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An unusual oxygen-donor adduct of triphenyltin saccharin: crystal structure of [*N*-triphenylstannyl 1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide] · [*O*-glycolic acid]

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Abstract

Glycolic acid forms a 1/1 complex with triphenyltin saccharin through its hydroxylic rather than its carboxylate oxygen. The adduct is almost isomorphous with triphenyltin saccharin ethanol.

Introduction

Treatment of triphenyltin hydroxide with saccharin in ethanol gives the ethanol complex of triphenyltin saccharin [1], an adduct in which ethanol is coordinated to tin. We planned next to make *N*-saccharin acetic acid and examine its triphenylstannyl ester crystallographically in a comparative study. This acid is reported in the literature to separate out upon acidification of a dimethylformamide solution of sodium saccharin and sodium bromoacetate [2]. In our hands, use of this procedure in water followed by subsequent treatment of the organic product with triphenyltin hydroxide in ethanol yielded the title adduct rather than the stannyl ester.

Experimental

Addition of concentrated hydrochloric acid to an aqueous solution of equimolar amounts of sodium saccharin and sodium chloroacetate resulted in precipitation of a white solid which was recrystallized from ethanol. The compound, incorrectly assumed to be saccharin acetic acid and so assumed to have the molecular weight of the latter, was heated with a supposed equimolar amount of triphenyltin hydroxide

Table 1

Atomic coordinates for triphenyltin saccharin glycolic acid

Atom	<i>x</i>	<i>y</i>	<i>z</i>
Sn	0.24201(5)	0	0.09626(5)
S	0.1887(3)	-0.0605(1)	0.4240(2)
O(1)	0.0805(7)	-0.0003(7)	0.4206(6)
O(2)	0.3482(7)	-0.0472(5)	0.5280(7)
O(3)	0.1146(7)	-0.1869(4)	0.0818(7)
N	0.1767(7)	-0.0803(4)	0.2508(7)
C(1)	0.1262(8)	-0.1588(5)	0.4530(9)
C(2)	0.1083(10)	-0.1874(10)	0.5817(10)
C(3)	0.0618(10)	-0.2695(7)	0.5747(11)
C(4)	0.0332(10)	-0.3159(6)	0.4468(11)
C(5)	0.0529(9)	-0.2857(6)	0.3193(10)
C(6)	0.1016(8)	-0.2054(5)	0.3257(8)
C(7)	0.1312(8)	-0.1593(5)	0.2051(8)
O(4)	0.3105(7)	0.0774(4)	-0.0814(6)
O(5)	0.6402(11)	0.1861(8)	-0.0778(13)
O(6)	0.3974(12)	0.1325(9)	-0.3031(11)
C(8)	0.4889(9)	0.1417(5)	-0.1395(9)
C(9)	0.4359(12)	0.1134(8)	-0.0483(11)
C(10)	0.0011(7)	-0.0088(5)	-0.2369(8)
C(11)	-0.1457(9)	-0.0119(7)	-0.3559(8)
C(12)	-0.2739(9)	-0.0149(7)	-0.3218(9)
C(13)	-0.2555(8)	-0.0126(6)	-0.1707(10)
C(14)	-0.1102(8)	-0.0107(6)	-0.0522(9)
C(15)	0.0189(7)	-0.0078(6)	-0.0856(7)
C(16)	0.4506(10)	0.1250(6)	0.3344(9)
C(17)	0.4928(11)	0.2020(7)	0.3968(10)
C(18)	0.3871(11)	0.2645(7)	0.3590(10)
C(19)	0.2370(11)	0.2519(6)	0.2537(10)
C(20)	0.1904(10)	0.1750(6)	0.1830(10)
C(21)	0.2975(9)	0.1106(5)	0.2256(8)
C(22)	0.4476(9)	-0.1043(7)	-0.0204(10)
C(23)	0.5726(10)	-0.1519(7)	-0.0064(12)
C(24)	0.6823(10)	-0.1734(8)	0.1350(12)
C(25)	0.6674(11)	-0.1451(8)	0.2602(12)
C(26)	0.5444(10)	-0.1009(7)	0.2536(10)
C(27)	0.4325(8)	-0.0766(5)	0.1118(8)

