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TITLE: mer-[InCl₃(C₅D₅N)₃]₂·C₅D₅N: A New d₅-pyridine Derivative of Indium(III) Chloride Complex

YEAR: 2025

DOI: <https://doi.org/10.18596/jotcsa.1590991>

VERSION: Publishers PDF

CITATION: Tuna A, Papageorgiou A, Peuronen A, Peljo P, Knör G. mer-[InCl₃(C₅D₅N)₃]₂·C₅D₅N: A New d₅-pyridine Derivative of Indium(III) Chloride Complex. JOTCSA. 2025;12(1):15-22.

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***mer*-[InCl₃(C₅D₅N)₃]₂·C₅D₅N: A New D₅-pyridine Derivative of Indium(III) Chloride Complex**

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Abstract: In this study, we report the crystal structure of a meridional octahedral pyridine-indium(III) chloride adduct, *mer*-trichlorotris(*d*₅-pyridine)indium(III)·0.5(*d*₅-pyridine), InCl₃(C₅D₅N)₃·0.5(C₅D₅N), which exhibits an unreported molecule-to-solvent ratio compared to other substituted and unsubstituted pyridine-indium(III) chloride adducts described in earlier literature. The bonding characteristics of this *d*₅-pyridine complex are compared to analogous complexes that have already been reported. Furthermore, the role of this metal complex in indium insertion into corrole ligands is discussed.

Keywords: indium; *d*₅-pyridine; *mer*-isomer; solvated-complex; indium corrole

Submitted: November 26, 2024. **Accepted:** February 1, 2025.

Cite this: Cite Tuna A, Papageorgiou A, Peuronen A, Peljo P, Knör G. *mer*-[InCl₃(C₅D₅N)₃]₂·C₅D₅N: A New D₅-pyridine Derivative of Indium(III) Chloride Complex. JOTCSA. 2025;12(1):15-22.

DOI: <https://doi.org/10.18596/jotcsa.1590991>.

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1. INTRODUCTION

Geometric isomerism is a fundamental aspect in coordination complexes where the complex molecules have the same empirical formula, but where the arrangement of the ligand molecules around the metal center are distinctively different. Isomers may exhibit different physical, chemical and even biological properties due to different geometries, such as *cis*, *trans*, facial (*fac*), and meridional (*mer*) isomerism (1). In *fac*-isomerism, three identical ligands are positioned on one face of the octahedron, creating a triangular arrangement around the central metal atom. The *mer*-isomer features three identical ligands and a metal atom in a single plane, forming a T-shaped arrangement around the central metal atom (2). This configuration reduces intra-ligand repulsions, making it more suitable for smaller metal complexes, such as high-valent metal centers (1,3). The distinct spatial arrangements of ligands in *fac*- and *mer*-isomers result in differences in chemical reactivity,

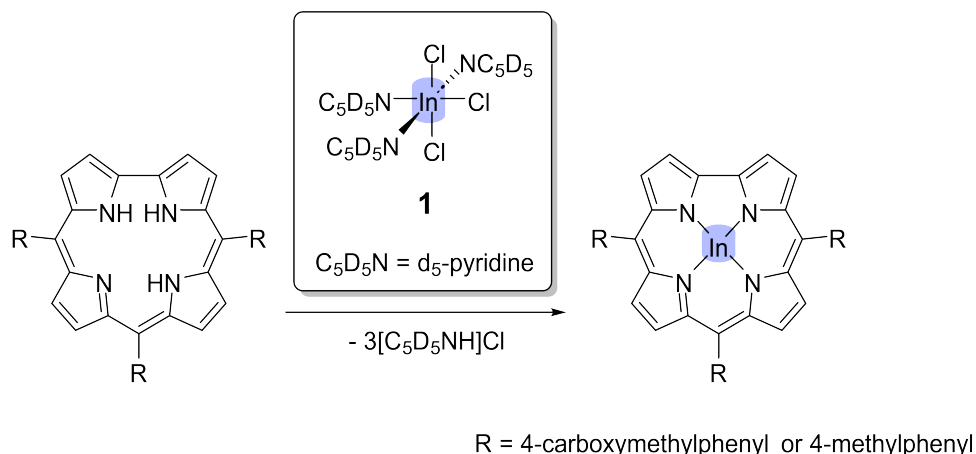
physical properties, and biological activity, highlighting their importance in the study and application of coordination and inorganic chemistry (4-6).

