Structure of the Cycloaddition Products of Pyrido[2,1-*a*]isoindole with Maleimide Derivatives: X-ray Diffraction Analysis and ^{1}H NMR Variable-Temperature Spectra

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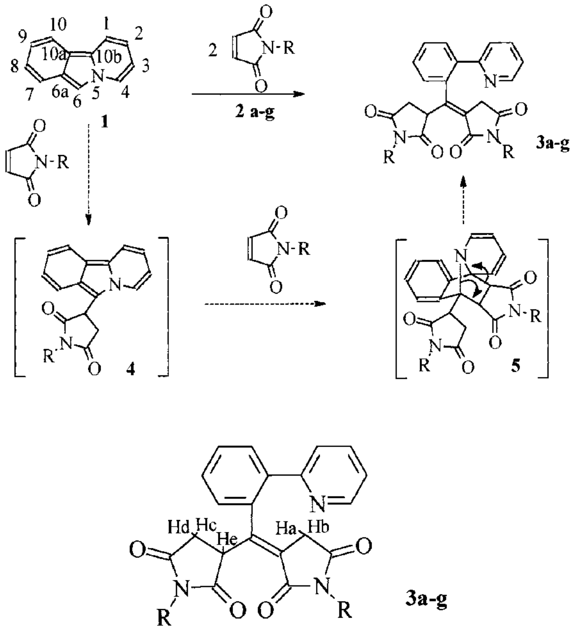
Investigation of the cycloaddition reaction under thermodynamic control of pyrido[2,1-*a*]isoindole with maleimide derivatives revealed a new rearrangement leading to 2-{2′-[(1*R*)-2,5-dioxopyrrolidinylidene]-2′-[(1*R*)-2,5-dioxopyrrolidinyl)-methyl]}phenylpyridine. Their structure was confirmed by X-ray diffraction analysis. The atropisomeric properties of the obtained compounds were examined in a detailed NMR spectroscopic study. Dependence of the spectra on temperature was investigated, and coalescence temperatures and conversion barriers between the corresponding atropisomers determined.

Introduction

Isoindoles, despite being aromatic heterocycles, easily undergo Diels−Alder reaction at the pyrrole ring.[1−3] For 1,2-disubstituted isoindoles, Michael reaction at position 3 may be a competing process.[2,4] Such reactions of simple isoindoles[1−5] have been well studied, but those of annelated isoindole systems with a nodal nitrogen atom less so.[6−8] Calculations on pyrido[2,1-*a*]isoindole **1** show that it can react either in a Diels−Alder reaction with the diene fragment of the pyrrole ring, or additionally in Michael fashion at position 6.[9] Examples of cycloaddition reactions between pyrido[2,1-*a*]isoindole and dienophiles containing both double and acetylene bonds[10,11] are known in the literature. Reactions take place in 1,4- or 1,6-fashion, and formation of Michael adducts at position 6 are described.

Only one example involving maleimide derivatives is known; treatment of **1** with *p*-tolylmaleimide under mild conditions afforded the Michael adduct, 6-[1-(*p*-tolyl)succinimidyl]-pyrido[2,1-*a*]isoindole.[11]

In a preliminary study, we demonstrated that the reaction between pyrido[2,1-*a*]isoindole (**1**) and maleimide derivatives **2a−g** under thermodynamic control resulted in the formation of (1*R*)-3-{(1*R*)-2,5-dioxotetrahydro-1*H*-pyrrol-3-ylidene-[2-(2-pyridyl)phenyl]methyl}pyrrolidine-2,5-dione (**3**) by a previously unknown rearrangement (Scheme 1).[12]



Scheme 1. R = Ph (a); *p*-$CH_3C_6H_4$ (b); *p*-$CH_3OC_6H_4$ (c); *p*-$NO_2C_6H_4$ (d); H (e); CH_2Ph (f); 2-naphthyl (g).

The following mechanism seems likely: the first molecule of maleimide adds to the 6 position of the pyrido[2,1-*a*]isoindole in a Michael reaction; the newly formed isoindole **4** then incorporates the second molecule of maleimide, through positions 6 and 10b, in a Diels−Alder reaction. A subsequent rearrangement of the adduct **5** (1:2 ratio) produces the end product **3**. This process is thermodynamically favoured because it includes the cleavage of two bridge bonds C−C and C−N in a constrained cycle, the aromatization of the pyridine ring and the formation of an exocyclic double bond (Scheme 1). Moreover, this mechanism should result only in the *E* isomer, the cleavage of the C−N and C−C bonds in **5** to obtain **3** being unequivocal (Scheme 1).

