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Syntheses and characterization of $\text{Pb}(\text{trz})_n\text{X}_2$ ($\text{X} = \text{CH}_3\text{COO}^-$, NCS^- , and $n = 1, 2$) complexes, and crystal structure of $[\text{Pb}(\text{trz})_2(\text{MeOH})](\text{ClO}_4)_2 \cdot \text{H}_2\text{O}$

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Syntheses and characterization of $\text{Pb}(\text{trz})_n\text{X}_2$ ($\text{X} = \text{CH}_3\text{COO}^-$, NCS^- , ClO_4^- and $n = 1, 2$) complexes, and crystal structure of $[\text{Pb}(\text{trz})_2(\text{MeOH})](\text{ClO}_4)_2 \cdot \text{H}_2\text{O}$

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Lead(II) complexes with 2,4,6-tris(2-pyridyl)-1,3,5-triazine (trz) have been synthesized using a direct synthetic method and characterized by IR and ^{207}Pb NMR spectroscopy and CHN elemental analysis. The structure of $[\text{Pb}(\text{trz})_2(\text{MeOH})](\text{ClO}_4)_2 \cdot \text{H}_2\text{O}$ was confirmed by X-ray crystallography. Single-crystal X-ray data for $[\text{Pb}(\text{trz})_2(\text{MeOH})](\text{ClO}_4)_2 \cdot \text{H}_2\text{O}$ show the complex to be monomeric with the Pb having an unsymmetrical seven-coordinate geometry, coordinated by six nitrogen atoms of the trz ligands and one oxygen atom of MeOH. The arrangement of the ligands in the $[\text{Pb}(\text{trz})_2(\text{MeOH})](\text{ClO}_4)_2 \cdot \text{H}_2\text{O}$ complex exhibits a coordination gap around the Pb(II), occupied possibly by a stereoactive lone pair of electrons on lead(II); the coordination around the lead atoms is hemidirected.

Keywords: Lead(II); Triazine; Stereoactive lone pair; Seven-coordinate complex

1. Introduction

The presence of hard donor atoms such as nitrogen and oxygen in the coordination sphere of Pb(II) ion may allow significant involvement of the 6s orbital in the valence shell. This lone pair will contain p orbital character and, accordingly, produce a gap in the coordination sphere where the lone pair is stereochemically active. Understanding the factors that control the stereoactivity of the lone pair in lead(II) chemistry may be important in the design of complexing agents that can remove toxic metals selectively from biological systems [1]. Details of the coordination chemistry of lead(II) compounds with N- and O-donor ligands regarding the coordination and stereoactivity of the valence-shell lone electron pairs have been reported [1–5]. At various coordination numbers and coordination geometries, the lone pair of electrons has influence on the structure of the complexes. Recent structural studies of lead(II) compounds [1], in particular, have provided a basis for detailed analysis of the evidence for coordination sphere distortions as a consequence of such electron pairs. The lone pair activity

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depends on the following factors: (1) low *versus* high coordination number, (2) hard *versus* soft ligands, (3) attractive *versus* repulsive interactions among ligands, (4) whether or not the lone pair has p character, and (5) electron donation from ligands to metal [1]. In the present article, we describe the syntheses and characterization of 1 : 1 and 1 : 2 lead(II) complexes of the 2,4,6-tris(2-pyridyl)-1,3,5-triazine (trz) ligand with different counterions, $\text{Pb}(\text{trz})_n\text{X}_2$ ($\text{X} = \text{CH}_3\text{COO}^-$, NCS^- , ClO_4^- and $n = 1, 2$).

2. Experimental

2.1. Physical measurements

IR spectra were recorded as nujol mulls using Perkin-Elmer 597 and Nicolet 510P spectrophotometers. Microanalyses were carried out using a Heraeus CHN-O Rapid analyzer. Melting points were measured on an Electrothermal 9100 instrument and are uncorrected. ^{207}Pb solution NMR spectra were recorded on a Bruker DRX-500 Avance spectrometer at 104.6 MHz using a 5-mm broad-band probe. Lead chemical shifts are reported (ppm) downfield from tetramethyllead using PbPh_4 ($\delta_{\text{Pb}} = -178.0$ ppm, saturated in CDCl_3) as an external standard, 0.01 M at 25°C. Each lead spectrum was obtained in 3–10 h.

