

Short Note

(E)-1-(5-Methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-3-(naphthalen-2-yl)prop-2-en-1-one

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Abstract: A reaction of equimolar equivalents of 2-naphthaldehyde (**1**) and 1-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)ethan-1-one (**2**) in ethanolic sodium hydroxide at 20 °C for 4 h gave (*E*)-1-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-3-(naphthalen-2-yl)prop-2-en-1-one (**3**) in 92% yield. Nuclear magnetic resonance spectroscopy and single-crystal X-ray diffraction were used to establish the structure of **3**.

Keywords: synthesis; 1,2,3-triazole; X-ray crystal structure; 2-naphthaldehyde; chalcone; structure characterization



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1. Introduction

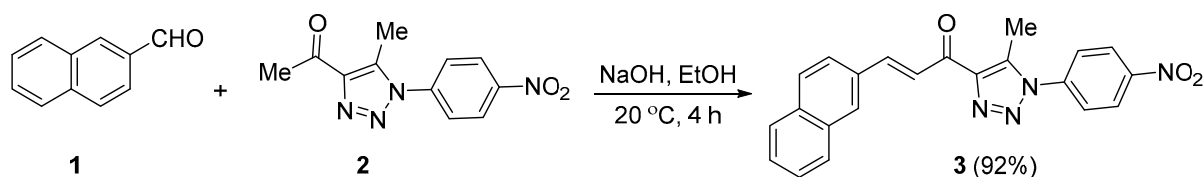
Chalcones are important intermediates in synthetic and medicinal chemistry [1–3]. Compounds containing chalcone skeletons that display a variety of pharmacological properties have been developed [4–6]. Possible applications of the compounds include the treatment of viral disorders, cardiovascular diseases, parasitic infections, and stomach cancer [7–12]. In addition, they have been used as food additives and in the production of cosmetic formulations [13]. Chalcones are also common natural pigments as well as acting as intermediaries in the biosynthesis of flavonoids [14]. Chalcones are mainly synthesized by Claisen–Schmidt condensation, which involves a cross Aldol reaction of aldehydes and ketones in the presence of an acidic or basic catalyst followed by the elimination of water [15–17].

Heterocycles containing 1,2,3-triazole residues present a variety of biological activities [18–22]. 1,2,3-Triazoles can be synthesized through click chemistry, which involves a simple procedure and provides high yields of many substituted derivatives [23]. 1,3-Cycloaddition of substituted nitriles containing active methylene groups and aromatic azides is an efficient procedure for production of 1,2,3-triazoles [24,25]. Additionally, 1,2,3-triazoles can be synthesized via reactions of diazo compounds with several active species, including amines, amides, azides, and alkynes [26–30]. Reported here is research into the synthesis and structural characterization of a heterocycle containing the 1,2,3-triazole unit. Recently, we established the structures of related heterocycles [31–34].

2. Results and Discussion

2.1. Synthesis of **3**

Claisen–Schmidt condensation of 2-naphthaldehyde (**1**) and 1-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)ethan-1-one (**2**) in ethanolic sodium hydroxide at 20 °C for 4 h gave (*E*)-1-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-3-(naphthalen-2-yl)prop-2-en-1-one (**3**) in 92% yield (Scheme 1). Nuclear magnetic resonance (NMR) spectroscopy (Section 2.2.) and single-crystal X-ray diffraction (Section 2.3) were used to confirm the identity of **3**.



Scheme 1. Synthesis of **3**.

2.2. NMR Spectroscopy

The ^1H NMR spectrum of **3** shows the presence of two characteristic doublets ($J = 16.2$ Hz) at 8.06 and 8.20 ppm due to the $\text{CH}=\text{CH}$ protons. In addition, it shows a singlet at high field (2.66 ppm), corresponding to the methyl protons, along with 13 aromatic protons. The ^{13}C NMR spectrum of **3** shows the presence of a signal at high field (10.4 ppm) due to the carbon of the methyl group. In addition, the signal due to the carbon atom of the carbonyl group is observed at very low field (184.3 ppm). The IR spectrum of **3** shows a band at 1662 cm^{-1} indicating the presence of a carbonyl group. The Supplementary Material contains the FTIR and NMR spectra of **3**.