Metal activation through the coordination of pyridine or other solvents occurs when metal complexes interact with the coordinated solvent molecules, thereby affecting the reactivity and properties of the metals involved whereas coordinated solvent pushes the electrons through the metal center to make it more active as the metal center becomes more electro-positive (7-9). Such activation is very useful in metalation, where it can aid the metal centers in forming new complexes with higher yields while preventing the formation of mixtures of products (9,10).

Corroles are conjugated 18π-electron aromatic ring system carrying one direct pyrrole-pyrrole linkage. They can be considered as ring-contracted porphyrin

derivatives. Many metallocorroles have been successfully synthesized and investigated in the last decades. The first attempts at the synthesis of an indium corrole compound were made in the late 80s, but complete characterization of this compound was unsuccessful for many years (11,12). Recently, the first successful indium insertion into the 5,10,15-tris(pentafluorophenyl)corrole was achieved and the obtained indium corrole complex was successfully characterized by relevant analytical techniques (13). Remarkably, the observed Soret- and Q-band patterns of the formed indium corrole complex in the absorption spectrum shows a hypsochromic shift

which indicates a significant change of HOMO-LUMO gap as well as an extent of conjugated π -system in order to structural modifications influenced the electronic transitions in the direction of successful metal insertion. We believe that when the metalation process of indium corrole is conducted in d_5 -pyridine, an *in situ* formed intermediate, trichlorotrakis(d_5 -pyridine)indium(III) plays a key role in the metalation process. Here, we explore this claim, by isolating and characterizing *mer*-trichlorotrakis(d_5 -pyridine)indium(III) (**1**) and by using it as a metal precursor in the metalation of two different corrole ligands (Scheme 1).



Scheme 1. *mer*-Trichlorotrakis(d_5 -pyridine)indium(III) (**1**) and its role in indium insertion to corroles.

2. MATERIALS AND METHODS

2.1. Materials

Indium(III) chloride (anhydrous, 99.95%) was purchased from ABCR Chemie, Germany. d_5 -pyridine (99.8 atom%D) was purchased from ARMAR Isotopes, Germany. Anhydrous pyridine (99.9%) and other supplements were obtained from VWR, Finland. Free-base corrole ligands were synthesized at the University of Rome "Tor Vergata" (Italy) according to the literature (18).

2.2. Preparation of Single Crystals and Powder

300.4 mg of anhydrous indium(III) chloride was dissolved in an excess amount of d_5 -pyridine (5-5.1 mL) and the mixture was refluxed for 30 min under an Ar atmosphere until the solution became transparent. The solution was then divided into two vials of relatively equal amount (2.5 mL) in a fume hood. One vial was covered with a lid and small holes were opened to allow slow evaporation of the solution at room temperature. The other vial was poured into a 25 mL single-neck round-bottom flask, and d_5 -pyridine was removed under reduced pressure at 50 °C to give a white powder. Crystals (**1**) were obtained from the first vial.

2.3. Preparation of Indium Corroles

mer-Trichlorotrakis(d_5 -pyridine)indium(III) obtained from the second vial as a white powder was added in small portions to a solution of the free-base ligand (50

mg each, 1 eq) in anhydrous pyridine at reflux, until indium insertions were completed. The metalation reaction progress was monitored by UV-Vis spectroscopy where the spectra were measured with a Cary 60 spectrophotometer.

2.4. Single Crystal Measurement

A suitable crystal of **1** was selected and immobilized on a polyamide loop using a small amount of paratone oil. X-ray diffraction data were collected on a Rigaku Micromax-007 HF generator equipped with a HyPix-6000HE hybrid photon counting detector. The crystal was kept at room temperature during data collection. The X-ray data were processed with CrysAlisPro (18).

Crystal Data for 1: monoclinic, space group $P2_1$ (no. 4), $a = 13.3862(5)$ Å, $b = 8.9223(3)$ Å, $c = 17.0910(7)$ Å, $\beta = 101.666(4)^\circ$, $V = 1999.11(13)$ Å³, $Z = 4$, $T = 293(2)$ K, $\mu(\text{Cu K}\alpha) = 13.188$ mm⁻¹, $D_{\text{calc}} = 1.713$ g/cm³, 12904 reflections measured ($5.28^\circ \leq 2\theta \leq 136.498^\circ$), 5305 unique ($R_{\text{int}} = 0.0839$, $R_{\text{sigma}} = 0.0626$) which were used in all calculations. The final R_1 was 0.0991 ($I > 2\sigma(I)$) and wR_2 was 0.2776 (all data).

