Pyridine-type nitrogen atoms in azoles or azines are readily available for adduct formation with various boranes [1]. Depending on steric requirements, there can be fast exchange between the azole-(azine)-borane adduct and excess of either the azole (azine) or the borane. Recently, it was shown that this situation applies to azole-BEt$_3$ adducts if a Me$_3$Sn group is present adjacent to the two-coordinated nitrogen atom [2]. Such adducts are intermediates preceding the elimination of Me$_3$SnEt and the formation of azaboles [3]. The molecular structures of 3 in the solid state and in solution are very similar.

2-Stannylpyridine-Borane Adducts – Multinuclear Magnetic Resonance Study and X-Ray Structure Determination of a 1,4-Dihydro-4a,1,4-azastannabora-naphthalene Derivative

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2-Trimethylstannlyphridine, Borane Adducts, Heterobicycle, X-Ray, NMR Spectra

The adduct formation between 2-Trimethylstannlyphridine (1) and triethylborane, leading to 2a, and 9-borabicyclo[3.3.1]nonane, leading to 2b, was studied by $^1$H, $^{11}$B, $^{13}$C, $^{15}$N and $^{119}$Sn NMR in solution. Changes in the magnitude of the coupling constants $^2$J($^{119}$Sn,$^{13}$C) with respect to the data for 1, were analysed. The absolute signs have been determined [all coupling constants $^2$J($^{119}$Sn,$^{13}$C) to methyl and pyridine carbon atoms in 1 to 3 possess a negative sign and the same is true for $^2$J($^{119}$Sn,$^2$H) of the pyridine hydrogen atoms] by various two-dimensional NMR experiments, and attributed to the influence of the lone pair of electrons at the nitrogen atom in 1. The NMR spectroscopic results for the 1,4-dihydro-4a,1,4-azastannabora-naphthalene derivative 3, in which structural fragments are present analogous to those in the borane adducts 2, correspond to those for 1 and 2a,b. The molecular structure of 3 has been determined by X-ray analysis [orthorhombic; $P_2_1_2_1_2_1$; $a$ = 713.9(2), $b$ = 1566.0(2), $c$ = 1578.4(2) pm]. Solid-state $^{13}$C and $^{119}$Sn CP/MAS NMR spectra prove that the molecular structures of 3 in the solid state and in solution are very similar.

$^1$H, $^2$B, $^{13}$C, $^{14}$N and $^{119}$Sn NMR) using two-dimensional (2D) techniques for the determination of absolute signs of the coupling constants $^2$J($^{119}$Sn,$^2$H) and $^2$J($^{119}$Sn,$^{13}$C). A kinetically more stable N–B bond is encountered in 2b, the 9-borabicyclo[3.3.1]nonane-adduct of 1, and in the fused heterobicycle 3. Compound 3 was also studied by $^{13}$C and $^{119}$Sn CP/MAS NMR in the solid state, and its molecular structure was determined by single crystal X-ray analysis.

Results and Discussion

Synthesis

Reactions between 1 and Et$_3$B or 9-borabicyclo[3.3.3]nonane (9-BBN) were studied on a small scale in NMR tubes. 2-Trimethylstannlyphridine (1) reacts with Et$_3$B to give an adduct which, at room temperature, readily exchanges with an excess of 1 or Et$_3$B. In contrast, 1 reacts very slowly

* Reprint requests to Prof. Dr. B. Wrackmeyer.

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at room temperature with the dimeric 9-BBN. Fast and irreversible symmetrical cleavage of the BH₂B moiety takes place at temperatures >50 °C and the adduct 3 is formed. Under these conditions, only a trace of Me₃SnH can be detected ($^{119}$Sn NMR). Any exchange between 3 and an excess of either 1 or dimeric 9-BBN is slow on the NMR time scale.

