

REACTIONS OF (TRIPHENYLSTANNYL)CARBODIIMIDES AND -CYANAMIDE WITH ORGANIC HALIDES AND ISOTHIOCYANATES

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SUMMARY

Although the reaction of bis(triphenylstannyl)carbodiimide (I) with alkyl halides was not found to be a general route to dialkylcarbodiimides, some interesting reactions of (I) with alkyl halides were uncovered. Whereas the reaction of (I) with trityl chloride afforded ditritylcarbodiimide in high yield, the reaction of (I) with benzyl bromide afforded a novel dibenzylcyanamide/triphenyltin bromide complex (IV). The reaction of *N*-(triphenylstannyl)-*N'*-tritylcarbodiimide (II) with trityl chloride also afforded ditritylcarbodiimide, but the reaction of (II) with benzyl bromide afforded a benzyltritylcyanamide/triphenyltin bromide complex (V). The complexes (IV) and (V) were prepared also by the reaction of triphenyltin bromide with the corresponding dialkylcyanamide. Some evidence that the nitrile nitrogen, rather than the amino nitrogen, is bonded to tin in these complexes was found. The reaction of (triphenylstannyl)cyanamide (III) with trityl chloride and triethylamine gave ditritylcarbodiimide along with a small amount of (II).

The reaction of (I) with phenyl isothiocyanate afforded *N*-phenyl-*N'*-(triphenylstannyl)-*N'*-cyano-*S*-(triphenylstannyl)isothioureas. (III) was found to react with organic isothiocyanates to give *N*-substituted *N'*-cyano-*S*-(triphenylstannyl)isothioureas. The order of reactivity of organic isothiocyanates in this reaction was determined. *N*-Phenyl-*N'*-cyano-*S*-(triphenylstannyl)isothioureas was found to decompose in refluxing benzene to give bis(triphenyltin) sulfide and a 1,3,5-thiadiazine derivative. Some evidence for the mechanism of this novel reaction was found.

INTRODUCTION

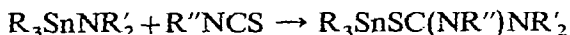
We recently described¹ some methods of preparation and some reactions of bis(triphenylstannyl)carbodiimide(I), *N*-(triphenylstannyl)-*N'*-tritylcarbodiimide(II), and (triphenylstannyl)cyanamide (III). The only other organotin carbodiimides reported in the literature are bis(triethylstannyl)carbodiimide^{2,3} and bis(trimethylstannyl)carbodiimide⁴. No other organotin cyanamides have been reported. Like other organotin-nitrogen compounds⁵, organotin-carbodiimides and -cyanamides are potentially useful as synthetic reagents, which makes a study of their reactions a

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most worthwhile endeavor. In addition to the reactions of (I), (II), and (III) described in our previous paper¹, only the reactions of bis(triethylstannyl)carbodiimide with cyanamide², triethyltin chloride², and salts of some metals⁶ have been described.

Pommier *et al.*⁷ have reported that the reactions of (diethylamino)tributyltin with alkyl halides yield the corresponding tributyltin halide and diethylalkylamine. Cleavage of the tin–nitrogen bond in (dialkylamino)butylstannanes by alkyl halides was also observed by Lorberth⁸. These results suggested to us that the reactions of (I) with alkyl halides might possibly afford a general route to dialkylcarbodiimides, a very important class of compounds⁹. The reactions of (II) and (III) with alkyl halides were also considered of interest.

Organotin–nitrogen compounds are known to react with organic isothiocyanates^{10,12}.

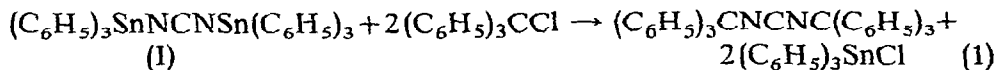


Organotin–oxygen compounds react with isothiocyanates in a similar fashion¹¹. It was of interest to study the reactions of (I), (II), and (III) with isothiocyanates to determine if analogous products containing the cyano group could be obtained and, if so, to determine, in a preliminary way, if such products might have some synthetic utility.