2.2. Preparation of $[\text{Pb}(\text{trz})_2(\text{MeOH})](\text{ClO}_4)_2 \cdot \text{H}_2\text{O}$

The $[\text{Pb}(\text{trz})_2(\text{MeOH})](\text{ClO}_4)_2$ complex was prepared by dissolving lead(II) acetate (0.36 g, 1 mmol) and sodium perchlorate (0.367 g, 3 mmol) in distilled water and adding an alcoholic (MeOH) solution of trz (0.927 g, 3 mmol). The resulting solution was stirred for 2 h at room temperature, and then allowed to stand for 2–3 days in a refrigerator (about 6°C). Yellow crystals (m.p. > 320°C) of the product precipitated out. The product was filtered, washed with acetone and ether and air dried (0.739 g, yield 70%) Anal. Calcd for $\text{C}_{43}\text{H}_{35}\text{N}_6\text{Cl}_2\text{O}_{10}\text{Pb}$ (%): C, 48.00; H, 3.25; N, 7.81. Found: C, 48.23; H, 3.25; N, 7.7%. Selected IR(cm^{-1}) bands: 620 (s), 740 (s), 1010 (vs), 1545 (s), 1620 (s), 3040 (w). ^{207}Pb NMR (DMSO, δ): -1590.2 ppm.

2.3. Preparation of $[\text{Pb}(\text{trz})(\text{O}_2\text{CCH}_3)_2]$

Lead(II) acetate (0.36 g, 1 mmol) was dissolved in hot water and added dropwise with stirring to an aqueous solution of trz (0.31 g, 1 mmol). The resulting solution was stirred for 2 h at room temperature. On standing, the product precipitated out and was collected by filtration, washed with a little ice-cold water and recrystallized from a concentrated aqueous solution. The pure product was washed with ice-cold ethanol then diethylether before drying in air. Yield 0.328 g, 50%; m.p. 180°C. Anal. Calcd for $\text{C}_{25}\text{H}_{22}\text{N}_3\text{O}_4\text{Pb}$ (%): C, 47.16; H, 3.46; N, 6.60. Found: C, 47.20; H, 3.50; N, 6.75. IR(cm^{-1}) selected bands: 740 (s), 1010 (s), 1360 (vs), 1545 (vs), 1620 (s), 2991 (w), 3040 (w). ^{207}Pb NMR (DMSO, δ): -886.2 ppm.

2.4. Preparation of $[\text{Pb}(\text{trz})(\text{NCS})_2]$

The $[\text{Pb}(\text{trz})(\text{NCS})_2]$ complex was prepared by the branch tube method: trz (0.31 g, 1 mmol) was placed in one arm of the branched tube and a mixture of lead(II)

acetate (0.36 g, 0.5 mmol) and potassium thiocyanate (0.097 g, 1 mmol) in the other. Methanol was carefully added to fill both arms, then the tube was sealed and the ligand-containing arm immersed in a bath at 60°C, while the other arm was at ambient temperature. After 15 days, white crystals (m.p. 209°C) had deposited in the cooler arm. Yield: 0.294 g, 45%. Anal. Calcd for $C_{23}H_{16}N_3PbS_2$ (%): C, 42.20; H, 2.44; N, 10.70. Found: C, 42.70; H, 2.40; N, 10.50. Selected IR(cm^{-1}) bands: 820 (m), 1150 (m), 1460 (s), 1540 (s), 1620 (s), 2025 (vs), 3040 (w). ^{207}Pb NMR (DMSO, δ): -1427.06 ppm.