The reaction of 2-pyridyllithium [6] with Me₃SnCl affords 2-trimethylstannyl-pyridine (1), and with (E)-2-chloro(dimethyl)stannyl-3-diethylboryl-2-pentene [7] the heterobicycle 3 is obtained [eq. (1)] as a colourless, crystalline, moisture-sensitive solid. The reaction according to eq. (1) has been mentioned previously [8].

\[
\begin{align*}
\text{Me}_3\text{SnCl} + \text{LiNMe}_2 & \rightarrow \text{Me}_3\text{SnLi} + \text{NMe}_3 \\
\text{Me}_3\text{SnLi} + \text{Cl}^{-} & \rightarrow \text{Me}_3\text{SnCl} \\
\text{Me}_3\text{SnCl} + \text{C}(\text{Cl}) & \rightarrow \text{Me}_3\text{SnCl}_2
\end{align*}
\]

\[\text{eq. (1)}\]

**X-ray structure determination of 3**

Data relevant to the X-ray analysis of 3 are given in Table I [9]. The molecular structure of 3 is shown in Fig. 1 together with selected bond lengths and angles. The skeleton of the heterobicycle 3 is not exactly planar. Starting from the plane of the pyridine ring, the tin atom is located 15 pm above and the boron atom 18 pm below that plane. The atoms B, C(5), C(3), Sn form a plane within the experimental error. There are distorted tetrahedral surroundings for both the tin and the boron atom, with endocyclic bond angles being small \([\text{C(3)SnC(12)} = 101.8(1)^\circ]\) and fairly large \([\text{NBC(5)} = 118.5(2)^\circ]\). The latter bond angle together with the rather long BN distance causes a wide bond angle \(\text{BC(5)C(3)} = 132.7(3)^\circ\). The BN bond \([168.9(5) \text{ pm}]\) is longer than all BC distances in 3, as in other pyridine-trialkylborane [10] or in trialkylamine-dialkylborane adducts [11]. The arrangement of the ethyl groups at the boron atom is noteworthy (see Fig. 1). The non-bonding distances \(\text{BC(9)}\) and \(\text{BC(11)}\) (266 pm) suggest that interactions between the hydrogen atoms at C(9) and C(11) and the boron atom are conceivable which could account for the long BN distance and deviations from ideal bond angles.

![Fig. 1. Molecular structure of the heterobicycle 3. Selected bond lengths [pm] and angles [°]: Sn-C(1) 214.8(4), Sn-C(2) 214.5(4), Sn-C(3) 209.7(4), Sn-C(12) 214.9(3), N-B 168.9(5), B-C(5) 163.0(5), B-C(8) 163.9(5), B-C(10) 164.7(5), C(3)-C(5) 134.6(5); C(1)-Sn-C(2) 110.2(2), C(3)-Sn-C(12) 101.8(1), Sn-C(3)-C(5) 118.7(3), Sn-C(12)-N 101.8(1), Sn-C(12)-N 120.1(2), N-B-C(5) 118.5(2), B-N-C(12) 127.5(3), B-C(5)-C(3) 132.7(3), B-C(8)-C(9) 114.5(3), B-C(10)-C(11) 114.1(3).](image-url)

### Table I. Experimental data related to the single crystal X-ray analysis of 3.

<table>
<thead>
<tr>
<th>Formula (molecular mass)</th>
<th>C₁₄H₂₈B₃N₄Sn (363.9)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crystal; Size</td>
<td>colourless platelet; 0.24×0.24×0.14 mm³</td>
</tr>
<tr>
<td>Crystal system; space group; Z</td>
<td>orthorhombic; P₂₁₂₁₂₁; 4</td>
</tr>
<tr>
<td>Unit cell dimensions [µm]</td>
<td>a = 713.9(2), b = 1566.0(2), c = 1578.4(2)</td>
</tr>
<tr>
<td>V [Å³]</td>
<td>1764.6(6)</td>
</tr>
<tr>
<td>Absorption coefficient [mm⁻¹]</td>
<td>1.437</td>
</tr>
<tr>
<td>Diffractometer</td>
<td>Siemens P4</td>
</tr>
<tr>
<td>Radiation [µm]</td>
<td>MoKα, λ = 71.073, graphite-monochromator</td>
</tr>
<tr>
<td>Temperature [K]</td>
<td>173</td>
</tr>
<tr>
<td>2θ-range</td>
<td>4° ≤ 2θ ≤ 55°</td>
</tr>
<tr>
<td>Scan type</td>
<td>2θ-θ plus Kα-separation</td>
</tr>
<tr>
<td>Scan range (ω)</td>
<td>1.30°</td>
</tr>
<tr>
<td>Measured reflections</td>
<td>3308</td>
</tr>
<tr>
<td>Independent/observed reflections</td>
<td>3101 (F &gt; 0.0σ(F), no reflections omitted)</td>
</tr>
<tr>
<td>Refined parameters</td>
<td>173</td>
</tr>
<tr>
<td>Solution</td>
<td>direct methods (SHELXTL PLUS)</td>
</tr>
<tr>
<td>Weighting scheme</td>
<td>w⁻¹ = σ²(F) + 0.0000F²</td>
</tr>
<tr>
<td>R-w/R-value</td>
<td>0.029/0.024</td>
</tr>
<tr>
<td>Max./min. residual electron density [e Å⁻³]</td>
<td>0.49/ -0.47</td>
</tr>
</tbody>
</table>

