

# $(\mu 2$ - $\eta 4$ -N-(2-Butynyl)phthalimide)(hexacarbonyl)dicobalt

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Short Note

## $(\mu_2-\eta^4-N-(2-Butynyl)phthalimide)(hexacarbonyl)dicobalt$

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**Abstract:** The reaction of  $[Co_2(CO)_8]$  with an equimolar amount of the internal alkyne N-(2-butynyl)phthalimide (1-Phthalimido-2-butyne) **1** in heptane solution yields the title compound  $[Co_2(CO)_6(\mu-phthalimidoCH_2C\equiv CMe)]$  **2**. Compound **2** has been characterized using IR,  $^1H$  and  $^{13}C$  NMR spectroscopy; the tetrahedrane-type cluster framework has been ascertained using a single-crystal X-ray diffraction study performed at 100 K.

Keywords: cobalt; carbonyl; triphenylphosphite; crystal structure; molecular cluster

#### 1. Introduction

Since the first reports dating from the 1950s on the reaction of dicobalt octacarbonyl with alkynes yielding dicobaltatetrahedranes  $[\text{Co}_2(\text{CO})_6(\mu\text{-RC}\equiv\text{CR}')]$  [1,2], there is nowadays a plethora of articles dealing with this organometallic reaction [3]. The interest in this research is not only driven by merely synthetic aspects, i.e., spectroscopic and structural characterization of these cage-like species, but also by the use of these compounds as precursors for further modifications in material sciences [4] and organic syntheses [5,6]. An important example for the latter domain is the Pauson–Khand reaction, in which  $[\text{Co}_2(\text{CO})_8]$  reacts with an alkyne, an alkene, and carbon monoxide to form an  $\alpha,\beta$ -cyclolopentenone [7–11]. In addition to the applications using dicobaltatetrahedranes as synthetic tools, this class of dinuclear organometallic compounds is emerging in bio-organometallic chemistry [12–21]. Some examples of dicobaltatetrahedranes, ligated using terminal functionalized alkynes and displaying biological activity, are depicted in Scheme 1.

Among the organic heterocyclic compounds showing a promising biological and medicinal activity, there are also some representatives based on the phthalimido scaffold [22–24]. In 2005, Gust et al. also reported the reaction of propargyl phtalimide with  $[Co_2(CO)_8]$  and determined the cytotoxicity and DNA binding efficiency of the resulting complex N-(2-propynyl)phthalimide]hexacarbonyldicobalt (see compound V of Scheme 1) [21]. In the context of our research on Co–Co carbonyl complexes towards various alkynes producing dicobaltatetrahedranes [25,26], we attempted to synthetize a Co–Co complex ligated using an internal alkyne, namely 1-Phthalimido-2-butyne 1, for upcoming biological studies. Apart from the purely synthetic aspect, one of the objectives was to compare the impact of the replacement of a terminal alkyne by an internal one (Phtal-C $\equiv$ CH vs. Phtal-C $\equiv$ CMe).



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**Scheme 1.** Examples of some dicobaltatetrahedranes displaying biological activity (**I**, **III–V**: antiproliferative activity against cancer cells; **II**: hormonally active compound).

#### 2. Results and Discussion

The title compound **2** was obtained via treatment of  $[Co_2(CO)_8]$  with an equimolar amount of N-(2-butynyl)phthalimide **1** in heptane solution at 60 °C as shown in Scheme **2**. Upon cooling, small orange-red needle-shaped crystals of air-stable **2** were isolated, and elemental analysis confirmed their composition as  $[Co_2(CO)_6(\mu\text{-phthalimidoCH}_2C\equiv CMe)]$ .

**Scheme 2.** Synthesis of the title compound **2**.

The IR spectrum of this product in cyclohexane, shown in Figure 1, reveals the signals of terminal carbonyl with intense  $\nu(CO)$  vibrations at 2093, 2055, and 2028 cm<sup>-1</sup>. In addition, two further absorptions at 1775 and 1727 cm<sup>-1</sup> are attributed to the carbonyls' stretching modes (symmetric and antisymmetric) of the imide function [27]. These values fit well with those measured in cyclohexane for  $[Co_2(CO)_6(\mu\text{-phthalimidoCH}_2C\equiv CH)]$  at 2094, 2057, and 2030 as well as 1775 and 1727 cm<sup>-1</sup> (Figure S1). The latter complex has already been described and characterized using IR spectroscopy in solid state as KBr pellets (2095, 2054, 2036, 2016 (Co-CO); 1712 (C=O) cm<sup>-1</sup>) [21]. The infrared spectrum of 2 in the solid state was also recorded in attenuated total reflectance (ATR) mode and is presented in the Supplementary Materials (Figure S2). Despite being a monocrystalline solid, the carbonyl absorptions were not fully resolved (2093, 2052, 2030, and 2008 as well as 1766 and 1708 cm<sup>-1</sup>).