2.5. Determination of the structure

Crystallographic measurements were carried out at 120(2) K using a Siemens R3m/V diffractometer. The intensity data were collected within the range $1.94^\circ \leq \theta \leq 30.06^\circ$ using graphite-monochromated $Mo K\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$). Accurate unit cell parameters and the orientation matrix for data collection was obtained from a least-squares refinement. Intensities of 46 443 unique reflections were measured, from which 9045 with $I > 2\sigma(I)$ were used in the refinement. The structure was solved by direct methods and refined by full-matrix least-squares techniques on F^2 . The positions of hydrogen atoms were idealized and included in the calculations of the structure factors as fixed contributions. Each hydrogen atom was assigned an isotopic thermal parameter. R and R_w , with goodness of fit on F^2 of 1.099, are 0.0577 and 0.1387, respectively. The final difference density map showed a maximum peak and hole of 2.410, -2.400. Corrections for the Lorentz and polarization effects as well as the empirical correction for absorption using the Sadabs programs were applied. All structural calculations were carried out with a PDP-11/23+ computer using the SDP-PLUS program package [6, 7].

Crystal data and structure refinement are given in table 1. Selected bond lengths and angles are given in table 2. Anisotropic thermal parameters, observed and calculated structure factors, full lists of bond distances, bond angles and torsion angles are given in the supplementary material. ORTEP diagrams and a perspective view of the packing in the unit cell are shown in figures 1 and 2, respectively.

3. Discussion

3.1. Synthesis

Reaction between $Pb(CH_3CO_2)_2$ and trz , $Pb(CH_3COO)_2$ and trz and potassium thiocyanate, and also $Pb(CH_3CO_2)_2$ with trz and sodium perchlorate provided powdery materials that analyzed as $[Pb(trz)(CH_3CO_2)_2]$, $[Pb(trz)(NCS)_2]$ and $[Pb(trz)_2(MeOH)](ClO_4)_2 \cdot H_2O$, respectively.

The IR spectrum of the $[Pb(trz)_2(MeOH)](ClO_4)_2 \cdot H_2O$ complex shows $\nu(ClO_4)$ at about 1010 cm^{-1} . The characteristic bands of the acetate anions appear at about $1545 \nu_{as}(C-O)$ and $1360 \nu_{sym}(C-O) \text{ cm}^{-1}$ for the $[Pb(trz)(CH_3CO_2)_2]$ complex. The Δ value ($\nu_{as} - \nu_{sym} = 185 \text{ cm}^{-1}$) indicates that the acetate anions are coordinated to the Pb^{II} center in a bidentate mode [8, 9]. The IR spectrum of the $[Pb(trz)(NCS)_2]$ complex shows $\nu(SCN)$ at ca 2025 cm^{-1} , similar to other lead(II) complexes including $[Pb(phen)_2(NO_3)(NCS)]$ (2020 cm^{-1}) [10], $[Pb(phen)(O_2CCH_3)(NCS)]$ (2040 cm^{-1}) [11] and $[Pb(phen)_2(O_2CCH_3)(NCS)]$ (2040 cm^{-1}) [12]. Significant differences are observed

Table 1. Crystal data and structure refinement for $[\text{Pb}(\text{trz})_2(\text{MeOH})](\text{ClO}_4)_2 \cdot \text{H}_2\text{O}$.

Empirical formula	$\text{C}_{43}\text{H}_{36}\text{N}_6\text{O}_{10}\text{Pb}$
Formula weight	1074.87
Temperature	120(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	$a = 10.0687(17)$ Å $b = 21.018(4)$ Å $c = 18.845(3)$ Å $\beta = 96.061(4)^\circ$ 3665.8(12) Å ³
Volume	
Z	4
Density (calculated)	1.800 Mg m ⁻³
Absorption coefficient	4.460 mm ⁻¹
$F(000)$	2128
Crystal size	0.50 × 0.50 × 0.30 mm ³
Theta range for data collection	1.94 to 30.06°
Index ranges	$-14 \leq h \leq 14$, $-29 \leq k \leq 29$, $-26 \leq l \leq 26$
Reflections collected	46 443
Independent reflections	11 572 [$R(\text{int}) = 0.0476$]
Completeness to theta	99.6%
Absorption correction	Semiempirical
Max. and min. transmission	1.000000 and 0.600900
Refinement method	Full-matrix least-squares on F^2
Data/Restraints/Parameters	11 572/38/569
Goodness-of-fit on F^2	1.099
Final R [$I > 2\sigma(I)$]	$R1 = 0.0577$, $wR2 = 0.1387$
R indices (all data)	$R1 = 0.0749$, $wR2 = 0.1448$
Largest diff. peak, hole	2.410, -2.400 e Å ⁻³

with the mercury(II) complexes, for example $[\text{Hg}(\text{bq})(\text{SCN})_2]$ (2070 cm⁻¹) [13] and $[\text{Hg}(\text{DABTZ})(\text{SCN})_2]$ (2065 cm⁻¹) [14], suggesting that the thiocyanate anions in the Pb(II) complex are different than in the mercury(II) complexes coordinated through the nitrogen atom.