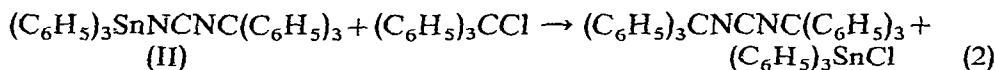
RESULTS AND DISCUSSION

A. Reactions with alkyl halides

The reaction of (I) with trityl chloride (1/2 mole ratio) in ethyl ether at 26° did, indeed, afford a 74% yield of ditritylcarbodiimide; the other product was triphenyltin chloride (78%):



Ditritylcarbodiimide was prepared previously¹⁴ by the reaction of trityl chloride with cyanamide in pyridine for 6 days at 37°. The reaction of (I) with trityl chloride (1/1 mole ratio) gave ditritylcarbodiimide (11%), (II) (63%), and triphenyltin chloride (59%). The reaction of (II) with trityl chloride gave ditritylcarbodiimide (85%) and triphenyltin chloride (61%):



No reaction was observed between (I) and benzyl bromide (1/1 mole ratio) in ethyl ether at 26°. The reaction of (I) with benzyl bromide (1/2 mole ratio) in refluxing acetonitrile gave a 57% yield of a novel complex (IV) composed of one mole of dibenzylcyanamide and one mole of triphenyltin bromide; the other product was triphenyltin bromide (98%):

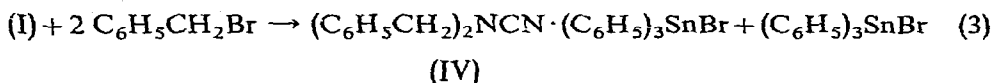


TABLE 1

N-SUBSTITUTED N'-CYANO-S-(TRIPHENYLSTANNYL)ISOTHIUREAS^a

No.	R	Reaction time (h)	Yield (%) ^b	M.p. (°C) ^c	Elemental analysis, found (calcd.) (%)				
					C	H	N	S	Sn
(VII)	<i>p</i> -O ₂ NC ₆ H ₄	24	79	159–162 ^d	54.66 (54.67)	3.45 (3.53)	9.76 (9.81)	5.76 (5.61)	21.11 (20.78)
(VIII)	C ₆ H ₅	72	55 ^e	147–148	59.36 (59.34)	4.09 (4.02)	7.90 (7.99)	6.35 (6.09)	22.48 (22.55)
(IX)	C ₆ H ₅ CH ₂	72	24	156–157	60.04 (60.03)	4.44 (4.29)	7.93 (7.78)	5.67 (5.93)	21.86 (21.97)
(X)	<i>p</i> -C ₂ H ₅ OC ₆ H ₄	72	22	154–156	58.68 (58.97)	4.51 (4.42)	7.28 (7.37)	5.69 (5.62)	21.25 (20.81)
(XI)	C ₂ H ₅	120 ^f	42 ^g	155–157	55.02 (55.26)	4.44 (4.43)	8.81 (8.79)	6.62 (6.71)	24.55 (24.82)

^a All reactions were carried out in acetonitrile; except where noted, the reaction temperature was 26°, and the product was recrystallized from chloroform/*n*-pentane. ^b Based on the analytical sample. ^c Refers to the analytical sample; melting occurred with decomposition. ^d Recrystallized from tetrahydrofuran/*n*-pentane. ^e The yield of material, m.p. 139–143°, was 74%. ^f The reaction temperature was 44°; no reaction occurred at 26° for 120 h. ^g The yield of (XI), m.p. 153–155°, was 50%.

TABLE 2

IR SPECTRA OF N-SUBSTITUTED N'-CYANO-S-(TRIPHENYLSTANNYL)ISOTHIUREAS^a

No.	NH	C≡N	C=N ^b	C ₆ H ₅ ring vibration	SnS	SnC ₆ H ₅	
						<i>v</i> _{as}	<i>v</i> _s
(VII)	3378 m	2203 s	1534 s	455 s	359 m	276 s	231 m ^c
(VIII)	3356 m	2193 s	1522 s	455 s	345 m	277 s	225 m
(IX)	3367 m	2183 s	1515 s	451 s	357 w	274 s	231 m ^d
(X)	3367 m	2188 s	1515 s	455 s	377 w	273 s	217 m (br)
(XI)	3401 m	2193 s	1522 s	455 s	349 s	275 s	221 s (br)

^a Values are expressed in cm⁻¹. ^b This assignment is uncertain due to the presence of C₆H₅ bands in this region. ^c A band at 220 cm⁻¹ (m) was also present which may be due to the δ (SnC₆H₅) vibration²⁵. ^d A band at 219 cm⁻¹ (s) was also present.

stabilize the negative charge on nitrogen in the transition state both inductively and by a resonance effect.

