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Crystal structures of the 1/1 adducts of diphenylcyclopropenone with 3-triphenylstannyloxy-4,5-dimethylisothiazole 1,1-dioxide and 3-triphenylstannyloxybenzisothiazole 1,1-dioxide

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Abstract

Triphenyltin hydroxide condenses with 4,5-dimethylisothiazol-3(2*H*)-one 1,1-dioxide in the presence of one equivalent of diphenylcyclopropenone in ethanol to give $(C_6H_5)_3SnOC{=}NS(O_2)C(CH_3){=}CCH_3 \cdot O{=}CC(C_6H_5)CC_6H_5$. In the adduct the cyclic ketone (C=O = 1.245(3) Å) is linked coordinately (Sn ← O = 2.372(2) Å; Sn ← O=C = 122.7(2)°C) to the Lewis acid (Sn–O = 2.164(2) Å). The geometry of the tin atom is *trans*-trigonal bipyramidal (O–Sn ← O = 178.5(1)°; ΣC–Sn–C = 357.7(3)°). Triphenyltin hydroxide reacts with saccharin and diphenylcyclopropenone in ethanol to give the isostructural $(C_6H_5)_3SnOC{=}NS(O_2)C_6H_4 \cdot O{=}CC(C_6H_5)CC_6H_5$ complex. Selected bond distances and angles in this complex are: Sn–O = 2.164(3), Sn ← O = 2.421(3), C=O = 1.230 (4) Å, Sn ← O=C = 129.3(2)°; O–SN ← O = 173.2(1)°; ΣC–Sn–C = 358.5(3)°.

1. Introduction

Triphenyltin hydroxide condenses with 4,5-dimethylisothiazole 1,1-dioxide in ethanol to give the *N*-stannyl derivative as an air-stable product. The preparation of the analogous compound from 1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide (saccharin) requires the use of toluene as the reaction medium [1] since when the reaction is conducted in ethanol it yields exclusively the ethanol adduct [2]. Several 1/1 *O*-donor adducts of *N*-triphenylstannyl 1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide also have been prepared and the *trans*-C_3SnNO trigonal bipyramidal environments established by crystal structure analysis for the *N*,*N*-dimethylformamide [3], quinoline *N*-oxide [4] and dibenzylsulfoxide [5] complexes. A similar structure has been assigned to the diphenylcyclopropenone adduct [3] on the basis of its Mössbauer spectral values. Crystal structure analysis of this adduct and of the 4,5-dimethylisothiazole, 1,1-dioxide complex reveals that the Lewis acid exists as an *O*-bonded stannyloxy-isothiazole. Their crystal structures are described in this report.

2. Experimental section

Stoichiometric amounts of triphenyltin hydroxide, 4,5-dimethylisothiazol-3(2*H*)-one 1,1-dioxide and diphenylcyclopropenone were briefly heated in a small volume of ethanol to yield 3-triphenylstannyloxy-4,5-dimethylisothiazole 1,1-dioxide · diphenylcyclopropenone. The product was recrystallized from ethanol. [**Caution:** Diphenylcyclopropenone may cause severe allergic reactions on the skin! See caution note in H.L. Ammon, *J. Am. Chem. Soc.*, 95 (1973) 7093.]

Unit-cell dimensions were calculated from the 25 strongest reflections in the $\Theta = 12$–$14°$ shell. Data were measured with the Enraf-Nonius CAD4 diffractometer (Mo Kα, 0.71073 Å) operating in the ω–2Θ scan mode up to $2\Theta_{max} = 50°$ (collection range: $0 \le h \le 11$, $-13 \le k \le 12$, $-18 \le l \le 18$). Peak profiles were calculated [6]. Of the 6094 data, 4762 of the 5348 unique data