**NMR spectroscopic results**

Relevant $^{11}$B, $^{13}$C, $^{14}$N and $^{119}$Sn NMR data of the compounds 1 to 3 are given in Table II. The mutual assignment of 1 to 3 is based on 2D $^{13}$C/$^1$H heteronuclear shift correla-
Table II. \( ^{11}B, ^{13}C, ^{14}/^{15}N \) and \(^{119}Sn\) NMR data\(^a\) of 2-trimethylstannyl-pyridine (1) and the borane adducts 2 and of the heterobicycle 3.

<table>
<thead>
<tr>
<th>Compound No.</th>
<th>( \delta ^{11}B )</th>
<th>( \delta ^{13}C )</th>
<th>C(3)</th>
<th>C(4)</th>
<th>C(5)</th>
<th>C(6)</th>
<th>SnMe</th>
<th>( \delta ^{15}N )</th>
<th>( \delta ^{119}Sn )</th>
</tr>
</thead>
<tbody>
<tr>
<td>1(^b)</td>
<td>173.4</td>
<td>131.6</td>
<td>133.4</td>
<td>122.4</td>
<td>150.6</td>
<td>-9.3</td>
<td>-37.6</td>
<td>-52.9</td>
<td></td>
</tr>
<tr>
<td></td>
<td>[−605.5]</td>
<td>[−93.2]</td>
<td>[−37.2]</td>
<td>[−12.0]</td>
<td>[−68.7]</td>
<td>[−346.6]</td>
<td>[+108.2]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2a(^c)</td>
<td>+5.0(^d)</td>
<td>171.3</td>
<td>134.5</td>
<td>134.9</td>
<td>123.0</td>
<td>148.6</td>
<td>-4.1</td>
<td>-100(^e)</td>
<td>-30.4</td>
</tr>
<tr>
<td></td>
<td>[−500.0]</td>
<td>[−66.4]</td>
<td>[−35.0]</td>
<td>[−9.8]</td>
<td>[−36.0]</td>
<td>[−369.8]</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2b(^f)</td>
<td>+3.6</td>
<td>173.7</td>
<td>133.9</td>
<td>135.7</td>
<td>123.5</td>
<td>146.7</td>
<td>-5.6</td>
<td>-109.8</td>
<td>-53.5</td>
</tr>
<tr>
<td></td>
<td>[−491.6]</td>
<td>[−61.0]</td>
<td>[−34.1]</td>
<td>[−8.1]</td>
<td>[−26.0]</td>
<td>[−381.5]</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3(^g)</td>
<td>+4.2</td>
<td>173.7</td>
<td>133.4</td>
<td>134.9</td>
<td>123.5</td>
<td>147.0</td>
<td>-8.5</td>
<td>-109.3</td>
<td>-119.7</td>
</tr>
<tr>
<td></td>
<td>[−354.0]</td>
<td>[−39.5]</td>
<td>[−22.5]</td>
<td>[−6.3]</td>
<td>[−21.5]</td>
<td>[−325.6]</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3(^h)</td>
<td></td>
<td>174.0</td>
<td>134.0</td>
<td>136.9</td>
<td>125.3</td>
<td>148.0</td>
<td>-8.6</td>
<td></td>
<td>-122.3</td>
</tr>
</tbody>
</table>