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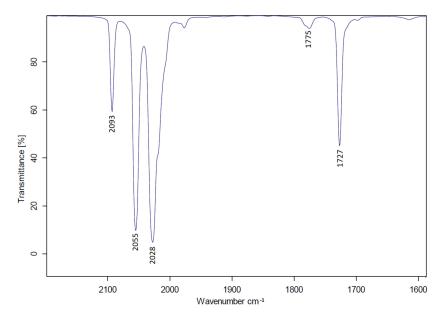
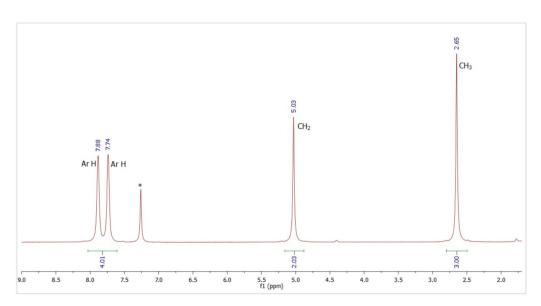


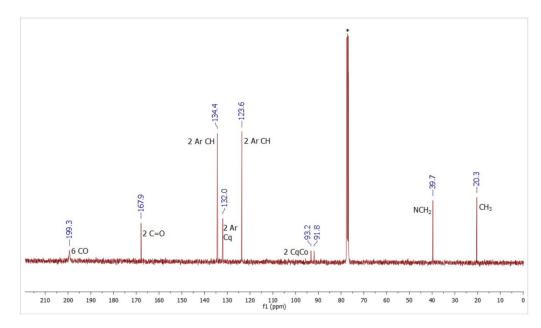
Figure 1. IR spectrum of compound 2 recorded in cyclohexane.

The  $^1$ H-NMR recorded in CDCl $_3$  reveals a singlet at  $\delta$  2.65 due to the acetylenic methyl group; a further singlet at  $\delta$  5.03 is attributed to the enantiotopic methylene hydrogens. The remaining resonances at  $\delta$ 7.74 and 7.88 are assigned to the aromatic cycle (Figure 2). In the proton-decoupled  $^{13}$ C-NMR spectrum depicted in Figure 3, only a broadened resonance centered at  $\delta$  199.3 could be observed for the six Co-bound carbonyl groups, suggesting a fluxional behavior of the COs. In contrast, the resonance of the two chemically equivalent imido C=O carbonyl groups at  $\delta$  167.9, the two acetylenic carbons at  $\delta$  93. 2 and 91.8, as well as those of the CH $_2$  and CH $_3$  carbons gave rise to well-resolved signals.



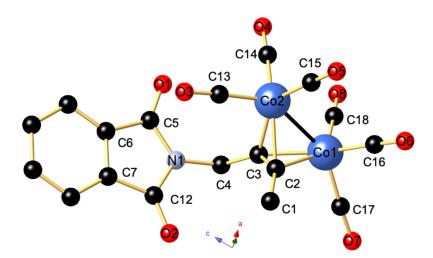
**Figure 2.**  $^{1}$ H-NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound **2** at 25  $^{\circ}$ C. The asterisk \* denotes the chloroform resonance.

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**Figure 3.**  $^{13}$ C{ $^{1}$ H}-NMR spectrum (100.62 MHz, CDCl<sub>3</sub>) of compound **2** at 25 °C. The asterisk \* denotes the CDCl<sub>3</sub> resonance.

In addition to the spectroscopic characterization of **2** in solution, the complex was also unambiguously analyzed using X-ray diffraction performed at 100 K. Complex **2** crystallizes in the triclinic space group P-1; the asymmetric unit contains two independent molecules with slightly different bond lengths and angles. As shown in Figure 4, in this organometallic species the former alkyne is almost orthogonally  $\mu_2$ - $\eta^4$ -coordinated with respect to the two crystallographically non-equivalent Co1 and Co2 centers, which are linked through a metal–metal bond of 2.4688(14) Å.

