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4. The standard procedure for preparation of the dialkyl-amino-substituted derivatives of $\mathrm{LiAlH}_{4}$ is described in ref. 3 a. The ${ }^{27} \mathrm{AI}-\mathrm{NMR}$ spectra of LTDEA, LTDBA, and LTDHA showed a broad singlet at $\delta 120,128$ and 129.5 ppm , respectively, relative to $\mathrm{Al}\left(\mathrm{H}_{2} \mathrm{O}\right)_{6}{ }^{3+}$.

## Synthesis of Homoleptic Ionic Indium(III) Thiolates and Molecular Structure of $\left(\mathbf{N M e}_{4}\right)\left[\mathbf{I n}\left(\mathrm{SC}_{6}\right.\right.$ $\left.\left.\mathrm{H}_{3}-\mathbf{2 , 6}-\mathrm{Me}_{2}\right)_{4}\right]$

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Indium and its compounds have been widely used in various fields ranging from nuclear medicine to microelectronics. ${ }^{1-6}$ Particularly, sulfur-containing indium complexes found their use in both extremes. Indium(III) complexes of thiocarbamate, alkylxanthate and dialkyldithiophosphate ligands have been exploited as blood cell labelling agents with reduced cell toxicity.' On the other hand, organoindium precursors involving sulfur have been employed in the improved preparation of thin transparent conducting indium oxide film. ${ }^{8}$ Prompted by the foregoing significance of sulfur-containing indium complexes and the lack of X-ray crystallographically characterized homoleptic indium thiolate, the tetramethylammonium salts of $\left[\mathrm{In}(\mathrm{SR})_{4}\right]^{-}\left(1: \mathrm{SR}=\mathrm{SC}_{6} \mathrm{H}_{5} ; 2\right.$ : $\mathrm{SR}=\mathrm{SC}_{6} \mathrm{H}_{3}-2,6-\mathrm{Me}_{2}$ ) were prepared and structurally characterized, accounts of which are described in this report.

The methanolic solution of NaSR, generated in situ by the reaction of sodium metal ( 2 mmol ) in $c a .20 \mathrm{ml}$ of $\mathrm{CH}_{3} \mathrm{OH}$

Table 1. Summary of Crystal Data, Intensity Collection, and Final Structure Refinement for $\left(\mathrm{NMe}_{4}\right)\left[\operatorname{In}\left(\mathrm{SC}_{6} \mathrm{H}_{3}-2,6-\mathrm{Me}_{2}\right)_{4}\right]$

| formular | $\mathrm{C}_{36} \mathrm{H}_{48} \mathrm{InNS}_{4}$ |
| :---: | :---: |
| f.w. | 737.8 |
| crystal system | monoclinic |
| space group | $P 2_{1} / n$ |
| $a, \AA$ | 10.894(3) |
| b, A | 19.313(3) |
| c, $\AA$ | 17.393(3) |
| $\boldsymbol{\beta}$. deg | 91.74(2) |
| $V, \AA^{3}$ | 3657.7(12) |
| $Z$ | 4 |
| temp, ${ }^{\circ} \mathrm{C}$ | 23 |
| $F(000)$ | 1524.0 |
| radiation source | $\mathrm{MoK}_{\mathrm{a}}(\lambda=0.71073 \AA)$ |
| scan mode | $\omega / 2 \theta$ |
| $2 \theta$ limit, deg | 2-50 |
| no. of collected data | 4970 |
| no. of unique data | 4785 |
| no. of obsd data ( $F>6 \sigma(F)$ ) | 4363 |
| no. of variables | 436 |
| $R$ | 0.0376 |
| $\boldsymbol{R}^{\boldsymbol{\omega}}$ | 0.0410 |