2.5. Refinement

With Olex2 as the graphical interface, the structure was solved using intrinsic phasing in the SHELXT (19,20) structure solution program and refined by employing Least Squares minimization. All D atoms

were refined isotropically using idealized C-D sp^2 geometries and distances with $U_{iso(H)} = 1.2U_{eq(C)}$, whereas non-D atoms were refined anisotropically. In the final model, the atoms of the aromatic rings of the disordered d_5 -pyridine groups could not be refined without geometrical restriction and were instead subjected to a rigid refinement procedure. Details of data collection and structure refinement are summarized in Table 1.

3. RESULTS AND DISCUSSIONS

The indium(III) chloride complex of d_5 -pyridine (**1**) crystallizes in the monoclinic space group $P2_1$ with two *mer*-trichlorotris(d_5 -pyridine)indium(III) molecules and one d_5 -pyridine molecule within the asymmetric unit resulting in a 2:1 ratio between the complex units and non-coordinated solvate molecules (Figure 1). The three d_5 -pyridine ligands are coordinated meridionally in a T-shape geometry around the metal center. One of the d_5 -pyridine ligands was found to be disordered in two sites rotated by approximately 23° over the C_5N plane.

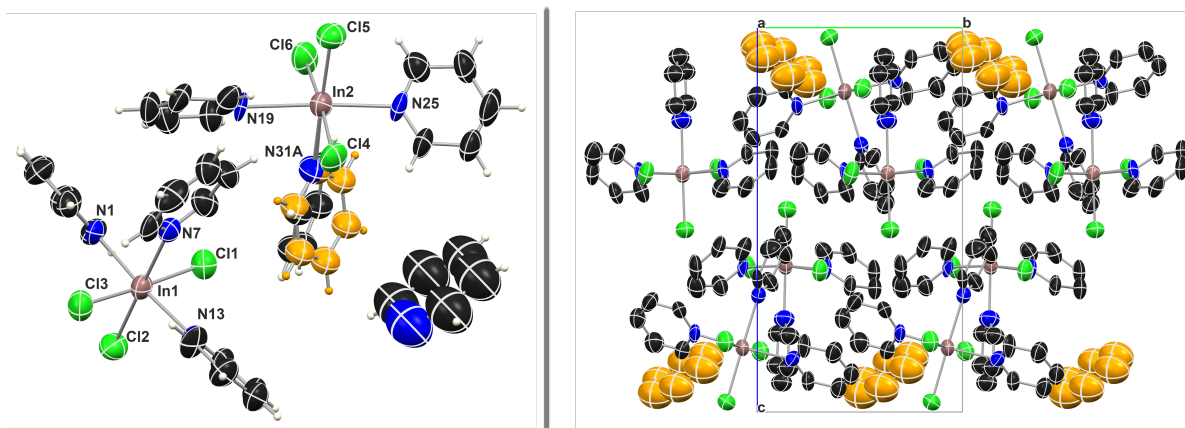


Figure 1 Left: Illustration of the asymmetric unit of the crystal structure of **1** where the disordered d_5 -pyridine ligand at In2 is also shown. Right: Crystal packing of **1** viewed along the crystallographic *a*-axis (D atoms and disordered d_5 -pyridine ligands omitted). The displacement ellipsoids are drawn at the 50% probability level.

Table 1 shows a list of crystallographic parameters of analogous trichlorotris(pyridine)indium(III) structures that have already been introduced in the literature and contain pyridine (**2**) (14), 4-methylpyridine (**3**) (15), and 4-ethylpyridine (**4**) (16) as ligands (Figure 2). Despite their similar molecular structures, **1** and **2** crystallize in different space groups and exhibit a different number of solvent molecules in their crystal lattices. Compounds **1** and **2** are crystallized with surrounding solvate molecules whereas the **3** and **4** are crystallized without any solvate molecules (Table 1). As mentioned above, compound **1** crystallizes in the monoclinic $P2_1$ space group with 0.5 non-coordinated d_5 -pyridine solvate molecules per complex unit in the crystal lattice. This indicates a more complex packing arrangement, potentially influenced by the deuterium atoms in d_5 -pyridine, which can affect intermolecular interactions and packing density.