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The structure of **3** was confirmed by CHN elemental analysis, assorted spectral data,[12] and in the cases of **3a** and **3b** by single-crystal X-ray study (Figure 1; Table 1−3).

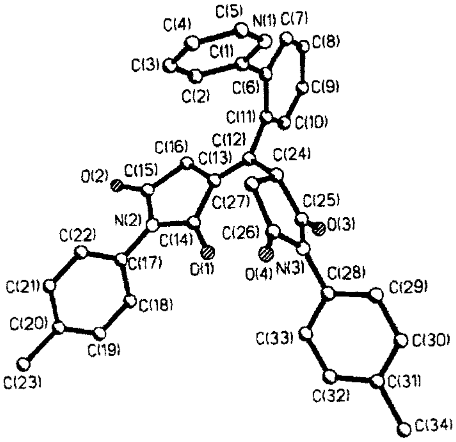

Figure 1. X-ray structure of compound **3b**

Table 1. Selected bond lengths [Å] of **3a** ($C_{32}H_{23}N_3O_4$) and **3b** ($C_{34}H_{27}N_3O_4$)

	3a	**3b**
O(1)−C(14)	1.212(7)	1.253(4)
O(2)−C(15)	1.208(0)	1.249(4)
O(3)−C(25)	1.212(1)	1.242(4)
O(4)−C(26)	1.206(8)	1.239(4)
N(2)−C(14)	1.405(1)	1.440(4)
N(2)−C(15)	1.400(7)	1.442(5)
N(2)−C(17)	1.438(3)	1.467(5)
N(3)−C(26)	1.399(6)	1.423(5)
N(3)−C(25)	1.390(0)	1.437(5)
N(3)−C(28)	1.429(6)	1.481(5)
C(1)−C(6)	1.490(5)	1.516(5)
C(11)−C(12)	1.498(1)	1.548(5)
C(12)−C(13)	1.346(1)	1.376(5)
C(12)−C(24)	1.520(1)	1.569(5)
C(13)−C(14)	1.488(0)	1.506(5)
C(13)−C(16)	1.501(0)	1.549(5)
C(15)−C(16)	1.507(8)	1.517(5)
C(20)−C(23)	−	1.562(6)
C(24)−C(25)	1.521(4)	1.554(5)
C(24)−C(27)	1.539(4)	1.588(5)
C(26)−C(27)	1.513(1)	1.545(5)
C(31)−C(34)	−	1.566(6)

In the crystals, the five-membered rings C(13)−C(14)−N(2)−C(15)−C(16) and C(24)−C(25)−N(3)−C(26)−C(27) are in an envelope conformation (in **3b**, for example, the deviation of the C(13) atom from the root-mean-square plane of the other atoms of the cycle amounts to 0.17 Å) and in twist conformation [the C(24) and C(25) atoms in **3b** deviate from the plane of the other atoms by 0.12 Å and −0.09 Å respectively].

The tolyl substituent remains nearly perpendicular to the five-membered rings [torsion angles in **3b** are −71.05° for C(14)−N(2)−C(17)−C(18) and 85.20° for C(26)−N(3)−C(28)−C(33)]. In contrast, the phenyl substituents in **3a** are more highly conjugated with their adjacent nitrogen atoms [torsion angles are −171.09° for C(14)−N(2)−C(17)−C(18) and 119.39° for C(25)−N(3)−C(28)−C(33)], and one of them is almost planar to the succinimide ring.

