Reactions of Tetraalkyldiindane(4), $R_2In{-}InR_2$ ($R = CH(SiMe_3)_2$), with *tert*-Butyl and Phenyl Isonitriles. Formation of Adducts with Retention of the In−In Bond[†]

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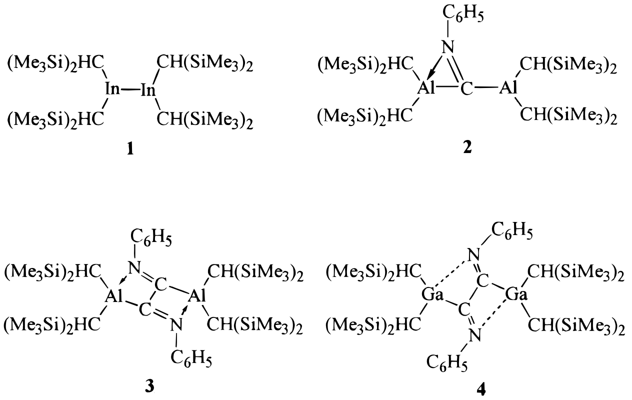
*Summary: Tetrakis[bis(trimethylsilyl)methyl]diindane(4) (**1**) reacts with tert-butyl or phenyl isonitrile not by the insertion of the isonitrile carbon atom into its In−In single bond but by the formation of weak adducts with one isonitrile molecule coordinated to each of the coordinatively unsaturated In atoms. While the tert-butyl isonitrile adduct **5** is almost centrosymmetric in the solid state with the largest possible separation between the bulky tert-butyl groups, the phenyl groups are neighbored and parallel in the corresponding phenyl derivative **6**. The In−In distances of both compounds (2.8483 Å on average for **5** and 2.8482(6) Å for **6**) are identical and only slightly lengthened compared with the starting compound **1** (2.828(1) Å).*

Introduction

Organoelement compounds which contain a single bond between the heavier elements of the third main group aluminum,[1] gallium,[2] and indium (**1**)[3] were synthesized and characterized the first time 10 years ago by our group, and subsequently, many important derivatives have been published by others.[4] These compounds showed a remarkable and unprecedented chemical reactivity, and many secondary products were synthesized during the past decade.[5] A very important type of reaction is the insertion of atoms or whole molecules such as isonitriles into the element−element bonds. The insertion of one isonitrile molecule into the Al−Al bond of the aluminum analogue of **1** was easily achieved by the reaction with 1 equiv of the corresponding isonitrile.[6] A three-membered heterocycle was formed by the interaction of the nitrogen lone pair with one of the coordinatively unsaturated Al atoms (**2**), Scheme 1. Only use of phenyl isonitrile in excess resulted in the insertion of a second isonitrile molecule to yield compound **3**, in which a carbon−carbon single bond was formed and each nitrogen atom was coordinated to an Al atom with short Al−N bonds to give two anealated four-membered heterocycles.[6] In contrast, an excess of *tert*-butyl isonitrile yielded a trimeric dialkylaluminum cyanide by the cleavage of the N−C(*tert*-butyl) bond.[7] The gallium analogue of **1** with a Ga−Ga single bond on treatment with isonitriles gave exclusively the products of the two fold insertion (**4**). The insertion of only one RNC molecule has up to now not been realized, even by systematically enlarging the steric demand of the isonitrile or by changing the

Scheme 1



[†] Dedicated to Prof. Dr. P. Jutzi on the occasion of his 60th birthday.

