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# The bent metallocene geometries of potassium polyalkyl cyclopentadienyl THF solvates: monosolvated [(THF)K(μ-$C_5Me_5$)]$_n$, disolvated [$(THF)_2$K(μ-$C_5Me_5$)]$_n$ and the tethered olefin complex [(THF)K(μ-$C_5Me_4SiMe_2CH_2CH{=}CH_2$)]$_n$ ☆

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## Abstract

THF-solvated $KC_5Me_5$ and $KC_5Me_4SiMe_2CH_2CH{=}CH_2$ complexes crystallize with extended structures that provide information on bent metallocenes containing potassium, [(μ-$C_5R_5$)K(μ-$C_5R_5$)]$^-$. The substructures of [(THF)K(μ-$C_5Me_5$)]$_n$, [$(THF)_2$K(μ-$C_5Me_5$)]$_n$, and [(THF)K(μ-$C_5Me_4SiMe_2CH_2CH{=}CH_2$)]$_n$ display (μ-$C_5R_5$)K(μ-$C_5R_5$) angles of 137.9–143.7°. The olefin substituent in [(THF)K(μ-$C_5Me_4SiMe_2CH_2CH{=}CH_2$)]$_n$ approaches potassium with K···C distances of 3.58 and 4.20 Å. © 2002 Elsevier Science B.V. All rights reserved.

*Keywords:* Potassium; Cyclopentadienide; Bent metallocenes; Tethered olefins

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## 1. Introduction

Potassium cyclopentadienides are commonly used reagents for forming organometallic complexes with metal halide precursors, Eq. (1) [1]. The potassium halide byproduct has a large

$$n\mathrm{KC_5R_5} + \mathrm{MCl}_x \rightarrow n\mathrm{KCl} + (\mathrm{C_5R_5})_n\mathrm{MCl}_{x-n} \qquad (1)$$

lattice energy and frequently can be separated from the desired organometallic product more efficiently than, e.g. LiCl, which often forms adducts such as $(C_5R_5)_nMCl_{x-n+1}Li(solvate)_2$ [2,3]. Although $KC_5R_5$ ligands are heavily used synthetically, little is known about their structures particularly in the presence of the common solvent THF. This is not surprising since even the pentamethylcyclopentadienyl complexes are neither very soluble nor easy to crystallize. To date, the only potassium alkyl/silyl cyclopentadienide complexes that have been structurally characterized have either involved strongly coordinating bases, (18-crown-6)K($C_5Me_5$) [4] and [$(pyridine)_2$K($C_5Me_5$)]$_n$ [5], or alkyl groups that enhance solubility, [K{$C_5H_2(SiMe_3)_3$-1,2,4}]$_n$ [6], $(THF)_3$K[$C_5(CH_2C_6H_5)_5$] [7], [K($C_5H_4SiMe_3$)]$_n$ [8], and {(THF)K[($C_5Me_4$)$SiMe_2NH(^tBu)$]}$_n$ [9]. The structural data on unsubstituted potassium cyclopentadienyl systems is even more sparse. Structures of [($Et_2O$)K($C_5H_5$)]$_n$ [10] and (18-crown-6)K($C_5H_5$) [11] have been reported and powder data on K($C_5H_5$) [12] have been obtained.

In the course of our studies of organometallic chemistry using $KC_5R_5$ in THF, we have been able to obtain single crystals of three THF solvates, namely [(THF)K(μ-$C_5Me_5$)]$_n$ (**1**), [$(THF)_2$K(μ-$C_5Me_5$)]$_n$ (**2**), and [(THF)K(μ-$C_5Me_4SiMe_2CH_2CH{=}CH_2$)]$_n$ (**3**). Since each of these compounds crystallizes with extended structures via bridging cyclopentadienyl rings, this generates ring–metal–ring metallocene substructures. Although, considerable experimental and theoretical effort has been devoted to studying the structures of bent metallocenes and particularly the bent metallocenes of the

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lanthanide elements and the alkaline earth metals [13–18], the alkali metals have not received as much attention [19–21]. This is reasonable since these singly charged metals do not form neutral molecular bent metallocenes. Nevertheless, structural comparisons are of interest since the radius of $K^+$ is similar to that of $Ba^{2+}$ [22].