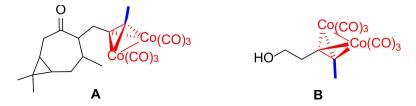


**Figure 4.** Molecular structure of one of two independent molecules of **2**. All H atoms are omitted for clarity. Selected bond lengths (Å) and angles (deg): Co1–Co2 2.4688(14), Co1–C2 1.980(6), Co1–C3 1.952(6), Co2–C2 1.984(6), Co2–C3 1.954(7), Co1–C16 1.812(7), Co1–C17 1.784(8), Co1–C18 1.831(7), Co2–C13 1.794(7), Co2–C14 1.812(7), Co2–C15 1.805(7), C2–C3 1.323(9); Co1–Co2–C3 50.75(15), Co2–Co1–C2 50.83(19), Co1–C2–Co2 77.0(2), Co1–C3–Co2 78.4(2).

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The resulting tetrahedral skeleton, reminiscent of that of organic tetrahedranes (https://en.wikipedia.org/wiki/Tetrahedrane, accessed on 26 December 2022), allows for considering this 40-electron species an organometallic cluster of dimetallatetrahedranes, in which two  $Co(CO)_3$  units of the parent cluster  $[\{Co_4(\mu_2-CO)_3(CO)_9]\}$  are replaced by isolobal C–R fragments, forming the edges of the cluster core. The former alkyne unit lost its linearity; the torsion angle C1–C2–C3–C4 changed to  $-3.1(18)^\circ$ . The coordination sphere around each Co atom is completed by three terminal carbonyls. The mean Co–C bond length of the four pseudo-equatorial carbonyls is somewhat longer than that of the two pseudo-axial C $\equiv$ O ligands (1.815(7) vs. 1.789(7) Å). The C2–C3 bond distance of 2 is much shorter than that of a C–C single bond, but considerably elongated with respect to the C $\equiv$ C bond reported for [phthalimidoCH $_2$ C $\equiv$ CCH $_2$ phthalimido] (CSD refcode ECUFOB) (1.323(9) vs. 1.191(4) Å) [28].

Although there are numerous examples of crystallographically characterized hexacarbonyldicobalt compounds which are  $\mu_2$ - $\eta^2$ , $\eta^2$  capped by an internal alkyne, a survey of the CSD data base revealed only two other examples containing a -CH<sub>2</sub>-C $\equiv$ C-Me motif as is present in 2, namely (*trans*-5,8,8-trimethyl-*trans*-1,4,5,7-tetrahydro-*cis*-4-(2-butynyl)bicyclo(5.1.0)octan-3-one)(hexacarbonyl)dicobalt (**A**) and (pent-3-yn-1-ol)(hexacarbonyl)dicobalt (**B**) (Figure 5) [29,30]. The most relevant averaged bond lengths (comprising molecules 1 and 2) of **2** are listed in Table 1 and compared with those of compounds **A** and **B** (averaged bond lengths).



**Figure 5.** Presentation of the two structurally characterized dicobaltatetrahedranes (**A**) and (**B**) incorporating a -CH<sub>2</sub>-C $\equiv$ C-Me motif.

**Table 1.** Comparison of relevant bond lengths (Å) and angles (°) in **2** and the crystallographically characterized  $[Co_2(CO)_6(\mu\text{-RCH}_2C\equiv CCH_3)]$  tetrahedranes **A** and **B**.

	2	A	В
Co-Co	2.4679(14)	2.4688(9)	2.4690(9)
C-C <sub>alkyne</sub>	1.330(9)	1.325(6)	1.326(6)
$C_{alkyne}-C_{Me}$	1.481(9)	1.486(6)	1.492(6)
$C_{alkyne}$ – $C_{H2}$	1.499(9)	1.500(7)	1.493(6)
Co-C <sub>alkyne</sub>	1.972(6)	1.974(5)	1.972(4)
Co-C <sub>pseudo-equatorial</sub>	1.815(7)	1.823(5)	1.820(5)
Co-C <sub>pseudo-axial</sub>	1.797(7)	1.785(6)	1.787(5)
CSD reference	This work	DOTKEC [29]	HUKCIA [30]

In the molecular cell of **2** with Z = 4, there are no intermolecular contacts deserving any discussion (Figure 6).

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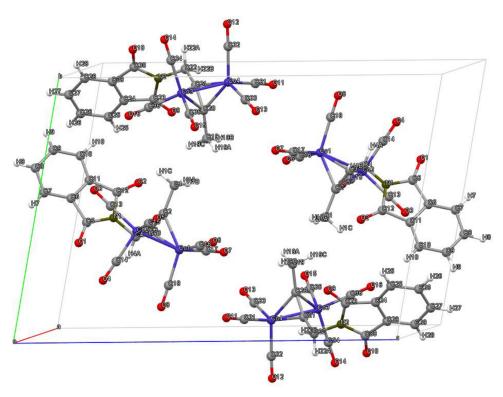


Figure 6. View of the molecular cell of 2 showing the two pairwise arranged independent molecules.

