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Short Note

$(\mu_2-\eta^4-N-(2\text{-Butynyl})\text{phthalimide})(\text{hexacarbonyl})\text{dicobalt}$

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Abstract: The reaction of $[\text{Co}_2(\text{CO})_8]$ with an equimolar amount of the internal alkyne *N*-(2-butynyl)phthalimide (1-Phthalimido-2-butyne) **1** in heptane solution yields the title compound $[\text{Co}_2(\text{CO})_6(\mu\text{-phthalimidoCH}_2\text{C}\equiv\text{CMe})]$ **2**. Compound **2** has been characterized using IR, ¹H and ¹³C 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 dicobalttetrahedranes $[\text{Co}_2(\text{CO})_6(\mu\text{-RC}\equiv\text{CR}')] [1,2]$, 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 α,β -cyclopentenone [7–11]. In addition to the applications using dicobalttetrahedranes as synthetic tools, this class of dinuclear organometallic compounds is emerging in bio-organometallic chemistry [12–21]. Some examples of dicobalttetrahedranes, 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 phthalimide with $[\text{Co}_2(\text{CO})_8]$ 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 dicobalttetrahedranes [25,26], we attempted to synthesize 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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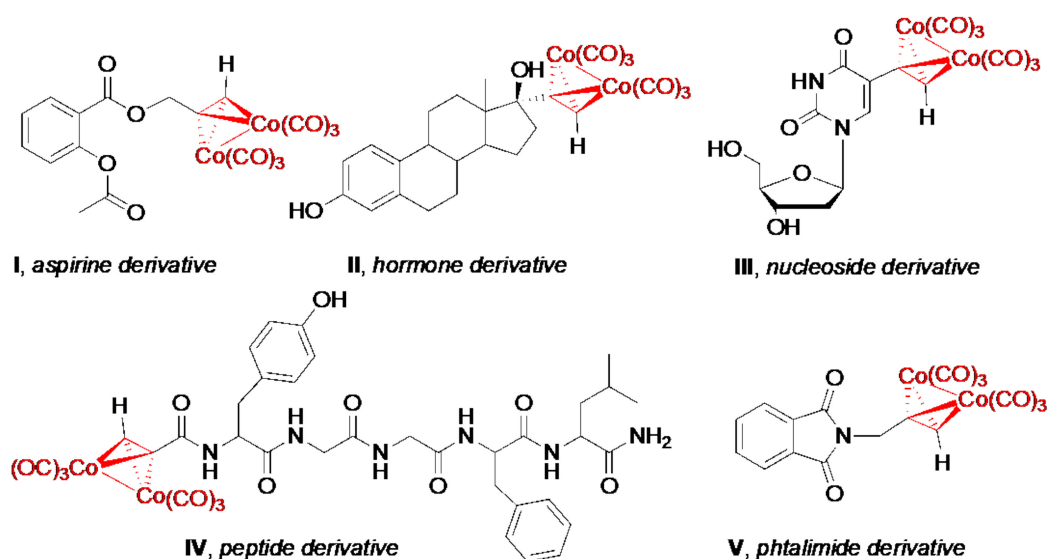
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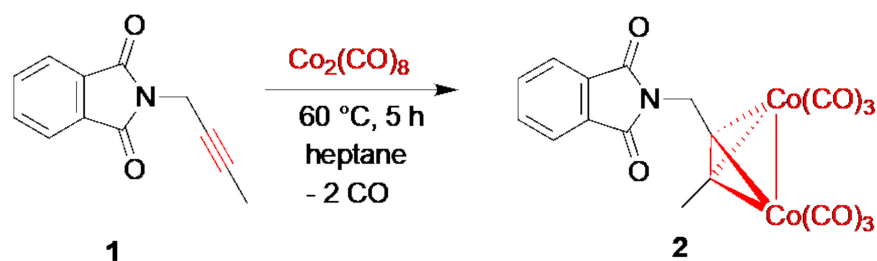
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Scheme 1. Examples of some dicobaltatetrahedranes displaying biological activity (I, III–V: antiproliferative activity against cancer cells; II: hormonally active compound).

