Synthesis of bis-oxathiaaza[3.3.3]propellanes via nucleophilic addition of (1,ω-alkanediyl)bis(N'-organylthioureas) on dicyanomethylene-1,3-indanedione

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Abstract
A concise and efficient route for synthesis of bis-oxathiaaza[3.3.3]propellanes by reaction of N,N,-N''-(1,ω-alkanediyl)bis-(N''-organylthioureas) with (1,3-dioxo-2,3-dihydro-1H-inden-2-ylidene)propanedinitrile is reported. The structures of the products have been confirmed by using NMR as well as single crystal X-ray analysis for one product. A plausible mechanism for formation of the products is presented.

Keywords: (1,ω-Alkanediyl)bis(N''-organylthioureas), dicyanomethylene-1,3-indanedione, bis-oxathiaaza[3.3.3]propellanes, nucleophilic addition, X-ray analysis

Introduction
Propellane is considered as annulated tricyclic systems,1 with skeletons occupying a privileged place in synthetic organic chemistry. The propellane moiety is present in various biologically active and natural products.2-5 The natural products including propellanes have been evaluated for their anticancer6 and antifungal7 activities.

Sequential reactions of ninhydrin, malononitrile, primary amines and dialkyl acetylenedicarboxylates has been used for synthesis of polysubstituted heterocyclic[3.3.3]propellanes.3 Upon mixing arylisothiocyanates, ninhydrin and malononitrile in the presence of NaH in DMF, oxaaaza[3.3.3]propellanes have been formed.8 Using simple regioselective multicomponent reactions of ninhydrin, malononitrile, hydrazine derivatives and β-ketoesters or
dimethyl acetylenedicarboxylate, oxaaaza[3.3.3]propellanes were synthesized.\textsuperscript{9} Oxathiaaza[3.3.3]propellane derivatives were prepared by reaction of symmetrical thioureas with ninhydrin and malononitrile.\textsuperscript{10}

Alizadeh et al.\textsuperscript{11} reported that the reaction of ninhydrin with malononitrile gave a Knoevenagel adduct, which was trapped \textit{in situ} by various ketene aminals through conjugate addition and cyclization to give oxaaaza[3.3.3]propellanes 3 and 4 (Scheme 1).\textsuperscript{11}

\begin{center}
\includegraphics[width=0.7\textwidth]{scheme1}
\end{center}

\textbf{Scheme 1.} Reaction between diamines, primary amines, nitroketene, dithioacetal(1,1-bis(methylsulfanyl)-2-nitroethene)ninhydrin and malononitrile.\textsuperscript{11}

Thiourea derivatives are extremely versatile starting materials for the synthesis a wide variety of heterocyclic compounds. Among the most important heterocyclization reactions of thioureas are the condensations with $\alpha$-halocarbonyl compounds to give substituted 1,3-thiazoles.\textsuperscript{12-14} The reaction between aroylthiourea derivatives with dicyanomethylene indane-1,3-dione furnished indenothiazepines.\textsuperscript{15}

Symmetrical and unsymmetrical 2,5-dithiobiureas act as a key for the synthesis of many organic heterocyclic ring systems. Pyrazole, thiadiazole and thiazepine derivatives were isolated during the reaction between dithiourea and thioureidoethylthiourea derivatives with tetracyanoethylene.\textsuperscript{16} Thioureidoethyl- and propylthioureas reacted with mercury bis(phenylacetylide) afforded imidazolidine derivatives.\textsuperscript{17} 1,3,6-Thiadiazepane-3-thione can be formed via interaction thioureidothioureas with chloranil or bromanil.\textsuperscript{18}

Recently, a new series of bis-thiazoilidin-4-ones were synthesized from $N,N,N''$-(1,ω-alkanediyl)bis($N''$-organyltiourea) derivatives with dimethyl acetylenedicarboxylate.\textsuperscript{19}

\section*{Results and Discussion}

As part of our investigations of the interaction of carbothioamides with $\pi$-deficient compounds\textsuperscript{20-22} we herein report a tandem method for synthesis of bis-oxathiaaza[3.3.3]-propellanes by the reaction of $N,N,N''$-(1,ω-alkanediyl)bis($N''$-organyltiourea) derivatives 5a-e with dicyanomethylene-1,3-indanedione 6.
Treatment of 5a-e with two molecular equivalents of 6 in THF as solvent under reflux, gave nearly quantitative conversion after 10-14 hours. Bisoxathiaaza[3.3.3]propellanes 7a-e were precipitated as colourless major products 67-74\% (Scheme 2). From the filtrate, imidazolothione or primidinethione (9-12\%), together with 1,3-dihydroxyindan-2-ylidenepropandinitrile was formed in yields varying between 5-7\%.