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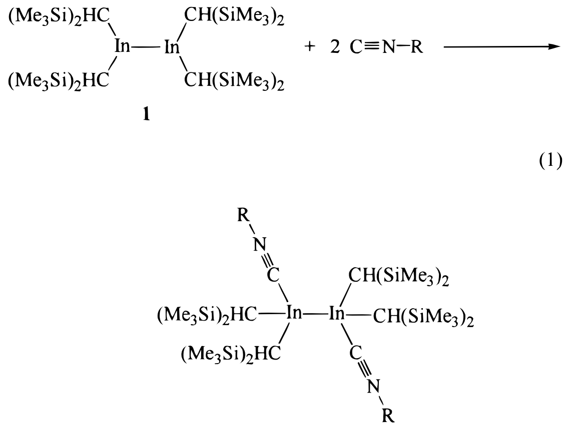
stoichiometric ratios of the reactants.[8] The different behavior of the dialuminum and digallium compounds may be caused by the better steric shielding of the gallium compound due to the shorter Ga−Ga bond, which, once opened, favors the attack of a second isonitrile molecule, or by the weaker Lewis-acidic character of the digallium compound. The molecular structure of **4** showed an important difference in comparison with the corresponding aluminum derivative **3** in that only weak interactions between the nitrogen and the gallium atoms were observed, as deduced from the long Ga−N distances and from temperature-dependent NMR experiments.[8] It is now of particular interest to investigate the behavior of the diindium(4) derivative **1** in its reaction with isonitriles. Surprisingly, a third type of reaction was encountered and is reported here.

Results and Discussion

Treatment of the orange-red diindane(4) compound **1** with an excess of *tert*-butyl or phenyl isonitrile gave bright yellow mixtures, from which, after evaporation of the solvent and the excess of isonitrile, yellow solid residues were isolated. Dissolution of these residues in pure *n*-pentane for recrystallization resulted, however, in orange-red solutions characteristic of **1**, and the NMR spectra in benzene showed the resonances of **1** in the $SiMe_3$ region as well as signals due to the corresponding free isonitriles. Integration of the ^1H NMR spectra revealed a ratio of **1** to the isonitrile of 1:2. The yellow reaction products could only be obtained in a crystalline form when crystallized from a pentane solution in the presence of an excess of the respective isonitrile. For this reason we were unable to characterize the products by NMR spectroscopy, and as expected, the determination of the molar mass in benzene gave only one-third of the calculated formula weight owing to the complete dissociation into three independent molecules. The formulation of the molecular structure of the products **5** (*tert*-butyl isonitrile) and **6** (phenyl isonitrile) as adducts with an intact In−In bond given in eq 1 is thus derived mainly from the results of the crystal structures discussed below. The slow transformation of **5** and **6** into the orange-red diindium(4) compound **1** is observed, even in the solid state at room temperature, but yellow crystals of **5** and **6** can be stored at −30 °C in the dark for several months without decomposition. The melting points (**5**, 78 °C; **6**, 84 °C accompanied by slow decomposition) are lowered compared with the decomposition point of **1** (154 °C), which rapidly decomposes on melting by the precipitation of elemental indium.[3] Insertion of the isonitriles into the In−In bond of the diindium(4) compound **1** was not achieved, even when mixtures of the components were heated in *n*-hexane under reflux. Instead, owing to the low thermal stability of **1**, slow decomposition of **1** was observed after some hours by precipitation of elemental indium and formation of bis(trimethylsilyl)methane.



1

(1)