2. Results and Discussion

The title compound **2** was obtained via treatment of $[\text{Co}_2(\text{CO})_8]$ with an equimolar amount of *N*-(2-butyne)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 $[\text{Co}_2(\text{CO})_6(\mu\text{-phthalimidoCH}_2\text{C}\equiv\text{CMe})]$.



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 $\nu(\text{CO})$ vibrations at 2093, 2055, and 2028 cm^{-1} . In addition, two further absorptions at 1775 and 1727 cm^{-1} 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 $[\text{Co}_2(\text{CO})_6(\mu\text{-phthalimidoCH}_2\text{C}\equiv\text{CH})]$ at 2094, 2057, and 2030 as well as 1775 and 1727 cm^{-1} (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^{-1}) [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^{-1}).

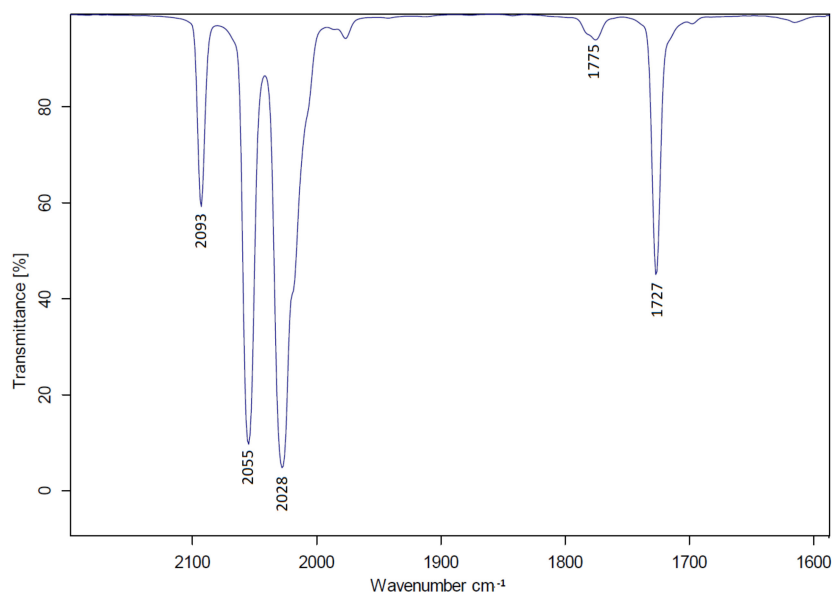


Figure 1. IR spectrum of compound **2** recorded in cyclohexane.

The ^1H -NMR recorded in CDCl_3 reveals a singlet at δ 2.65 due to the acetylenic methyl group; a further singlet at δ 5.03 is attributed to the enantiotopic methylene hydrogens. The remaining resonances at δ 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 δ 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 $\text{C}=\text{O}$ carbonyl groups at δ 167.9, the two acetylenic carbons at δ 93.2 and 91.8, as well as those of the CH_2 and CH_3 carbons gave rise to well-resolved signals.