The molecular structures of products 7a-e were elucidated from their mass spectrometric analyses, IR, $^1$H NMR and $^{13}$C NMR spectra, for example 7a. The mass spectrum of 7a displayed the molecular ion peaks at $m/z$ 746 which is in agreement with the proposed structure and which clearly shows the addition of one molecule of 5a to one molecule of 6 without any elimination. The IR spectrum of 7a shows absorption bands at 3321 and 3208 cm$^{-1}$ due to NH$_2$, sharp band at 2186 cm$^{-1}$ and four absorption bands 1734, 1624, 1589 and 1099 cm$^{-1}$ relating to C≡N, C=O, C=N, Ar-C=C and C-O-C stretching frequencies clearly indicated on the most significant functional groups of 7a.

In the $^1$H NMR spectrum of 7a, the NH$_2$ protons appeared as broad signal with two protons at 8.36 ppm, one multiplet at 3.58 due to NCH$_2$-CH$_2$N. Aromatic protons gave rise to characteristic signals in the aromatic region of the spectrum at 7.05-8.05 ppm.

<table>
<thead>
<tr>
<th>5,7</th>
<th>R</th>
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<th>Yield of 7a-e</th>
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<tr>
<td>a</td>
<td>Ph</td>
<td>2</td>
<td>74 %</td>
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<tr>
<td>b</td>
<td>Allyl</td>
<td>2</td>
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<tr>
<td>c</td>
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<td>3</td>
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<td>d</td>
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<td>3</td>
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<tr>
<td>e</td>
<td>Ethyl</td>
<td>3</td>
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**Scheme 2.** (Synthesis of bis-oxathiaaza[3.3.3]propellanes).

In the $^{13}$C NMR spectrum of 7a, thiazole-C5 resonated at 71.11 ppm, further peaks are at $\delta$c 53.29 (furan-C3), 165.76 (furan-C2) are in accordance with the observed trends in $\delta$ values for carbon atoms in push-pull alkenes,$^{23,24}$ $^{13}$C NMR shows signals at 115.82 (CN), 192.44 (indene-C=O).

Compounds 5a-e may react at least with their Sulphur atom, and NH's as nucleophilic sites. Several alternative structures **could** be excluded on the basis of $^{13}$C NMR spectrum and absence
of C=S signal in 7a-e. Without reference compounds, it would not be easy to compare the $^1$H NMR or $^{13}$C NMR chemical shifts for possible sets of isomers 7 and it would not be easy to assess the correct structure just from spectroscopic data.

The structure of 7a with crystallographic C$_i$-symmetry was unequivocally resolved by X-ray crystallography (Fig. 1) and Tables S1-S7 in the supplementary data (note that the crystallographic numbering does not correspond to systematic IUPAC numbering rules). The C4-C(12) bond length of 1.550(2) Å has a C-C single bond character and is shared by three different rings (C4-C12-S1-C2-N3/C4-C12-O15-C14-C13/and C4-C12-C5-C10-C11) in the three dimensional structure to form the propellane system. The angles between the planes S1-C2-N3-C4-C12/C4-C12-O15-C14-C13 62.5(1)$^\circ$ and C4-C12-C13-C15-O15/C4-C12-C11-C10-C5 64.4(1)$^\circ$ and C4-C12-S1-C2-N3/C4-C12-C11-C10-C5 53.6(1)$^\circ$.

**Figure 1.** Molecular structure of 7a in the crystal (displacement parameters are drawn at 50% probability level). The crystallographic numbering does not reflect the systematic IUPAC numbering.