5: R = CMe_3
6: R = C_6H_5

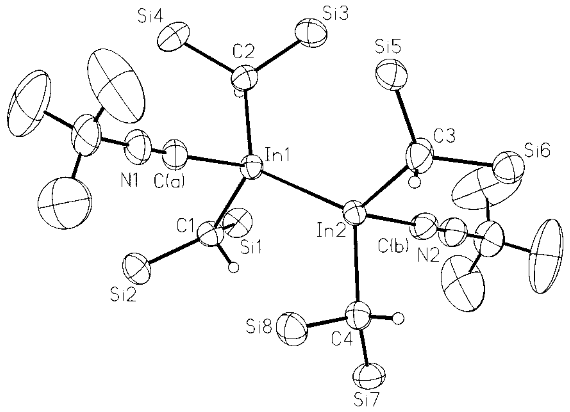


Figure 1. Molecular structure of **5**. The thermal ellipsoids are drawn at the 40% probability level. Methyl groups are omitted for clarity. Important bond lengths (Å) and angles (deg) (corresponding figures of the second molecule in brackets): In1−In2 2.8469(4) [2.8497(5)], In1−C(a) 2.418(5) [2.433(5)], In2−C(b) 2.454(5) [2.444(5)], In1−C1 2.248(4) [2.234(4)], In1−C2 2.242(4) [2.249(4)], In2−C3 2.243(4) [2.245(5)], In2−C4 2.256(4) [2.256(4)], C(a)−N1 1.142 [1.144(5)], C(b)−N2 1.134(5) [1.146(6)], In1−In2−C(b) 100.6(1) [97.7(1)], In2−In1−C(a) 98.3(1) [99.3(1)], In1−In2−C3 125.8(1) [114.2(1)], In1−In2−C4 122.1(1) [125.5(1)], In2−In1−C1 114.3(1) [124.4(1)], In2−In1−C2 126.8(1) [122.2(1)].

The crystal structures of both compounds are depicted in Figures 1 (**5**) and 2 (**6**). Both isonitriles did not insert into the In−In bond but formed adducts in which each indium atom is terminally coordinated by one isonitrile ligand and the In−In bond is not cleaved. Both compounds show identical In−In bond lengths (2.8483 Å on average for **5** and 2.8482(6) Å for **6**), which are only slightly lengthened compared with the starting compound **1** (2.828(1) Å).[3] The In−C bonds to the bis(trimethylsilyl)methyl substituents are, however, significantly influenced by the increase of the coordination number of the indium atoms and are lengthened from 2.196(5) Å of the starting compound **1** to 2.247 and 2.246 Å (on average) in compounds **5** and **6**. The distances between indium and the carbon atoms of the isonitrile groups are very long (2.437 Å on average in **5** and 2.449(3) Å in **6**). They are lengthened by ca. 0.2 Å compared to normal In−C single bonds and possibly indicate a weak coordinative interaction, which may cause the facile dissociation observed in solution. In contrast, the B−C and Al−C distances detected in adducts of isonitriles with boron and aluminum com-

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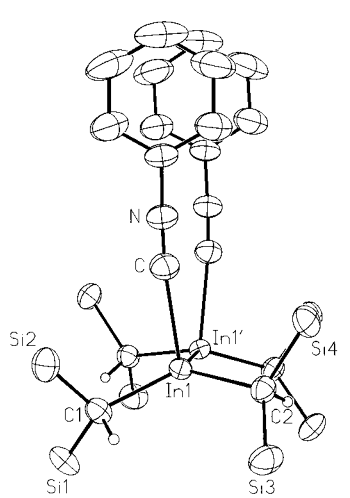

Figure 2. Molecular structure of **6**. The thermal ellipsoids are drawn at the 40% probability level. Methyl groups are omitted for clarity. Important bond lengths (Å) and angles (deg): In1−In1′ 2.8482(6), In1−C 2.449(3), In1−C1 2.235(2), In1−C2 2.257(3), C−N 1.145(3), In1′−In1−C1 124.96(8), In1′−In1−C2 114.17(8), In1′−In1−C 97.66(8), C1−In1−C2 113.4(1), C1−In1−C 100.0(1), C2−In1−C 99.9(1) (In′ calculated by $-x + 2$, y, $-z + 0.5$).

pounds[9] are only slightly lengthened with respect to the corresponding element−carbon single bonds, which further supports the description of **1** as a very weak Lewis acid. The C−N triple bond lengths (1.142 Å on average for **5** and 1.145(3) Å for **6**) are similar to those found in uncoordinated isonitrile groups (about 1.15−1.16 Å).[10] Similar short distances were observed in compounds in which isonitrile groups act as pure σ-donors, as in adducts with boranes or organoaluminum derivatives.[9] Isonitrile ligands bound to transition metals show differing C−N distances, which seem to depend, in part, on the extent of the π-back-bonding.[11] The In−C−N−C groups of the coordinated isonitrile ligands are almost ideally linear with In−C−N and C−N−C angles in compound **5** of 176.8° and 178.5° (on average) and in compound **6** of 171.9(3)° and 177.4(3)°, respectively.