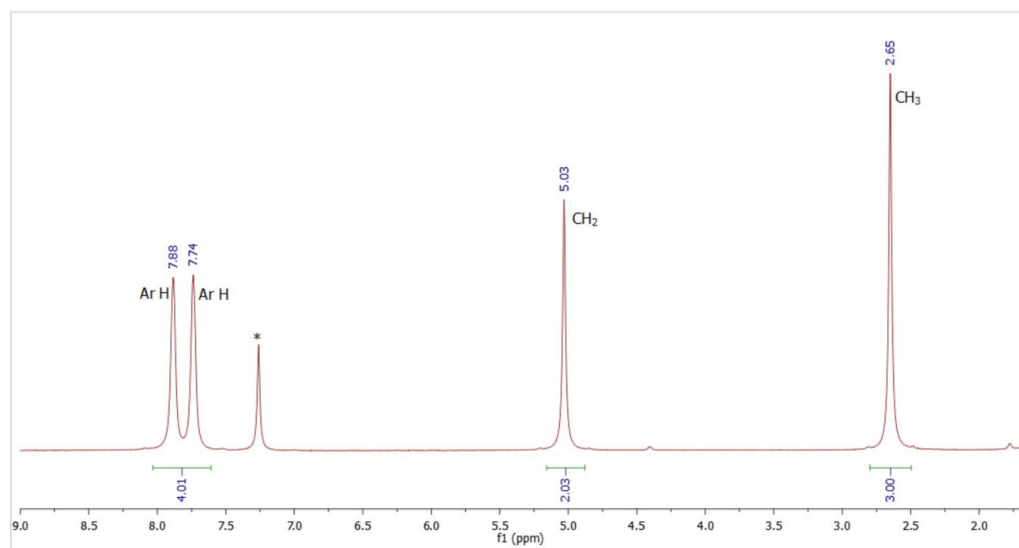


Figure 2. ^1H -NMR spectrum (400 MHz, CDCl_3) of compound **2** at 25 °C. The asterisk * denotes the chloroform resonance.

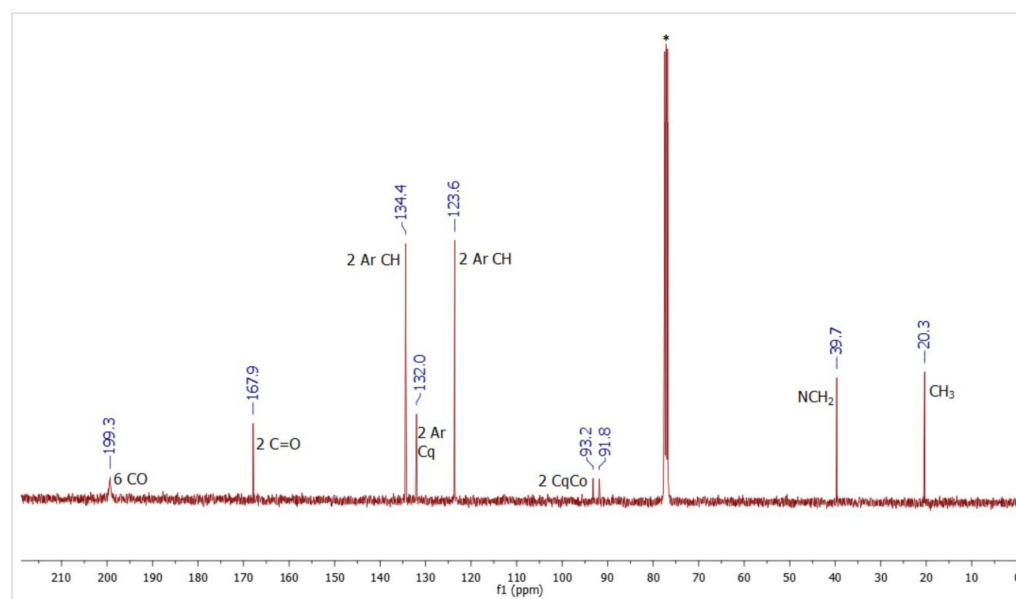


Figure 3. $^{13}\text{C}\{^1\text{H}\}$ -NMR spectrum (100.62 MHz, CDCl_3) of compound **2** at 25 °C. The asterisk * denotes the CDCl_3 resonance.

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 $P\bar{1}$; 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 $\mu_2\text{-}\eta^4$ -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.

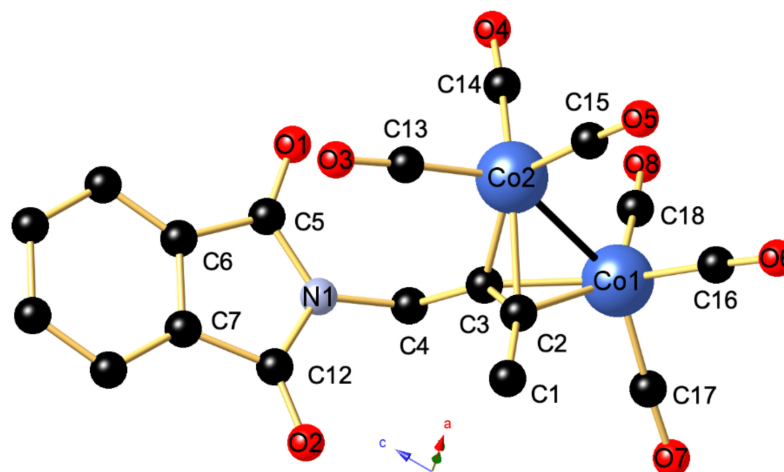


Figure 4. Molecular structure of one of two independent molecules of **2**. All H atoms are omitted for clarity. Selected bond lengths (Å) and angles (deg): Co1–Co2 2.4688(14), Co1–C2 1.980(6), Co1–C3 1.952(6), Co2–C2 1.984(6), Co2–C3 1.954(7), Co1–C16 1.812(7), Co1–C17 1.784(8), Co1–C18 1.831(7), Co2–C13 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).