The complexes reported here allow a comparison of simple $C_5R_5$ systems solvated by THF and by a THF with another potentially coordinating ligand, an alkene. These data are compared with the potassium metallocene substructures in $[K\{C_5H_2(SiMe_3)_3\text{-}1,2,4\}]_n$ [6], $[(pyridine)_2K(C_5Me_5)]_n$ [5], $(THF)K[(C_5Me_4)SiMe_2NH(^tBu)]_n$ [9], $[K(C_5H_4SiMe_3)]_n$ [8], and the mixed metal species $[(C_5Me_5)Sm(OAr)(C_5Me_5)K(THF)_2]_n$, ($Ar = C_6H_3{}^tBu_2$-2,6, $C_6H_2{}^tBu_2$-2,6-Me-4 [23]).

## 2. Experimental

### 2.1. General

The chemistry described below was performed under nitrogen with rigorous exclusion of air and water by using Schlenk, vacuum line, and glovebox techniques. Solvents were purified as previously described [24]. NMR spectra were collected using a Bruker DRX400 spectrometer. KH was purchased from Strem and washed with hexanes before use. $C_5Me_4HSiMe_2CH_2CH{=}CH_2$ was prepared as previously described [25].

Table 1
Crystal data and structure refinement parameters for $[(THF)K(\mu\text{-}C_5Me_5)]_n$ (**1**), $[(THF)_2K(\mu\text{-}C_5Me_5)]_n$ (**2**), and $[(THF)K(\mu\text{-}C_5Me_4SiMe_2CH_2CH{=}CH_2)]_n$ (**3**)

| | 1 | 2 | 3 |
|---|---|---|---|
| Empirical formula | $[C_{14}H_{23}KO]$ | $[C_{18}H_{31}KO_2]$ | $[C_{18}H_{31}KOSi]$ |
| Formula weight | 246.42 | 318.53 | 330.62 |
| Temperature (K) | 158(2) | 168(2) | 158(2) |
| Crystal system | Orthorhombic | Orthorhombic | Orthorhombic |
| Space group | *Pbca* | *Pnma* | *Pbca* |
| Unit cell dimensions | | | |
| $a$ (Å) | 10.422(3) | 10.509(2) | 18.5716(16) |
| $b$ (Å) | 16.450(5) | 18.811(5) | 10.5683(9) |
| $c$ (Å) | 17.029(5) | 9.737(2) | 19.9257(17) |
| $V$ (Å$^3$) | 2919.3(16) | 1925.0(7) | 3910.8(6) |
| $Z$ | 8 | 4 | 8 |
| $\rho_{calc}$ (Mg m$^{-3}$) | 1.121 | 1.099 | 1.123 |
| Diffractometer [a] | Bruker CCD | Siemens P4 | Bruker CCD |
| $\mu$ (mm$^{-1}$) | 0.345 | 0.279 | 0.331 |
| Refinement [b]: $R_1$ $I>2\sigma(I)$ | 0.1632 | 0.0766 | 0.0600 |
| Reflections with $I>2.0\sigma(I)$ | 1194 | 765 | 2811 |
| $wR_2$ (all data) | 0.4226 | 0.2112 | 0.1804 |

[a] Radiation: Mo–$K_\alpha$ ($\lambda = 0.710730$ Å). Monochromator: highly oriented graphite.

[b] $R_1 = \Sigma\|F_o|-|F_c\|/\Sigma|F_c|$; $wR_2 = [\Sigma[w(F_o^2-F_c^2)^2]/\Sigma[w(Fo^2)^2]]^{1/2}$.

### 2.2. X-ray data collection and solution and refinement for $[(THF)K(\mu\text{-}C_5Me_5)]_n$ (1)

A colorless crystal of approximate dimensions 0.10 × 0.20 × 0.20 mm obtained from a reaction of $KN(SiMe_3)_2$ with $(C_5Me_5)_2Y(ONC_5H_{10})$ [26] was mounted on a glass fiber and transferred to a Bruker CCD platform diffractometer. The SMART [27] program package was used to determine the unit-cell parameters (Table 1) and for data collection (30 s frame$^{-1}$ scan time for a sphere of diffraction data). The raw frame data were processed using SAINT [28] and SADABS [29] to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL [30] program. The diffraction symmetry was *mmm* and the systematic absences were consistent with the orthorhombic space group *Pbca* which was later determined to be correct. The structure was solved by direct methods and refined on $F^2$ by full-matrix least-squares techniques. Unfortunately, the data are weak and only connectivity could be established in this structure.