The IR spectra of the compounds in Table 1 are summarized in Table 2. The assignments are based largely on the work of others^{23,25-30}.

(VIII) was found to be stable in acetic acid at 26°. (VIII) was found to decompose in refluxing benzene to give bis(triphenyltin) sulfide (84%) and a compound which we have tentatively assigned structure (XII) (63%), eqn. (7). The structural assignment is supported by IR, NMR, and mass spectrometric data. The IR spectrum

nitrogen and -oxygen compounds also form 1/1 adducts with organic isocyanates^{12,13,31}. Whether the tin is bonded to nitrogen or to oxygen in these compounds has not been definitely established.

(VI) was found to decompose in refluxing benzene to give bis(triphenyltin) sulfide (63%) and a yellow solid, m.p. 165–172° dec., which contained C, H, N, and Sn. The IR spectrum of this substance contained bands at 2188 s (CN), 1493 s (br), 731 m and 695 s (C₆H₅), 455 s (C₆H₅ ring vibration), 274 s [$\nu_{as}(\text{SnC}_6\text{H}_5)$] and 229 m [$\nu_s(\text{SnC}_6\text{H}_5)$] cm⁻¹. This substance was not identified.

EXPERIMENTAL

Melting points were determined with a Mel-Temp capillary melting point apparatus and are uncorrected. The infrared data were obtained using KBr pellets with a Perkin-Elmer Infracord or Beckman IR 8 infrared spectrophotometer. The far infrared data were obtained with a Perkin-Elmer Model FIS-3 far infrared spectrophotometer (polyethylene pellets) and with a Perkin-Elmer Model 21 double beam infrared spectrophotometer which was fitted with a cesium bromide prism and purged with nitrogen (KBr pellets). The NMR spectra were determined with a Varian Model A-60 NMR spectrometer using tetramethylsilane as the internal standard. The mass spectral data were obtained with a Hitachi RMU-6D mass spectrometer by Morgan-Schaffer Corporation, Montreal 252, Quebec, Canada. Elemental analyses were performed by Schwarzkopf Microanalytical Laboratory, Woodside, New York.

Reaction of bis(triphenylstannyl)carbodiimide (I) with trityl chloride

(1). *1/2 Mole ratio.* A mixture of (I) (1.48 g, 0.002 mol), trityl chloride (1.12 g, 0.004 mol), and ethyl ether (15 ml) was stirred at 26° for 23 h. The mixture was cooled to about 5° and filtered to give 0.97 g (92%) of crude ditritylcarbodiimide, m.p. 180–205°. Recrystallization from benzene/ethanol gave 0.78 g (74%) of ditritylcarbodiimide, m.p. 208–211° (lit.¹⁴ m.p. 210°), IR spectrum superimposable upon that of an authentic sample: 2123 (NCN), 755 and 697 (C₆H₅) cm⁻¹.

Evaporation of the solvent from the original filtrate gave 1.24 g (81%) of crude triphenyltin chloride, m.p. 88–96°. Recrystallization from isopropanol gave 1.18 g (78%) of triphenyltin chloride, m.p. 104–106°, no depression on admixture with an authentic sample, IR spectrum superimposable on that of an authentic sample.

In another run carried out in refluxing acetonitrile for 2 h there was obtained ditritylcarbodiimide (70%), m.p. 206–209°, and triphenyltin chloride (53%), m.p. 103–107°.

(2). *1/1 Mole ratio.* A mixture of (I) (3.70 g, 0.005 mol), trityl chloride (1.40 g, 0.005 mol), and ethyl ether (20 ml) was stirred at 26° for 24 h. The mixture was filtered to give 0.29 g (11%) of ditritylcarbodiimide, m.p. 200–206°.

Evaporation of the filtrate left a clear viscous liquid which was stirred with n-pentane (50 ml). The solid which formed was collected on a filter to give 2.0 g (63%) of *N*-(triphenylstannyl)-*N'*-tritylcarbodiimide (II), m.p. 97–104°. Recrystallization from ethanol gave (II) (0.92 g, 29%), m.p. 106–108° (lit.¹ m.p. 107–109°).