followed by the addition of $2 \mathrm{mmol} \mathrm{HSR}\left(\mathrm{SR}=\mathrm{SC}_{6} \mathrm{H}_{5} ; \mathrm{SC}_{6} \mathrm{H}_{3}{ }^{-}\right.$ $2,6-\mathrm{Me}_{2}$ ), was anaerobically treated with a solution of $\mathrm{InCl}_{3}$ ( 0.5 mmol ) in $\mathrm{CH}_{3} \mathrm{OH}$. The reaction mixture was stirred for 3 hrs at ambient temperature and then was treated with a solution of $\mathrm{NMe}_{4} \mathrm{Br}(0.5 \mathrm{mmol})$ in $\mathrm{CH}_{3} \mathrm{OH}$, resulting in the precipitation of white microcrystalline solids which were collected and washed firstly with $\mathrm{CH}_{3} \mathrm{OH}$ and with $\mathrm{Et}_{2} \mathrm{O}$ several times. Recrystallization of the dried solid product from $\mathrm{DMF} / \mathrm{Et}_{2} \mathrm{O}$ afforded analytically pure white crystalline ( $\mathrm{NMe}_{4}$ ) 1 and ( $\mathrm{NMe}_{4}$ ) 2 in the yield of 51 and $60 \%$, respectively. ${ }^{9}$ Analytical and spectroscopic data suggest $1: 1: 4$ ratio of ( $\left.\mathrm{NMe}_{4}\right)^{+}: \operatorname{In}(\mathrm{III}):(\mathrm{SR})^{-}$and single crystal X-ray diffraction study reveals the composition of $\left(\mathrm{NMe}_{4}\right)\left[\operatorname{In}\left(\mathrm{SC}_{6} \mathrm{H}_{3}-2,6\right.\right.$ $\left.\left.\mathrm{Me}_{2}\right)_{4}\right]^{10.11}$ In Table 1 and 2, crystallographic data and the atomic coordinates of non-hydrogen atoms for ( $\mathrm{NMe}_{4}$ ) $\cdot 2$ are listed, respectively.
The crystal structure of $\left(\mathrm{NMe}_{4}\right) \cdot 2$ consists of well-separated cations and anions. The structure of $\mathrm{NMe}_{4}{ }^{+}$is not unusual and will not be discussed any further. As the ORTEP view in Figure 1 and the molecular model indicate, the geometry of the entire anion 2 lacks in symmetry, leading to $C_{1}$ point group symmetry. The $C_{1}$ nature of 2 is rather unusual since $S_{4}$ symmetry is common for many $\mathrm{R}_{4} \mathrm{M}$ type compounds. ${ }^{13}$ In fact, the $\left[\mathrm{M}(\mathrm{SR})_{4}\right]$ units in $\left(\mathrm{PPh}_{4}\right)_{2}\left[\mathrm{Fe}(\mathrm{SPh})_{4}\right],{ }_{4}^{14}$ $\left(\mathrm{NE}_{4}\right)\left[\mathrm{Fe}\left(\mathrm{SPh}_{4}\right)_{4}\right]^{15}$ and $\left(\mathrm{NEt}_{4}\right)\left[\mathrm{Fe}\left(\mathrm{SC}_{6} \mathrm{H}-2,3,5,6-\mathrm{Me}_{4}\right)_{4}\right]^{16}$ are known to have $S_{4}$ point group symmetry although the same unit in $\left(\mathrm{NEt}_{4}\right)\left[\mathrm{Ga}\left(\mathrm{SPh}_{4}\right)\right]^{17}$ has a pseudo $C_{2}$ axis. Four $\mathrm{SC}_{6} \mathrm{H}_{3}-$ $2,6-\mathrm{Me}_{2}$ groups in 2 are arranged such that only two groups are related by two-fold axis while the others are not. For example, for a given pseudo $C_{2}$ axis defined by S1InS2 and S3InS4 planes, the Sl-aryl and the S2-aryl groups are only symmetrically related. Similarly, only the 51 -aryl and the S3-aryl groups are symmetric with respect to a $C_{2}$ axis that bisects the $\mathrm{S} 1-\mathrm{In}-\mathrm{S} 2$ and the $\mathrm{S} 1-\mathrm{In}-\mathrm{S} 3$ angles. Taken alone,

Table 2. Atomic Coordinates for $\left(\mathrm{NMe}_{4}\right)\left[\operatorname{In}\left(\mathrm{SC}_{6} \mathrm{H}_{3}-2,6-\mathrm{Me}_{2}\right)_{4}\right]$