2.3. X-ray Crystal Structure

The molecule from the crystal structure is shown in Figure 1. The molecule comprises naphthalene (**A**: C1–C10), methyltriazole (**C**: C14–C16, N1–N3), and nitrobenzene (**D**: C17–C22, N4, O2, O3) ring systems. Groups **A** and **C** are linked by a propanal moiety (**B**: C11–C13, O1).

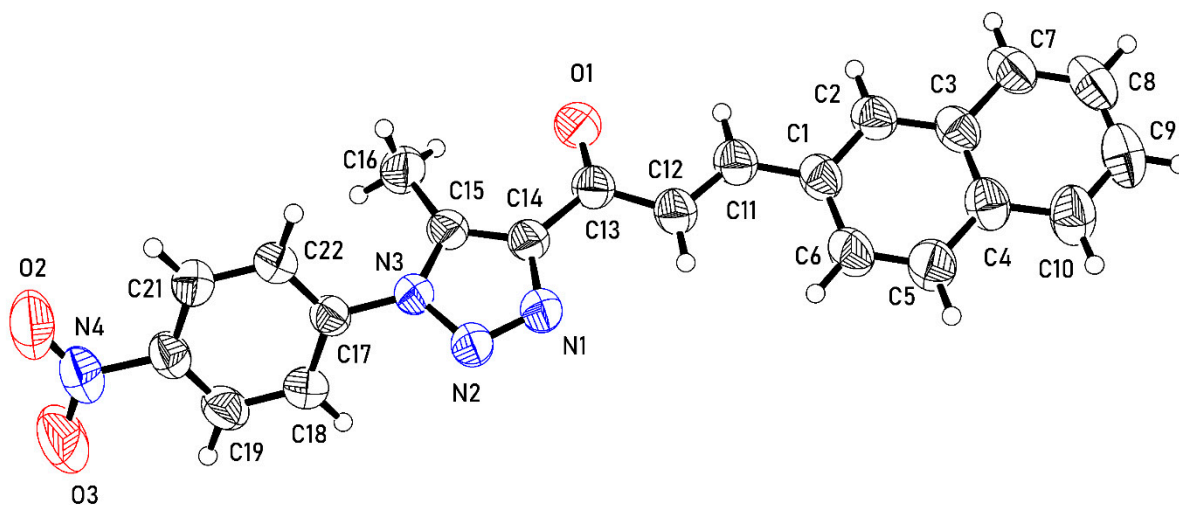


Figure 1. An ortep representation of the molecule of **3** showing 50% probability atomic displacement ellipsoids.

In the crystal structure, groups **A–C** of the molecule are almost co-planar. This is indicated by the twist angles of the planes through groups **A–C**, namely $6.11(17)^{\circ}$ and $8.42(21)^{\circ}$ for **A/B** and **B/C**, respectively. The orientation of the nitrobenzene group deviates significantly from the **A–C** plane, as shown by a **C/D** twist angle of $57.1(8)^{\circ}$.

In the crystal, the nitrobenzene groups (**D**) of all the molecules are parallel to the (1 3 $\bar{2}$) crystallographic plane, as is clearly visible in Figure 2a. Additionally, the planes of the **A–C** groups are aligned parallel to the (1 0 $\bar{2}$) crystallographic plane of the crystal (Figure 2b).