The pentane filtrate was concentrated to about 10 ml, allowed to stand over-

night at 4°, and filtered to give 1.93 g of solid, m.p. 83–90°. Recrystallization from isopropanol afforded 1.13 g (59%) of triphenyltin chloride, m.p. 93–103°. A second recrystallization from isopropanol gave 0.94 g (49%) of triphenyltin chloride, m.p. 102–105°.

Reaction of N-(triphenylstannyl)-N'-tritylcarbodiimide (II) with trityl chloride

A mixture of (II) (1.27 g, 0.002 mol), trityl chloride (0.56 g, 0.002 mol), and ethyl ether (13 ml) was stirred at 26° for 22 h. The mixture was cooled to about 5° and filtered to give 0.89 g (85%) of ditritylcarbodiimide, m.p. 204–208°. Recrystallization from benzene/ethanol gave 0.82 g (78%) of ditritylcarbodiimide, m.p. 207–209°.

Evaporation of the original filtrate gave 0.90 g of solid, m.p. 98–103°. Recrystallization from isopropanol gave 0.47 g (61%) of triphenyltin chloride, m.p. 104–106°.

Reaction of (I) with benzyl bromide

A mixture of (I) (8.33 g, 0.0113 mol), benzyl bromide (3.85 g, 0.0225 mol), and acetonitrile (25 ml) was refluxed for 23 h. The solution was concentrated to about one-third of its original volume, cooled to about 5°, and filtered to give 4.74 g (98%) of triphenyltin bromide, m.p. 120–123° (lit.³² m.p. 121–122°).

Evaporation of the filtrate left 7.20 g of a yellow, tacky solid, m.p. 63–70°. The solid was extracted with boiling n-hexane (80 ml), the hexane extract was cooled to about 5°, and the colorless crystals which separated were collected on a filter to give 4.16 g (57%) of dibenzylcyanamide/triphenyltin bromide complex, m.p. 82–86°. Recrystallization from n-pentane gave 2.39 g (33%) of the complex, m.p. 88–90°. (Found: C, 60.76; H, 4.54; Br, 12.34; N, 4.18; Sn, 18.48. C₃₃H₂₉BrN₂Sn calcd.: C, 60.77; H, 4.48; Br, 12.25; N, 4.30; Sn, 18.20%.) IR: 2227 s (CN), 729 s and 696 s (C₆H₅), 595 w, 532 w, 485 w, 474 w, 451 s (C₆H₅ ring vibration)²³, 271 s [ν_{as} (Sn-C₆H₅)]^{23,25,26}, 254 m (SnBr)²⁵, 221 w [ν_s (SnC₆H₅)]²⁵, 178 w [δ (SnC₆H₅)]²⁵ cm⁻¹; NMR (CCl₄): τ 6.01 (singlet, 4 H, CH₂) and 2.90–2.27 (multiplet, 25 H, C₆H₅).

In another run, in which twice the amounts of materials given above were employed, the yield of triphenyltin bromide, m.p. 122–125°, was 97%, and the yield of the complex, m.p. 82–87°, was 60%. Recrystallization of the complex from isopropanol gave triphenyltin bromide (71%), m.p. 121–123°, and dibenzylcyanamide (79%), m.p. 43–50° (lit.³³ m.p. 53.5°). A single recrystallization of the dibenzylcyanamide from n-hexane afforded a 64% yield of this substance, m.p. 50–53°.

Reaction of (II) with benzyl bromide

A mixture of (II) (1.90 g, 0.003 mol), benzyl bromide (0.51 g, 0.003 mol), and acetonitrile (5 ml) was refluxed for 21 h. The solution was allowed to stand at 4° overnight and then was filtered to give 0.081 g of solid, m.p. 180–195°, whose IR spectrum indicated it to be a mixture of ditritylcarbodiimide and (II).