| Atom | * | $y$ | 2 | $U_{\text {an }}\left(\AA^{\text {a }}\right.$ ) |
| :---: | :---: | :---: | :---: | :---: |
| In | 0.2082(<1) | 0.1932(<1) | 0.0269(<1) | 0.0339 |
| S1 | $0.0659(1)$ | $0.1858(1)$ | $-0.0857(1)$ | 0.0496 |
| S2 | $0.2483(1)$ | $0.0756(1)$ | $0.0771(1)$ | 0.0511 |
| S3 | 0.4072(1) | $0.2439(1)$ | $-0.0083(1)$ | 0.0469 |
| S4 | 0.1107(1) | $0.2664(1)$ | 0.1226(1) | 0.0448 |
| C 11 | 0.1448(4) | 0.1315(3) | -0.1521(3) | 0.0375 |
| C 12 | $0.1278(5)$ | $0.0597(3)$ | -0.1496(3) | 0.0453 |
| C13 | 0.1801(6) | $0.0193(3)$ | -0.2054(4) | 0.0594 |
| C14 | 0.2459(6) | 0.0478(4) | -0.2643(4) | 0.0684 |
| C 15 | 0.2621 (5) | $0.1183(4)$ | -0.2669(3) | 0.0599 |
| Cl 6 | $0.2130(5)$ | $0.1619(3)$ | $-0.2103(3)$ | 0.0449 |
| C17 | 0.0579(6) | 0.0249(4) | -0.0865(4) | 0.0654 |
| C18 | $0.2346(7)$ | 0.2392(3) | $-0.2141(4)$ | 0.0714 |
| C21 | 0.3318(5) | $0.0886(3)$ | $0.1654(3)$ | 0.0424 |
| C 22 | 0.46206) | 0.0855(3) | 0.1681(4) | 0.0600 |
| C23 | $0.5231(7)$ | $0.0945(4)$ | 0.2365 (5) | 0.0796 |
| C24 | 0.4604(9) | $0.1057(4)$ | 0.3040(5) | 0.0954 |
| C25 | 0.3328(8) | 0.1057(4) | 0.3031(4) | 0.0809 |
| C26 | 0.2667(6) | $0.0975(3)$ | $0.2344(3)$ | 0.0529 |
| C27 | $0.532066)$ | $0.0743(4)$ | 0.0953 (5) | 0.0817 |
| C28 | 0.1295(6) | 0.0963 (4) | 0.2357(4) | 0.0714 |
| C31 | 0.4062(4) | $0.3276(3)$ | 0.0357(3) | 0.0419 |
| C32 | 0.3782(5) | $0.3858(3)$ | -0.0106(4) | 0.0548 |
| C33 | 0.3840(6) | 0.4058(3) | $0.0239(5)$ | 0.0702 |
| C34 | 0.4184(6) | 0.4590(4) | 0.0987(5) | 0.0805 |
| C35 | 0.4463(6) | $0.4016(4)$ | $0.1435(4)$ | 0.0723 |
| C36 | 0.4385(5) | $0.3354(3)$ | 0.1126(3) | 0.0539 |
| C37 | $0.3429(8)$ | 0.3795(5) | -0.0957(4) | 0.0865 |
| C38 | $0.4624(7)$ | $0.2737(4)$ | $0.1640(4)$ | 0.0717 |
| C41 | -0.0292(4) | 0.2961 (3) | $0.0781(3)$ | 0.0376 |
| C42 | $-0.0385(5)$ | 0.3646(3) | 0.0523(3) | 0.0512 |
| C43 | -0.1513(5) | $0.3884(3)$ | 0.0234(3) | 0.0590 |
| C44 | $-0.2530(5)$ | 0.3468(4) | 0.02023 ) | 0.0591 |
| C45 | $-0.2436(5)$ | 0.2795(3) | 0.0444(3) | 0.0540 |
| C46 | -0.1322(4) | 0.2527(3) | 0.0737(3) | 0.0408 |
| C47 | 0.0706 (7) | $0.4130(4)$ | 0.0582(6) | 0.0902 |
| C48 | -0.1269(6) | $0.1785(3)$ | $0.1013(3)$ | 0.0584 |
| N01 | $0.1708(4)$ | 0.3555(2) | 0.3417(2) | 0.0382 |
| C01 | 0.1923 (10) | $0.2801(4)$ | 0.3416(5) | 0.1035 |
| C02 | $0.1944(8)$ | 0.3801(4) | 0.4206(4) | 0.0903 |
| C03 | $0.0454(7)$ | $0.3696(5)$ | $0.3169(6)$ | 0.1097 |
| C 04 | $0.2575(7)$ | $0.3887(4)$ | 0.2895(4) | 0.0935 |

the $\ln \mathrm{S}_{4}$ unit is distorted from $T_{d}$ symmetry by a slight compression along the noncrystallographic $S_{4}$ axis as indicated by the presence of the S1-In-S3 (111.8(1) ${ }^{\circ}$ ) and S2-In-S4 ( $110.9(1)^{\circ}$ ) angles greater than $109.5^{\circ}$ and other S-In-S angles similar (S1-In-S2, 109.4(1) ${ }^{\circ}$; S3-In-S4, 109.8(1) ${ }^{\circ}$ ) to or smaller (S1-In-S4, 107.3(1) ${ }^{\circ}$; S2-In-S3, 107.7(1) ${ }^{\circ}$ ) than $109.5^{\circ}$. The average value ( $88.2^{\circ}$ ) of three dihedral angles between two SInS planes that define $C_{2}$ axis also reflects the extent of distrotion of the $\operatorname{InS}_{4}$ unit.