#### 3. Materials and Methods

N-(2-butynyl)phthalimide 1 was commercially purchased from Aldrich.  $^{1}$ H- and  $^{13}$ C-NMR spectra were recorded on a Brucker AC 400 (Bruker, Wissembourg, France) at 400 and 100.62 MHz, respectively, using CDCl<sub>3</sub> as solvent. Infrared spectra were recorded on a Vertex 70 spectrometer (Bruker, Wissembourg, France) in ATR mode or in solution.

 $(\mu_2-\eta^4-N-(2-butynyl)phthalimide)$ (hexacarbonyl)dicobalt: Alkyne 1 (199.2 mg, 1 mmol) was added to a stirred solution of Co<sub>2</sub>(CO)<sub>8</sub> (342.0 mg, 1 mmol) in heptane (10 mL). An immediate gas evolution was observed. The reaction mixture was then heated to 60 °C for 5 h. The solution was cooled to room temperature prior to lowering its temperature to 4 °C. Product 2 crystallized as orange-red needles which were collected via filtration. Yield: 78%. Anal. Calc. for C<sub>18</sub>H<sub>9</sub>Co<sub>2</sub>NO<sub>8</sub> (M.W = 485.14 g.mol<sup>-1</sup>): C, 44.56; H, 1.87; N, 2.89%. Detected: C, 44.64; H, 1.92; N, 2.98 %. <sup>1</sup>H-NMR (CDCl<sub>3</sub>) at 298 K: δ 2.65 (s, CH<sub>3</sub>), 5.03 (s, NCH<sub>2</sub>), 7.74 (br, CH Ar), 7.88 (br, CH Ar) ppm. <sup>13</sup>C{<sup>1</sup>H}-NMR (CDCl<sub>3</sub>) at 298 K: δ 20.3 (CH<sub>3</sub>), 39.7 (NCH<sub>2</sub>), 91.8 (Cq), 93.2 (Cq), 123.6 (CH), 132.0 (Cq), 134.4 (CH), 167.9 (C=O), 199.3 (br, CO) ppm.

Since the grown single-crystals of 2 used for the determination of the crystal structure were quite small,  $CuK_{\alpha}$  radiation was employed instead of  $MoK_{\alpha}$  radiation. Moreover, the crystals were twinned. The B-alert found in the checkcif is due to the high electronic residual around the Co–Co bonds, which could not be further refined due to the low resolution at  $138.0^{\circ}$ .

Crystal data for  $C_{18}H_9Co_2NO_8$ ,  $M=485.12~g.mol^{-1}$ , orange-red needles, crystal size  $0.377\times0.194\times0.15~mm^3$ , triclinic, space group P-1: a=7.4317(4)Å, b=13.4351(7) Å, c=19.2794(11) Å,  $\alpha=78.462(3^\circ,\ \beta=84.011(3)^\circ,\ \gamma=89.156(3)^\circ,\ V=1875.73(18)$  Å<sup>3</sup>, Z=4,  $D_{calc}=1.718~g/cm^3$ , T=100~K,  $h=-8\le h\le 8$ ,  $k=-15\le k\le 16$ ,  $I=0\le 1\le 16$ , GOF=1.077,  $R_1=0.0855$ , wR2=0.2428 for 6962 reflections with  $I>=2\sigma$  (I) and 11958 independent reflections. Largest diff. peak/hole/e Å<sup>-3</sup> 1.35/-1.02. The structure was solved using direct methods and refined using full-matrix least-squares against  $F^2$  (SHELXL, 2015 [31–33]).

Data were collected using graphite-monochromated  $CuK_{\alpha}$  radiation l = 1.54178 Å and were deposited at the Cambridge Crystallographic Data Centre as CCDC 2219351.

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(Supplementary Materials). The data can be obtained free of charge from the Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk/getstructures.

#### 4. Conclusions

We have demonstrated that addition of the internal alkyne N-(2-butynyl)phthalimide 1 to  $[Co_2(CO)_8]$  straightforwardly yields the stable complex  $[Co_2(CO)_6(\mu\text{-phthalimidoCH}_2C\equiv CMe)]$  2 featuring a dimetallatetrahedrane framework. The investigation of the biological activity of 2 and other related compounds will be the topic of a forthcoming study.

**Supplementary Materials:** The following are available online, CIF file, Check-CIF report, and ATR-IR spectrum.

**Author Contributions:** I.J. and T.C. prepared the compound; C.S. and J.-L.K. collected the X-ray data and solved the structure; I.J., M.K. and M.O. designed the study; I.J. and M.K. analyzed the data and wrote the paper. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The X-ray data are at CCDC as stated in the paper.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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