### 2.3. X-ray data collection and solution and refinement for $[(THF)_2K(\mu\text{-}C_5Me_5)]_n$ (2)

A colorless crystal of approximate dimensions 0.20 × 0.25 × 0.30 mm obtained from a reaction of $KC_5Me_5$ with $Nd(NO_3)_3$ [31] was mounted on a glass fiber and transferred to a Siemens P4 diffractometer. The XSCANS [32] program package was used to determine the Laue symmetry, crystal class, unit-cell parameters and for data collection. Intensity data were collected at 168 K using a $2\theta-\omega$ scan technique with Mo–$K_\alpha$ radiation. The raw data were processed with a local version of CARESS [33] which employs a modified version of the Lehman–Larsen algorithm to obtain intensities and standard deviations from the measured 96-step profiles. Subsequent calculations were carried out using the SHELXTL [30] program. All data were corrected for Lorentz and polarization effects and placed on an approximately absolute scale. The diffraction symmetry was *mmm* and the systematic absences were consistent with the orthorhombic space groups $Pna2_1$ and *Pnma*. The centrosymmetric space group *Pnma* was assigned and later determined to be correct.

The structure was solved by direct methods and refined on $F^2$ by full-matrix least-squares techniques. The analytical scattering factors [34] for neutral atoms

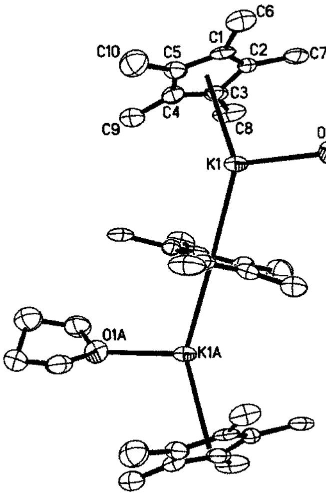

Fig. 1. Thermal ellipsoid plot of $[(THF)K(\mu\text{-}C_5Me_5)]_n$ (**1**), drawn at the 50% probability level.

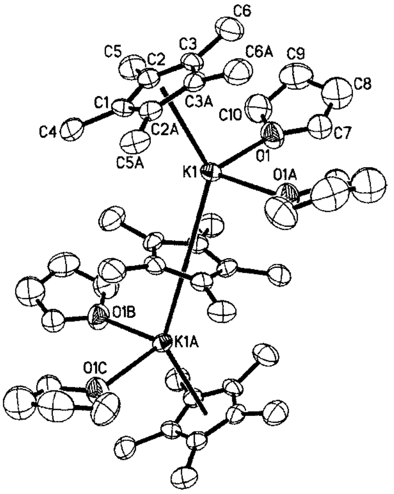

Fig. 2. Thermal ellipsoid plot of $[(THF)_2K(\mu\text{-}C_5Me_5)]_n$ (**2**), drawn at the 50% probability level.

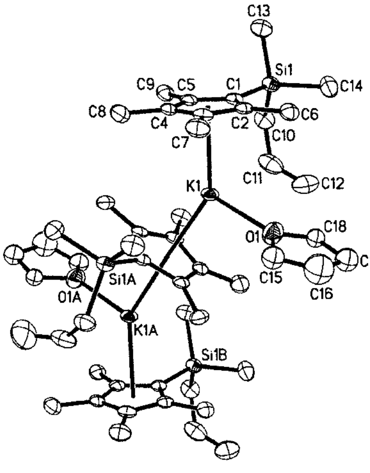

Fig. 3. Thermal ellipsoid plot of $[(THF)K(\mu\text{-}C_5Me_4SiMe_2CH_2CH{=}CH_2)]_n$ (**3**), drawn at the 50% probability level.

were used throughout the analysis. The polymeric structure was located on a mirror plane. Carbon atoms C(7) and C(8) were disordered and included using multiple components with partial site-occupancy-factors. Hydrogen atoms were included using a riding model. At convergence, $wR_2 = 0.2112$ and GOF = 1.028 for 98 variables refined against 1754 data. As a comparison for refinement on $F$, $R_1 = 0.0766$ for those 765 data with $I > 2\sigma(I)$.