The arylthiolate ligands in the $\left[\mathrm{M}(\mathrm{SR})_{4}\right]$ unit are known to adopt a unique M-S-Aryl conformation in which the planes


Figure 1. ORTEP diagram of the anion (2) of $\left(\mathrm{NMe}_{4}\right)\left[\operatorname{In}\left(\mathrm{SC}_{6} \mathrm{H}_{3}\right.\right.$. $\left.\left.2,6 \mathrm{Me}_{2}\right)_{4}\right]$.

Table 3. Selected Bond Distances ( $\AA$ ) and Bond Angles (deg) for ( $\mathrm{NMe}_{4}$ ) $\left[\operatorname{In}\left(\mathrm{SC}_{6} \mathrm{H}_{3}-2,6-\mathrm{Me}_{2}\right)_{4}\right]$

| In-S1 | $2.452(1)$ | In-S2 | $2.474(1)$ |
| :--- | :--- | :--- | :--- |
| In-S3 | $2.467(1)$ | In-S4 | $2.461(1)$ |
| S1-C11 | $1.798(5)$ | S2-C21 | $1.779(5)$ |
| S3-C31 | $1.790(5)$ | S4-C41 | $1.785(5)$ |
| C11-C12 | $1.398(7)$ | C12-C13 | $1.384(8)$ |
| C13-C14 | $1.382(9)$ | C14-C15 | $1.373(10)$ |
| C15-C16 | $1.412(8)$ | C11-C16 | $1.407(7)$ |
| C12-C17 | $1.512(8)$ | C16-C18 | $1.514(8)$ |
| C21-C22 | $1.415(8)$ | C22-C23 | $1.365(10)$ |
| C23-C24 | $1.387(12)$ | C24-C25 | $1.391(12)$ |
| C25-C26 | $1.379(9)$ | C21-C26 | $1.417(8)$ |
| C22-C27 | $1.517(10)$ | C26-C28 | $1.504(9)$ |
| C31-C32 | $1.405(8)$ | C32-C33 | $1.396(9)$ |
| C33-C34 | $1.343(12)$ | C34-C35 | $1.386(11)$ |
| C35-C36 | $1.385(9)$ | C31-C36 | $1.382(8)$ |
| C32-C37 | $1.527(10)$ | C36-C38 | $1.510(10)$ |
| C41-C42 | $1.398(7)$ | C42-C43 | $1.392(8)$ |
| C43-C44 | $1.373(9)$ | C44-C45 | $1.371(9)$ |
| C45-C46 | $1.402(7)$ | C41-C46 | $1.403(7)$ |
| C42-C47 | $1.510(9)$ | C46-C48 | $1.505(7)$ |
| N01-C01 | $1.475(9)$ | N01-C02 | $1.467(8)$ |
| N01-C03 | $1.446(9)$ | N01-C04 | $1.475(9)$ |
| S1-In-S2 | $109.4(<1)$ | S1-In-S3 | $111.8(<1)$ |
| S1-In-S4 | $107.4(<1)$ | S2-In-S3 | $107.7(<1)$ |
| S2-In-S4 | $110.9(<1)$ | S3-In-S4 | $109.8(<1)$ |
| In-S1-C11 | $103.8(2)$ | In-S2-C21 | $105.1(2)$ |
| In-S3-C31 | $103.8(2)$ | In-S4-C41 | $105.6(2)$ |

