ANTIMONY(III) COMPLEXES OF 2-(N-SALICYLIDENE)-5-CHLOROBENZOPHENONE: SYNTHESIS, SPECTROSCOPIC STUDIES AND REACTIVITY WITH RESPECT TO SULFUR DONOR LIGANDS

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ABSTRACT

*Dichloro, chlorobis and tris [2-(N-salicylidene)-5-chlorobenzophenone] derivatives of antimony(III) have been synthesised by the reactions of antimony trichloride with the sodium salt of 2-(N-salicylidene)-5-chlorobenzophenone (prepared *insitu*) in 1:1, 1:2 and 1:3 molar ratios, respectively. The substitution reactions of these newly synthesised chloro derivatives have been studied with alkyl xanthates. All these compounds are characterised by elemental analyses, molecular weight measurements and spectroscopic (IR, ^1H and ^{13}C NMR) studies.*

INTRODUCTION

Complexes of antimony containing oxygen and sulfur donor ligands have been known for many years^{1,2}, and are found to be biologically active³ and carcinostatic⁴. A few antimony compounds are known for their technical uses in catalytic processes^{5,6}. The metal complexes derived from 2-(N-salicylidene)-5-chlorobenzophenone are reported to act as potential antianxiety^{7,8} and antiepileptic⁹ agents. In a recent study the, Cu(II) and Zn(II) complexes of this moiety have been shown to possess antiinflammatory¹⁰ activity also. Pursual of above literature and in continuation of our earlier work^{11,12}, we have undertaken a systematic investigation on these and report here synthesis, characterisation and structural features of a few complexes of antimony(III) and their substitution reactions.

RESULTS AND DISCUSSION

Dichloro, chlorobis and tris-antimony(III) derivatives of 2-(N-salicylidene)-5-chlorobenzophenone are synthesised by the reaction of antimony trichloride with the sodium salt of the ligand in 1:1, 1:2 and 1:3 molar ratios, respectively.

Where $n = 1$, Compd. (I); $n = 2$, Compd. (II); $n = 3$, Compd. (III).

These derivatives were prepared by refluxing the reactants in benzene for about 4-5 hours

to ensure the completion of the reaction. The desired product could be isolated by evaporation of the solvent under reduced pressure, after filtering off the precipitated sodium chloride. These were further purified by crystallisation from benzene-petroleum ether (40-60°) mixtures. The mixed derivatives are synthesised by the reaction of dichloro (I) and chlorobis(II) compounds with potassium alkyldithiocarbonates in carbon disulfide benzene in 1:2 and 1:1 molar ratios, respectively.

Where $n = 2$, $R = \text{Me}$, Compd. (1); $n = 1$, $R = \text{Me}$, Compd. (2); $n = 2$, $R = \text{Et}$, Compd. (3); $n = 1$, $R = \text{Et}$, Compd. (4); $n = 2$, $R = i\text{-Pr}$, Compd. (5); $n = 1$, $R = i\text{-Pr}$, Compd. (6); $n = 2$, $R = n\text{-Bu}$, Compd. (7); $n = 1$, $R = n\text{-Bu}$, Compd. (8); $n = 2$, $R = i\text{-Bu}$, Compd. (9); $n = 1$, $R = i\text{-Bu}$, Compd. (10).

The mixed derivatives were synthesised by stirring the reactants in dry carbon disulfide/benzene for about 3 hours. The KCl was filtered off and the compound was isolated and purified by crystallisation from benzene/carbon disulfide mixtures. All these complexes are yellow crystalline solids, soluble in common organic solvents and are monomeric in benzene.

IR spectral data

The infrared spectra of these complexes have been recorded on a Perkin-Elmer 580B spectrometer, using KBr pellets. The spectrum of the ligand shows bands in the regions $3380\text{--}3260\text{ cm}^{-1}$, 1680 cm^{-1} and 1620 cm^{-1} which have been assigned to $\nu(\text{OH})$, $\nu(\text{C}=\text{O})$ and $\nu(\text{C}=\text{N})$ respectively¹³. The disappearance of $\nu(\text{OH})$ in the complexes indicate deprotonation of phenolic -OH and consequent coordination to antimony, which is further substantiated by the appearance of new bands in the region $465\text{--}445\text{ cm}^{-1}$ and 350 cm^{-1} assignable to $\nu(\text{Sb-O})$ and $\nu(\text{Sb-Cl})$ respectively.¹¹ The band present at 1620 cm^{-1} due to $\nu(\text{C}=\text{N})$ is found to be shifted for about $20\text{--}25\text{ cm}^{-1}$ to lower wave number in the complexes suggesting coordination of $\text{C}=\text{N}$ to antimony. A band at 1680 cm^{-1} in the ligand, which can be assigned to keto carbonyl does not show any discernible shift in the spectra of the complexes suggesting the non-participation of the keto group in coordination.