### *2.4. $[(THF)K(\mu\text{-}C_5Me_4SiMe_2CH_2CH{=}CH_2)]_n$ (3)*

Solid KH (22 mg, 0.55 mmol) was added slowly to a light yellow solution of $Me_2Si(C_5Me_4H)CH_2CH{=}CH_2$ (100 mg, 0.45 mmol) in 10 ml of THF. The reaction was stirred for 24 h during which time the solution changed to a dark yellow color. The reaction mixture was centrifuged to remove insoluble material and the THF was removed by rotary evaporation leaving a light yellow/white solid. The solid was washed with hexanes (2 × 5 ml) and dried under vacuum yielding **3** as a white solid (96 mg, 64%). X-ray quality crystals were obtained by cooling a saturated solution of **3** in pyridine overnight. $^1$H-NMR (pyridine-$d_5$): $\delta$ 6.16 (m, 1H, $\mathrm{CH_2C}H\mathrm{{=}CH_2}$), 5.00 (d, $J = 16$ Hz, 1H, $\mathrm{CH_2CH{=}C}H_2$), 4.80 (d, $J = 12$ Hz, 1H, $\mathrm{CH_2CH{=}C}H_2$), 3.63 (m, THF), 2.48 (s, 6H, $\mathrm{C_5}Me_4$), 2.30 (s, 6H, $\mathrm{C_5}Me_4$), 2.02 (d, $J = 8$ Hz, 2H, $\mathrm{C}H_2\mathrm{CH{=}CH_2}$), 1.59 (m, THF), 0.48 (s, 6H, $\mathrm{Si}Me_2$).

### *2.5. X-ray data collection and solution and refinement for $[(THF)K(\mu\text{-}C_5Me_4SiMe_2CH_2CH{=}CH_2)]_n$ (3)*

A colorless crystal of approximate dimensions 0.13 × 0.21 × 0.38 mm was handled as described above for **1**. The diffraction symmetry was *mmm* and the systematic absences were consistent with the orthorhombic space group *Pbca* which was later determined to be correct. The structure was solved by direct methods and refined on $F^2$ by full-matrix least-squares techniques. The analytical scattering factors [34] for neutral atoms were used throughout the analysis. Hydrogen atoms were included using a riding model. The THF carbon atoms were disordered and included using the PART [30] command with partial site-occupancy-factors (0.50; isotropic). At convergence, $wR_2 = 0.1804$ and GOF = 1.024 for 186 variables refined against 3993 unique data (0.80 Å cutoff). (As a comparison for refinement on $F$, $R_1 = 0.0600$ for those 2811 data with $I > 2\sigma(I)$.)

## 3. Results and discussion

Complexes **1**–**3** crystallize with the extended structures shown in Figs. 1–3. Due to the bridging nature of the polyalkylcyclopentadienyl ligands in the structures of **1**–**3**, each potassium has a bent metallocene coordination environment much like those found with higher valent metals which form $(C_5R_5)_2ML_x$ structures.

$[(THF)K(\mu\text{-}C_5Me_5)]_n$ (**1**), and $[(THF)_2K(\mu\text{-}C_5Me_5)]_n$ (**2**), were crystallized as byproducts of organometallic reactions involving $KC_5Me_5$. It was fortunate to crystallize both mono-THF and bis-THF adducts of $KC_5Me_5$: generally there is one optimum degree of solvation which is favored and which provides crystals suitable for X-ray analysis. However, the diffraction data on **1** were weak and only connectivity could be determined. Isolation of crystals with better diffraction was not achieved. $[(THF)K(\mu\text{-}C_5Me_4SiMe_2CH_2\text{-}CH{=}CH_2)]_n$ (**3**), was crystallized to examine possible interactions between potassium and the tethered allyl substituent. This ligand is being investigated to examine metal olefin interactions in metallocene systems that are active in olefin polymerization reactions [25]. Surprisingly, **3** crystallized from pyridine as a THF adduct.

