2 molar ratio of aniline to benzyl bromide is the best reaction condition with about 88 % yield.

Also, since the yields of the dibenzylation reactions were good, the catalyst was not used.

**Table 1.** Optimization of the reaction of aniline with benzyl bromide

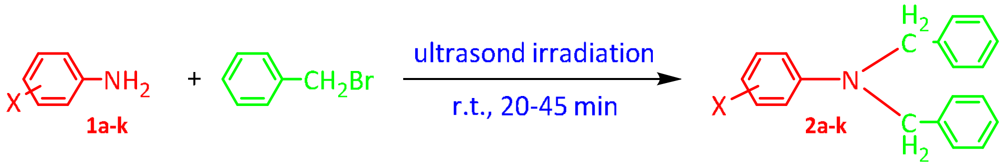
| Molar ratio of aniline to benzyl bromide | Time, min | Yield, % |
|---|---|---|
| 1:1 | 80 | 68 |
| **1:2** | **20** | **88** |
| 1:3 | 40 | 53 |

### 3.2. Synthesis of Dibenzylated Compounds (2a-k)

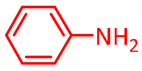
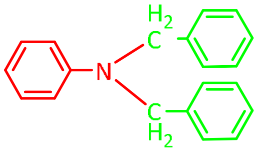
According to the obtained results regarding the reaction optimization, diverse dibenzylated compounds were prepared by the reaction of different aromatic amines (**1a-k**) with benzyl bromide using ultrasound irradiation at solvent- and catalyst-free conditions (Table 2). The products were characterized by different spectroscopic methods and melting points and the physical data of known compounds were found to be identical with those reported in the literature.

All applied amines, regardless of possessing the electron releasing or electron withdrawing groups, are potent to give the good yields, which shows the capability of the applied method. In the absence of ultrasonic irradiation, the reaction will not be completed even after 24 h.

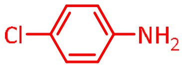
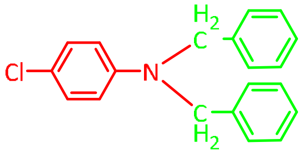
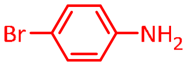
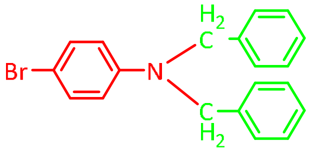
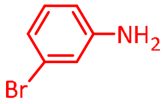
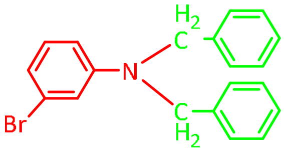
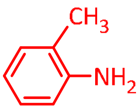
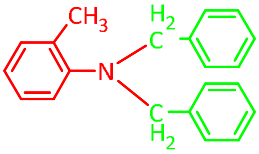
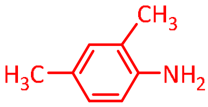
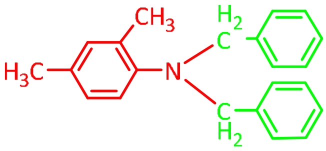
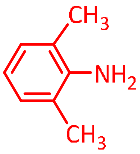
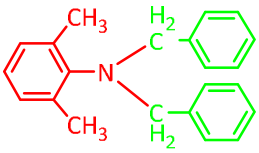
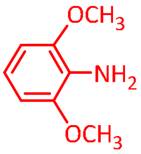
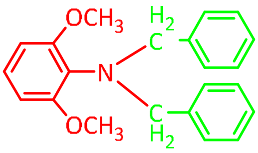
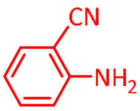
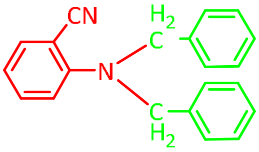
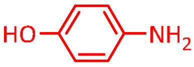
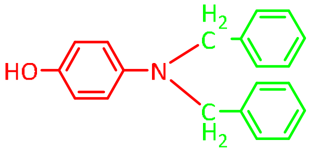
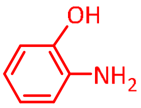
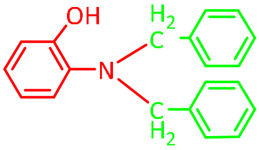
**Table 2.** Synthesis of diverse dibenzylated compounds (2a-k)