Infrared spectra of all the mixed ligand complexes of antimony(III) show bands in the regions $1030\text{--}1005\text{ cm}^{-1}$ and $330\text{--}290\text{ cm}^{-1}$ due to $\nu(\text{C}=\text{S})$ and $\nu(\text{Sb-S})$ vibrations respectively^{14,15}. The presence of a strong band in the region $1030\text{--}1005\text{ cm}^{-1}$ due to $\nu(\text{C}=\text{S})$ indicates bidentate nature of the xanthate moiety¹⁶.

NMR spectral data

The ^1H NMR spectrum of the ligand is characterised by appearance of a signal at $\delta 12.40$ ppm for phenolic -OH group, at $\delta 10.60$ ppm for azomethine group and a multiplet at $\delta 6.84\text{--}7.86$ ppm attributable to protons of phenyl moiety. The resonance at $\delta 12.40$ ppm is absent in the spectra

of the complexes suggesting deprotonation of phenolic - OH and its subsequent involvement in coordination. The coordination through C=N is supported by the down field shift of the free ligand signal at δ 10.60 ppm to δ 11.55 ppm in the complexes¹⁷. The resonance due to phenyl moiety remains unaffected. The xanthate moieties also exhibited the expected proton resonances^{14, 15} due to their respective alkoxy protons.

¹³C NMR spectra of these derivatives have been recorded. The aromatic carbon resonances of these derivatives were assigned by using incremental rules for aromatic substitution. The number of ¹³C NMR signals correspond to the number of chemically different carbon atoms present in these compounds. There is no significant chemical shift for the various ¹³C signals present in the mixed ligand complexes of antimony(III) as compared to the corresponding tris (alkyldithiocarbonate) antimony(III) and tris [2-(N-(salicylidene)-5-chlorobenzophenone)] antimony(III) complexes.

Thus based on the elemental analysis, molecular weight measurements and spectroscopic studies a bidentate mode of attachment of the both types of ligands to the metal atom is proposed. The dichloro derivative possess trigonal bipyramidal geometry with the lone pair in equatorial site(a), and bis derivative has octahedral geometry with the lone pair occupying an axial position(b) while tris derivative and mixed derivatives show a distorted octahedral geometry if the presence of the stereochemically active lone pair is also considered in the capping position (c, d, e) with pseudo seven coordinated structures.

(a)

(b)

(c)

(d)

(e)

EXPERIMENTAL

All reactions and manipulations were performed under an inert atmosphere. All solvents were freshly distilled over appropriated drying agents immediately prior to use¹⁸. All the chemicals used were of reagent grade. Antimony trichloride was distilled under reduced pressure before use. 2-(N-(salicylidene)-5-chlorobenzophenone¹⁰ and potassium alkylxanthates¹⁹ were prepared using a previously described procedure. Antimony was estimated iodometrically²⁰ after converting it to antimony(V); sulfur was estimated gravimetrically as barium sulphate²¹ and nitrogen was estimated by Kjeldahl's method²². The IR spectra were recorded on a Perkin-Elmer 580B instrument. The ¹H NMR spectra were recorded on a Perkin-Elmer R-32 and ¹³C NMR spectra, on a JEOL FX-90, using TMS as an external standard.

Reaction between SbCl₃ and the sodium salt of 2-(N-salicylidene)-5-chlorobenzophenone in a 1:1 molar ratio :

To an excess of isopropanol-benzene mixture (20 ml) a weighed quantity of Na metal (0.14g, 6 mmole) was added and refluxed for about half an hour till a clear solution of the sodium isopropoxide was obtained. After cooling 2.01g (6 mmole) of 2-(N-salicylidene)-5-chlorobenzophenone and about

15 ml of benzene was added and the mixture was refluxed for 2 hours on an oil bath. After cooling, 1.36g (6 mmole) of SbCl_3 was added and the mixture was further refluxed for 2-3 hours to ensure the completion of the reaction. The desired product was isolated after filtering off the precipitated NaCl . The product was further purified by crystallisation from benzene-petroleum ether (40-60°C) mixture.