Complex **3** can be considered an intermediate in solvation between mono-solvate **1** and di-solvate **2** in that only one THF is coordinated, but the olefinic portion of the side arm is oriented toward the potassium, Fig. 3. Although this positioning could be due to crystal packing effects, it seems that the location of the olefin near the open part of the metallocene wedge is more than coincidence. The position of the $Me_2SiCH_2CH{=}CH_2$ side arm, among the five possibilities in a cyclopentadienyl ring in a metallocene wedge structure, and the position of the $CH_2CH{=}CH_2$ substituent, among the three possibilities on the silicon attached to the ring, suggest that a long-range metal olefin interaction is present.

The K···C(alkene) distances in **3**, K–C(11), 3.58(3) Å, and K–C(12), 4.20(3) Å are long compared to the $K\text{–}C(C_5Me_4R)$ distances, 2.998(3)–3.042(3) Å as expected for a weakly interacting moiety. These K···C(alkene) can be compared with long-range K···C(arene) distances in the literature: $KBPh_4$, 3.191(5) Å [35], $\{K[(\mu\text{-}C_5H_5)_2Nd(\mu\text{-}OC_6H_3Me_2\text{-}2,6)_2]\}_n$ 3.17(2)–3.93(2) Å [36], and $\{K[Nd(OC_6H_3^iPr\text{-}2,6)_4]\}_n$, 3.097(10)–3.473(11) Å [37]. These numbers can also be compared with the 3.723 Å K···N distance to the side arm donor atom in $\{(THF)K[Me_2Si(C_5Me_4)NH(^tBu)]\}_n$ [9]. The two olefinic carbon atoms are not both in the plane bisecting the ring–metal–ring wedge. The C(12) atom is located 0.3599 Å below the plane defined by C(11)–K(1)–O(1). The olefin moiety is oriented to put the internal carbon C(11) closer to the potassium. This orientation does not seem to be due to any intramolecular geometric requirement. Overall, the position of the olefin seems to be more than random and functions to occupy a coordination position, albeit at a long distance.

Table 2 presents a comparison of the structural parameters of **2** and **3** with those of other potassium alkylcyclopentadienyl complexes whose extended structures form metallocene subunits, $[K\{C_5H_2(SiMe_3)_3\text{-}1,2,4\}]_n$ (**4**), $[(pyridine)_2K(C_5Me_5)]_n$ (**5**), $[K(C_5H_4\text{-}SiMe_3)]_n$ (**6**), $\{(THF)K[Me_2Si(C_5Me_4)NH(^tBu)]\}_n$ (**7**), and $[(C_5Me_5)Sm(OAr)(C_5Me_5)K(THF)_2]_n$, (Ar = $C_6H_3^tBu_2$-2,6 (**8**), $C_6H_2^tBu_2$-2,6-Me-4 (**9**)). The table is arranged to present the data in decreasing order of (ring centroid)–K–(ring centroid) angle. The angles decrease over a wide range from 172.7 to 137.9°. The decrease is regular as the formal coordination number increases: the eight coordinate structures are more bent (smaller angle) to accommodate the extra ligands. On the basis of the angle ordering, complex **3** falls at the borderline between structures which are clearly seven and eight coordinate. This is consistent with its structure, which is seven coordinate for ligands at normal distances, and could be considered eight coordinate if the distant tethered olefin was counted as a ligand.

For all of the complexes the K–(ring centroid)–K-angles and, in the case of the mixed metal Sm–ring–K–ring–Sm structures, **8** and **9**, the K–(ring centroid)–Sm angles are close to linear, 172–179°. The K–(ring centroid) distances do not follow the same pattern as the (ring centroid)–K–(ring centroid) angles. The mixed metal complexes **8** and **9** have distinctly longer distances, 2.905–2.929 Å, compared to all the other entries, which fall in the relatively narrow range 2.751–2.821 Å. Evidently, the presence of a metal different from potassium on the other side of the ring in the (ring)–K–(ring) substructure strongly affects the K–(ring) distances. Examination of the other K–(ring centroid) distances shows no regular variation of these distances with the (ring centroid)–K–(ring centroid) angles. Apparently, the (ring centroid)–K–(ring centroid) angle is more sensitive to coordination number and ring substitution than the K–(ring centroid) distance. The six K–O(THF) distances also show no regular trend consistent with the (ring centroid)–K–(ring centroid) angles. The K–O(THF) distances for **2**, **3**, and **7** increase with increasing K–(ring centroid) distances, but the THF distances in mixed metal complexes **8** and **9** do not fit this pattern.