\(^{a}\) In C\(_6\)D\(_6\) (\(= 10\%\)–20\%, 25 ± 1 °C); coupling constants \( ^{11}J(^{119}Sn, ^{13}C) (\pm 1 \text{ Hz}) \) together with the sign (if experimentally determined) are given in square brackets; \(^b\) See also Ref. [16] for \( \delta ^{13}C \) values and coupling constants \( ^{11}J(^{119}Sn, ^{13}C) \); the values \( ^{11}J(^{119}Sn, ^{13}C) \) given in [16] differ considerably from those reported here, probably because of a marked dependence on concentration; most of the data and signs of coupling constants given here stem from Ref. [15], however the data for C(3) and C(4) in Table I of [15] have to be exchanged. \(^c\) In [14] the data for C(3) and C(4) in Table I of [15] have to be exchanged. \(^d\) Other \( \delta ^{13}C \) values from solid-state CP/MAS measurements: 127.2 (Sn-C); 172.1 (broad, BCH); 37.3, 29.5 (CH\(_2\)); 24.7, 23.6 (–CH\(_3\)–); 19.8 (broad), 10.6 (BEt); 26.3, 19.8 (Et); 21.2, 18.1 (Me); e Other \( \delta ^{13}C \) values from solid-state CP/MAS measurements: 127.2 (Sn-C); 172.1 (broad, BCH); 37.3, 29.5 (CH\(_2\)); 24.7, 23.6 (–CH\(_3\)–); 19.8 (broad), 10.6 (BEt); 26.3, 19.8 (Et); 21.2, 18.1 (Me); f Other \( \delta ^{13}C \) values from solid-state CP/MAS measurements: 127.2 (Sn-C); 172.1 (broad, BCH); 37.3, 29.5 (CH\(_2\)); 24.7, 23.6 (–CH\(_3\)–); 19.8 (broad), 10.6 (BEt); 26.3, 19.8 (Et); 21.2, 18.1 (Me); g Other \( \delta ^{13}C \) values from solid-state CP/MAS measurements: 127.2 (Sn-C); 172.1 (broad, BCH); 37.3, 29.5 (CH\(_2\)); 24.7, 23.6 (–CH\(_3\)–); 19.8 (broad), 10.6 (BEt); 26.3, 19.8 (Et); 21.2, 18.1 (Me); h Other \( \delta ^{13}C \) values from solid-state CP/MAS measurements: 127.2 (Sn-C); 172.1 (broad, BCH); 37.3, 29.5 (CH\(_2\)); 24.7, 23.6 (–CH\(_3\)–); 19.8 (broad), 10.6 (BEt); 26.3, 19.8 (Et); 21.2, 18.1 (Me).
the broadened $^{15}$N NMR signals observed for 2b and 3 it was not possible to determine the coupling constant $^{2}J(^{119}\text{Sn},^{15}\text{N})$ from the $^{15}$N NMR spectra.

The $^{119}$Sn NMR spectrum of 1 shows a single broad resonance, and it was possible to measure $^{2}J(^{119}\text{Sn},^{15}\text{N}) = +108.2$ Hz from the $^{15}$N satellites in the $^{119}$Sn NMR spectra by using Hahn-echo extended (HEED) INEPT pulse sequences [15,20]. In contrast, the $^{119}$Sn NMR signal of 2b is rather sharp ($\delta_{1/2} < 10$ Hz) which means that the magnitude of $|^{2}J(^{119}\text{Sn},^{14}\text{N})|$ and therefore also that of $|^{2}J(^{119}\text{Sn},^{15}\text{N})|$ must be small. The $^{119}$Sn NMR signal of 3 is somewhat broader than for 2b. However this broadening is due to non-resolved scalar $^{119}$Sn–$^{11}$B coupling across three bonds rather than to $^{2}J(^{119}\text{Sn},^{14}\text{N})$.