All the other antimony(III) derivatives of 2-(N-salicylidene)-5-chlorobenzophenone were synthesised similarly.

Reaction between Chlorobis [2-(N-salicylidene)-5-chlorobenzophenone] antimony(III) and potassium methyl dithiocarbonate in a 1:1 molar ratio :

Potassium methyl dithiocarbonate and chlorobis [2-(N-salicylidene)-5-chlorobenzophenone] antimony(III) were stirred in benzene-carbon disulfide mixture (20 ml each) in 1:1 molar ratio for ~3 hours at room temperature. The precipitated KCl was removed by filtration, and the desired compound could be obtained by removing the solvent under reduced pressure. The compound was further purified by crystallisation from benzene-carbondisulfide mixture.

The other mixed derivatives were synthesised similarly. The characterisation of the compounds is given below.

Compound I : $\text{C}_{20}\text{H}_{13}\text{NO}_2\text{SbCl}_2$:

Yield, 96%; Mol. Wt. $[\text{F}(\text{C})]$: 525 (527.55) yellow solid; M.P., 129-130°C.

Analysis [%F(C)] : Sb, 20.75(23.07); Cl, 18.13 (20.18); N 2.42 (2.65)

IR (cm^{-1}) : ν (C=O), 1660; ν (C=N), 1610; ν (Sb-O), 510

PMR (δ ppm) : 6.65-7.60, m, 12H (Ph); 3.60, s, 1H (CH)

^{13}C NMR (δ ppm) : 197.4, C-8; 164.30, C-7; 134.11, C-1; 135.12, C-2; 138.0, C-3; 129.15, C-4; 133.09, C-5; 129.0, C-6; 135.50, C-1'; 116.15, C-2'; 129.01, C-3'; 118.90, C-4'; 128.10, C-5'; 117.50, C-6'; 151.56, C-1"; 137.00, C-2"; 132.11, C-3"; 127.19, C-4"; 127.95, C-5"; 130.59, C-6".

Compound II : $[\text{C}_{20}\text{H}_{13}\text{NO}_2]_2\text{SbCl}$:

Yield, 94%; Mol. Wt. $[\text{F}(\text{C})]$: 830 (826.85) yellow solid; M.P., 135-137°C.

Analysis [%F(C)] : Sb, 14.78 (14.72); Cl, 11.51 (12.88); N 3.22 (3.38)

IR (cm^{-1}) : ν (C=O), 1655; ν (C=N), 1615; ν (Sb-O), 515

PMR (δ ppm) : 6.60-7.62, m, 24H (Ph); 3.70, s, 2H (CH)

^{13}C NMR (δ ppm) : 196.4, C-8; 165.30, C-7; 134.22, C-1; 136.12, C-2; 138.9, C-3; 130.15, C-4; 133.29, C-5; 130.0, C-6; 136.50, C-1'; 116.55, C-2'; 129.31, C-3'; 118.90, C-4'; 128.40, C-5'; 116.50, C-6'; 150.56, C-1"; 137.40, C-2"; 132.61, C-3"; 126.79, C-4"; 127.95, C-5"; 130.59, C-6".

Compound III : $[\text{C}_{20}\text{H}_{13}\text{NO}_2]_3\text{Sb}$:

Yield, 97%; Mol. Wt. $[\text{F}(\text{C})]$: 1127 (1125.15) yellow solid; M.P., 136-138°C.

Analysis [%F(C)] : Sb, 10.32 (10.82); N 3.61 (3.73)

IR (cm^{-1}) : ν (C=O), 1650; ν (C=N), 1610; ν (Sb-O), 520

PMR (δ ppm) : 6.80-7.85, m, 36H (Ph); 3.85, s, 3H (CH)

^{13}C NMR (δ ppm) : 195.01, C-8; 163.74, C-7; 135.54, C-1; 136.32, C-2; 137.3, C-3; 129.54, C-4; 132.43, C-5; 128.65, C-6; 135.1, C-1'; 116.80, C-2'; 128.15, C-3'; 119.74, C-4'; 128.65, C-5'; 117.8, C-6'; 153.1, C-1"; 136.52, C-2"; 131.13, C-3"; 127.95, C-4"; 128.09, C-5"; 131.13, C-6".

