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The synthesis and characterization of heterocyclic compound 4-Bromo-3-butyl-5-(dibromomethylene) furan-2(5H)-one

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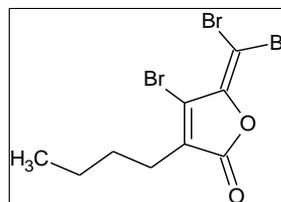
Abstract

In this paper, a heterocyclic compound 4-bromo-3-butyl-5-(dibromomethylene) furan-2(5H)-one has been prepared by using simple organic reactions like acetoacetic ester synthesis. After the synthesis, the compound was characterized by solubility, melting point, conductivity and spectral analysis. The analyses confirmed that the compound is five membered oxygen containing heterocyclic compound.

Keywords: Furan, ethylacetoacetate, spectral analysis

Introduction

IUPAC suggests the systematic names of the compound as 4-bromo-3-butyl-5-(dibromomethylene) furan-2(5H)-one. The structure may be shown as:



There are two C-C double bonds, a butyl chain and three bromo substituent, two of them bonded to an exocyclic double bond to the five member heterocycle.

The 4-bromo-3-butyl-5-(dibromomethylene)furan-2(5H)-one, my target is one of a halogenated secondary metabolite which has been isolated ^[1-3] from a red alga nearby Sydney called *Delisea pulchra* 4(Bonnemaisoniaceae), now synonymous of *fimbriata*. The interest was stimulated by the significant *in vivo* antifungal activity of this alga ^[4, 5]. After freeze-drying of freshly collected material Dworjany ^[6] obtained about 5% (dry weight) of a complex mixture of dichloromethane soluble material.

Experimental

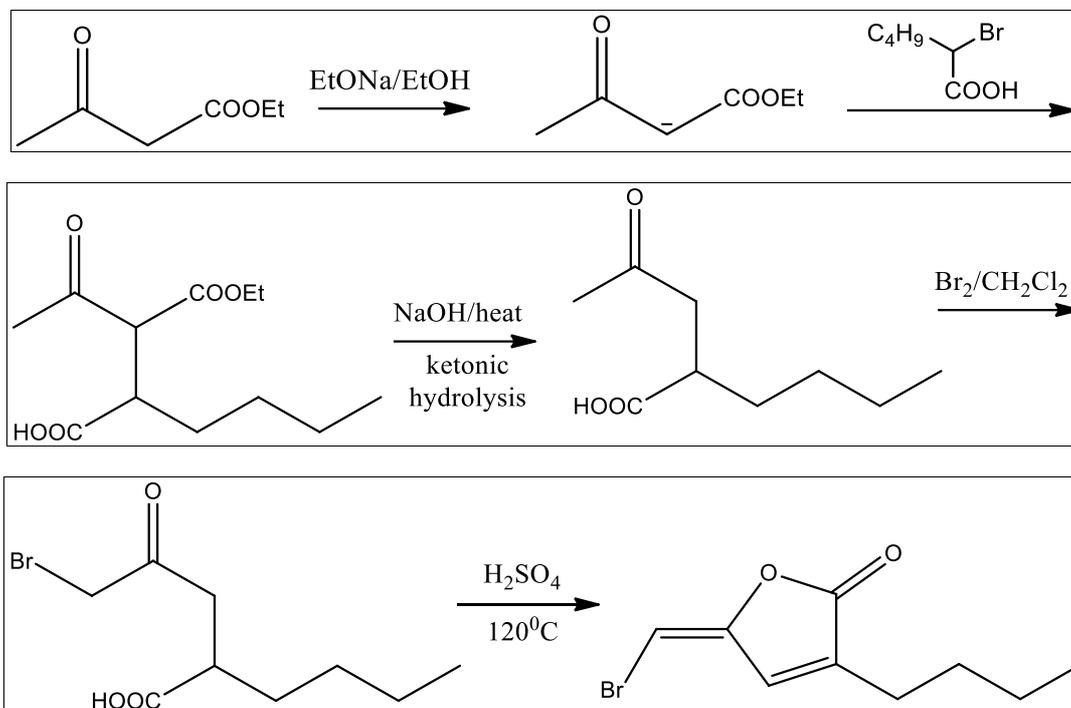
Materials

All glassware used to air sensitive experiments were oven-dried at least 1hr before use. Prior to use, THF and Et₂O were pre-dried over anhydrous CaCl₂ and then reflux over sodium benzophenone ketyl under nitrogen atmosphere. Dioxane was distilled under reduced pressure over anhydrous CaCl₂ and stored over molecular sieves. Toluene, dichloromethane, pentane and methanol were used as delivered.

Now, the synthesis of the compound was completed by using following procedure.

One equivalent of alkylated bromofuran dissolved in the dichloromethane to prevent decomposition was introduced in the reactor. Then a catalytic amount of polystyrene bounded Rose Bengal and finally two equivalent of base. The oxygen flow was started to obtain a fine stream of bubble. The reactor was placed the Dewar vase. The cooling water was put on and the lamp lighted. To follow the reaction by GC I have to turn off the lamp, cut off the oxygen flow and remove the cooling system of the reactor in order to get out a sample. The reaction scheme may be shown as:

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Thereafter, further, simple bromination was performed to obtain target molecule. The product was oil that could not be crystallized, which was characterized by ^{13}C NMR was then the only way to determinate its structure. The product is the 3-bromo-4-butyl-5-hydroxyfuran-2(5H)-one and yield after incomplete purification are in the range 50%-65%.