Attempts to isolate 1 : 2 adducts of $[\text{Pb}(\text{trz})_2\text{X}_2]$ ($\text{X} = \text{CH}_3\text{COO}^-$ and NCS^-) were not successful, only the 1 : 1 adduct was isolated, but we were successful in the preparation of a 1 : 2 adduct for $\text{X} = \text{ClO}_4^-$. This is consistent with the assumption that acetate and thiocyanate anions are stronger binding ligands than perchlorate, resulting in the acetate and thiocyanate anions coordinating to lead(II) preventing formation of 1 : 2 adducts with these anions.

3.2. Crystal structure of $[\text{Pb}(\text{trz})_2(\text{MeOH})](\text{ClO}_4)_2 \cdot \text{H}_2\text{O}$

The crystal structure of this compound consists of monomeric units of $[\text{Pb}(\text{trz})_2(\text{MeOH})](\text{ClO}_4)_2 \cdot \text{H}_2\text{O}$. Each lead atom is chelated by the nitrogens of the trz ligands with Pb–N distances of 2.604(4), 2.645(4), 2.687(4), 2.720(4), 2.771(4) and 2.807(4) Å, and also by the oxygen atom of methanol with a Pb–O distance of 2.639(5) Å. The coordination number in this complex is seven (six from trz, one from methanol). The perchlorate anions are disordered and there are also weak interactions of lead(II) with O6 and O8 of the perchlorate [$\text{Pb}(1)–\text{O}(6\text{S})\#1 = 3.006(5)$ Å and $\text{Pb}(1)–\text{O}(8\text{S})\#1 = 3.251(8)$ Å]. Each Pb atom in this structure has seven normal bonds and two “weak” Pb ⋯ O bonds, giving nine (PbN₆O₃) coordination.

Table 2. Selected bond lengths (Å) and angles (°) for $[Pb(trz)_2(MeOH)](ClO_4)_2 \cdot H_2O$.

Pb(1)–N(1)	2.604(4)	N(1)–Pb(1)–O(1)	84.6(1)
Pb(1)–O(1)	2.639(5)	N(1)–Pb(1)–N(2)	62.50(13)
Pb(1)–N(2)	2.645(4)	O(1)–Pb(1)–N(2)	74.97(14)
Pb(1)–N(5)	2.687(4)	N(1)–Pb(1)–N(5)	83.26(13)
Pb(1)–N(4)	2.720(4)	O(1)–Pb(1)–N(5)	140.88(14)
Pb(1)–N(3)	2.771(4)	N(2)–Pb(1)–N(1)	129.41(12)
Pb(1)–N(6)	2.807(4)	N(1)–Pb(1)–N(4)	80.59(14)
Pb(1)–O(1')	2.911(10)	O(1)–Pb(1)–N(4)	151.49(14)
Pb(1)–O(6S)#1	3.006(5)	N(2)–Pb(1)–N(4)	76.59(13)
Pb(1)–O(8S)#1	3.251(8)	N(5)–Pb(1)–N(4)	61.15(12)
		N(1)–Pb(1)–N(3)	122.49(13)
		N(3)–Pb(1)–O(1)	78.17(14)
		N(2)–Pb(1)–N(3)	60.08(12)
		N(5)–Pb(1)–N(3)	138.50(12)
		N(4)–Pb(1)–N(3)	89.38(12)
		N(1)–Pb(1)–N(6)	79.60(14)
		O(1)–Pb(1)–N(6)	82.13(14)
		N(2)–Pb(1)–N(6)	136.88(13)
		N(5)–Pb(1)–N(6)	59.13(12)
		N(4)–Pb(1)–N(6)	118.56(12)
		N(3)–Pb(1)–N(6)	148.27(13)
		N(1)–Pb(1)–O(1')	139.6(3)
		O(1)–Pb(1)–O(1')	64.4(3)
		N(2)–Pb(1)–O(1')	126.3(2)
		N(5)–Pb(1)–O(1')	104.1(12)
		N(4)–Pb(1)–O(1')	138.1(2)
		N(3)–Pb(1)–O(1')	77.8(2)
		N(1)–Pb(1)–O(6S)#1	141.68(13)
		O(1)–Pb(1)–O(6S)#1	133.59(13)
		N(2)–Pb(1)–O(6S)#1	121.63(12)
		N(5)–Pb(1)–O(6S)#1	65.39(12)
		N(4)–Pb(1)–O(6S)#1	65.39(12)
		N(3)–Pb(1)–O(6S)#1	76.29(12)
		N(6)–Pb(1)–O(6S)#1	100.66(12)
		O(1')–Pb(1)–O(6S)#1	72.7(2)