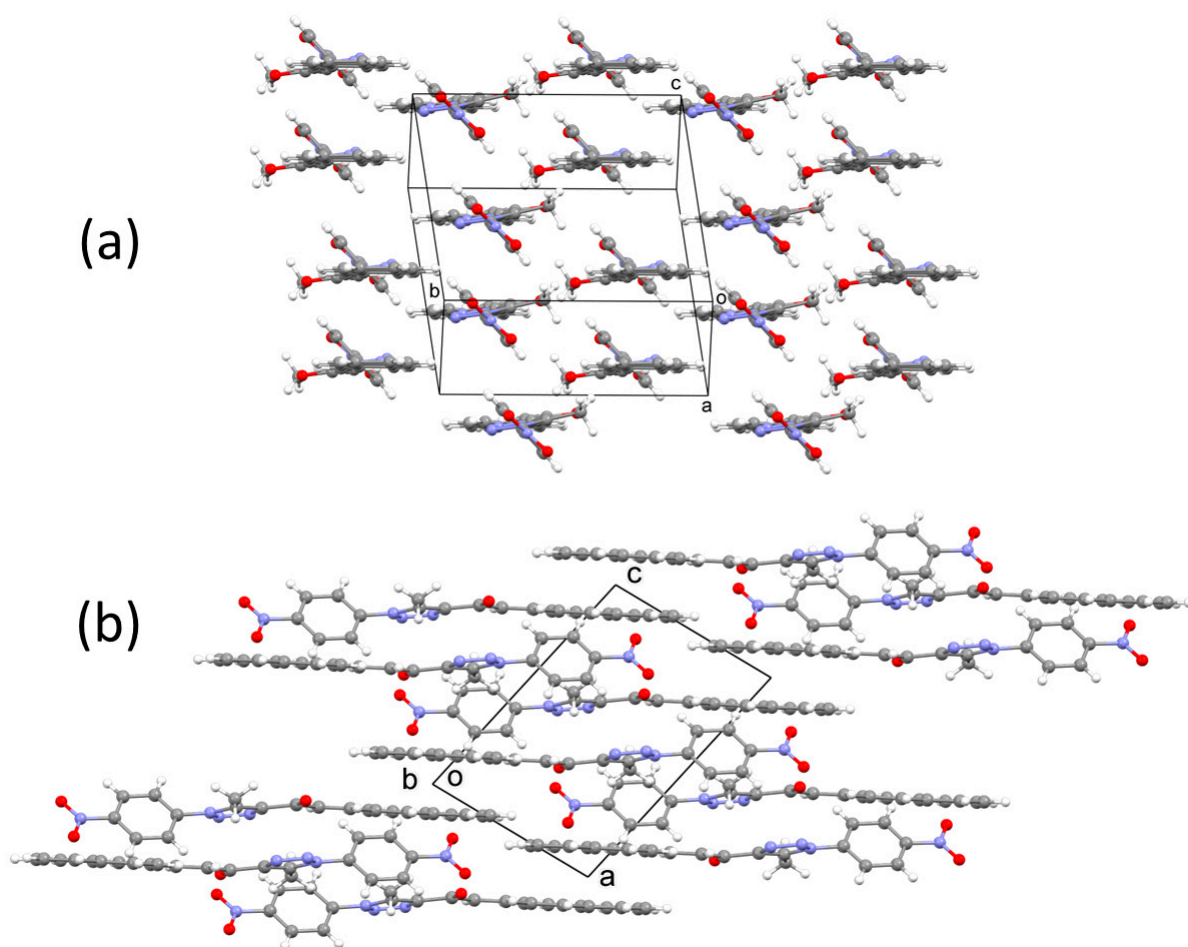


Figure 2. Views of the crystal structure (a) down the length of the molecule and (b) along the *b* axis.

3. Materials and Methods

3.1. General

A Bruker Tensor 27 FTIR spectrometer (Zürich, Switzerland) was used to record the IR spectrum of **3**. A JEOL NMR spectrometer (Tokyo, Japan) was used to record the ^1H (500 MHz) and ^{13}C NMR (125 MHz) spectra. Chemical shifts (δ) are reported in ppm and the coupling constants (*J*) in Hz. Compound **2** was produced based on a literature procedure [35].