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obeyed the $I \geq 3\sigma$ (I) criterion. The data were corrected for decay of 3.80% over 84.3 h, and for absorption (transm. factors = 0.8869–0.9582) effects [7]. The structure was solved by using the heavy atom method. Non-H atoms were refined anisotropically; H-atoms were located and refined with $B = 5$ Å^2. Refinement on F converged for the 499 variables with a shift-to-error ratio of 0.07 for non-H atoms (0.12 for all atoms) to final unweighted R and weighted R_w indices of 0.026 and 0.029, respectively; $w = [\sigma^2 |F_o| + (0.02|F_o|)^2 + 1]^{-1}$ [8] and $S = 0.305$. In the final difference map extrema ranged between $-0.22(3)$ and 0.32(3) eÅ^{-3} against a diffuse background. Analytical expressions of neutral scattering factors and anomalous-dispersion coefficients were taken from Tables 2.2B and 2.3.1. of the *International tables for X-ray crystallography,* Vol. IV (1974). The MOLEN package [9] running on a MicroVAX minicomputer was used for all calculations. The atomic coordinates are listed in Table 1; the molecular structure is depicted in Fig. 1. Supplementary data are available from the authors.

2.1. Crystal data

$C_{38}H_{31}NO_4SSn$, FW = 716.43, triclinic, $P\bar{1}$ (No. 2), $a = 9.6537(4)$, $b = 11.0826(5)$, $c = 15.7283(5)$ Å, $\alpha = 95.696(3)$, $\beta = 91.314(3)$, $\gamma = 102.651(4)°$, $V = 1632.1(1)$ Å^3, $F(000) = 728$, $D_x = 1.458$, $\mu = 8.85$ cm^{-1} for $Z = 2$.

3-Triphenylstannyloxybenzisothiazole 1,1-dioxide · diphenylcyclopropenone was prepared from triphenyltin hydroxide, saccharin and diphenylcyclopropenone in ethanol [3] and recrystallized from ethanol. Diffraction data were measured up to $2\Theta_{max} = 55°$ with the Siemens P4 instrument (Mo Kα, 0.71073 Å) operating in the ω-scan mode (collection range: $0 \leq h \leq 21$, $0 \leq k \leq 13$, $-31 \leq l \leq 31$). The 9658 unique data were processed with a profile-fitting procedure [10] and were corrected for absorption (transm. factors = 0.585–0.644) [11]; 6322 having $|F_o| \geq 3\sigma|F_o|$ were used in the structure solution and refinement. The structure was solved by using the heavy atom method. The three phenyl rings bonded to tin and the two phenyl rings belonging to the cyclic ketone were found to be disordered, and these rings had to be constrained into regular hexagons (C–C = 1.395 Å). Non-H atoms were refined anisotropically; H-atoms were generated geometrically (C–H = 0.96 Å, $U = 0.080$ Å^2) and were included in the structure-factor calculations. Refinement on F for the 214 variables gave a final R value of 0.042; $R_w = 0.057$ ($w = [\sigma^2|F_o| + 0.0015|F_o|^2]^{-1}$, $S = 1.06$). No peaks smaller than -0.68 eÅ^{-3} or larger than 0.83 eÅ^{-3} were found in the final difference map. Computations were performed with the SHELXTL-PLUS [12] package on a MicroVAX II minicomputer. The atomic coordinates are listed in Table 2; the structure is illustrated in Fig. 2. Supplementary data are available from the authors.

TABLE 1. Atomic coordinates and isotropic [a] temperature factors for $(C_6H_5)_3SnOC{=}NS(O_2\,C(CH_3)CCH_3 \cdot O{=}CC(C_6H_5)CC_6H_5$