#1 $-x+1, -y+1, -z$.

The arrangement of *trz* ligands and methanol anions suggests a gap or hole in the coordination geometry around the metal ion [the O(1)–Pb–N(4) angle is 151.49(14)° and the N(3)–Pb–N(6) angle is 148.27(13)°], possibly occupied by a stereoactive lone pair of electrons on lead(II). The shortening of the Pb–N bonds on the side of Pb(II) opposite to the putative lone pair [2.604(4) and 2.639(5) Å compared with 2.807(4) and 2.771(4) Å adjacent to the lone pair] supports the presence of a stereoactive lone pair [15]. Hence, the geometry of the nearest coordination environment of every lead atom is most likely determined by the geometrical constraints of the coordinated *trz* ligand and methanol and by the influence of a stereochemically active lone pair of electrons in a hybrid orbital on the metal atom (figure 1). The possible stereochemical activity of the lone pair in divalent lead compounds has recently been discussed by Shimoni-Livny *et al.* based on a thorough review of crystal data available in the Cambridge Structural Database (CSD) [1]. They classify lead coordination as holo-directed, which refers to complexes in which the bonds to ligand atoms are directed throughout the surface of an encompassing sphere, while hemidirected refers to those cases in which the bonds to ligand atoms are directed to only a part of the coordination

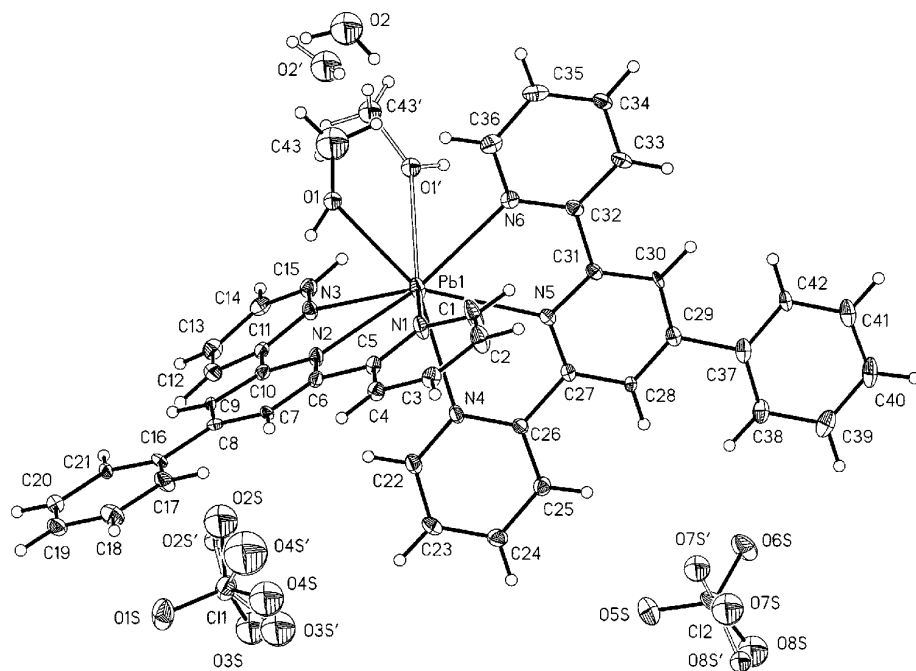


Figure 1. ORTEP diagram of the $[\text{Pb}(\text{trz})_2(\text{MeOH})](\text{ClO}_4)_2 \cdot \text{H}_2\text{O}$ complex.