As a result, it was found that solvent, temperature and molar ratio of reactants may all play a critical role on the reaction efficiency. Different solvents were used and studied their effect on the
reaction pathway, tetrahydrofuran (THF) was a superior solvent compared to ethyl acetate, DMF, CH$_3$CN and ethyl alcohol.

Two molecules of 6 were necessary to obtain the products in high yield. Traces of the products were formed upon applying this reaction at room temperature for long time. Subjecting the reaction under reflux in THF for 14 hours, satisfied yield of the products were observed.

Based on these results, a plausible mechanism for the formation of products 7a-e has been proposed (Scheme 3). In order to rationalize the formation of propellanes 7a-e, the bithioureas with initially attack the C=C of 6 via nucleophilic sulfur atom to form the intermediate 8, followed by addition another molecule of 6 afforded the adduct 9. Attack of NH on C=O gave the intermediate 10. Attack of the formed OH on one of the cyano groups followed by tautomerism of the imine to enamine gave the more stable propellanes 7a-e.

![Scheme 3. A plausible mechanism for the formation of 7a-e.](image)

**Conclusions**

We report on a novel series bis-oxathiaaza[3.3.3]propellanes via nucleophilic addition of (1,ω-alkanediyl)bis(N'-organylthioureas) on (1,3-dioxo-2,3-dihydro-1H-inden-2-ylidene)propane-dinitrile. The symmetrical 2,5-dithiobiureas required the availability of four NH's as well as two sulfur atoms as nucleophilic sites.
Experimental Section

General. Melting points (uncorrected) and were determined using open glass capillaries on Gallenkamp melting point apparatus. IR spectra: as KBr pellets on Shimadzu 408 or Alpha, Bruker FT-IR instruments, ν (cm⁻¹) 1H NMR (400 MHz) and 13C NMR (100 MHz) spectra: Bruker AM 400 spectrometer with tetramethyldisilane as internal standard, s = singlet, m = multiplet, t = triplet, q = quartet, br = broad. The 13C NMR signals were assigned on the basis of DEPT 135/90 spectra. Chemical shifts were expressed as δ (ppm). EI-Mass was recorded on Finnigan-MAT 8430 mass spectrometer at an ionization potential of 70 eV. Elemental analyses for C, H, N and S: Carried out at the Microanalytical Center, Cairo University, Egypt. Preparative layer chromatography was run at 48 cm wide and 20 cm tall glass plates covered with a 1.0 mm thick layer of slurry, air dried silica gel Merck PF254.

N,N"-(1,ω-Alkanediyl)bis(N"-organylthiourea) derivatives 5a-e were prepared by the reaction of the diamine (1,2-diaminoethane, 1,3-diaminopropane) with ethyl-, phenyl-, benzyl-, or allyl-isothiocyanate in DMF according to published procedures in literature: 5a, 5b, 5c, 5d, 5e (1,3-Dioxo-2,3-dihydro-1H-inden-2-ylidene)propanedinitrile (6) was prepared by using method by Chatterjee. 20

Reaction of N,N,N" -(1,ω-Alkanediyl)bis-(N"-organylthioureas) 5a-e with (6). A solution of (1,ω-alkanediyl)bis(N"-organylthioureas) 5a-e (5a: 0.330 g, 1.0 mmol, 5b: 0.258 g, 1.0 mmol, 5c: 0.344 g, 1.0 mmol, 5d: 0.372 g, 1.0 mmol, 5e: 0.248 g, 1.0 mmol) in dry tetrahydrofuran (THF) (25 ml) was added dropwise with stirring at room temperature to 6 (0.416 g, 2.0 mmol) in THF (20 ml). The reaction mixture was gently refluxed with stirring for 12 h (in case of 7a, 7e), 14 h, (in case of 7d) and 10 h (in case of 7b, 7e). The resulting colourless precipitate containing compounds (7a-e) was filtered off, washed with THF and recrystallized from suitable solvent. The filtrate was concentrated and the residue was then separated by preparative layer chromatography (plc), using toluene / ethyl acetate (10:7) in case of (5a, 5c, 5e with 6) and (10:8) in case of (5d, 5b with 6) as eluent to give numerous coloured zones, the most migrating zone, which quenched all indicators fluorescence upon exposure to 254 nm UV light, contained compounds (Imidazolidine-2-thione and 1,3-diazinane-2-thione). The slowest migrating zone contained (1,3-dihydroxyindan-2-ylidene)propanedinitrile. Extraction of the zones with acetone gave pure compounds.