Atom	x	y	z	B_{eq} (Å^2)
Sn	0.06442(2)	0.18761(2)	0.25937(1)	2.898(4)
S	0.1549(1)	0.65144(9)	0.32758(6)	4.61(2)
O1	0.1694(2)	0.0312(2)	0.1914(1)	3.45(4)
O2	−0.0307(2)	0.3284(2)	0.3245(2)	4.38(5)
O3	0.1339(4)	0.7145(3)	0.2547(2)	6.97(8)
O4	0.2850(3)	0.6982(3)	0.3773(2)	7.31(8)
N	0.1343(3)	0.5023(3)	0.3001(2)	4.25(6)
C1	0.2286(3)	0.2026(3)	0.3558(2)	3.31(6)
C2	0.3190(3)	0.3118(3)	0.3916(2)	4.08(7)
C3	0.4197(4)	0.3071(4)	0.4555(2)	5.20(9)
C4	0.4300(4)	0.1974(4)	0.4843(3)	5.8(1)
C5	0.3402(5)	0.0895(4)	0.4502(3)	5.9(1)
C6	0.2404(4)	0.0913(3)	0.3858(2)	4.75(8)
C7	−0.1208(3)	0.0480(3)	0.2755(2)	3.35(6)
C8	−0.2414(4)	0.0796(3)	0.3083(2)	4.68(8)
C9	−0.3623(4)	−0.0098(4)	0.3192(3)	5.8(1)
C10	−0.3665(4)	−0.1315(4)	0.2980(3)	6.1(1)
C11	−0.2499(5)	−0.1643(4)	0.2657(3)	6.8(1)
C12	−0.1275(4)	−0.0769(3)	0.2539(3)	4.99(9)
C13	0.0609(3)	0.2454(3)	0.1335(2)	3.01(6)
C14	0.1196(3)	0.3622(3)	0.1089(2)	3.45(6)
C15	0.1008(4)	0.3887(3)	0.0254(2)	4.07(7)
C16	0.0222(4)	0.2984(3)	−0.0347(2)	4.52(8)
C17	−0.0346(4)	0.1830(3)	−0.0116(2)	4.43(8)
C18	−0.0160(3)	0.1555(3)	0.0711(2)	3.75(7)
C19	0.2966(3)	0.0512(3)	0.1730(2)	3.13(6)
C20	0.4096(3)	0.0046(3)	0.1406(2)	3.26(6)
C21	0.4292(3)	0.1298(3)	0.1648(2)	3.37(6)
C22	0.4633(3)	−0.1016(3)	0.1058(2)	3.26(6)
C23	0.3807(3)	−0.2195(3)	0.1096(2)	3.95(7)
C24	0.4313(4)	−0.3220(3)	0.0774(3)	4.91(9)
C25	0.5613(4)	−0.3065(3)	0.0418(2)	4.77(8)
C26	0.6426(3)	−0.1902(3)	0.0376(2)	4.40(7)
C27	0.5951(3)	−0.0865(3)	0.0695(2)	3.89(7)
C28	0.5247(3)	0.2510(3)	0.1714(2)	3.34(6)
C29	0.6642(4)	0.2656(3)	0.1471(3)	4.75(8)
C30	0.7523(4)	0.3810(4)	0.1515(3)	5.36(9)
C31	0.7042(4)	0.4832(4)	0.1817(3)	5.6(1)
C32	0.5659(4)	0.4711(3)	0.2062(3)	5.29(9)
C33	0.4766(3)	0.3555(3)	0.2007(2)	4.12(7)
C34	0.0179(3)	0.4461(3)	0.3333(2)	3.68(7)
C35	−0.0615(3)	0.5254(3)	0.3866(2)	3.47(6)
C36	0.0061(4)	0.6431(3)	0.3921(2)	4.02(7)
C37	−0.0224(5)	0.7580(4)	0.4397(3)	6.3(1)
C38	−0.1967(4)	0.4700(4)	0.4248(2)	5.05(8)

[a] $B_{eq} = 4/3(a^2B_{1,1} + b^2B_{2,2} + c^2B_{3,3} + ab(\cos\gamma)B_{1,2} + ac(\cos\beta)B_{1,3} + bc(\cos\alpha)B_{2,3})$.

2.2. Crystal data:

$C_{40}H_{29}NO_4SSn$, FW = 738.4, monoclinic, $P2_1/c$ (No. 14), $a = 15.485(3)$, $b = 9.548(2)$, $c = 22.449(3)$ Å, $\beta = 90.59(1)°$, $V = 3319(1)$ Å^3, $F(000) = 1496$, $D_x = 1.478$, $\mu = 8.76$ cm^{-1} for $Z = 4$