Table 2. Selected angles [°] of **3a** and **3b**

	3a	**3b**
C(14)−N(2)−C(15)	112.4(7)	112.0(3)
C(14)−N(2)−C(17)	124.2(0)	124.7(3)
C(15)−N(2)−C(17)	123.2(4)	123.3(3)
C(26)−N(3)−C(25)	112.5(9)	112.6(3)
C(26)−N(3)−C(28)	123.8(3)	124.6(3)
C(25)−N(3)−C(28)	122.9(2)	122.5(3)
C(13)−C(12)−C(11)	120.2(5)	120.7(3)
C(13)−C(12)−C(24)	124.6(2)	124.6(3)
C(11)−C(12)−C(24)	114.9(1)	114.6(3)
C(12)−C(13)−C(14)	125.5(9)	127.5(3)
C(12)−C(13)−C(16)	126.6(7)	125.0(3)
O(1)−C(14)−N(2)	123.9(0)	123.6(4)
O(2)−C(15)−N(2)	124.0(7)	123.7(3)
C(25)−C(24)−C(12)	112.5(8)	112.5(3)
C(25)−C(24)−C(27)	104.0(7)	104.2(3)
C(12)−C(24)−C(27)	119.4(3)	120.0(3)
O(3)−C(25)−N(3)	124.8(8)	124.6(3)
O(4)−C(26)−N(3)	124.4(7)	123.4(4)
C(29)−C(28)−N(3)	119.6(3)	119.7(4)

Table 3. Selected torsion angles [°] **3a** and **3b**

	3a	**3b**
N(1)−C(1)−C(6)−C(11)	−120.9(2)	127.0(4)
C(1)−C(6)−C(11)−C(12)	12.4(3)	−8.9(5)
C(10)−C(11)−C(12)−C(13)	59.0(1)	−66.6(4)
C(6)−C(11)−C(12)−C(24)	58.1(0)	−66.6(4)
C(11)−C(12)−C(13)−C(16)	7.4(1)	−9.8(5)
C(14)−N(2)−C(17)−C(22)	−131.2(6)	110.9(4)
C(15)−N(2)−C(17)−C(22)	52.4(7)	−71.0(5)
C(13)−C(12)−C(24)−C(25)	−59.5(4)	66.0(4)
C(11)−C(12)−C(24)−C(25)	115.1(5)	−111.0(4)
C(13)−C(12)−C(24)−C(27)	62.9(4)	−57.1(5)
C(12)−C(24)−C(25)−O(3)	−39.6(4)	39.0(5)
C(12)−C(24)−C(25)−N(3)	143.0(7)	−144.5(3)
C(28)−N(3)−C(26)−O(4)	−6.6(9)	2.4(7)
C(12)−C(24)−C(27)−C(26)	−136.9(9)	137.5(4)
C(25)−N(3)−C(28)−C(29)	−59.6(6)	91.4(4)
C(26)−N(3)−C(28)−C(33)	−50.6(5)	85.2(5)

The double bond C(12)−C(13) in **3a** and **3b** is twisted [torsion angles C(16)−C(13)−C(12)−C(11) are 7.41° and −9.80°, respectively] and hence lengthened to 1.346 Å in **3a** and 1.376 Å in **3b**, against a standard double bond length of 1.322 Å.[13] The conjugation of the π-systems of the benzene and pyridine rings is mainly independent, due to rotation around the bonds C(11)−C(12) and C(1)−C(6) (torsion angle C(6)−C(11)−C(12)−C(13) is 116.2° in **3a** and −126.9° in **3b**; torsion angle C(11)−C(6)−C(1)−C(2) is −53.3° in **3a** and 59.5° in **3b**.

It is noticeable that both the degree of torsion in the C=C bond and the whole molecular geometry are sensitive to the *p*-substituent in the benzene ring of the maleimide moiety. Consequently, similar structures offer promise for research into twisted double bond compounds.[14]

Finally, only the *E* isomers are present in the crystalline state, thus reinforcing the mechanism proposed for the rearrangement.

Regarding the behaviour of compounds **3** in solution, ^1H NMR spectra present complicated patterns and variations with solvents and temperature, indicating the presence of an exchange processes. A priori, two mechanisms − atropisomerism and/or *E*/*Z* isomerization − may be in operation, although an *E*/*Z* isomerization at low temperature was thought less probable. The former process may involve rotations around C(1)−C(6), C(11)−C(12) and C(12)−C(24) bonds, whereas the latter concerns the double bond C(12)−C(13).

