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*Short Note*

# 9-Methyl-3,5-dioxo-4-azatricyclo[5.2.2.0$^{2,6}$]undec-8-ene-1,8-diyl Diacetate

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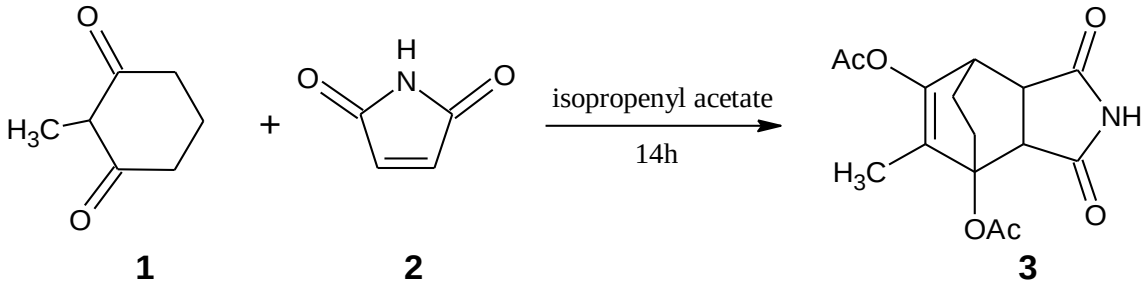
**Abstract:** 9-Methyl-3,5-dioxo-4-azatricyclo[5.2.2.0$^{2,6}$]undec-8-ene-1,8-diyl diacetate was synthesized from 2-methylcyclohexane-1,3-dione and 1*H*-pyrrole-2,5-dione. The title compound was characterized by $^{1}$H NMR, $^{13}$C NMR, elemental analysis and MS.

**Keywords:** cyclic imide; 4-azatricyclo[5.2.2.0$^{2,6}$]undecene

## 1. Introduction

Cyclic imides are extensively used as analgesic [1] and antinociceptive agents [2], or as reactants for polymer synthesis [3]. An imide nucleus can be also found in a structure of anxiolytic [4], antimicrobial [5], anticancer and anti-inflammatory substances [6,7]. They are also the objects of quantum chemical studies [8]. Several techniques to produce cyclic imides were described. Thus, unsubstituted cyclic anhydrides are successfully subjected to the reaction with ammonia, urea, formamide, lithium nitride or ammonium carbonate under mild conditions [9–11]. Substituted polycyclic imide rings are usually prepared in the Diels-Alder reaction [12,13]. Currently, catalyzed syntheses conducted under microwave irradiation have been described [14]. This work describes a conventional method for the synthesis of the substituted 4-azatricyclo-[5.2.2.0$^{2,6}$]undecene derivative.

**Scheme 1.** Synthesis of 9-methyl-3,5-dioxo-4-azatricyclo[5.2.2.$0^{2,6}$]undec-8-ene-1,8-diyl diacetate.




## 2. Experimental

### *2.1. General*

All chemicals and solvents were purchased from Sigma-Aldrich (Vienna, Austria). Melting points were determined on an Electrothermal Digital Melting Point Apparatus (Essex, UK) and are uncorrected. The NMR spectra were recorded on a Bruker (Rheinstetten, Germany) spectrometer. The chemical shift values are expressed in ppm relative to TMS as an internal standard. Elemental analysis was recorded on a CHN model 2400 Perkin-Elmer (Hitachi, Tokyo, Japan). Mass spectra were performed on MARINER PE Biosystems instrument (Foster City, USA) with TOF detector. Methanol was used as a solvent. The spectra were performed in the positive ion mode with a declustering potential 140–300 V. TLC was carried out using silica gel 60 $F_{254}$, layer thickness 0.25 mm (E. Merck, Darmstadt, Germany) and the results were visualized using UV lamp at 254 nm.

### *2.2. Synthesis of 9-methyl-3,5-dioxo-4-azatricyclo[5.2.2.$0^{2,6}$]undec-8-ene-1,8-diyl diacetate* ***(3)***

A mixture of 2-methylcyclohexane-1,3-dione **(1)** (5 g, 0.040 mol), 1*H*-pyrrole-2,5-dione **(2)** (4.62 g, 0.048 mol), and 4-methylbenzenesulfonic acid (0.05 g, 0.0003 mol) was dissolved in 15 mL of isopropenyl acetate and refluxed for 14 h. The solvent was evaporated. The crude product was crystallized from a hexane:ethyl acetate mixture (1:1 vol.) to afford a colourless solid.

Yield: 58%.

M.P. 161–162 °C.

$^{1}$H NMR (400 MHz, DMSO-$d_6$): δ = 11.18 (s, 1H, NH), 4.00 (d, $J$ = 8.4 Hz, 1H, CH-C=O), 3.07 (dd, $J_1$ = 2.8 Hz, $J_2$ = 8.4 Hz, 1H, CH-C=O), 2.81 (d, $J$ = 2.4 Hz, 1H, CH-C=C), 2.45 (m, 1H, $\underline{CH_2}CH_2$), 2.14 (s, 3H, $CH_3$-C=O), 2.09 (s, 3H, $CH_3$-C=O), 1.73 (m, 2H, $CH_2\underline{CH_2}$), 1.55 (s, 3H, $CH_3$), 1.47 (dd, $J_1$ = 4.4 Hz, $J_2$ = 12.0 Hz, 1H, $\underline{CH_2}CH_2$).

$^{13}$C NMR (100 MHz, DMSO-$d_6$): δ = 178.60 (C=O), 176.94 (C=O), 169.67 (C=O), 168.58 (C=O), 142.46 (C=C), 123.18 (C=C), 81.07 (C), 45.35 (CH), 44.41 (CH), 35.36 (CH), 27.64 ($CH_2$), 23.18 ($CH_2$), 21.87 ($CH_3$), 21.16 ($CH_3$), 9.45 ($CH_3$).

HR ESI-MS: *m/z* [%]: 330.0954 $[M + Na]^+$ 100.

Anal. Calcd. (found) for $C_{15}H_{17}NO_6$ (307.30): C, 58.63 (58.68); H, 5.58 (5.62); N, 4.56 (4.58).

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