The second structure (**2**), crystallizing in the orthorhombic $C222_1$ space group, differs from **1** by having an equivalent number of complex molecules to pyridine solvate molecules in the structure. The presence of one complex and one solvent molecule in the asymmetric unit as well as the higher crystallographic symmetry suggest a more straightforward packing arrangement in **2** compared to **1**. The use of d_5 -pyridine instead of non-deuterated pyridine could lead to subtle differences in the crystal packing and intermolecular forces due to the slightly

different mass and vibrational properties of deuterium compared to hydrogen. These differences may contribute to the observed differences in the space group and stoichiometry between the two structures. Overall, the choice of solvate molecule and the resulting crystal packing appear to significantly influence the crystallographic properties and symmetry of the resulting structures.

The In-N bond lengths in **1** are systematically longer compared to the other analogous complexes **2-4** (Table 2). This difference is most evident for the N-In bond located trans to the Cl atom, while the N-In-N bond lengths are, in comparison, rather similar. The different In-N bond lengths between the complexes indicate subtle differences in the N-donor capabilities of the ligands, while crystal packing effects can also play a role. For example, in **2** - the closest analogue to **1** - the crystal packing is mostly dictated by the intermolecular interactions between the complex molecules and the solvent, while in **1** several intermolecular C-H \cdots π and $\pi\cdots\pi$ contacts can be observed between the In complexes. This difference between these two crystal structures most likely arises from the higher complex-to-lattice solvent ratio in **2**. The observed differences in the In-N distances between the two crystal structures can also be related to isotopic effects. Isotopic substitution, in which atoms of different isotopes are present, may influence bond lengths owing to variations in atomic masses

and vibrational frequencies. Heavier isotopes tend to vibrate at lower frequencies, leading to slightly different bond lengths. Therefore, the minor variations in the bond lengths observed in the structures could be attributed to the presence of different isotopes of hydrogen, such as deuterium, resulting in subtle

changes in the local bonding environment. A further comparison of all the four structures **1-4** shows that the In-Cl bonds appear to be less sensitive to changing the N-donor, although there is some variation in the In-Cl bonds lengths which does not seem to be systematical.

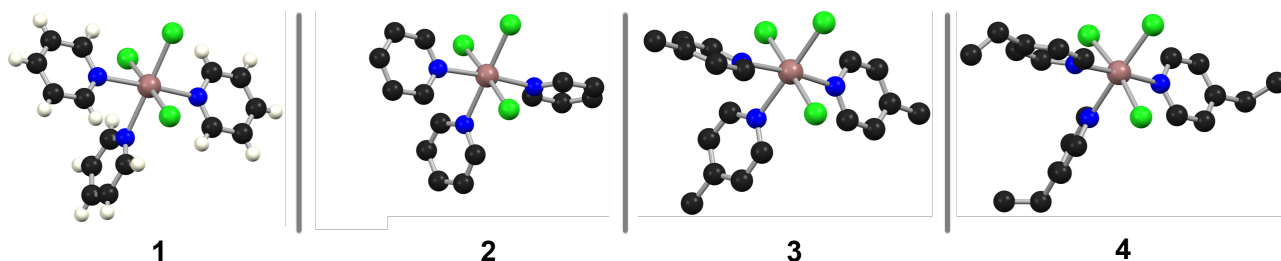


Figure 2 The complex molecule of structure **1** (this work) compared to **2-4** drawn using crystallographic data obtained from the Cambridge Structural Database (17). The ^2H atoms of **1** are highlighted while ^1H atoms are omitted from the figure.

Table 1 Crystal data for compound **1** (this work) compared to different *mer*-trichlorotrakis(pyridine)indium(III) derivatives (**2-4**) obtained from previous literature.

