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Microwave-assisted facile synthesis and crystal structure of cis-9,10,11,15-tetrahydro-9,10[3'4']-furanoanthracene-12,14-dione

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Abstract

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Keywords

Microwave, assisted, facile, synthesis, crystal, structure, cis, tetrahydro, furanoanthracene, dione, CMMB

Disciplines

Life Sciences | Physical Sciences and Mathematics | Social and Behavioral Sciences

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Short communication

Microwave-assisted facile synthesis and crystal structure of *cis*-9,10,11,15-tetrahydro-9,10[3',4']-furanoanthracene-12,14-dione

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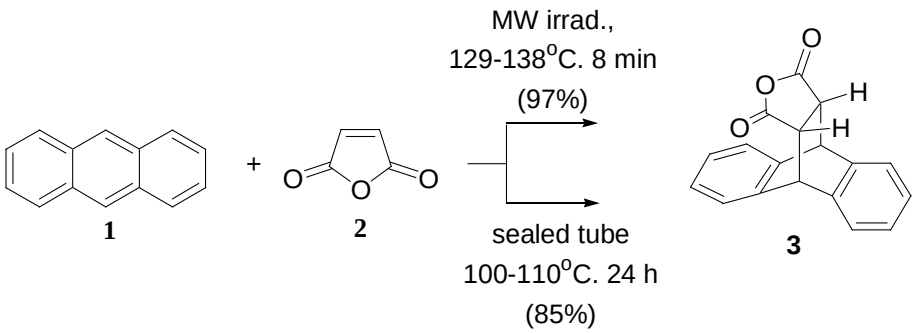
Abstract

A facile synthesis and crystal structure of *cis*-9,10,11,15-tetrahydro-9,10[3',4']-furanoanthracene-12,14-dione from the reaction of anthracene and maleic anhydride in xylene in a short time and high yield using a modified commercial domestic microwave oven is reported.

Short communication

Microwave reactors are becoming increasingly popular and effective for applications in organic synthesis.[1] The reported preparation of *cis*-9,10,11,15-tetrahydro-9,10[3',4']-furanoanthracene-12,14-dione (**3**) involves heating a mixture of anthracene (**1)** and maleic anhydride (**2)** in a high boiling point solvent at reflux temperature (Scheme 1).[2] This process requires long reaction times to achieve a satisfactory yield. We report here the synthesis and the crystal structure of *cis*-9,10,11,15-tetrahydro-9,10[3',4']-furanoanthracene-12,14-dione (**3**) by using a modified domestic microwave oven.[3]

When a mixture of anthracene (**1**) and maleic anhydride (**2**) in xylene, was irradiated (800 W) in a microwave oven for 8 min, the cycloadduct **3** was obtained in 97% yield. The temperature during microwave irradiation was recorded using an infrared thermometer (129-138 C^o). For comparison, this reaction was repeated in a sealed tube in an oil bath maintained at 100-110 C^o for 24 h. This reaction gave the desired product (**3**) in 85% yield (Scheme 1). The structure of compound **3** was confirmed by single crystal X-ray structural analysis as shown in Figure 1.



Scheme 1. Diels-Alder reactions of anthracene (**1**) and maleic anhydride (**2**) under different reaction conditions.