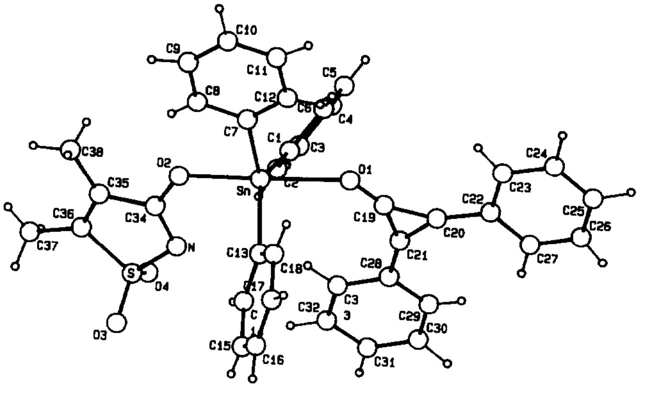

Fig. 1. Numbering scheme for $(C_6H_5)_3SnO\overline{C{=}NS(O_2)C(CH_3)C}CH_3\cdot O{=}\overline{CC(C_6H_5)C}C_6H_5$. Selected bond distances (Å) and angles (°): Sn–O1 = 2.372(2), Sn–O2 = 2.164(2), Sn–C1 = 2.136(3), Sn–C7 = 2.131(3), Sn–C13 = 2.142(3), C19–O1 = 1.245(3), C19–C20 = 1.392(4), C19–C21 = 1.398(4), C20–C21 = 1.373(4), C24–O2 = 1.278(4) Å; O1–Sn–O2 = 178.49 (9), O1–Sn–C1 = 83.9(1), O1–Sn–C7 = 88.9(1), O1–Sn–C13 = 83.4(1), O2–Sn–C1 = 95.1(1), O2–Sn–C7 = 90.6(1), O2–Sn–C13 = 98.2(1), C1–Sn–C7 = 113.6(1), C1–Sn–C13 = 133.7(1), C7–Sn–C13 = 110.4(1), Sn–O1–C19 = 122.7(2), O1–C19–C20 = 148.4(3), O1–C19–C21 = 152.6(3), C19–C20–C21 = 60.8(2), C19–C21–C20 = 60.3(2), C20–C19–C21 = 58.9(2), Sn–O2–C34 = 127.6(2)°.

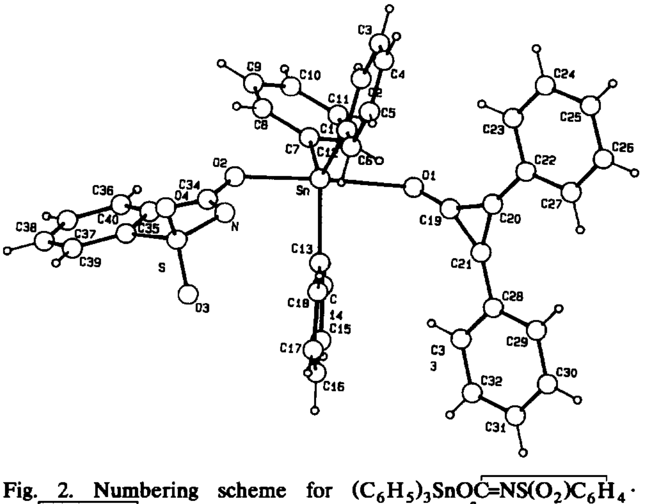

Fig. 2. Numbering scheme for $(C_6H_5)_3SnO\overline{C{=}NS(O_2)C_6H_4}\cdot O{=}\overline{CC(C_6H_5)C}C_6H_5$. Selected bond distances (Å) and angles (°): Sn–O1 = 2.421(3), Sn–O2 = 2.164(3), Sn–C1 = 2.117(3), Sn–C7 = 2.128(2), Sn–C13 = 2.115(3), C19–O1 = 1.230(4), C19–C20 = 1.401(5), C19–C21 = 1.392(5), C20–C21 = 1.368(5), C24–O2 = 1.272(4) Å; O1–Sn–O2 = 173.2(1), O1–Sn–C1 = 85.9(1), O1–Sn–C7 = 87.2(1), O1–Sn–C13 = 84.8(1), O2–Sn–C1 = 100.8(1), O2–Sn–C7 = 89.6(1), O2–Sn–C13 = 91.3(1), C1–Sn–C7 = 113.6(1), C1–Sn–C13 = 127.1(3), C7–Sn–C13 = 117.8(1), Sn–O1–C19 = 128.8(2), O1–C19–C20 = 149.3(4), O1–C19–C21 = 152.0(4), C19–C20–C21 = 60.3(3), C19–C21–C20 = 61.0(3), C20–C19–C21 = 58.7(2), Sn–O2–C34 = 129.3(2)°.