Our previous calculations (Hyperchem),[15] examination of Dreiding molecular models, and the X-ray diffraction analysis data mentioned above demonstrate that steric hindrances to rotation exist, even in the C(1)−C(6) bond if we take into account the tendency of a biphenyl or a bipyridyl molecule to adopt a position of maximum conjugation between the rings.[16]

^{1}H NMR spectra of **3a**−**g** in solution at room temperature demonstrate the presence of two diastereomers, and so only two combinations of torsion angles among all the possible ones are appreciably stable. This is illustrated by examination of the aliphatic part of the signals in the spectra. These are more complex than should be expected from only one diastereomer. The H_a and H_b proton signals (two doublets with geminal spin-spin interaction constants) appear twice (Table 4, Figure 2). The H_c, H_d and H_e protons give a complex picture too. Theoretically, one might expect three doublets of doublets (dd) for each isomer, but in both isomers the signals of H_c and H_d superimpose in the 2.94−3.14 ppm region. As well as this, the H_c and H_d protons give two dd signals for one of the isomers, while only one doublet signal is present for the other isomer, probably due to the values of the torsion angles resulting in magnetic equivalency of the protons.

The H_e proton gives a signal at lower field, as a triplet or multiplet, with $\delta = 4.2$ for one of the isomers and at $\delta = 6$ for the other. The sums of the integral intensity of these two signals correspond to one proton. Both of these peaks are broadened, maybe as a result of dynamic processes. Such a profound shift between these two signals is surprising. It is likely that one of the H_e protons lies in the plane of a benzene or pyridine ring, the corresponding signal thus being deshielded.

Irradiation of the H_e proton at $\delta = 4.2$ results in the conversion of the doublet common to H_c and H_d into a singlet, while irradiation of H_e for the other isomer at $\delta = 6$ results in the transformation of the two dd for H_c and H_d into two doublets. Therefore, it is possible to ascertain which signals belong to the same isomer (see Table 4)

Hence, compound **3** probably exists in solution as a mixture of two isomers (α) and (β). For a more thorough investigation of this phenomenon, we studied the ^{1}H NMR spectra at variable temperature. We first confirmed that the ^{1}H NMR spectrum does not change with time at room temperature. Spectra do change dramatically, however, if the temperature is lowered or raised. This investigation was carried out for **3a**−**d** in deuteriochloroform at 400 MHz in a temperature range of −10 to + 50 °C and in dimethyl sulfoxide and/or pyridine at 200 MHz for temperatures between 60 and 100 °C. At − 10 °C, all compounds displayed the complete or virtual disappearance of one of the isomers (β). Figure 2 demonstrates that the H_a and H_b protons of the thermodynamically favoured isomer (α) of **3b** at −10 °C gave rise to only two doublets, with a geminal spin-spin constant of 21.9 Hz. When the temperature was increased, concentration of the other isomer (β) increased markedly; up to 30−35% at 50 °C. Chemical shift differences between H_a−H_d decreased somewhat, being still less at 60 °C.

Strongly downfield-shifted H_a−H_d proton signals, together with the α-proton of the pyridine ring, are the most sensitive to isomerism. These protons were used to determine coalescence temperatures (see Figure 3 and Table 5).

Coalescence temperatures were checked by using internal reference compounds with known shift/temperature dependencies, and corrected values were used to calculate free energies of activation for the processes of conversion between the isomers. The calculations were made according to the formula:[17]

$$\Delta G^{\neq} = 19.14\ T(9.97 + \log T/\Delta\nu)\ \text{kJ/mol}$$

Table 4. ^{1}H NMR values of compounds **3a**−**c** ($CDCl_3$, 250 MHz, 25 °C)