While the bond lengths and angles in both molecules are quite similar, the molecular conformations differ significantly. As expected for a compound that is isoelectronic to a distannane derivative, the *tert*-butyl adduct **5** adopts an ethane-like staggered conformation across the In−In bond with the isonitrile groups almost ideally in an antiperiplanar position. The torsion angles C−In−In−C between the isonitrile carbon atoms are 178.5° and −179.4°. This structure is to be expected the most sterically favorable conformation because it ensures the best separation of the bulky substituents. However, all substituents of the phenyl isonitrile adduct **6** are in the sterically unfavorable eclipsed position, and the torsion angles across the In−In bond are C1−In1−In1′−C2′ = 14.8°, C2−In1−In1′-C1′ = 14.8°, and C−In1−In1′−C′ = 11.3°. This unusual conformation may be caused by an interaction between both of the parallel phenyl groups. Such arene−arene interactions have been verified by theoretical calculations[12] and cause the unusual packing observed in many solid-state structures of aromatic compounds. The shortest separation between the phenyl rings in **6** is 3.710 Å (C3−C3′), and this gradually increases to 3.967 Å (C6−C6′). This is exactly the range for which significant interactions between phenyl groups were discussed.

The IR spectra made an important contribution to the discussion of the bonding situation in both molecules. The stretching vibration of the C−N triple bond increases upon coordination to the indium atoms in comparison to the corresponding vibrations of the free isonitriles by 57 (**5**) and 46 cm^{-1} (**6**) to 2187 and 2168 cm^{-1}, respectively. Such shifts to higher wavenumbers are common for isonitriles, which act exclusively as σ-donors with little or no contribution by back-donation of π electrons. Examples of such a bonding situation are cationic transition-metal complexes[11] or adducts of isonitriles with coordinatively unsaturated boron or aluminum compounds.[9] Similar effects have been observed recently in cationic transition-metal carbonyl complexes.[13]

Experimental Section

All procedures were carried out under purified argon. *n*-Pentane was dried over $LiAlH_4$. Commercially available *tert*-butyl isonitrile (Aldrich) was used without further purification, and phenyl isonitrile was synthesized from aniline and diphosgene according to the literature[14] and stored at −50 °C to prevent decomposition. Diindane(4) **1** was prepared as recently published.[3]

$R_2In-InR_2\cdot 2Me_3CNC$ (5). Solid diindane(4) **1** (612 mg, 0.707 mmol) was treated with 3 mL of *tert*-butyl isonitrile without any other solvent. The suspension was stirred at room temperature, and a yellow solution was obtained after 10 min. The excess isonitrile must be cautiously distilled off under reduced pressure because the amorphous residue decomposed on thorough drying in vacuo to yield the orange-red starting compound **1**. Recrystallization from 1.5 mL of a 1 M solution of *tert*-butyl isonitrile in *n*-pentane gave the product (**5**) as yellow crystals. Yield: 594 mg (81%). Dec. (argon, sealed

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Table 1. Crystal Data, Data Collection Parameters, and Structure Refinement for Compounds 5 and 6

	5	**6**
formula	$C_{38}H_{94}In_2N_2Si_8 \cdot 0.125$*n*-pentane	$C_{42}H_{86}In_2N_2Si_8$
cryst syst	monoclinic	monoclinic
space group	*I*2/*a*; No. 15[15]	*C*2/*c*; No. 15[15]
Z	16	4
temp, K	293(2)	293(2)
D_{calcd}, g/cm^3	1.138	1.194
a, Å	21.108(3)	19.563(3)
b, Å	21.520(2)	14.264(2)
c, Å	53.861(6)	21.443(3)
β, deg	96.430(10)	93.83(2)
V, 10^{-30} m^3	24312(5)	5970(2)
μ, mm^{-1}	0.940	0.959
cryst dimens, mm	0.67 × 0.44 × 0.81	0.48 × 0.37 × 0.41
radiation	Mo Kα; graphite monochromator	
2θ range, deg	$4.0 \leq 2\theta \leq 48.1$	$3.8 \leq 2\theta \leq 48.2$
index ranges	$-24 \leq h \leq 21$	$-22 \leq h \leq 22$
	$-22 \leq k \leq 24$	$-16 \leq k \leq 16$
	$-61 \leq l \leq 61$	$-24 \leq l \leq 23$
no. of unique rflns	18999	4684
no. of params	977	256
R1 ($I > 2\sigma(I)$)	0.0331 (11632)	0.0254 (3549)
wR2 (all data)	0.0661	0.0482
max/min residual electron density, 10^{30} e/m^3	0.64/−0.67	0.57/−0.30