in ethanol and the mixture then cooled to give crystals of the title complex. For the diffraction study, a total of 5048 ($2\theta_{\max}$ 60°) reflections was collected on a Nicolet R3m/V four-circle diffractometer using graphite-monochromatized $\text{Mo-K}\alpha$ radiation (λ 0.71073 Å) radiation. The structure was solved by the heavy-atom method and refined to an R_F factor of 0.040 for 3095 independent absorption-corrected reflections obeying the $|F_0| > 4\sigma(|F_0|)$ criterion. The hydroxyl and carbonyl H atoms did not appear on the final difference map. The atomic coordinates are listed in Table 1. Listings of calculated and observed structure factors, anisotropic temperature factors, and bond distances and angles are available from the authors. Crystal data: $\text{C}_{25}\text{H}_{19}\text{NO}_3\text{SSn} \cdot \text{HOCH}_2\text{CO}_2\text{H}$, M 608.23, monoclinic, space group $P2_1$, a 9.467(2), b 16.331(3), c 9.572(2) Å, β 113.85(2)°, V 1353.5(4) Å³, D_c 1.492 g cm⁻³ for $Z = 2$.

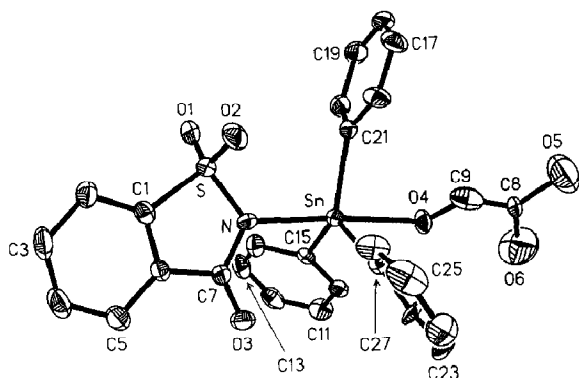


Fig. 1. Molecular structure of the triphenyltin saccharin·glycolic acid adduct. Selected bond distances and angles: Sn–N 2.242(8), Sn–O(4) 2.409(7), Sn–C(15) 2.129(5), Sn–C(21) 2.132(8), Sn–C(27) 2.150(8) Å; N–Sn–O(4) 175.7(2), N–Sn–C(15) 93.3(3), N–Sn–C(21) 100.4(3), N–Sn–C(27) 93.5(3), O(4)–Sn–C(15) 85.2(3), O(4)–Sn–C(21) 83.8(3), O(4)–Sn–C(27) 84.0(3), C(15)–Sn–C(21) 117.1(3), C(15)–Sn–C(27) 123.3(3), C(21)–Sn–C(27) 116.7(3)°.

Discussion

Figure 1 depicts the molecular structure of triphenyltin saccharin·glycolic acid. The tin atom is five-coordinate and its geometry *trans*-C₃SnNO trigonal bipyramidal, with the nitrogen atom of saccharin and the alcoholic oxygen atom of glycolic acid in the apical positions. The values of the N–Sn–O(4) (175.7(2)°) angle and the Sn–N (2.242(8) Å), Sn–O(4) (2.409(7) Å) bond lengths are similar to those in the triphenyltin saccharin·ethanol adduct (176.6°, 2.240 and 2.394 Å [1], respectively). A significant feature of the compound, compared to other organostannylamides, is its unusual stability to protic cleavage: the glycolic acid adopts a coordinating mode, binding to the tin uniquely through the alcoholic function. In the infrared (KBr) spectrum of the title compound, the broad CO₂ band appears at ca. 1650 (vs) cm⁻¹.

Interestingly, the glycolic acid and ethanol adducts of triphenyltin saccharin are nearly isomorphous, with closely matching unit-cell and atomic coordinates. The formation of the former adduct without contamination by the latter, suggests that glycolic acid presumably arises by hydrolytic cleavage of saccharin acetic acid under our reaction conditions.

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References

- 1 S.W. Ng, Chen Wei, V.G. Kumar Das and T.C.W. Mak, *J. Organomet. Chem.*, 373 (1989) 21.
- 2 C.S. Giam and R.A. Lockhart, *Org. Prep. Proced. Int.*, 6 (1974) 1.