3.2. Synthesis of **3**

Compound **2** (0.49 g, 2 mmol) was added to a solution of NaOH (0.40 g, 10 mmol) in a mixture of H_2O (10 mL) and EtOH (30 mL). The mixture was stirred at 20 °C for 30 min followed by the addition of **1** (0.31 g, 2 mmol). The mixture was stirred for an additional 3.5 h and poured into iced water (60 mL) with stirring for 30 min. The solid obtained was filtered, washed with H_2O (2×20 mL), and recrystallized from DMF to give colorless crystals of **3**. Yield 92%, mp 244–246 °C. IR (KBr): 3058 (CH), 1662 (C=O), 1601 (C=C), 1496 (C=N) cm^{-1} . ^1H NMR (CDCl_3): 2.66 (s, 3H, Me), 7.25–7.29 (m, 2H, H6/H7 of naphthyl), 7.45–7.48 (m, 2H, H3/H4 of naphthyl), 7.50–7.52 (m, 2H, H5/H8 of naphthyl), 7.82–7.87 (m, 4H, H2/H4 and H-/H5 of Ar), 8.06 (d, $J = 16.2$ Hz, 1H, CH), 8.09 (s, 1H, H1 of naphthyl), 8.20 (d, $J = 16.2$ Hz, 1H, CH). ^{13}C NMR (CDCl_3): 10.4 (Me), 116.8 (C3 of naphthyl), 117.0 (C3/C5 of Ar), 123.2 (C6 of naphthyl), 124.3 (C7 of naphthyl), 126.8 (C5 of naphthyl), 127.4 (C1 of naphthyl), 127.5 (C8 of naphthyl), 127.9 (C4 of naphthyl), 128.8 (C2/C6 of Ar), 130.0 (C4a of naphthyl), 131.6 (C8a of naphthyl), 132.6 (C2 of naphthyl), 133.5 (C4 of triazolyl), 134.6 (C5 of triazolyl), 143.9 (CH), 144.2 (C1 of Ar), 162.3 (CH), 164.3 (C4 of Ar), 184.3 (C=O).

3.3. Data Collection and Structure Refinement Details

An Agilent SuperNova Dual Atlas diffractometer using mirror monochromated MoK α radiation was used to collect single-crystal diffraction data. The structure was solved by direct methods using SHELXS [36] and refined by full-matrix least-squares methods on F² with SHELXL-2014 [37]. C₂₂H₁₆N₄O₃, FW = 384.39, T = 293(2) K, λ = 0.71073 Å, triclinic, P $\bar{1}$, a = 7.7175(10) Å, b = 11.1752(18) Å, c = 11.6839(15) Å, α = 73.654(13)°, β = 75.259(11)°, γ = 76.247(13)°, V = 920.0(2) Å³, Z = 2, calculated density = 1.388 Mg/m³, absorption coefficient = 0.095 mm^{−1}, F(000) = 400, crystal size = 0.410 × 0.150 × 0.080 mm³, reflections collected = 7864, independent reflections = 4310, R(int) = 0.0155, parameters = 263, goodness of fit on F² = 1.056, R1 = 0.0635, wR2 = 0.1621 for (I > 2 σ (I)), R1 = 0.0993, wR2 = 0.1858 for all data, largest difference peak and hole = 0.212 and −0.243 e.Å^{−3}. The X-ray crystallographic data for compound **3** have been deposited in the Cambridge Crystallographic Data Center under CCDC reference number 2205568.

4. Conclusions

(E)-1-(5-Methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-3-(naphthalen-2-yl)prop-2-en-1-one was synthesized in excellent yield in a single step, and its structure was determined using single-crystal X-ray diffraction and nuclear magnetic resonance.

Supplementary Materials: The following are available online. IR, ¹H, and ¹³C NMR spectra, CIF and checkcif reports for the title compound.

Author Contributions: Conceptualization, B.M.K. and G.A.E.-H.; methodology: B.M.K., B.F.A.-W., H.A.M. and G.A.E.-H.; software, B.M.K.; validation, B.M.K., B.F.A.-W., H.A.M. and G.A.E.-H.; formal analysis: B.M.K., B.F.A.-W., H.A.M. and G.A.E.-H.; investigation: B.M.K., B.F.A.-W., H.A.M. and G.A.E.-H.; resources: B.M.K. and G.A.E.-H.; data curation: B.M.K., B.F.A.-W., H.A.M. and G.A.E.-H.; writing—original draft preparation, B.M.K., B.F.A.-W., H.A.M. and G.A.E.-H.; writing—review and editing, B.M.K., B.F.A.-W., H.A.M. and G.A.E.-H.; visualization, B.M.K.; supervision: B.F.A.-W. and H.A.M.; project administration, B.F.A.-W. and H.A.M.; funding acquisition: B.M.K. and G.A.E.-H. All authors have read and agreed to the published version of the manuscript.

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