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Structure Crystal of (*N*-Methyl benzyl Dithiocarbamate) dibutyl Tin (IV).

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ABSTRACT

(*N*-methylbenzylTin(IV) dithiocarbamate has been synthesized and characterized, the compound is prepared from the reaction between the 4-methylbenzyl amine dissolved in absolute ethanol with carbon disulfide in absolute ethanol with the same ratio of 0.2 mmol, the mixture of this compound was added to the metal dibutyl tin (IV) dichloride in an ethanol solvent, then distirer for 2 hours. resulting in the formation of precipitate, then filtered and recrystallized with a mixture of ethanol with chloroform with a ratio of 1: 1. The crystals formed were chelate octahedrons with 6 coordinates with a dithiocarbamate league binding of aniso bidentat, the results of the crystallographic test show that the compound formed is octahedron distorted, resulting in a new compound N-methylbenzyl ditiocarbamate dibutyl Tin (IV).
Keywords: benzyl dtc, methyl , crystal structure.

The title compound [$Sn(C_4H_9)_2(C_6H_8NS_4)(CH_3)_2$], has a long-range intramolecular Sn----S interaction of 3.0950 (10) Å, which allows the geometry of the Sn atom to be close to octahedral, with an axial bond angle of C(19)-Sn1-C19, C(19)-Sn1-S2 and C19-Sn1-S2 angles of 140.06 (18)°, 105.28 (9)° and 103.84 (9)°. The structural dimensions of the molecule are comparable with those of other dibutyltindithiocarbamate complexes.

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COMMENT

Several dibutyltin-dithiocarbamate complexes have been reported to have a geometry of the central Sn atom between tetrahedral and octahedral due to the presence of a long interaction between the Sn atom and the uncoordinated S atom of the dithiocarbamate ligand. The latest six-octahedron organotin (IV) dithiocarbamate complexes are found as biological activities, as anti-inflammatory, antimicrobial, antiviral, anti-cancer and anti-oxidant (Adeyemi, Onwudiwe, Ekennia, Uwaoma, & Hosten, 2018), then the N-methyl complex -n-phenyl-diorganotin (IV), is found in distorted octahedron form, also has 6 coordinates (Adeyemi, Onwudiwe, & Hosten, 2017) (*N*-Cyclohexyl-*N*-methyldithiocarbamato) triphenyltin(IV) (Awang *et al*., 2003), (*N*-butyl-*N*-methyldithiocarbamato) triphenyltin(IV) (Kana *et al*., 2001), (diethyldithiocarbamato) triphenyltin(IV) (Lindley & Carr, 1974) and (*N*-cyclohexyl-*N*-ethyldithiocarbamato)- triphenyltin(IV) (Awang *et al*., 2003) are typical examples where the geometry of the central Sn atom in the complexes is close to octahedral. The title compound, (I) (Fig. 1), is analogous to those complexes and shows similar structural dimensions (Table 1) with a long intramolecular Sn1---S2 interaction of 3.0950 Å (10) , comparable with those in (*N*-cyclohexyl-*N*-methyldithiocarbamato) triphenyltin(IV)[3.0134(8)Å].The geometry of the Sn atom is also close to octahedral, with C2-Sn1-C19, C19-Sn1-S1 and C12-Sn1-S1 angles of 116.49 (3), 120.29 (7) and 110.95 (7),respectively, in the equatorial positions. The C18-Sn1-S2 angle for the axial position is 156.50 (6). Other bond lengths and angles of the ligand are in the normal ranges (Allen et al.,The title compound was synthesized by the addition of carbon disulfide (1.2 ml, 0.02 mol) to an ethanol solution of 4-methylbenzylamine(1.743 g, 0.02 mol); the resulting solution was stirred for 1 h at 269^{o} K.

Figure 1: ORTEP plot of Bu_2Sn(Mebzdtc) in the equatorial positions.