Results and Discussion

It was a challenge to set up an apparatus for the photooxidation [7] with the material available in the laboratory. It was disposition a photoreactor [8] and a mercury lamp without any specification about the power. I decided to mount on it a homemade tube to introduce pure O_2 to the bottom of the reactor. The reaction tube was immersed in a long Dewar vase [9] with dry ice and acetone to reach the -78°C required. Constant checking of the cooling bath was necessary to ensure the good temperature since the lamp develops a lot of heat which was aggravated because of the reflex ion of the mirror inside of the Dewar vase.

IR Spectrum

IR spectrum of the compound was recorded as KBr pellets in the region of $4000\text{-}400\text{ cm}^{-1}$ on a Perkin-Elmer Spectrophotometer. The spectrum was interpreted considering few main peaks observed.

Table 1: IR Frequencies (in cm^{-1}) of 4-bromo-3-butyl-5-(Dibromomethylidene) furan-2(5H)-one

$\nu_{\text{C=O}}$	$\nu_{\text{C-O}}$	$\nu_{\text{C=C}}$	ν_{CH_2}	ν_{CH_3}
1745	1245	1650	1460	1380

The IR spectrum of the compound showed a strong band at 1745 cm^{-1} indicating [10] the presence of carbonyl group of five membered lactone ring. The lactone ring was further confirmed [11] by the presence of two strong bands at 1250

cm^{-1} and 1050 cm^{-1} .

The IR spectrum of the compound shows a medium band at 1650 cm^{-1} indicating [12] the presence of olefinic C=C group. The presences of a medium band at 1460 cm^{-1} indicates [13] the presence of a methylene (CH_2) group in the molecule. There is another medium band at 1380 cm^{-1} indicates⁴ the presence of a methyl (CH_3) group in the molecule.

NMR Spectrum

The $^1\text{H-NMR}$ spectra were recorded on a Hitachi FT-NMR model R-600 spectrometer using CDCl_3 as the solvent. The chemical shifts are given in ppm relative to tetramethylsilane.

Table 1: NMR signal (in δ ppm) of 4-bromo-3-butyl-5-(dibromomethylidene) furan-2(5H)-one

CH_2	CH_2	CH_2	CH_3
2.8(t)	1.3(quin.)	1.1(sex)	0.8(t)

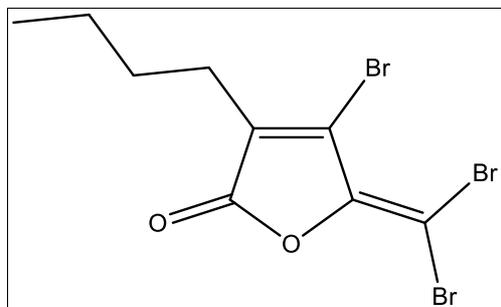
The NMR signal of the molecule shows a triplet at 0.8 ppm respectively integrating corresponding to three protons. These signals indicate [14] the presence of a methyl group adjacent to CH_2 unit.

The molecule shows a triplet at 2.8 ppm integrating corresponding to two protons. This signal indicates [15] the presence of a CH_2 group adjacent to another CH_2 unit.

The NMR spectrum of the molecule shows a coupled quintet signal at 1.3 ppm integrating corresponding to two protons. This signal indicates [16] the presence of a CH_2 group adjacent to two CH_2 units.

There is a coupled sextet signal at 1.1 ppm integrating two protons indicating [17] the presence of another CH_2 unit adjacent of one CH_3 unit and one CH_2 unit.

On the basis of above spectral analyses of the molecule confirm the structure of 4-bromo-3-butyl-5-(dibromomethylidene) furan-2(5H)-one.



References

1. O'Toole George, Kaplan Heidi B, Kolter Roberto. Annual Review of Microbiology. 2000;54:49-79.
2. Fraser GM, Hughes C. Swarming motility. Current opinion in microbiology. 1999, 630-5.
3. Gram Lone, De Nys Rocky, Maximilien Ria, Givskov Michael, Steinberg Peter, Kjelleberg Staffan. Applied and Environmental Microbiology. 1996;62(11):4284-4287.
4. Kazlauskas R, Murphy PT, Quinn RJ, Wells RJ. Tetrahedron Letters. 1977;(1):37-40.
5. Manefield Michael, De Nys Rocky, Kumar Naresh, Read Roger, Givskov Michael. Microbiology (Reading, United Kingdom). 1999;145(2):283-291.
6. Dworjanyn SA, de Nys R, Steinberg PD. Marine Ecology: Progress Series. 2006, 318.
7. Beechan Curtis M, Sims James J. Tetrahedron Letters. 1979, 1649-52.
8. Wells PR. Australian Journal of Chemistry. 1963;16:165-9.
9. Kjelleberg J, Staffan Kumar Naresh, de Nys Rocky, Read Roger W, Steinberg, Peter, Tetrahedron. 1997;53:15813-15826.
10. Allan IR, Baillie GM, Baired ND. Journal of Organic Chemistry. 1984;13(2):83-88.
11. Sousa EH, Basso LA, Sanots DS, Diogenes IC, Longhinotti E, Lopes LG, *et al.* J Biol Inorg. Chem. 2012 Feb;17(2):275-283.
12. Bhatia S, Kaushik NK, Sodhi GS. J Inorg. Biochem. 1999 Mar;29(3):181-186.
13. Banwell CN, McCash E. Fundamentals of Molecular Spectroscopy, 4th ed. 2002, 86.
14. Bruice PY. Org. Chem., 3rd ed. 2004, 710.
15. Altreuther P. Data on Chemistry and Toxicology of Baytri, Veterinary Medical, Review. 1987;2:87-89.
16. Zur Hausen H. Viruses in Human Cancers, Science. 1991;254:1167-1173.
17. Augusto O, Cavalieri EL, Rogan EG. J Biol. Chem. 1990;256:22093-22096.