(3aS,3a'S,8bR,8b'R,10Z,10'Z)-9,9'-((Ethane-1,2-diyl)bis(2-amino-4-oxo-10-(phenylimino)-4H-3a,8b-(epithiomethanoimino)indeno-[1,2-b]furan-3-carbonitrile) (7a). Recrystallization with (acetonitrile/DMF) gave colourless crystals, (0.553 g, 74%); mp 260-262 °C. IR (KBr, ν, cm⁻¹): 3321, 3208 (NH₃), 2186 (CN), 1733 (C=O), 1624 (C≡N), 1589 (Ar-C≡C), 1099 (C-O-C) cm⁻¹. δH (400 MHz, DMSO-d₆) 3.58 (s, 4H, 2 CH₂N), 7.05-7.10 (m, 4H, Ar-H), 7.20-7.26 (m, 4H, Ar-H), 7.40-7.45 (m, 4H, Ar-H), 7.82-7.85 (m, 2H, Ar-H), 7.97-8.05 (m, 4H, Ar-H), 8.36 (br, s, 4H, 2 NH₂). δC (100 MHz, DMSO-d₆) 38.88 (CH₂N), 53.29 (furan-C₃), 71.11 (thiazolidine-C₅), 106.86 (furan-C₅), 115.82 (CN), 121.53, 124.51, 125.20, 125.74, 127.88, 129.42, 131.74 (Ar-CH),
137.74, 142.78, 149.32 (Ar-C), 155.69 (C=N), 165.76 (furan-C2), 192.44 (indenone-CO). m/z (%) 746 (M+, 9), 718 (17), 694 (12), 614 (26), 564 (37), 135 (76), 91 (52), 77 (100). Anal. Calcd for C_{40}H_{26}N_{9}O_{8}S_{2} (746.82), C, 64.33; H, 3.51; N, 15.00; S, 8.59. Found: C, 64.19; H, 3.62; N, 14.87; S, 8.43%.

(3aS,3a'S,8bR,8b'R,10Z,10'-Z)-9,9'-((Ethane-1,2-diyl)bis(10-allylimino)-2-amino-4-oxo-4H-
3a,8b-(epithiomethanoimino)indenol[1,2-b]furan-3-carbonitrile) (7b). Recrystallization with (acetonitrile/DMF) gave colourless crystals, (0.470 g, 70%); mp 286-288 °C. IR (KBr, cm⁻¹): 3328, 3268 (NH₂), 2212 (CN), 1734 (C=O), 1620 (C=N), 1592 (Ar-C=C), 1093 (O-C-O) cm⁻¹. δH (400 MHz, DMSO-d₆) 3.52 (s, 4H, 2 CH₂N), 4.28-4.30 (br, 4H, 2 allyl-CH₂N), 5.22-5.24 (m, 4H, 2 allyl-CH₂=), 5.91-5.93 (m, 4H, 2 allyl-CH=), 7.30-7.36 (m, 2H, Ar-H), 7.54-7.61 (m, 2H, Ar-H), 7.81-7.88 (m, 2H, Ar-H), 7.98-8.08 (m, 2H, Ar-H), 8.51 (br, s, 4H, 2 NH₂). δC (100 MHz, DMSO-d₆) 38.73 (CH₂N), 44.67 (allyl-CH₂N), 53.16 (furan-C3), 71.27 (thiazolidine-C5), 107.05 (furan-C5), 116.14 (CN), 117.93 (allyl-CH₂=), 124.62, 127.55, 128.91, 129.45 (Ar-CH), 135.27 (allyl-CH=), 137.51, 142.25 (Ar-C), 155.76 (C=N), 165.64 (furan-C2), 192.51 (indenone-CO). m/z (%) 674 (M⁺, 13), 646 (8), 622 (23), 564 (35), 542 (28), 104 (62), 55 (37), 41 (100). Anal. Calcd for C_{34}H_{26}N_{9}O_{8}S_{2} (674.75), C, 60.52; H, 3.88; N, 16.61; S, 9.50. Found: C, 60.71; H, 3.96; N, 16.44; S, 9.39%.