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 $\text{Co}(\text{CO})_3$ units of the parent cluster $[\text{Co}_4(\mu_2\text{-CO})_3(\text{CO})_9]$ 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 $\text{C}\equiv\text{O}$ 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 $\text{C}\equiv\text{C}$ bond reported for [phthalimido $\text{CH}_2\text{C}\equiv\text{CCH}_2$ 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 $\mu_2\text{-}\eta^2, \eta^2$ capped by an internal alkyne, a survey of the CSD data base revealed only two other examples containing a $-\text{CH}_2\text{C}\equiv\text{C}-\text{Me}$ 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).

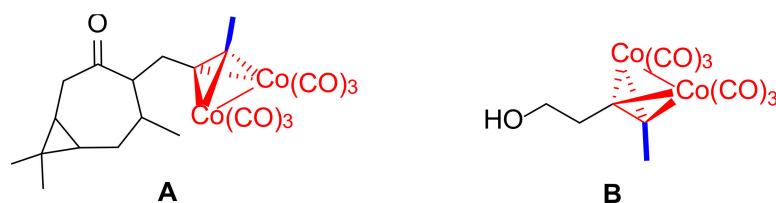


Figure 5. Presentation of the two structurally characterized dicobaltatetrahedranes (**A**) and (**B**) incorporating a $-\text{CH}_2\text{C}\equiv\text{C}-\text{Me}$ motif.

Table 1. Comparison of relevant bond lengths (Å) and angles ($^\circ$) in **2** and the crystallographically characterized $[\text{Co}_2(\text{CO})_6(\mu\text{-RCH}_2\text{C}\equiv\text{CCH}_3)]$ tetrahedranes **A** and **B**.

	2	A	B
Co–Co	2.4679(14)	2.4688(9)	2.4690(9)
C–C _{alkyne}	1.330(9)	1.325(6)	1.326(6)
C _{alkyne} –C _{Me}	1.481(9)	1.486(6)	1.492(6)
C _{alkyne} –CH ₂	1.499(9)	1.500(7)	1.493(6)
Co–C _{alkyne}	1.972(6)	1.974(5)	1.972(4)
Co–C _{pseudo-equatorial}	1.815(7)	1.823(5)	1.820(5)
Co–C _{pseudo-axial}	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).