Compounds **3a**−**c**		H_a		H_b		H_c and H_d			H_e	
Atropisomers α (major) and β		α	β	α	β	α H_c	α H_d	β H_c+H_d	β	α
3a	δ	2.72 d	2.78 d	3.04 d	2.98 d	2.96 dd	3.16 dd	3.14 d	4.2	6.0
	Coupling constants, Hz	21.9	21.8	21.9	21.8	6.1; 18.7	9.1; 18.7	8.3		
	Irradiation at *δ = 4.20							3.14 s		
	Irradiation at *δ = 6.00					2.96 d	3.16 d			
	Coupling constants, Hz					18.7	18.7			
3b	δ	2.70 d	2.76 d	3.02 d	2.96 d	2.95 dd	3.14 dd	3.11 d	4.2	6.0
	Coupling constants, Hz	21.9	21.7	21.9	21.7	6.2; 18.6	9.4; 18.6	8.3		
	Irradiation at *δ = 4.20							3.11 s		
	Irradiation at *δ = 6.00					2.95 d	3.14 d			
	Coupling constants, Hz					18.6	18.6			
3c	δ	2.69 d	2.75 d	3.01 d	2.95 d	2.93 dd	3.13 dd	3.10 d	4.2	6.0
	Coupling constants, Hz	22.0	21.8	22.0	21.8	6.2; 18.6	9.2; 18.6			
	Irradiation at *δ = 4.20							3.10 s		
	Irradiation at *δ = 6.00					2.93 d	3.13 d			
	Coupling constants, Hz					18.6	18.6			

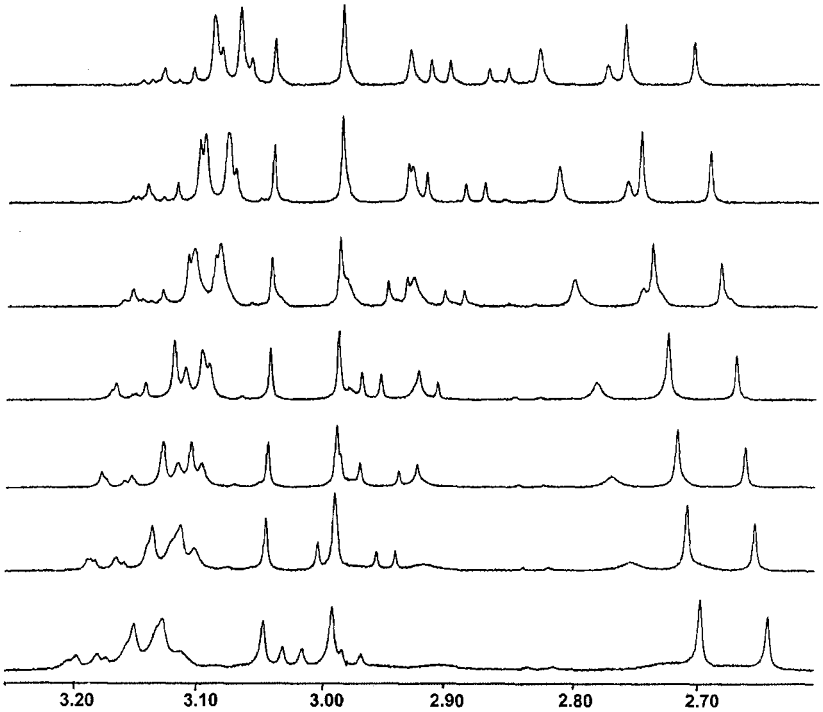

Figure 2. $H_{a\text{-}d}$ protons of compound **3b** at varying temperatures [−10, 0, 10, 20, 30, 40, 50 °C from bottom to top]

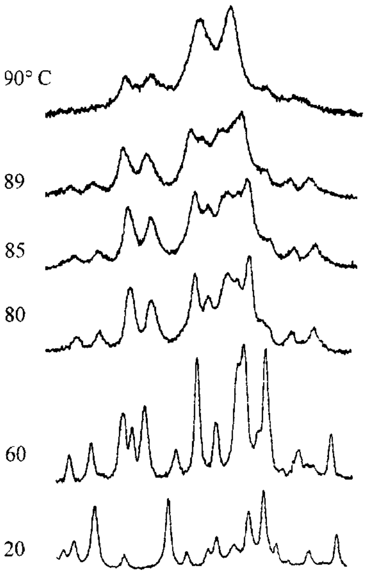

Figure 3. Compound **3c** at varying temperature: $H_{a\text{-}b}$ protons between $\delta = 3.5$ and 3.9 in C_5D_5N

T is the coalescence temperature; $\Delta\nu$ is the chemical shift difference of merging signals measured far below coalescence temperature.

$\Delta G^{\neq}$ values for interconversions of isomers **3** (see Table 5) are all in the range of 78−80 kJ/mol (18−19 kcal/mol).