(3aS,3a'S,8bR,8b'R,10Z,10'-Z)-9,9'-((Propane-1,3-diyl)bis(2-amino-4-oxo-10-(phenylimine)-4H-
3a,8b-(epithiomethanoimino)indenol[1,2-b]furan-3-carbonitrile) (7c). Recrystallization with (acetonitrile/DMF) gave colourless crystals (0.563g, 73%); mp 270-272 °C. IR (KBr, cm⁻¹): 3332, 3274 (NH₂), 2210 (CN), 1728 (C=O), 1632 (C=N), 1589 (Ar-C=C), 1090 (O-C-O) cm⁻¹. δH (400 MHz, DMSO-d₆) 2.54 (m, 2H, propane-CH₂), 3.83 (t, 4H, CH₂N, J 7.66), 7.12-7.16 (m, 4H, Ar-H), 7.24-7.29 (m, 4H, Ar-H), 7.44-7.49 (m, 4H, Ar-H), 7.86-7.89 (m, 2H, Ar-H), 8.02-8.07 (m, 4H, Ar-H), 8.48 (br, s, 4H, 2 NH₂). δC (100 MHz, DMSO-d₆) 29.05 (propane-CH₂), 41.12 (CH₂N), 52.96 (furan-C3), 71.27 (thiazolidine-C5), 106.61 (furan-C5), 116.08 (CN), 122.12, 125.41, 125.83, 126.52, 127.68, 129.53, 130.88 (Ar-CH), 136.92, 141.93, 148.56 (Ar-C), 155.33 (C=N), 165.44 (furan-C2), 191.96 (indenone-CO). m/z (%) 760 (M⁺, 9), 704 (14), 628 (26), 490 (46), 135 (83), 77 (100). Anal. Calcd for C_{41}H_{28}N_{9}O_{8}S_{2} (760.84), C, 64.72; H, 3.71; N, 14.73; S, 8.43. Found: C, 64.86; H, 3.62; N, 14.59; S, 8.55%.

(3aS,3a'S,8bR,8b'R,10Z,10'-Z)-9,9'-((Propane-1,3-diyl)bis(2-amino-10-(benzylimino)-4-oxo-
4H-3a,8b-(epithiomethanoimino)indenol[1,2-b]furan-3-carbonitrile) (7d). Recrystallization with (acetonitrile/DMF) gave colourless crystals, (0.567g, 72%); mp 295-298 °C. IR (KBr, cm⁻¹): 3332, 3269 (NH₂), 2196 (CN), 1730 (C=O), 1625 (C=N), 1600 (Ar-C=C), 1086 (O-C-O) cm⁻¹. δH (400 MHz, DMSO-d₆) 2.52 (m, 2H, propane-CH₂), 3.77 (t, 4H, 2 CH₂N, J 7.69), 4.83 (s, 4H, CH₂Ph), 7.05-7.09 (m, 4H, Ar-H), 7.16-7.21 (m, 2H, Ar-H), 7.38-7.45 (m, 4H, Ar-H), 7.55-7.62 (m, 2H, Ar-H), 7.68-7.76 (m, 2H, Ar-H), 7.96-8.06 (m, 4H, Ar-H), 8.46 (br, s, 4H, 2 NH₂). δC (100 MHz, DMSO-d₆) 28.33 (propane-CH₂), 41.68 (CH₂N), 47.83 (CH₂Ph), 53.09 (furan-C3), 71.14 (thiazolidine-C5), 106.93 (furan-C5), 116.22 (CN), 122.86, 124.53, 125.89, 127.64, 128.33, 129.74, 131.18 (Ar-CH), 134.22, 137.66, 142.19 (Ar-C), 155.22 (C=N), 165.52 (furan-C2), 192.37 (indenone-C=O). m/z (%) 788 (M⁺, 11), 746 (14), 656 (26), 490 (46), 149 (76), 91 (100), 77 (100).