TABLE 2. Atomic coordinates and equivalent isotropic [a] temperature factors for $(C_6H_5)_3SnO\overline{C{=}NS(O_2)C_6H_4}\cdot O{=}\overline{CC(C_6H_5)C}C_6H_5$

Atom	x	y	z	U_{eq} (Å^2)
Sn	0.20989(1)	0.24636(3)	0.20946(1)	0.0334(1)
S	0.24467(6)	0.27362(9)	0.43585(4)	0.0373(3)
O1	0.2803(2)	0.2923(3)	0.1156(1)	0.0431(1)
O2	0.1494(2)	0.1793(3)	0.2911(1)	0.048(1)
O3	0.3331(2)	0.2361(3)	0.4478(2)	0.058(1)
O4	0.2126(2)	0.3940(3)	0.4665(1)	0.051(1)
N	0.2277(2)	0.2910(3)	0.3640(1)	0.042(1)
C1	0.1905(2)	0.4656(3)	0.2141(1)	0.039(1)
C2	0.1239(2)	0.5191(3)	0.1785(1)	0.054(1)
C3	0.1066(2)	0.6625(3)	0.1785(1)	0.067(1)
C4	0.1559(2)	0.7524(3)	0.2141(1)	0.072(1)
C5	0.2225(2)	0.6989(3)	0.2497(1)	0.076(1)
C6	0.2398(2)	0.5555(3)	0.2497(1)	0.062(1)
C7	0.1127(2)	0.1393(3)	0.1596(1)	0.033(1)
C8	0.0346(2)	0.1077(3)	0.1870(1)	0.049(1)
C9	−0.0288(2)	0.0325(3)	0.1561(1)	0.059(1)
C10	−0.0141(2)	−0.0110(3)	0.0978(1)	0.058(1)
C11	0.0639(2)	0.0207(3)	0.0703(1)	0.050(1)
C12	0.1273(2)	0.0959(3)	0.1012(1)	0.038(1)
C13	0.3241(2)	0.1352(3)	0.2313(1)	0.036(1)
C14	0.3356(2)	0.0079(3)	0.2019(1)	0.053(1)
C15	0.4075(2)	−0.0750(3)	0.2151(1)	0.063(1)
C16	0.4679(2)	−0.0306(3)	0.2576(1)	0.060(1)
C17	0.4564(2)	0.0967(3)	0.2869(1)	0.058(1)
C18	0.3845(2)	0.1976(3)	0.2738(1)	0.046(1)
C19	0.3474(2)	0.3573(4)	0.1064(2)	0.035(1)
C20	0.3886(2)	0.4830(4)	0.0916(2)	0.036(1)
C21	0.4367(2)	0.3647(4)	0.1008(2)	0.036(1)
C22	0.3837(1)	0.6292(2)	0.0763(1)	0.036(1)
C23	0.3028(1)	0.6936(2)	0.0763(1)	0.046(1)
C24	0.2948(1)	0.8337(2)	0.0595(1)	0.057(1)
C25	0.3677(1)	0.9094(2)	0.0428(1)	0.056(1)
C26	0.4485(1)	0.8450(2)	0.0428(1)	0.051(1)
C27	0.4566(1)	0.7049(2)	0.0596(1)	0.043(1)
C28	0.5204(2)	0.2997(2)	0.1031(1)	0.040(1)
C29	0.5954(2)	0.3736(2)	0.0894(1)	0.045(1)
C30	0.6752(2)	0.3060(2)	0.0909(1)	0.054(1)
C31	0.6801(2)	0.1645(2)	0.1060(1)	0.057(1)
C32	0.6052(2)	0.0906(2)	0.1197(1)	0.057(1)
C33	0.5253(2)	0.1582(2)	0.1182(1)	0.048(1)
C34	0.1756(2)	0.1933(4)	0.3447(2)	0.038(1)
C35	0.1450(2)	0.0918(4)	0.3906(2)	0.036(1)
C36	0.0935(2)	−0.0253(4)	0.3832(2)	0.047(1)
C37	0.0750(3)	−0.1045(5)	0.4333(2)	0.059(2)
C38	0.1068(3)	−0.0656(5)	0.4889(2)	0.059(2)
C39	0.1585(3)	0.0505(5)	0.4970(2)	0.049(1)
C40	0.1771(2)	0.1273(4)	0.4462(2)	0.036(1)