Compound 1 : $[\text{C}_{20}\text{H}_{13}\text{NO}_2]_2\text{Sb}(\text{S}_2\text{COCH}_3)_2$:

Yield, 96%; Mol. Wt. $[\text{F}(\text{C})]$: 672 (670.55) yellow solid; M.P., 114-116°C.

Analysis [%F(C)] : Sb, 17.35 (18.16); S, 18.00 (19.09); N 1.99 (2.08)

IR (cm^{-1}) : ν (C=O), 1640; ν (C=N), 1590; ν (Sb-S), 320; ν (C=S), 1020

PMR (δ ppm) : 6.83-7.78, m, 12H (Ph); 3.25, s, 1H (CH); 0.98, s, 6H (CH_3)

^{13}C NMR (δ ppm) : 196.98, C-8; 163.44, C-7; 135.54, C-1; 134.94, C-2; 138.10, C-3; 129.15, C-4;

133.19, C-5; 128.55, C-6; 135.50, C-1'; 116.75, C-2'; 129.76, C-3'; 118.10, C-4'; 128.09, C-5'; 117.3, C-6'; 151.56, C-1"; 137.0, C-2"; 132.17, C-3"; 127.95, C-4"; 130.59, C-5"; 130.02, C-6"; 190.16, -C=S; 15.60, -OCH₃.

Compound 2 : [C₂₀H₁₃NO₂]₂ Sb S₂COCH₃ :

Yield, 91%; Mol. Wt. [F(C)] : 888 (898.35) yellow solid; M.P., 119-121°C.

Analysis [%F(C)] : Sb, 11.05 (13.55); S, 7.97 (7.12); N 2.97 (3.11)

IR (cm⁻¹) : ν (C=O), 1635; ν (C=N), 1590; ν (Sb-S), 315; ν (C=S), 1020

PMR (δppm) : 6.90-7.85, m, 24H (Ph); 3.20, s, 2H (CH); 1.05, s, 3H (CH₃)

¹³C NMR (δ ppm) : 197.3, C-8; 162.12, C-7; 132.19, C-1; 135.04, C-2; 138.06, C-3; 129.9, C-4; 133.02, C-5; 128.05, C-6; 133.60, C-1'; 117.8, C-2'; 130.0, C-3'; 117.11, C-4'; 130.09, C-5'; 119.3, C-6'; 151.4, C-1"; 138.14, C-2"; 133.7, C-3"; 127.6, C-4"; 128.34, C-5"; 130.02, C-6"; 190.66, -C=S; 15.98, -OCH₃.

Compound 3 : [C₂₀H₁₃NO₂]₂ Sb (S₂COC₂H₅)₂ :

Yield, 91%; Mol. Wt. [F(C)] : 678 (698.55) yellow solid; M.P., 116-117°C.

Analysis [%F(C)] : Sb, 17.12 (17.43); S, 17.58 (18.32); N 2.01 (2.00)

IR (cm⁻¹) : ν (C=O), 1635; ν (C=N), 1595; ν (Sb-S), 315; ν (C=S), 1015

PMR (δppm) : 6.90-7.80, m, 12H (Ph); 3.20, s, 1H (CH); 1.25, t, 6H (CH₃); 4.40, q, 4H (OCH₂)

¹³C NMR (δ ppm) : 196.9, C-8; 163.11, C-7; 132.79, C-1; 136.84, C-2; 139.21, C-3; 127.9, C-4; 133.52, C-5; 128.05, C-6; 133.64, C-1'; 117.52, C-2'; 129.92, C-3'; 117.84, C-4'; 129.00, C-5'; 119.76, C-6'; 152.54, C-1"; 137.00, C-2"; 132.8, C-3"; 128.6, C-4"; 128.0, C-5"; 130.76, C-6"; 188.32, -C=S; 63.4, -CH₂; 13.01, -CH₃.

Compound 4 : [C₂₀H₁₃NO₂]₂ Sb S₂COC₂H₅ :

Yield, 95%; Mol. Wt. [F(C)] : 915 (912.35) yellow solid; M.P., 113-115°C.