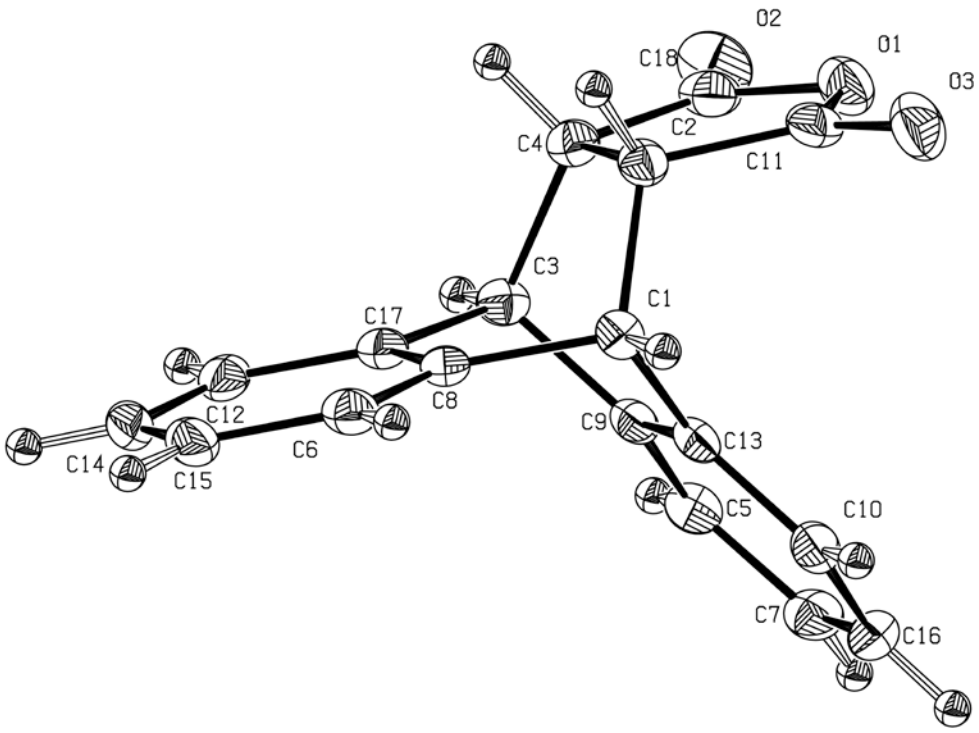


Figure 1. Molecular structure of *cis*-9,10,11,15-tetrahydro-9,10[3',4']-furanoanthracene-12,14-dione (**3**) showing 50% thermal ellipsoids.

Spectroscopic Procedures:

^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) experiments were carried out on a Bruker AM 400 spectrometer in $CDCl_3$ solution; IR spectrum was recorded on a Perkin Elmer FT-IR spectrometer; mass spectrum was recorded on a Perkin-Elmer GC/MS .

General Procedure for the Synthesis of* cis-*9,10,11,15-tetrahydro-9,10[3',4']-furanoanthracene-12,14-dione (3)

By using a modified commerical domestic microwave oven (http://www.science.mju.ac.th/chemistry/research/weerachai/reactor_eng.htm): A mixture of anthracene (1.00 g, 5.6 mmol), maleic anhydride (0.823 g, 8.4 mmol) and xylene (5 mL) contained in a 100 ml round bottom flask was placed in the modified microwave oven. A condenser was attached and the solution was subjected to irradiation of 800 Watt for 8 min. It was

then allowed to cool to room temperature. The product was purified according to the report of Bachmann *et al*.[2] After recrystallization from MeOH, colorless needles were obtained (1.512g, 97.85% yield), m.p. 262-265 ^{o}C (Lit.[2] 262-263 ^{o}C). IR (KBr) (ν_{max}, cm^{-1}): 1228 (C-O-C), 1475 and 1655 (C=C Ar.), 1783 (C=O), 3100 (=C-H Ar.); ^{1}H NMR δ: 3.51 (2H, s, 2CH), 4.80 (2H, s, 2CH), 7.16-7.39 (8H, m, Ar-H); ^{13}C NMR δ: 45.9 (CH), 48.2 (CH), 124.2-127.1 (ArCH), 138.2 (ArC), 140.1 (ArC), 170.1 (C=O); MS (EI) m/z : 276 (18), 203 (19), 178 (100), 149 (67)

Acknowledgements

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2. W. E. Bachmann and L. B. Scott, *J. Am. Chem. Soc*., **1948**, *70*, 1458.
3. Details of the microwave reactor are available on our Research Unit web page : http://www.science.mju.ac.th/chemistry/research/weerachai/reactor_eng.htm