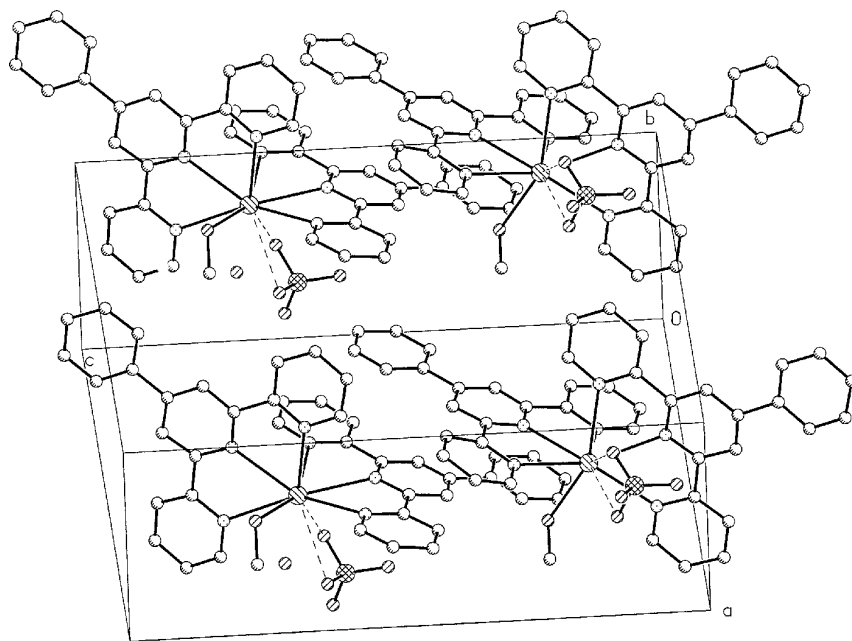


Figure 2. The unit cell of the $[\text{Pb}(\text{trz})_2(\text{MeOH})](\text{ClO}_4)_2 \cdot \text{H}_2\text{O}$ complex.

sphere, leaving a gap in the distribution of bonds to the ligand. The latter, present in all Pb(II) compounds with coordination number 2 to 5, is fairly common in Pb(II) compounds with coordination numbers 6, 7 and 8, but does not exist in lead complexes with higher coordination numbers, where holodirected geometry is the rule. For the structure described here, coordination around the lead atoms is hemidirected with a significant gap trans to the one chelating trz ligand. It would seem that the gap in the $[Pb(trz)_2(MeOH)](ClO_4)_2 \cdot H_2O$ complex is smaller than that of reported complexes [5–8]. This may relate to the high coordination number and overcrowding in $[Pb(trz)_2(MeOH)](ClO_4)_2 \cdot H_2O$.

The trz complexes of lead(II) are yellow, precipitating from colorless solution. The color depends not only on the nature of the aromatic nitrogen-donor ligand but also on the solid-state structures that involve charge-transfer-state packing. As shown in figure 2, there is a π – π stacking interaction [16, 17] between the parallel aromatic rings around the Pb(II) that may produce the yellow color of this complex in the solid state.

There is a notable lack of solvent bound to Pb(II) [1] and methanol coordinated to lead(II) is rare. This is in line with the low solubility of lead compounds in general. The $[Pb(trz)_2(MeOH)](ClO_4)_2 \cdot H_2O$ complex contains one disordered MeOH coordinated to lead(II) with two different bond lengths, Pb(1)–O(1) = 2.639(5) Å and Pb(1)–O(1) = 2.911(10) Å; this complex contains one water molecule that it is also disordered and not coordinated to the lead.

Supplementary data

Supplementary data including bond lengths and angles, coordinates and displacement parameters for $[Pb(trz)_2(MeOH)](ClO_4)_2 \cdot H_2O$ have been deposited at the Cambridge Crystallography Data Centre, CCDC No. 183932. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

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