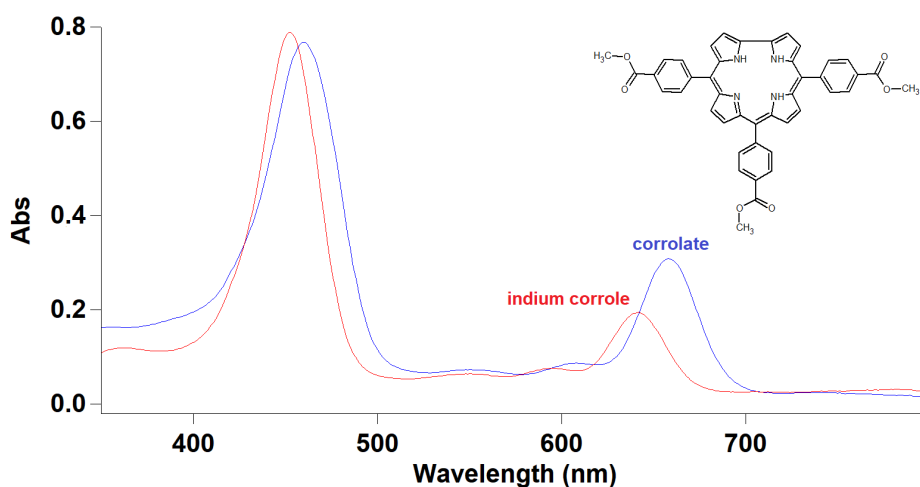
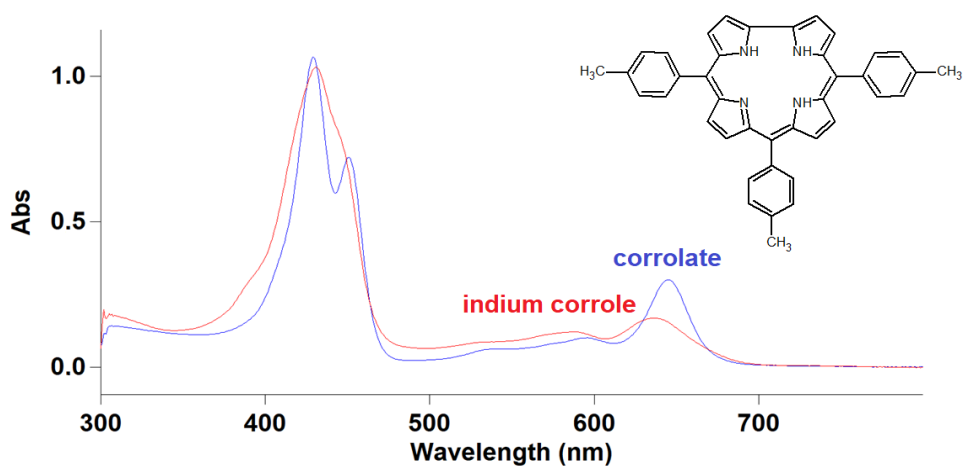
Parameters	1	2	3	4
pyridine derivative	d_5 -pyridine	pyridine	4-methylpyridine	4-ethylpyridine
empirical formula	$\text{C}_{17.5}\text{Cl}_3\text{D}_{17.5}\text{InN}_{3.5}$	$\text{C}_{20}\text{H}_{20}\text{Cl}_3\text{InN}_4$	$\text{C}_{18}\text{H}_{21}\text{Cl}_3\text{InN}_3$	$\text{C}_{21}\text{H}_{27}\text{Cl}_3\text{InN}_3$
molecular weight	515.63 Da	537.58 Da	500.56 Da	542.64 Da
crystal system	monoclinic	orthorhombic	triclinic	triclinic
space group	$P2_1$	$C222_1$	P_1	P_1
<i>a</i>	13.3862(5) Å	9.117(7) Å	9.3240(3) Å	9.7330(1) Å
<i>b</i>	8.9223(3) Å	16.83(2) Å	13.9580(6) Å	20.826(2) Å
<i>c</i>	17.0910(7) Å	14.66(1) Å	16.7268(7) Å	25.311(3) Å
α	90°	90°	84.323(2)°	74.970(1)°
β	101.666(4)°	90°	80.938(2)°	83.31(2)°
γ	90°	90°	78.274(3)°	89.26(2)°
<i>V</i>	1999.11(13) Å ³	2250(5) Å ³	2099.8(2) Å ³	4920.0(9) Å ³
<i>Z</i>	4	4	4	8
ρ_{calc}	1.713 g/cm ³	1.586 g/cm ³	1.58 g/cm ³	1.465 g/cm ³
temperature	293 K	293 K	203 K	293 K
X-ray source	Cu K α	Mo K α	Mo K α	Mo K α
<i>R</i> ₁	0.0991	0.050	0.035	0.0953
deposited on	this work	28/03/1985	28/12/2001	05/01/2001
published year	this work	1984	2013	2000
reference	this work	(14)	(15)	(16)
CSD reference	this work	CILYUR	WIBVEK	WOPYIJ