Evaporation of the filtrate left a viscous liquid to which was added acetonitrile (1 ml). The mixture was allowed to stand at 4° overnight and then was filtered to give 0.51 g (21%) of benzyltritylcyanamide/triphenyltin bromide complex (colorless crystals), m.p. 142–145°. Recrystallization from ethanol gave 0.32 g (13%) of the complex 146–148°. A second recrystallization from ethanol did not change the m.p. (Found: C, 67.34; H, 4.76; Br, 10.08; N, 3.45; Sn, 15.02. C₄₅H₃₇BrN₂Sn calcd.:

C, 67.19; H, 4.64; Br, 9.93; N, 3.48; Sn, 14.75 %.) IR: 2237 s (CN), 736 s, 712 s, 704 m, and 695 s (C_6H_5), 629 m (C_6H_5 ring vibration)²³, 565 w, 532 w, 521 w, 490 w, 451 s (C_6H_5 ring vibration), 356 w, 338 w, 314 w, 293 w, 277 s and 270 s [$\nu_{as}(SnC_6H_5)$], 258 m (sh) (SnBr), 224 s [$\nu_s(SnC_6H_5)$], 187 s [$\delta(SnC_6H_5)$], 157 m, 140 w, and 126 w cm^{-1} ; NMR (CCl_4): τ 6.34 (singlet, 2H, CH_2) and 2.87–2.31 (multiplet, 35H, C_6H_5).

Reaction of triphenyltin bromide with dibenzylcyanamide

A mixture of triphenyltin bromide (1.24 g, 0.00288 mol), dibenzylcyanamide³³ (0.64 g, 0.0029 mol), and acetonitrile (10 ml) was refluxed for 20 h. Evaporation of the clear solution left 1.82 g (97 %) of dibenzylcyanamide/triphenyltin bromide complex, m.p. 86–89°, IR spectrum superimposable on that of the analytical sample described above. A single recrystallization from petroleum ether (b.p. 30–60°) gave 1.26 g (67 %) of the complex, m.p. 88–90°.

Reaction of triphenyltin bromide with benzyltritylcyanamide

A mixture of triphenyltin bromide (0.75 g, 0.0017 mol), benzyltritylcyanamide¹⁴ (0.65 g, 0.0017 mol), and acetonitrile (10 ml) was refluxed for 18 h. Evaporation of the pale-yellow solution left a tacky solid which was recrystallized from ethanol to give 0.40 g (29 %) of benzyltritylcyanamide/triphenyltin bromide complex, m.p. 144–146°. A second recrystallization from ethanol gave 0.34 g (24 %) of the complex, m.p. 147–148°, IR spectrum superimposable on that of the analytical sample described above.

N-Phenyl-N'-(triphenylstannyl)-N'-cyano-S-(triphenylstannyl)isothiourea (VI)

A mixture of (I) (0.74 g, 0.001 mol), phenyl isothiocyanate (0.142 g, 0.00105 mol), and benzene (10 ml) was stirred at 26° for 20 h. Evaporation of the benzene left a gum which solidified upon standing to give 0.69 g (78 %) of (VI) (colorless, crystalline solid), m.p. 134–136° dec. (Found: C, 60.23; H, 4.28; N, 4.79; S, 3.36; Sn, 27.08. $C_{44}H_{35}N_3SSn_2$ calcd.: C, 60.38; H, 4.03; N, 4.80; S, 3.66; Sn, 27.12 %.) IR: 2188 m (CN), 728 s and 696 s (C_6H_5), 453 s and 451 s (C_6H_5 ring vibration), 371 s (SnS), 326 m, 267 s [$\nu_{as}(SnC_6H_5)$], and 227 m [$\nu_s(SnC_6H_5)$] cm^{-1} .

Reaction of (VI) with water

A mixture of (I) (1.48 g, 0.002 mol), phenyl isothiocyanate (0.27 g, 0.002 mol), and benzene (25 ml) was stirred at 26° for 23 h. Water (0.36 ml, 0.02 mol) was added to the solution, and the mixture was stirred for 48 h. The mixture was filtered to give 0.55 g (52 %) of *N*-phenyl-*N'*-cyano-*S*-(triphenylstannyl)isothiourea (VIII), m.p. 144–145° dec., which was identical in every respect with an analytical sample prepared as described below.

Evaporation of the filtrate left a colorless gum which solidified upon being mixed with 95% ethanol (8 ml). Filtration afforded 0.83 g of a colorless solid, m.p. 112–129°, whose IR spectrum indicated it to be a mixture of (VIII) and triphenyltin hydroxide (SnOH, 914 and 900 cm^{-1}). The solid was mixed with methanol (6 ml), and the mixture was filtered to give 0.31 g (30%) of (VIII) m.p. 139–142°. Evaporation of the filtrate afforded 0.42 g (58 %) of a mixture of triphenyltin hydroxide (SnOH, 914 and 900 cm^{-1}) and bis(triphenyltin) oxide (SnOSn, 776 cm^{-1}), m.p. 109–120°. Recrystallization of this material from acetonitrile gave 0.30 g of the same mixture, m.p. 117–123°.