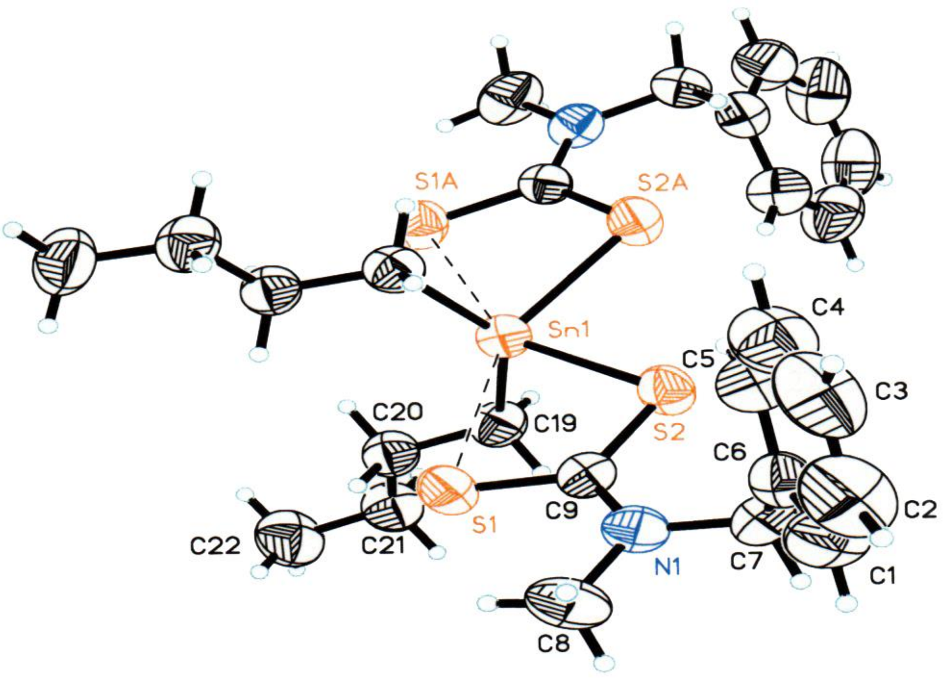

FIGURE 1. ORTEP plot of $Bu_2Sn(Mebzdtc)_2$

Dibutyltin(IV) chloride (3.84 g, 0.01 mol) was then added and the mixture was stirred for another 1 h. The white precipitate which formed was filtered off and washed with cold ethanol and dried in a vacuum. Good quality crystals were obtained by recrystallization from a mixture of ethanol and chloroform (1:2 v/v).

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Crystal data

$[Sn(C_4H_9)_2(C_6H_8NS_4)(CH_3)_2]$

Formula Empiric $C_{26}H_{38}N_2S_4Sn$

Mr = 625.515

Monoclinic, P21=n

a = 19.678 (10) Å

b = 6.972 (3) Å

c = 22.722(10) Å

$\beta(^0)$= 108.922 (5)

V 2949 (2) Å Ê 3

Z = 4

Dc = 1.409 Mg mÿ3

Mo K_ radiation

Cell parameters from 983

Reflections

_ = 2.2±26.5_
_ = 1.25 mm ÿ1

T = 273 (2) ^{0}K

R1= 0.0382, wR2= 0.0971

ΔP_{maxs} and ΔPmin($eÅ^{-3}$) = 0.954 dan -0.355

Block, colourless 0.38 _ 0.29 _ 0.16 mm

Data collection Bruker SMART APEX CCD area detector Diffractometer scans Absorption correction: multi-scan (SADABS; Sheldrick, 1996) Tmin = 0.648, Tmax = 0.825 13 102 measured reflections 4928 independent reflections 4481 reflections with I > 2_(I) Rint = 0.0382_max = 26.5_Rint = 0.017,ɵ_max = 2.1 - 26.0 h = -19→ 24,k = -8→8, l = -28→23

Refinement

Refinement on F2, R[F2 > 2_(F2)] = 0.028

wR(F2) = 0.0971 S = 1.144928 re¯ections

255 parameters H-atom parameters constrained. w = 1/[_2(Fo 2) + (0.0247P)2 + 0.9515P], where P = (Fo 2 + 2Fc 2)/3 (_/_)max < 0.001 __max = 0.45 e A Ê ÿ3
__min = ÿ0.23 e A Ê ÿ3.