## 4. Conclusions

The bent metallocene $[(\mu\text{-}C_5R_5)\text{–}K\text{–}(\mu\text{-}C_5R_5)]^-$ substructures, which occur in extended structures containing potassium polyalkylcyclopentadienyl moieties, can adopt a range of K–(ring centroid) distances and (ring centroid)–K–(ring centroid) angles depending on the coordination number, ring substituent, and the metal on the other side of the $[(\mu\text{-}C_5R_5)\text{–}K\text{–}(\mu\text{-}C_5R_5)]^-$ unit. Hence, potassium is found to be quite flexible in accommodating different bond lengths and angles in complexes stable enough to be crystallographically

Table 2
Selected bond lengths (Å) and bond angles (°) for $[(THF)_2K(\mu\text{-}C_5Me_5)]_n$ (**2**), $[(THF)K(\mu\text{-}C_5Me_4SiMe_2CH_2CH{=}CH_2)]_n$ (**3**), $[K\{\mu\text{-}C_5H_2(SiMe_3)_3\text{-}1,2,4\}]_n$ (**4**), $[(pyridine)_2K(\mu\text{-}C_5Me_5)]_n$ (**5**), $[K(\mu\text{-}C_5H_4SiMe_3)]_n$ (**6**), $(THF)K[Me_2Si(\mu\text{-}C_5Me_4)NH({}^tBu)]_n$ (**7**), and $[(C_5Me_5)Sm(OAr)(C_5Me_5)K(THF)_2]_n$ ($Ar = C_6H_3{}^tBu_2$-2,6 (**8**); $C_6H_2{}^tBu_2$-2,6-Me-4 (**9**)) arranged according to decreasing (ring centroid)–K–(ring centroid) angle

| Compounds | Formal metal coordination number | Centroid–K–Centroid (°) | K–Centroid–K (°) | K–Centroid (Å) | K–C range (Å) | K–C avg. (Å) | K–O (Å) | K–N (Å) | References |
|---|---|---|---|---|---|---|---|---|---|
| **4** | 6 | 172.73 | 176.16 | 2.751 | 2.955(5)–3.140(6) | 3.06 | – | – | [6] |
| **6** | 6 | 150.7 | 175.3 | 2.78 | 2.988–3.079 | 3.03 | – | – | [8] |
| **7** | 7 | 145.5 | 176.2 | 2.808 | 3.012–3.095 | 3.052 | 2.762(5) | 3.723 | [9] |
| **3** | 7 | 143.7 | 176.6 | 2.765 | 2.998(3)–3.042(3) | 3.013(3) | 2.702(3) | – | This work |
| **8** | 8 | 142.2(7) | 175.9 [a] | 2.929, 2.914 | 3.13(3)–3.157(15) | 3.146(19) | 2.79(2) | – | [23] |
| **9** | 8 | 140.2(3) | 175.0 [a] | 2.919, 2.905 | 3.006(7)–3.270((7) | 3.142(7) | 2.888(8),<br>2.787(9) | – | [23] |
| **5** | 8 | 138 | 172.4 | 2.79 | 2.962(2)–3.104(2) | 3.03 | – | 2.879(2),<br>2.974(2) | [5] |
| **2** | 8 | 137.9 | 179.0 | 2.821 | 3.048(5)–3.077(8) | 3.059(8) | 2.807(4) | – | This work |

[a] Data reported are for K–(ring centroid)–Sm angles.

characterized. The fact that potassium can be incorporated into such structures may be a result of this flexibility.

## 5. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC nos. 174255, 174256 and 174257 for compounds **1**–**3**, respectively. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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