Analysis [%F(C)] : Sb, 12.55 (13.34); S, 6.91 (7.01); N 2.95 (3.06)

IR (cm⁻¹) : ν (C=O), 1650; ν (C=N), 1595; ν (Sb-S), 320; ν (C=S), 1012

PMR (δppm) : 6.95-7.75, m, 24H (Ph); 3.25, s, 2H (CH); 1.20, t, 3H (CH₃); 4.45, q, 2H (OCH₂)

¹³C NMR (δ ppm) : 197.0, C-8; 162.11, C-7; 133.19, C-1; 136.04, C-2; 139.11, C-3; 128.9, C-4; 133.02, C-5; 128.95, C-6; 134.64, C-1'; 117.0, C-2'; 129.31, C-3'; 118.1, C-4'; 129.09, C-5'; 119.0, C-6'; 152.3, C-1"; 137.12, C-2"; 133.1, C-3"; 128.0, C-4"; 128.34, C-5"; 131.12, C-6"; 189.36, -C=S; 62.5, -CH₂; 13.41, -CH₃.

Compound 5 : [C₂₀H₁₃NO₂]₂ Sb (S₂COC₃H₇)₂ :

Yield, 90%; Mol. Wt. [F(C)] : 731 (726.55) yellow solid; M.P., 115-117°C.

Analysis [%F(C)] : Sb, 15.88 (16.76); S, 17.00 (17.62); N 1.77 (1.93)

IR (cm⁻¹) : ν (C=O), 1636; ν (C=N), 1605; ν (Sb-S), 310; ν (C=S), 1015

PMR (δppm) : 6.95-7.82, m, 12H (Ph); 3.28, s, 1H (CH); 1.40, d, 12H (CH₃); 5.10, m, 1H (OCH)

¹³C NMR (δ ppm) : 194.99, C-8; 165.36, C-7; 134.66, C-1; 135.33, C-2; 137.9, C-3; 130.0, C-4; 133.55, C-5; 130.0, C-6; 136.12, C-1'; 117.1, C-2'; 129.35, C-3'; 118.3, C-4'; 127.11, C-5'; 116.95, C-6'; 152.1, C-1"; 137.12, C-2"; 133.11, C-3"; 128.18, C-4"; 127.34, C-5"; 130.26, C-6"; 190.4, -C=S; 51.5, -OCH; 23.2, -CH₃.

Compound 6 : [C₂₀H₁₃NO₂]₂ Sb S₂COC₃H₇ :

Yield, 94%; Mol. Wt. [F(C)] : 921 (925.35) yellow solid; M.P., 118-121°C.

Analysis [%F(C)] : Sb, 14.82 (13.15); S, 5.21 (6.91); N 2.88 (3.02)

IR (cm⁻¹) : ν (C=O), 1660; ν (C=N), 1600; ν (Sb-S), 310; ν (C=S), 1020

PMR (δppm) : 7.00-7.95, m, 24H (Ph); 3.30, s, 2H (CH); 1.45, d, 6H (CH₃); 5.05, m, 1H (OCH)

¹³C NMR (δ ppm) : 195.22, C-8; 164.11, C-7; 133.92, C-1; 137.42, C-2; 137.15, C-3; 131.21, C-4; 134.31, C-5; 130.12, C-6; 136.5, C-1'; 117.95, C-2'; 130.02, C-3'; 121.12, C-4'; 127.04, C-5'; 117.22,

C-6'; 151.56, C-1"; 138.16, C-2"; 130.92, C-3"; 127.92, C-4"; 124.42, C-5"; 131.12, C-6"; 190.91, -C=S; 52.0, -OCH₃; 24.5, -CH₃.

Compound 7 : [C₂₀H₁₃NO₂]₂Sb(S₂COC₄H₉)₂ :

Yield, 87%; Mol. Wt. [F(C)] : 742 (738.55) yellow solid; M.P., 111-113°C.

Analysis [%F(C)] : Sb, 15.72 (16.49); S, 16.89 (17.33); N 1.69 (1.89)

IR (cm⁻¹) : ν (C=O), 1640; ν (C=N), 1580; ν (Sb-S), 315; ν (C=S), 1010

PMR (δppm) : 6.85-7.85, m, 12H (Ph); 3.15, s, 1H (CH); 0.90, t, 6H (CH₃); 1.75, m, 8H (CH₂) 4.40, t, 4H (OCH₂)

¹³C NMR (δ ppm) : 196.72, C-8; 165.81, C-7; 134.92, C-1; 136.92, C-2; 137.15, C-3; 130.11, C-4; 135.41, C-5; 129.12, C-6; 136.5, C-1'; 117.05, C-2'; 129.12, C-3'; 120.32, C-4'; 127.74, C-5'; 118.02, C-6'; 150.36, C-1"; 138.16, C-2"; 130.92, C-3"; 127.02, C-4"; 125.12, C-5"; 132.12, C-6"; 189.34, -C=S; 65.0, -OCH₂; 23.1, -CH₂; 12.1, -CH₃.