capillary): 78 °C. IR (CsBr plates, paraffin, cm^{-1}): 2187 w νNC; 2130 vw νNC of free isonitrile; 1462 vs, 1377 vs paraffin; 1255 w, 1246 s δCH$_3$; 1202 vw, 1170 vw, 1153 vw, 1113 vw, 1077 vw; 1020 m δCH; 916 w, 849 vs, 785 m, 761 m, 750 m, 723 m ρCH$_3$(Si); 685 w, 667 m ν_{as}SiC; 611 vw, 602 vw ν_sSiC; 521 vw, 467 w, 412 vw νInC; 338 vw δSiC.

$R_2In-InR_2 \cdot 2H_5C_6NC$ (6). Diindane(4) **1** (414 mg, 0.478 mmol) was dissolved in 40 mL of *n*-pentane and cooled to −55 °C. A solution of 181 mg (1.76 mmol) of phenyl isonitrile in 8 mL of pentane was added in 10 min. The mixture was warmed to room temperature and stirred for 45 min. After concentration to about 5 mL, the solution was cooled to −50 °C to obtain yellow crystals of the product (**6**). It was recrystallized from a mixture of cyclopentane (5 mL) with 150 mg of phenyl isonitrile. Yield: 396 mg (77%). Dec. (argon, sealed capillary): 84 °C. IR (CsBr plates, paraffin, cm^{-1}): 2168 m νNC; 2122 vw νNC of free isonitrile; 1589 w phenyl; 1458 vs, 1377 vs paraffin; 1292 w, 1244 s δCH$_3$; 1184 w, 1169 vw, 1115 vw, 1093 vw, 1069 vw; 1022 s δCH; 912 m, 849 vs, 787 m, 770 s, 748 s, 723 m ρCH$_3$(Si); 679 s, 665 vs ν_{as}SiC; 611 w, 600 m ν_sSiC; 509 m, 488 vw, 482 vw, 461 s, 449 m νInC; 362 vw, 342 vw δSiC.

Crystal Structure Determinations. Single crystals were obtained by slow cooling of a saturated solution in a mixture of *n*-pentane and *tert*-butyl isonitrile (**5**) or in a mixture of cyclopentane and phenyl isonitrile (**6**). The crystals were not dried in vacuo after isolation. Owing to the beginning of decomposition, the sealed, originally yellow crystals became orange-red after 24 h. The crystallographic data were collected with a STOE imaging plate diffractometer. Relevant crystal data, data collection parameters, and results of the structure refinement are summarized in Table 1. The structures were solved by direct methods and refined with the program SHELXL-93[16] by a full-matrix least-squares method based on F^2. Compound **5** has two independent molecules in the asymmetric unit, and its crystals include two molecules of *n*-pentane in the unit cell, which are disordered over a 2-fold rotational axis and were refined isotropically with fixed bond lengths and angles without consideration of hydrogen atoms. Two trimethylsilyl groups of **5** showed disorder (Si6 and Si10), and their atoms were isotropically refined with occupancy factors of 0.7 and 0.3 (Si6) and 0.53 and 0.47 (Si10) by the restriction of bond lengths and angles. **6** is located on a 2-fold crystallographic rotation axis perpendicular to the In−In bond.

Acknowledgment. We are grateful to the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie for generous financial support.

Supporting Information Available: Tables of atomic coordinates, isotropic and anisotropic displacement parameters, and all bond lengths and angles for **5** and **6** (22 pages). Ordering information is given on any current masthead page. Further details of the crystal structure determinations are available from the Fachinformationzentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen, Germany, on quoting the depository numbers CSD-408507 (**5**) and -408506 (**6**).

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