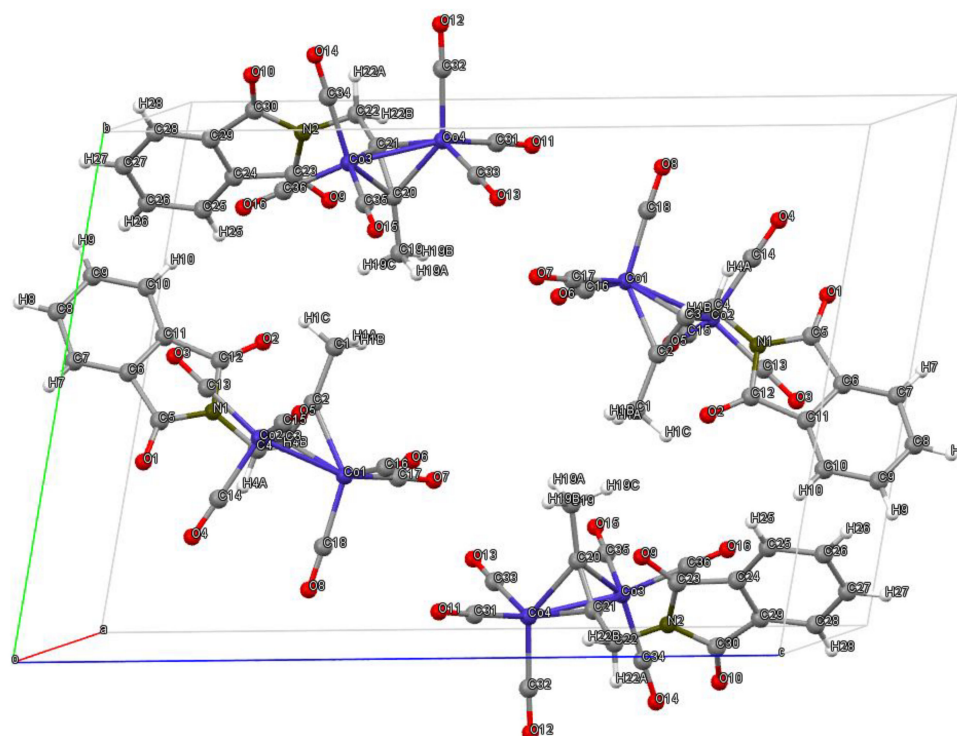


Figure 6. View of the molecular cell of **2** showing the two pairwise arranged independent molecules.

3. Materials and Methods

N-(2-butynyl)phthalimide **1** was commercially purchased from Aldrich. ^1H - and ^{13}C -NMR spectra were recorded on a Bruker AC 400 (Bruker, Wissembourg, France) at 400 and 100.62 MHz, respectively, using CDCl_3 as solvent. Infrared spectra were recorded on a Vertex 70 spectrometer (Bruker, Wissembourg, France) in ATR mode or in solution.

$(\mu_2\text{-}\eta^4\text{-N}-(2\text{-butynyl})\text{phthalimide})(\text{hexacarbonyl})\text{dicobalt}$: Alkyne **1** (199.2 mg, 1 mmol) was added to a stirred solution of $\text{Co}_2(\text{CO})_8$ (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 $\text{C}_{18}\text{H}_9\text{Co}_2\text{NO}_8$ (M.W = $485.14\text{ g}\cdot\text{mol}^{-1}$): C, 44.56; H, 1.87; N, 2.89%. Detected: C, 44.64; H, 1.92; N, 2.98 %. ^1H -NMR (CDCl_3) at 298 K: δ 2.65 (s, CH_3), 5.03 (s, NCH_2), 7.74 (br, CH Ar), 7.88 (br, CH Ar) ppm. $^{13}\text{C}\{^1\text{H}\}$ -NMR (CDCl_3) at 298 K: δ 20.3 (CH_3), 39.7 (NCH_2), 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, $\text{CuK}\alpha$ radiation was employed instead of $\text{MoK}\alpha$ 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° .

Crystal data for $\text{C}_{18}\text{H}_9\text{Co}_2\text{NO}_8$, $M = 485.12\text{ g}\cdot\text{mol}^{-1}$, orange-red needles, crystal size $0.377 \times 0.194 \times 0.15\text{ mm}^3$, triclinic, space group $P\bar{1}$: $a = 7.4317(4)\text{ \AA}$, $b = 13.4351(7)\text{ \AA}$, $c = 19.2794(11)\text{ \AA}$, $\alpha = 78.462(3)^\circ$, $\beta = 84.011(3)^\circ$, $\gamma = 89.156(3)^\circ$, $V = 1875.73(18)\text{ \AA}^3$, $Z = 4$, $D_{\text{calc}} = 1.718\text{ g}\cdot\text{cm}^{-3}$, $T = 100\text{ K}$, $h = -8 \leq h \leq 8$, $k = -15 \leq k \leq 16$, $l = 0 \leq l \leq 16$, GOF = 1.077, $R_1 = 0.0855$, $wR_2 = 0.2428$ for 6962 reflections with $I > 2\sigma(I)$ and 11958 independent reflections. Largest diff. peak/hole/ $e\text{ \AA}^{-3}$ 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 $\text{CuK}\alpha$ radiation $\lambda = 1.54178\text{ \AA}$ and were deposited at the Cambridge Crystallographic Data Centre as CCDC 2219351.

(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-butyne)phthalimide **1** to $[\text{Co}_2(\text{CO})_8]$ straightforwardly yields the stable complex $[\text{Co}_2(\text{CO})_6(\mu\text{-phthalimidoCH}_2\text{C}\equiv\text{CMe})]$ **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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