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NOTE

Microwave-Assisted Synthesis of Benzyl Benzotriazoles

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A series of substituted benzotriazoles were synthesized by substituted benzyl chloride in the presence of benzotriazole under the condition of microwave irradiation.

Key Words: Microwave irradiation, Benzotriazole, Solvent-free.

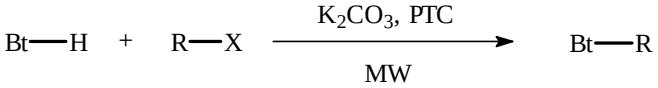
In recent reports, it was shown that benzotriazoles are very useful starting materials for the synthesis of various bioactive molecules[1]. Substituted benzotriazole are widely applied in medicine and agriculture as pesticides[2].

Several procedures are available for the one-step synthesis of benzotriazole derivative using benzotriazole and haloalkyl[3,4]. However, most the methods suffer from serious drawbacks which include the use of hazardous and expensive or commercially unavailable reagents, long reaction times, drastic reaction conditions and tedious procedure.

Microwave technique, meanwhile, has been widely used for a variety of organic reactions[5,6] such as Claisen, cyclization, oxidation, Diels-Alder reaction, hydrolysis, esterification, etherification, *etc*. Many reviews[7-9] have been published in favour of its considerable accelerations of the reaction rates and satisfactory yields.

Melting points were determined using a Yanaco MP-241 apparatus and are uncorrected. Infrared spectra were recorded on a Brucker Equinox55 FTIR spectrophotometer as potassium bromide tablets. ^{1}H NMR spectra were measured on a Brucker AC-P500 instrument (300 MHz) using tetramethylsilane as an internal standard and DMSO-d_6 as solvent. Elemental analyses were performed on a Yanaco MT-3CHN elemental analyzer.

In view of this, herein the preparation of a new series of substituted aromatic benzotriazole (Bt) using a microwave technique is reported with the objective of obtaining new biologically active compounds (**Scheme-I**).



Scheme-I

The reactants benzotriazole (0.01 mol), K_2CO_3 (0.045 mol), phase transfer catalyst (PTC) *i.e.*, Bu_4NBr (0.05 g) were grinded in a mortar with a pestle at room temperature for 10 min. Then it was irradiated in a microwave oven (200 W) for 5 min and add 40 % NaOH. The products was recrystalled from EtOH. Pured products enough for spectral characterization (IR, ^1H NMR).Yields are given in Table-1. All derived benzotriazoles are known compounds and their spectral data, as well as melting points of solids, were in agreement with reported values[10-15].

TABLE-1
PHYSICAL DATA OF BENZOTRIAZOLES UNDER MICROWAVE IRRADIATION

R	m.f.	Yield (%)	m.p. (ºC)	Elemental analysis % Calcd. (Found)		
				C	H	N
C_6H_5	$C_{13}H_{11}N_3$	91.2	113-114	74.62 (74.34)	5.30 (5.15)	20.08 (20.10)
p-$CH_3C_6H_4$	$C_{14}H_{13}N_3$	89.4	128-130	75.31 (75.13)	5.87 (5.56)	18.82 (19.01)
p-$OCH_3C_6H_4$	$C_{14}H_{13}N_3O$	90.1	83-84	70.28 (70.12)	5.48 (5.51)	17.56 (17.46)
o-$CH_3C_6H_4$	$C_{14}H_{13}N_3$	82.3	84-85	75.31 (75.22)	5.87 (5.66)	18.82 (18.99)
o-Cl-C_6H_4	$C_{13}H_{10}N_3Cl$	89.6	88-90	64.07 (64.32)	4.14 (4.01)	17.24 (17.04)
m-Cl-C_6H_4	$C_{13}H_{10}N_3Cl$	90.3	119-121	64.07 (63.95)	4.14 (3.89)	17.24 (17.51)
p-F-C_6H_4	$C_{13}H_{10}N_3F$	88.6	66-67	68.71 (68.56)	4.44 (4.43)	18.49 (19.34)
o-F-C_6H_4	$C_{13}H_{10}N_3F$	89.7	98-100	68.71 (68.45)	4.44 (4.56)	18.49 (18.32)
p-*t*-Bu·C_6H_4	$C_{17}H_{19}N_3$	84.6	116-118	76.95 (76.86)	7.22 (7.21)	15.84 (15.68)
o-CN·C_6H_4	$C_{14}H_{10}N_4$	81.6	133-135	71.78 (71.67)	4.30 (4.21)	23.92 (23.88)

REFERENCES

1. F.Q. He, B.L. Wang, Z.M. Li and H.B. Song, *Chin. J. Struct. Chem.*, **25**, 543 (2006).
2. A.R. Katritzzky and W. Jing, *Synthesis*, 597 (1994).
3. S. Ikeda, S. Murata, K. Ishii and H. Hamaguchi, *Bull. Chem. Soc. (Japan)*, **73**, 2783 (2000).
4. S.H. Mashraqui, S. Kumar and C.D. Mudaliar, *Bull. Chem. Soc. (Japan)*, **74**, 2133 (2001).
5. B. Baruah, D. Prajapati, A. Boruah and J.S. Sandhu, *Synth. Commun.*, **27**, 2563 (1997).
6. S.Y. Kim, P.S. Kwon and T.W. Kwon, *Synth. Commun.*, **27**, 533 (1997).

7. H.S. Ku, F. Siu, E. Siores, J.A.R. Ball and A.S.J. Blicblau, *Mater. Pro. Tech.*, **113**, 184 (2001).
8. H.S. Ku, E. Siores, A. Taube and J.A.R. Ball, *Comp. Ind. Eng.*, **42**, 281 (2002).
9. M.S. Venkatesh and G.S.V. Raghavan, *Bio. Eng.*, **88**, 1 (2004).
10. A.R. Katrizky, A.A.A. Abdel-Fattah, D.O. Tymoshenko and S.A. Belyakov, *Synthesis*, 1437 (1999).
11. K.Y. Ho and K. Kyongtae, *J. Heterocycl. Chem.*, **34**, 1741 (1997).
12. A.R. Katrizky, M.F. Gordeev, J.V. Greenhill and P.J. Steel, *J. Chem. Soc. Perkin Trans. I*, **9**, 1111 (1992).
13. J. Khalafy and R.H. Prager, *Aust. J. Chem.*, **51**, 925 (1998).
14. M. Paventi and A.S. Hay, *Tetrahedron Lett.*, **34**, 999 (1993).
15. K.Y. Ho and K. Kyongtae, *Tetrahedron*, **55**, 4271 (1999).

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