(3aS,3a'S,8b'R,8b'R,10Z,10'Z)-9,9'-(Propane-1,3-diyl)bis(2-amino-10(ethylimino)-4-oxo-4H-3a,8b-(epithiomethanoimino)indeno[1,2-b]furan-3-carbonitrile) (7e). Recrystallization with (acetonitrile/DMF) gave colourless crystals (0.458 g, 69 %); mp 238-240 °C. IR (KBr, ν, cm⁻¹): 3328, 3276 (NH₂), 2194 (CN), 1726 (C=O), 1630 (C=N), 1588 (Ar C=C), 1088 (C–O–C) cm⁻¹. δH (400 MHz, DMSO-d₆) 1.32 (t, 6H, 2 CH₃, J 7.64 Hz), 2.53 (m, 2H, propane-CH₂), 3.57 (q, 4H, 2 CH₂, J 7.60 Hz), 3.84 (t, 4H, 2CH₂N, J 7.60 Hz), 7.51-7.59 (m, 2H, Ar-H), 7.65-7.68 (m, 2H, Ar-H), 7.85-7.95 (m, 2H, Ar-H), 8.01-8.11 (m, 2H, Ar-H), 8.42 (br, s, 4H, 2NH₂). δC (100 MHz, DMSO-d₆) 15.84 (CH₃), 28.61 (propane-CH₂), 41.12 (propane-CH₂N), 49.63 (CH₂), 53.19 (furan-C₃), 71.34 (thiazolidine-C₅), 106.93 (furan-C₅), 116.23 (CN), 124.74, 127.61, 128.82, 129.64 (Ar-CH), 136.92, 141.86 (Ar-C), 155.88 (C=N), 165.58 (furan-C₂), 192.48 (indeno-CO). m/z (%) 664 (M⁺, 9), 606 (12), 532 (28), 490 (32), 104 (73), 87 (87), 43 (100). Anal. Calcd for C₃₃H₂₈N₈O₄S₂ (664.76): C, 59.62; H, 4.25; N, 16.86; S, 9.65. Found: C, 59.51; H, 4.32; N, 17.02; S, 9.76 %.

Imidazolidine-2-thione and 1,3-diazinane-2-thione were made according to literature procedures.¹³¹

**Single crystal X-ray structure determination of 7a.**

Single crystal X-ray diffraction study was carried out on an Agilent Super Nova diffractometer at 173 K with EOS-detector and MoKα radiation (λ, 0.71073 Å). Direct Methods (SHELXS-97) were used for structure solution and refinement was carried out using SHELXL-2013 (full-matrix least-squares on F²). Hydrogen atoms were localized by difference Fourier Synthesis map and refined using a riding model [H (N) free]. A semi-empirical absorption correction and an extinction correction were applied.

**Compound 7a.** C₄₀H₂₆N₈O₄S₂ ⋅ 2 (C₃H₇NO), Mr 893.00 gmol⁻¹, colorless plates, crystal size 0.30 × 0.02 × 0.10 mm, triclinic, P-1 (no. 2), a = 9.7515 (5) Å, b = 9.8024 (8) Å, c = 13.4038 (8) Å, α = 90.279 (6)°, β = 102.516 (5)°, γ = 118.268 (7), V = 1092.98(14) Å³, Z = 1, Dcalcd = 1.357 Mg m⁻³, F(000) = 466, μ = 0.184 mm⁻¹, T = 173 K, 7638 measured reflections (2θmax = 55°), 4954 independent reflections (Rint. = 0.016) 298 parameters, 2 restraints, R1 (for 3998 I > 2σ(I)) = 0.044, wR² (for all data) = 0.107, S = 1.03, largest diff. peak and hole = 0.35 eÅ⁻³, -0.300 eÅ⁻³. Crystallographic data (excluding structure factors) for the structure reported in this work have been deposited with Cambridge Crystallographic Data Center on supplementary publication no CCDC-1417181. Copies of the data can be obtained free of charge on publication to the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax:+44(1223)336033e-mail:deposit@ccdc.cam.ac.uk).
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