**a**: x=H, **b**: x=4-Cl, **c**: x=4-Br, **d**: x=3-Br, **e**: x=2-$CH_3$, **f:** x=2,4-di$CH_3$, **g**: x= 2,6-di$CH_3$, **h**: x=2,4-di$OCH_3$, **i**: x=2-CN, **j**: x=4-OH, **k**: x=2-OH

| Arylamine (**1a-k**) | Product (**2a-k**) | Time, min | Yield, % | Melting point, K | |
|---|---|---|---|---|---|
| | | | | Found | Ref.[11-19] |
| 1 | 2 | 3 | 4 | 5 | 6 |
|  **1a** |  **2a** | 20 | 88 | 339–341 | 339–342 |

Continuation of Table 2

| 1 | 2 | 3 | 4 | 5 | 6 |
|---|---|---|---|---|---|
|  **1b** |  **2b** | 20 | 83 | 378 | 377–379 |
|  **1c** |  **2c** | 35 | 82 | 397–399 | 397–398 |
|  **1d** |  **2d** | 30 | 84 | 375–377 | 375–377 |
|  **1e** |  **2e** | 20 | 86 | 315–316 | 315 |
|  **1f** |  **2f** | 30 | 83 | 418 | 417–419 |
|  **1g** |  **2g** | 35 | 84 | 421–423 | – |
|  **1h** |  **2h** | 40 | 85 | 365–367 | – |
|  **1i** |  **2i** | 45 | 86 | 377–380 | – |
|  **1j** |  **2j** | 20 | 85 | 385 | 400 |
|  **1k** |  **2k** | 30 | 83 | 353–355 | – |

As we know, the mixing caused by the ultrasound irradiation is more effective than the physical mixing that can be provided by different conventional energy sources. The ultrasound approach offers several advantages such as higher yields, enhanced organic reaction rates, milder reaction conditions, and waste minimization compared with traditional methods and saving money and energy. Compared to conventional heating which provides thermal energy in the macro system, ultrasound irradiation is able to activate many organic reactions by providing the activation energy in micro-environment.[20-23]

## 4. Conclusions

We were successful in the synthesis of diverse *N*,*N*-dibenzylated compounds from the reaction of various aromatic amines with benzyl bromide using the ultrasound irradiation in solvent- and catalyst-free conditions at room temperature.

## Acknowledgements

We are thankful to the Bu-Ali Sina University, Hamedan Iran, for the support of this work.

## References

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*Received: June 16, 2020 / Revised: August 20, 2020 / Accepted: October 12, 2020*

# "ЗЕЛЕНА" І ЕФЕКТИВНА ПРОЦЕДУРА УЛЬТРАЗВУКОВОГО ДИБЕНЗИЛУВАННЯ, БЕЗ РОЗЧИННИКІВ І КАТАЛІЗАТОРІВ

***Анотація**. Запропоновано екологічний синтез різноманітних N,N-дибензильованих сполук внаслідок реакції різних ароматичних амінів з бензилбромідом за допомогою ультразвукового опромінення без розчинників та каталізаторів. Вивчено реакції дибензилування в різних розчинниках та без них за умов ультразвукового опромінення і різних температур. При застосуванні розчиників одержано дуже низькі виходи, а при реакції без розчинників та за кімнатної температури виходи є високими. У зв'язку з одержанням високих виходів реакції каталізатор не використовували.*

***Ключові слова**: N, N-дибензилування, ароматичні аміни, опромінення ультразвуком, реакції без розчинників та каталізаторів, кімнатна температура.*