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Table 1

Selected geometric parameters (Å).

Sn1-S1 2.128 (3)
Sn1-S2 3.0950 (10)
Sn1-C12 2.132 (2)
Sn1-C19 2.134 (2)
Sn1-C18 2.154 (2)
S1-C1 1.757 (2)
S2-C1 1.679 (3)
N1-C1 1.328 (3)
N1-C4 1.469 (3)
N1-C2 1.478 (3)
C12-Sn1-C19 116.49 (9)
C12-Sn1-C18 106.03 (9)
C19-Sn1-C18 105.89 (9)
C18-Sn1-S1 93.23 (6)
C19-Sn1-S1 120.29 (7)
C12-Sn1-S1 110.95 (7)
C18-Sn1-S2 156.50 (6)

The crystal data and for complexes are: Bu_2Sn(Mebenzyldtc): crystal system, monoclinic; space group, *C*2/*c*; *a* = 19.678(10) Å, *b* = 6.972(3) Å, *c* = 22.722(10) Å, β = 108.922(5)º, *Z* = 4, *R* = 3.26 % for 2900 > 2σ(I) independent reflections.The ORTEP plot and numbering system of $Bu_2Sn(Mebzdtc)_2$ is depicted in Figure 1. The molecular structure of $Bu_2Sn(Mebzdtc)_2$ shows that the tin atom is bounded to two butyl groups and for sulphur atoms from two chelating bidentate dithiocarbamate. The Sn-S distances can be classed as the short [Sn(1)-S1 =3.0013 Å and long bonds Sn1 – S2A =3.0012Å]. The long Sn-S distances are however, significantly less than the sum of the van der Waals radii which is 4.0 Å [17] and as such the co-ordination number of the tin atom may be assigned as six [8,10]. The geometry of the Sn atom is close to a octahedron, with C(19)-Sn1-C19, C(19)-Sn1-S2 and C19-Sn1-S2 angles of 140.06 (18)°, 105.28 (9)° and 103.84 (9)° respectively, it can be concluded that the moieti ligand ditiocarbamate in the complex Bu_2Sn(Mebzdtc) has been anisobidentate, as has been reported by previous studies (Sanuddin, M. *et al*., 2004)

The complex structure $(C_4H_9)_2Sn\ [S_2CN\ (CH_2\text{-}C_6H_5)CH_3]_2$ formed is having six coordinates. two atoms of sulfur S1 and S1A are weakly coordinated with a central atom (Sn), this is caused a rather long distance between Sn atoms with S1 and Sn atoms with S1A, which are respectively. however long these two bonds do not extend beyond the van der waals (4.0 Å)

The reaction of diorganotin(IV) chloride with dithiocarbamates derived from the various amines used in this study afforded the 1:2 complexes with the eneral formula $R_2Sn(R'dtc)_2$ while the reaction of dibutyltin(IV) chloride with the dithiocarbamatesgave the 1:2 R_2Sn(R'dtc) type of complexes.

After their location in a difference map, all H atoms were positioned geometrically and llowed to ride on their parent C atoms, with CÐH = 0.93±0.97 Å , and Uiso(H) = 1.5Ueq(C) for CH_3 and 1.2Ueq(C) for CH_2 and CH. Data collection: SMART (Siemens, 1997); cell refenement: SAINT (Siemens, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 1997); program(s) used to refementner structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL, PARST (Nardelli,1995) and PLATON (Spek, 2003).The authors thank the Malaysian Government and Universiti Kebangsaan Malaysia for the research grant IRPA No. 09-02-02-0133. And KEMENRISTEKDIKTI Republic Indonesian PDP NOMOR : 074/L10/AK.04/KONTRAK-PENELITIAN/2019
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