N-Phenyl-N'-cyano-S-(triphenylstannyl)isothiourea (VIII)

A mixture of (III) (0.78 g, 0.002 mol), phenyl isothiocyanate (0.28 g, 0.0021 mol), and acetonitrile (10 ml) was stirred at 26° for 72 h. The mixture was cooled to 4° and filtered to give 0.78 g (74%) of (VIII), m.p. 139–143°. Recrystallization from chloroform/n-pentane gave 0.58 g (55%) of (VIII) (colorless crystals), m.p. 147–148° dec.

Decomposition of (VIII)

A mixture of (VIII) (0.90 g, 0.00171 mol) and benzene (10 ml) was refluxed for 20 h, allowed to cool to 26°, and filtered to give 0.17 g (63%) of a yellow solid, m.p. 256–260° dec. Recrystallization from acetonitrile gave 0.053 g (20%) of (XII), m.p. 274–275° dec. (Found: C, 60.01; H, 3.92; N, 26.48; S, 10.13; mol. wt. by mass spectrometry, 320. C₁₆H₁₂N₆S calcd.: C, 60.02; H, 3.78; N, 26.23; S, 10.00%; mol. wt., 320.378.) IR: 3472 m (NH), 3058 m (br) (aromatic CH, imino NH), 2193 s (CN), 1610 s (br), 1538 s, 1473 s, 758 m (C₆H₅), 741 m (C₆H₅), and 695 m (C₆H₅) cm⁻¹; NMR (DMSO-*d*₆): τ 7.93 (singlet, 2 H, NH₂), 2.58 (singlet, 5 H, C₆H₅), and 2.40 (singlet, 5 H, C₆H₅).

Evaporation of the original filtrate left a pale yellow solid which gave after recrystallization from acetonitrile 0.53 g (84%) of bis(triphenyltin) sulfide, m.p. 144–146°, no depression on admixture with an authentic sample, IR spectrum superimposable on that of an authentic sample.

Reaction of (I) with phenyl isothiocyanate and cyanamide

A mixture of (I) (2.22 g, 0.003 mol), phenyl isothiocyanate (0.81 g, 0.006 mol), cyanamide (0.126 g, 0.003 mol), and benzene (50 ml) was refluxed for 19 h, allowed to cool to 26°, and filtered to give 0.52 g of a yellow solid, m.p. 190–260°. The solid was stirred with methanol (12 ml), and the mixture was filtered to give 0.23 g (24%) of (XII), m.p. 255–258° dec. Recrystallization from acetonitrile/benzene gave 0.075 g (8%) of (XII), m.p. 275° dec.

Evaporation of the original filtrate left a yellow solid which afforded, after recrystallization from acetonitrile, 1.51 g (69%) of bis(triphenyltin) sulfide, m.p. 145–147°.

Reaction of (III) with phenyl isocyanate

A mixture of (III) (0.78 g, 0.002 mol), phenyl isocyanate (0.25 g, 0.0021 mol), and acetonitrile (10 ml) was stirred at 26° for 17 h and filtered to give 0.86 g (84%) of a 1/1 adduct, m.p. 154–160°. Two recrystallizations from tetrahydrofuran/n-pentane gave the analytical sample, m.p. 163–165°. (Found: C, 61.12; H, 3.91, N, 8.15; Sn, 23.44. C₂₆H₂₁N₃OSn calcd.: C, 61.21; H, 4.15; N, 8.24; Sn, 23.26%.) IR: 3401 m (NH), 2203 s (CN), 1603 s, 1567 s, 1481 s, 1431 s, 749 m (NC₆H₅), 729 s (SnC₆H₅), 682 s (NC₆H₅, SnC₆H₅), 453 s and 443 m (C₆H₅ ring vibration), 390 w, 329 s, 281 s and 272 s [*v*_{as}(SnC₆H₅)], and 235 s [*v*_s(SnC₆H₅)] cm⁻¹.

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