They do not depend greatly on the nature of the moieties at the *para* positions of the benzene rings of the succinimide fragments, probably because there is a large distance between moiety and rotating bond. For **3d**, a change in solvent results in an increase of the coalescence temperature to 83 °C in deuteriopyridine and 90 °C in $[D_6]$DMSO (corrected temperatures), but this difference is erased in $\Delta G^{\neq}$ calculations because of compensation by $\Delta\nu$.

The values of the conversion barriers are comparable to those for atropisomers of biphenyls (16−20 kcal/mol)[18,19] and significantly lower than *cis*/*trans* conversion barriers (40 kcal/mol for 2-butene).[18]

In conclusion, the compounds **3** obtained by the new rearrangement described in Scheme 1 exist as two isomers in solution. Their interconversion process proceeds through atropisomerism rather than *E*/*Z* exchange. Their structures were also confirmed by X-ray scattering, which further

Table 5. Coalescence temperatures and $\Delta G^{\#}$ values for **3a**, **3c** and **3e**

	[a]	Solvent	$\Delta\nu$ Hz	T_c °C[b]	T_c K	$\Delta G^{\#}$ kJ/mol	$\Delta G^{\#}$ kcal/mol
3a	H_a	C_5D_5N	9	83	356	78.81	18.83
3c	Hα(Py)	C_5D_5N	14	88	361	78.64	18.79
3e	H_a	C_5D_5N	6	83	356	80.01	19.12
3e	Hα(Py)	$[D_6]$DMSO	13	90	363	79.31	18.95

[a] Observed signals. − T_c corrected coalescence temperature.

demonstrated that these compounds are sensitive to the *p*-substitution pattern on the phenyl in the maleimide moiety. Thus they should serve as model examples for research into new distorted double bond compounds.

Experimental Section

Compounds **3** were obtained according to ref.[12] ^{1}H NMR spectra were recorded at 200, 250, 300 or 400 MHz. Temperature dependencies of ^{1}H NMR spectra were studied in $CDCl_3$ solutions of **3** at 400 MHz spectrometer for a temperature range of −10 to +50 °C, and in C_5D_5N and [D_6]DMSO solutions of **3** at 250 MHz spectrometer for a temperature range of +20 to +100 °C. When the coalescence point was reached, temperatures was measured and corrected. Corrected values were used for calculations of conversion barriers.

Crystallographic Data: Crystals of compound **3a** ($C_{32}H_{23}N_3O_4$) are triclinic. At 20 °C a = 10.260(2) Å, b = 11.165(2) Å, c = 11.289(2) Å, α = 97.63(3)°, β = 100.50(3)°, γ = 93.91(3)°, V = 1254.6(4) Å^3, $d_{calcd.}$ = 1.359 g/cm^3, space group P1(bar), Z = 2, F(000) = 536, μ = 0.09mm^{-1}. Intensities of 5763 unique reflection (R_{inf} = 0.064), were measured using Rigaku R05 diffractometer (Mo-K_α radiation, θ/2θscan, 2θmax = 60°).

Crystals of compound **3b** are triclinic ($C_{34}H_{27}N_3O_4$). At 20 °C a = 11.189(5) Å, b = 11.876(7) Å, c = 12.412(6) Å, α = 92.95(4)°, β = 100.73(4)°, γ = 104.77(4)°, V = 1558(1) Å^3, $d_{calcd.}$ = 1.154 g/cm^3, space group P1(bar), Z = 2, F(000) = 568, μ = 0.08 mm^{-1}.

Intensities of 5078 unique reflections (R_{inf} = 0.059), were measured using Siemens P3/PC diffractometer (Mo-K_α radiation, graphite monochromator, θ/2θ-scan, 2θmax = 60°).

Structures were found by direct method using programs SHELXL-97.[20]

Hydrogen atoms locations were calculated geometrically and refined with fixed $U_{iso} = nU_{eq}$ non-hydrogen atom connected to a given atom of hydrogen (n = 1,5 for methyl groups and n = 1,2 for the other hydrogen atoms).

Bond lengths, valence angles and the most important torsion angles are given in Table 1−3. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-152391 for **3a** and CCDC-147299 for **3b**. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB1 1EZ, UK [Fax: (internat.) + 44−1223/336−033; E-mail: deposit@ccdc.cam.ac.uk].

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