Compound 8 : [C₂₀H₁₃NO₂]₂Sb S₂COC₄H₉ :

Yield, 88%; Mol. Wt. [F(C)] : 941 (932.35) yellow solid; M.P., 114-116°C.

Analysis [%F(C)] : Sb, 13.45 (13.05); S, 5.37 (6.86); N 2.92 (3.00)

IR (cm⁻¹) : ν (C=O), 1653; ν (C=N), 1605; ν (Sb-S), 300; ν (C=S), 1010

PMR (δppm) : 6.90-7.80, m, 24H (Ph); 3.20, s, 2H (CH); 0.95, t, 3H (CH₃), 4.35, t, 2H (OCH₂); 1.80, m, 4H (CH₂)

¹³C NMR (δ ppm) : 195.72, C-8; 165.31, C-7; 134.62, C-1; 137.12, C-2; 137.95, C-3; 129.11, C-4; 134.01, C-5; 129.12, C-6; 136.0, C-1'; 117.55, C-2'; 129.72, C-3'; 119.32, C-4'; 127.04, C-5'; 118.42, C-6'; 151.36, C-1"; 138.1, C-2"; 131.92, C-3"; 127.42, C-4"; 126.32, C-5"; 132.12, C-6"; 189.34, -C=S; 65.39, -OCH₃; 23.6, -CH₂; 11.5, -CH₃.

Compound 9 : [C₂₀H₁₃NO₂]₂Sb (S₂COC₄H₉)₂ :

Yield, 92%; Mol. Wt. [F(C)] : 731 (738.55) yellow solid; M.P., 116-118°C.

Analysis [%F(C)] : Sb, 16.32 (16.49); S, 16.42 (17.33); N 1.71 (1.89)

IR (cm⁻¹) : ν (C=O), 1620; ν (C=N), 1575; ν (Sb-S), 320; ν (C=S), 1012

PMR (δppm) : 6.95-7.80, m, 12H (Ph); 3.30, s, 1H (CH); 0.95, d, 12H (CH₃); 1.65, m, 4H (OCH₂)

¹³C NMR (δ ppm) : 196.50, C-8; 166.30, C-7; 133.9, C-1; 134.9, C-2; 138.51, C-3; 129.7, C-4; 132.71, C-5; 129.65, C-6; 135.72, C-1'; 116.75, C-2'; 130.0, C-3'; 117.85, C-4'; 129.10, C-5'; 116.19, C-6'; 154.0, C-1"; 136.90, C-2"; 132.0, C-3"; 129.43, C-4"; 126.51, C-5"; 129.12, C-6"; 191.4, C=S; 67.2, -OCH₂; 31.2, -CH; 23.5, -CH₃.

Compound 10 : [C₂₀H₁₃NO₂]₂Sb S₂COC₄H₉ :

Yield, 95%; Mol. Wt. [F(C)] : 936 (932.35) yellow solid; M.P., 121-124°C.

Analysis [%F(C)] : Sb, 12.30 (13.05); S, 6.15 (6.86); N 2.87 (3.00)

IR (cm⁻¹) : ν (C=O), 1660; ν (C=N), 1595; ν (Sb-S), 305; ν (C=S), 1015

PMR (δppm) : 6.65-7.80, m, 24H (Ph); 3.20, s, 2H (CH); 0.90, d, 3H (CH₃); 1.70, m, 2H (OCH₂)

¹³C NMR (δ ppm) : 196.70, C-8; 166.50, C-7; 133.0, C-1; 134.5, C-2; 137.51, C-3; 129.7, C-4; 132.11, C-5; 129.15, C-6; 134.72, C-1'; 117.05, C-2'; 130.5, C-3'; 117.85, C-4'; 129.10, C-5'; 117.10, C-6'; 154.5, C-1"; 136.95, C-2"; 132.5, C-3"; 129.43, C-4"; 126.51, C-5"; 129.12, C-6"; 190.8, C=S; 68.1, -OCH₂; 30.9, -CH; 23.9, -CH₃.

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