The formed *mer*-trichlorotrakis(d_5 -pyridine)indium(III) structure is believed to play a key role in the metalation process of indium corrole formation in pyridine (11,13). To prove this claim, *mer*-trichlorotrakis(d_5 -pyridine)indium(III) was used as a

metal precursor in the metalation of corrole using different corrole ligands (Figure 3 and Figure 4). These UV-vis spectra suggest that the two selected corroles undergo metalation using the *mer*-trichlorotrakis(d_5 -pyridine)indium(III) as a metal precursor.

Table 2 The average bond lengths for *mer*-trichlorotrakis(pyridine-derivative)indium(III) structures containing different pyridine derivatives (**1-4**). For **1**, only one of the two distinct complex molecules in the asymmetric unit is shown.

Bond	1 ^a	2	3	4
In - N1	2.321(2) Å	2.302(7) Å	2.312(2) Å	2.279(1) Å
In - N2	2.410(2) Å	2.377(2) Å	2.324(2) Å	2.341(8) Å
In - N3	2.332(2) Å	2.302(7) Å	2.311(2) Å	2.304(1) Å
In - Cl1	2.479(5) Å	2.476(2) Å	2.482(2) Å	2.413(6) Å
In - Cl2	2.464(5) Å	2.471(8) Å	2.461(1) Å	2.468(6) Å
In - Cl3	2.472(6) Å	2.471(8) Å	2.475(4) Å	2.480(6) Å

^a This work.**Figure 3** UV-vis spectra of free-base corrole as a corrolate anion and formed indium corrole complex using the 5,10,15-tris(4-carboxymethylphenyl)corrole ligand in pyridine.**Figure 4** UV-Vis spectra of free-base corrole as a corrolate anion and formed indium corrole complex using the 5,10,15-tris(4-methylphenyl)corrole ligand in pyridine.

4. CONCLUSIONS

In conclusion, *mer*-trichlorotris(*d*₅-pyridine)indium(III) complex was crystallized and its crystal structure was determined and compared to analogous non-deuterated In complexes. The crystal structure indicates that changing H atoms to D atoms has a noticeable effect on the coordination environment of the In center, and a more profound effect on the crystal packing arrangements due to the crystallization of the complexes with different amounts of lattice solvent per complex molecule (0, 0.5, or 1). A further investigation showed that indium metal could be successfully transferred from the *mer*-trichlorotris(*d*₅-pyridine)indium(III) to different free-base corroles, 5,10,15-tris(4-carboxymethylphenyl)corrole and 5,10,15-tris(4-methylphenyl)corrole, respectively. The obtained UV-vis spectra were compared with the experimental data presented in a previous study on indium corrole synthesis as a result of successful indium corrole formations as significant spectral shifts on Soret and Q band patterns. In parallel, we are currently investigating the preparation of new indium corrole complexes with more detailed characterization data and further crystallization attempts to elucidate the first X-ray structure of an indium corrole.

5. ACKNOWLEDGMENT

We gratefully acknowledge the funding from the Research Council of Finland (Grant number 334828). We acknowledge the Materials Research Infrastructure (MARI) and Biocenter Finland infrastructure support for University of Turku. A.T. acknowledges EXACTUS travel grants (2022 and 2023) from University of Turku, Finland, Prof. Dr. Roberto Paolesse (University of Rome "Tor Vergata", Italy) for kind hospitality for multiple weeks in 2022 and 2023, and Prof. Dr. Bernhard Spingler (University of Zurich, Switzerland) for a preliminary discussion.

Supplementary Materials: The following supporting information can be downloaded at:.

Conflict of Interest: All authors declare that they have no conflicts of interest.

Data Availability Statement: CCDC 2392242 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html> (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336033; E-mail: deposit@ccdc.cam.ac.uk).

Author Contributions:

A. T.: conceptualization, data curation, formal analysis, investigation, methodology, validation, visualization, writing—original draft preparation, writing—review and editing.

A. C. P.: formal analysis, writing—original draft preparation, writing—review and editing.

A. P.: data curation, validation, visualization, writing—original draft preparation, writing—review and editing.

P. P.: resources, supervision, writing—review and editing.

G. K.: supervision, validation, writing—review and editing.

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