[a] U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

3. Results and discussion

Triphenyltin hydroxide condenses with 4,5-dimethylisothiazol −3(2*H*)-one 1,1-dioxide and with 1,2-benzisothiazol-3-(2*H*)-one, 1,1-dioxide in the presence of an equivalent quantity of diphenylcyclopropenone in ethanol to furnish 3-triphenylstannyloxy-4,5-dimethylisothiazole 1,1-dioxide · diphenylcyclopropenone and 3-triphenylstannyloxybenzisothiazole 1,1-dioxide · diphenylcyclopropenone, respectively. In both compounds the triphenyltin cation is covalently linked to the isothiazolyl ring by a tin–oxygen bond rather than the expected tin–nitrogen bond, so that the organotin moiety formally exists as a triphenyltin alkoxide. The diphenylcyclopropenone donor is linked through its oxygen end to the triphenyltin alkoxide molecule; the carbonyl bond distance is short however, implying that the *O*-donor exists as a ketone rather than as a dipolar compound in the complexes. The coordination behavior of the ligand parallels that found in its 1/1 complex with triphenyltin chloride (Sn ← O = 2.519(2) Å, ΣC–Sn–C = 354.3(3)°) [13]. The present compounds represent rare examples of air-stable organotin alkoxides, most of which are sensitive to moisture and carbon dioxide [14]. The organotin structural literature [15,16] contains only one documented example of an alkoxide: in the crystal structure of trimethyltin methoxide planar trimethyltin groups are linked symmetrically (Sn–O = 2.20(2), Sn ← O = 2.26(2) Å) by the methoxide ion to form a zig-zag chain [17].

In $(C_6H_5)_3SnO\overline{C{=}NS(O_2)C(CH_3){=}C}CH_3 \cdot O{=}\overline{CC(C_6H_5)C}C_6H_5$ the cyclic ketone (C=O = 1.245(3) Å) is linked coordinatively (Sn ← O = 2.372(2) Å; Sn ← O=C = 122.7(2)°) to the aryloxide (Sn–O = 2.164(2) Å) molecule. The oxygen–tin–oxygen skeleton is almost linear (O–Sn ← O = 178.5(1)°), but the geometry at tin is a distorted *trans*-C_3SnO_2 trigonal bipyramid arising from the widening (C1–Sn–C13 = 133.7(1)°) of one of the carbon–tin–carbon angles. The combined steric bulk of the isothiazolyl unit and a phenyl ring belonging to the diphenylcyclopropenone donor leads to some deformation of the two phenyl rings bonded to tin, as seen from the large tin–carbon$_{ipso}$–carbon$_{ortho}$ (Sn–C1–C2 = 126.1(3), Sn–C13–C14 = 127.4(2)°) angles. The tin atom is displaced out of the equatorial plane (ΣC–Sn–C = 357.7 (3)°) in the direction of the isothiazolyl oxygen atom by 0.184(1) Å. Other than for a lengthening of the carbon–oxygen bond, which is offset by a shortening of the carbon–nitrogen bond, other bond dimensions in the isothiazolyl unit do not differ much from those found in the parent imide [18] and in *N*-triphenylstannyl 4,5-dimethylisothiazol-3(2*H*)-one 1,1-dioxide [1].

The $(C_6H_5)_3SnO\overline{C{=}NS(O_2)C}_6H_4 \cdot O{=}\overline{CC(C_6H_5)C}C_6H_5$ complex is isostructural with that described above. The tin–oxygen coordination bond is somewhat longer (Sn ← O1 = 2.421(3) Å), although the covalent tin–oxygen (Sn–O2 = 2.164(3) Å) is not affected by the nature of the substituents in the isothiazolyl ring. Again, bond dimensions involving the saccharin unit do not differ much from those in the parent organic compound [19] or from those found in *N*-triphenylstannyl 1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide [1]. The carbon–oxygen (C–O = 1.272(4) Å) single-bond and carbon–nitrogen (C–N = 1.306(5) Å) double-bond both remain localized, in contrast to the situation found in the *N*,*N*-dimethyl-4-toluidinium salt of saccharin, which exists as an *N*,*N*-dimethyl-4-toluidium sulfobenzoimidate ion-pair (C–O = 1.252(4), C–N = 1.332(4) Å). In this case, the negative charge is delocalized over the O–C–N unit [20].

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