The presence or absence of the lone pair of electrons at nitrogen affects various coupling constants in pyridine, pyridine derivatives and in pyridinium cations [3–5]. The trends of $J(^{119}\text{Sn},^{1}\text{H})$ or of $J(^{119}\text{Sn},^{13}\text{C})$ in 1–3 should correspond to those found for $^{1}\text{H}–^{15}\text{C}$ or $^{1}\text{H}–^{1}\text{H}$ couplings in pyridine and in the pyridinium cation. Since all signs of the relevant coupling constants in 1–3 have been determined, this comparison is now feasible. Removal of a “cis lone pair” normally causes a decrease in $^{1}K$ [3]. This is not found in pyridine ($^{1}J(^{13}\text{C},^{1}\text{H}) = +177.4$ Hz) and in the pyridinium cation ($^{1}J(^{13}\text{C},^{1}\text{H}) = +190.7$ Hz) because of compensation of increased $\sigma$-inductive effects in the cation. However, in the borane adducts 2a, 2b and 3 this expectation is fulfilled since the

![Scheme 1](image-url)
Fig. 2. Contour plot of the 2D $^{13}C^2/{^1}H$ HETCOR spectrum of 3 based on the long range coupling constant $J(^{13}C^2, {^1}H_{SnMe})$ ($\approx 1.5$ Hz; see experiment A in Scheme 1). The negative tilt of the satellite cross peaks indicates that the signs of $^1K(^{119}Sn, {^1}H_{SnMe})$ (known to be <0) and $^4K(^{119}Sn, ^{13}C^2)$ (>0) are opposite.

Fig. 3. Contour plot of the 2D $^{13}C^2/{^1}H$ HETCOR spectrum of 3 based on the long range coupling constants $J(^{13}C^2, {^1}H_{6})$ ($= 6.0$ Hz; see experiment G in Scheme 1). The positive tilt of the satellite cross peaks indicates that the signs of $^1K(^{119}Sn, {^1}H_{6})$ (>0) and $^4K(^{119}Sn, ^{13}C^2)$ (>0) are alike.

$^1K(^{119}Sn, ^{13}C^2)$ values are all smaller than in 1. The $^2K(^{119}Sn, ^{15}N)$ value in 1 is large and positive, whereas it is small (sign and magnitude not determined) in 2b and 3. This trend agrees with that for $^2K(^{15}N, ^{1}H_{2,6})$ in pyridine and in the pyridinium cation. There is a markedly positive contribution of the lone pair of electrons at the nitrogen atom to $^3K(^{119}Sn, ^{13}C^3)$ in 1 as compared with 2 and 3, and this is again true for $^2K(^{13}C^3, ^{1}H^2)$ in pyridine and in the pyridinium cation [3]. There is only a small difference in the $^3K(^{119}Sn, ^{13}C^4)$ values between 1 and 2, similar to the situation for $^3K(^{13}C^4, ^{1}H^2)$ in pyridine and [C$_5$H$_5$NH]$^+$. The vicinal $^{119}Sn-C^4$ and $^{119}Sn-H^3$ couplings in 3 differ significantly from those in 2a and 2b. This points towards the influence of the deviation from the ideal planar bicyclic structure (vide supra). If the lone pair of electrons is attached to the intervening nitrogen atom (1), there is a strong positive contribution to $^3K(^{119}Sn, ^{13}C^6)$, in accord with the findings for $^3K(^{13}C^6, ^{1}H^2)$ in the parent compounds (pyridine: $3J(^{13}C^6, ^{1}H^2) = 8.5$ Hz; [C$_5$H$_5$NH]$^+$: $3J(^{13}C^6, ^{1}H^2) = 5.10$ Hz).

The changes in the $\delta^{13}C$(pyridine) values in 2a, 2b and 3 with respect to 1 are small and non-char-
acetic acid. The solid-state $\delta^{13}$C values of 3 are very similar to the data in solution. Nitrogen nuclear magnetic shielding increases significantly if the lone pair of electrons becomes engaged in the coordinate N–B bond. This is expected, considering the deshielding influence of B$_n$-induced circulation of charge connected with $\pi$*-transitions on nitrogen nuclear magnetic shielding [1c,21]. The $\delta^{119}$Sn value of 3 ($\delta$ = 119.7) is close to the range known for 1-stanna-2,5-cyclohexadienes [22] and must be regarded as typical of that particular structural unit. The $\delta^{119}$Sn value for a solid sample of 3 ($\delta$ = 122.3) differs little from the solution-state value, in agreement with the solution- and solid-state $\delta^{13}$C values for 3. Considering the great sensitivity of $^{119}$Sn nuclear shielding to small structural changes [22], this strongly suggests that there can be only minor differences between the surrounding of the tin atom in liquid and solid state. The similarity of the $\delta^{119}$Sn values of 1 ($\delta$ = 52.9) and 2b ($\delta$ = 53.9) cannot be explained as yet and the same is true for the $^{119}$Sn deshielding in the case of 2a ($\delta$ = 30.4). The values $^{1}$J$(^{119}$Sn,$^{13}$C$_{Me})$ in the liquid (325.6 Hz) and in the solid state (321 Hz) are similar, as expected from the chemical shift data. The proposed relationship between $^{1}$J$(^{119}$Sn,$^{13}$C$_{Me})$ and the bond angle C$_{Me}$SnC$_{Me}$ [23] does not include compound 3. We have already observed several failures of this relationship [24].