of the aryl rings are parallel ${ }^{14.15 .17}$ or perpendicular ${ }^{16.17}$ to the M-S bonds. The latter thiolate conformation observed in [Fe
$\left.\left.\left(\mathrm{SC}_{6} \mathrm{H}-2,3,5,6-\mathrm{Me}_{4}\right)_{4}\right]^{-(3)}\right)^{16}$ indicates the role of $o$-methyl groups in preventing the aryl rings from lying parallel to the $\mathrm{Fe}-\mathrm{S}$ bonds. In the anion 2, such a steric influence is operative resulting in rather random perpendicular conformations. For each In-S-Aryl group, the dihedral angle between the InSC plane and aryl ring is $93.6(2)^{\circ}$ (Sl case), 86.5 (2) ${ }^{\circ}$ (S2 case), $98.9(2)^{\circ}\left(\mathrm{S} 3\right.$ case) or $77.2^{\circ}$ ( S 4 case). The presence of $o$-substituents in the aryl ring also affects the In-S-C angless listed in the Table 3 and the average value of $104.6^{\circ}$ can be compared to the Fe-S-C angles (average $\left.102.4^{\circ}\right)^{16}$ of 3.
The average $\mathrm{In}-\mathrm{S}$ distance of $2.464 \AA$ in 2 is in good agreement with the sum of Shannon's ionic radii ${ }^{18}$ of tetrahedral $\operatorname{In}(I I I)(0.76 \AA)$ and $S^{2-}(1.70 \AA)$ as well as with that (average $2.450 \AA)$ of $\left(\mathrm{PPh}_{4}\right)\left[\mathrm{BrIn}(\mathrm{SPh})_{3}\right]{ }^{12}$ For the sake of comparison, it is also interesting to note that gallium(III), the lighter congener of indium(III), and high-spin iron(III), a similar transition element analogue of gallium(III), ${ }^{\text {b9 }}$ form M-S distances of $2.260 \AA(\mathrm{M}=\mathrm{Ga})$ and $2.297 \AA(\mathrm{M}=\mathrm{Fe})$ in $\left[\mathrm{M}(\mathrm{SPh})_{4}\right]^{-}$ anions.
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9. ( $\mathrm{NMe}_{4}$ ) 1: Anal. Found (Calc.) for $\mathrm{C}_{28} \mathrm{H}_{32} \mathrm{InNS}_{4}$ : C, 52.2 (53.8); H, 5.4 (5.2); N, 2.1 (2.2); S, 18.8 (20.5) \%; ${ }^{1} \mathrm{H}-\mathrm{NMR}$ ( $d_{6}$-DMSO), 3.1 ( $\mathrm{s}, 12 \mathrm{H}, M e_{4} \mathrm{~N}$ ), 6.9-7.3 ( $\mathrm{m}, 20 \mathrm{H}, \mathrm{C}_{6} H_{5}$ ) ppm. ( $\mathrm{NMe}_{4}$ ) 2: Anal. Found (Calc.) for $\mathrm{C}_{36} \mathrm{H}_{48} \mathrm{InNS}_{4}$ : C,
58.5 (58.6); H, 6.8 (6.6); N, 2.3(1.9); S, 17.6 (17.4) \%; ${ }^{1} \mathrm{H}-\mathrm{NMR}\left(d_{6}-\mathrm{DMSO}\right), 2.1$ ( $\mathrm{s}, 24 \mathrm{H}, \mathrm{S}-\mathrm{C}_{6} \mathrm{H}_{3}-2,6-\mathrm{Me} e_{2}$ ), 3.1 ( s , $12 \mathrm{H}, M e_{4} \mathrm{~N}$ ), 6.8 (m, 12H, S-C $\mathrm{C}_{6} H_{3}-2,6-\mathrm{Me}_{2}$ ) ppm.
10. Crystallographic data for ( $\mathrm{NMe}_{4}$ ) 2 is given in Table 1. Diffraction data were collected on an Enraf-Nonius CAD4 single crystal X-ray diffractometer at room temperature. The data did not show any significant decay during data collection. The unit cell was determined on the basis of accurately centered reflections that were widely separated in reciprocal space with $17^{\circ}<2 \theta<23^{\circ}$. The data was corrected for Lorentz, polarization and absorption effects. The indium atom was located by the Patterson map (SHELXS 86) and the remaining nonhydrogen atoms were located by use of block-matrices least-square procedures and $\Delta F$ syntheses (SHELX 76). All calculations were performed on the CRAY2S/4-128 Supercomputer. All phenyl groups were refined as non-rigid groups and all hydrogens included in calculated positions, $d_{\text {C. }}=1.08$ $\AA$ A. A final difference electron density was essentially featureless with maximum peak of $0.54 \mathrm{e}^{-3}$. The anisotropic thermal parameters and the final values of observed and calculated structural factors are available from the corresponding author upon request.
11. To our knowledge, no X-ray crystallographically characterized homoleptic $\operatorname{In}(I I I)$ tetrathiolate has been reported to date although $\mathrm{Na}^{+}$and $\mathrm{NEt}_{4}{ }^{+}$salts of $\left[\ln (\mathrm{SPh})_{4}\right]^{-}$ anion were previously prepared by reacting $\operatorname{In}\left(\mathrm{SPh}_{3}\right)_{3}$ with NaSPh and further with $\mathrm{NEt}_{4} \mathrm{Cl}^{12}$
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19. Gallium(III) and high-spin iron(III) are known to have similar coordination chemistry owing to their similar charges and ionic $\operatorname{radii}(\mathrm{Ga}(\mathrm{III})=0.61 \AA ; \mathrm{Fe}(\mathrm{III})=0.63 \AA){ }^{18}$