**Experimental**

All preparative work and handling of samples was carried out in an inert atmosphere (Ar or N$_2$). Compound 1 [16], Et$_3$B [25], dimeric 9-borabicyclo[3.3.1]nonane [26] and (E)-2-chlorodimethylstannyl-3-diethylboryl-2-pentene [7] were prepared according to literature procedures. NMR measurements in solution were performed using Bruker ARX 250, AC 300 and AM 500 instruments, all equipped with multinuclear units: $^{13}$C NMR [for values relative to Me$_4$Si: $^{1}$H(C$_6$D$_5$H) = 7.15, $\delta^{13}$C(C$_6$D$_5$) = 128.0]; $^{11}$B NMR [external standard: BF$_3$-OEt$_2$, $\Xi$(11B) = 22.145 MHz, $\Xi$(11B) = 32.083971 MHz]; $^{15}$N, $^{14}$N NMR [external standard: neat MeNO$_2$, $\Xi$(14N) = 7.226455 MHz; $\Xi$(15N) = 10.136767 MHz]; $^{119}$Sn NMR [external standard: Me$_3$Sn; $\Xi$(119Sn) = 37.290665 MHz].

Solid-state $^{13}$C and $^{119}$Sn CP/MAS NMR spectra of compound 3 were measured using a Bruker MSL 300 spectrometer, and the compound was packed into an airtight insert [27] fitting exactly into the commercial ZrO$_2$-7-mm-rotor for the double bearing probehead.

The 9-borabicyclo[3.3.1]nonane adduct 2b was isolated as a colourless powder (m.p. 88 °C) after recrystallization from pentane. – EI-MS (70 eV): m/e (%) = 365 M$^+$ (18), 228 C$_7$H$_{10}$Sn (100). – 500 MHz $^1$H NMR; see footnotes Table II.

3,4,4-Triethyl-1,4-dihydro-1,1,2-trimethyl-4a,l,4-azastannaborcino-naphthalene (3)

After cooling a solution of 0.79 g (5 mmol) of 2-bromopyridine in 30 ml of ether to –78 °C, 3.12 ml of a solution of n-BuLi in hexane (5 mmol) was added, and the reaction mixture was stored at –78 °C for 12 h. Then a solution of 1.6 g (5 mmol) of (E)-2-chloro(dimethyl)stannyl-3-diethylboryl-2-pentene [7] in 10 ml of THF was added at –78 °C. The reaction mixture was allowed to reach room temperature. After filtration and removal of all volatile material a yellowish solid was left. This was treated with hexane, and from the hexane-solution 1.65 g (91%) of compound 3 was obtained as a colourless, crystalline material (m.p. 67 °C). – 500 MHz $^1$H NMR, see footnotes Table II.

**Acknowledgements**

Support of this work by the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie is gratefully acknowledged. We thank Prof. Dr. R. Köster (Mülheim a. d. Ruhr) for a generous gift of triethylborane and Dr. A. Sebald (Bayerisches Geoinstitut, Bayreuth) for recording the $^{13}$C and $^{119}$Sn CP/MAS NMR spectra.

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B. Wrackmeyer et al. · 2-Stannylpyridine-Borane Adducts

[9] Further details of the crystal structure analysis are available on requests from the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-76344 Eggenstein-Leopoldshafen (FRG) on quoting the depository number CSD 401468, the names of the authors, and the journal citation.