The First Well-Defined Tellurastannoxanes: the X-ray Structure of *trans*-[$(Bu_3SnO)_2\{CH_2(Ph_2SnO)_2\}_2Te$]

Jens Beckmann,[a] Dainis Dakternieks,*[a] Jenny O'Connell,[a] Klaus Jurkschat,[b] and Markus Schürmann[b]

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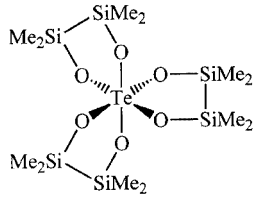
The syntheses of well-defined tellurastannoxanes, *mer*-$(Bu_3SnO)_3Te(OH)_3$ (**1**), and *trans*-$(Bu_3SnO)_2[CH_2(Ph_2SnO)_2]_2Te$ (**2**), are reported. The compounds were characterized by ^{119}Sn and ^{125}Te NMR spectroscopy and compound **2** also by single crystal X-ray diffraction.

Introduction

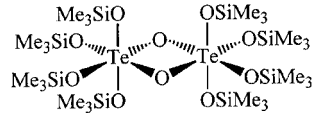
Currently there is great interest in molecular mixed-element oxides incorporating main group elemental oxide fragments such as $M(O^-)_3$ (M = B, Al, Ga), $MO(O^-)_3$ (M = P), or $M(O^-)_4$ (M = Si, Ge, Sn), mainly as a result of potential applications in material science and catalysis.[1,2] In most cases, the preferred valence numbers within these fragments preclude a degree of branching higher than four.

Although the synthesis of the first alkyl ester of orthotelluric acid, $Te(OH)_6$, was described early last century there have been only a few subsequent reports utilizing the acidic protons in $Te(OH)_6$ to build new organometallic complexes.[3] In one case molecular tellurasiloxanes, namely $[(Me_2SiO)_2]_3Te$ (**I**), and $(Me_3SiO)_8Te_2O_2$ (**II**)[4] were described and in another case a unique mixed-valence Te^{IV}-Te^{VI} species $\{OTe(C_8H_8)[(EtO)_2PS_2]\}_6Te$ (**III**) was reported.[5]

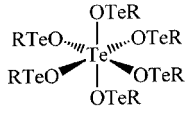
Here we present the syntheses of the first tellurastannoxanes *mer*-$[(Bu_3SnO)_3Te(OH)_3]$ and *trans*-$[(Bu_3SnO)_2\{CH_2(Ph_2SnO)_2\}_2Te]$. The former compound containing unsubstituted hydroxyl groups has potential as a building block for the preparation of ternary mixed-metal oxides, a class of compounds for which few examples are known.[6–8]



I



II

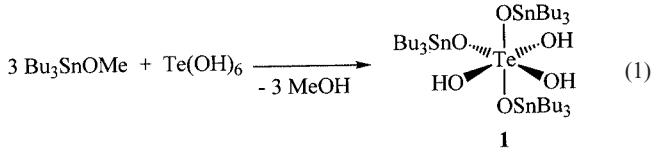


III

R = $(C_8H_8)(S_2P(OEt)_2)$

Results and Discussion

The reaction between $Te(OH)_6$ and three equivalents of Bu_3SnOMe quantitatively produced *mer*-$[(Bu_3SnO)_3Te(OH)_3]$ (**1**) [Equation (1)]:



(1)

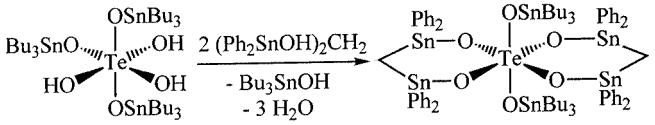
1

Compound **1** was obtained as a colorless, odorless low-melting waxy material that slowly crystallized over several weeks. The ^{119}Sn NMR spectrum ($CDCl_3$) of **1** reveals two signals at $\delta = 81.9$ [$^2J(^{119}Sn-O-^{125}Te) = 434$ Hz] and 93.6 [$^2J(^{119}Sn-O-^{125}Te) = 504$ Hz] with an integral ratio of 1:2. The number of signals confirms the exclusive formation of

[a] Centre for Chiral and Molecular Technologies, Deakin University,
Geelong, Victoria 3217, Australia
Fax: (internat.) +61-3/5227-1040
E-mail: dainis@deakin.edu.au
[b] Lehrstuhl für Anorganische Chemie II, Fachbereich Chemie der Universität Dortmund,
44221 Dortmund, Germany

the *mer*-isomer. The ^{125}Te NMR spectrum ($CDCl_3$) of **1** shows a signal at δ = 721.5 with two $^2J(^{125}Te-O-^{119}Sn)$ couplings of 501 and 432 Hz. Bearing in mind the natural abundance of the isotopes ^{117}Sn, ^{119}Sn, and ^{125}Te the integration of the satellites with respect to the main signal allows determination of the connectivity of the tin and tellurium atoms. This method has already been applied for the identification of other compounds showing heteronuclear couplings, such as ^{117}Sn/^{119}Sn,[9−11] ^{29}Si/^{119}Sn,[12] and ^{29}Si/^{125}Te.[4] The molecular weight determination of **1** ($CHCl_3$) unambiguously indicates that no condensation to higher aggregates had occurred and suggests that all three remaining hydroxyl groups are still present. The IR spectrum of **1** (KBr) further supports the presence of hydroxyl groups by revealing a very broad ν(OH) stretching vibration at 3372 cm^{-1}. The reason for selective formation of the *mer*-isomer under these reaction conditions is still not understood. Reactions of $Te(OH)_6$ with Bu_3SnOMe in stoichiometric ratios other than 1:3 provided mixtures of products with a varying degree of substitution, $[(Bu_3SnO)_nTe(OH)_{n-6}]$ $(n \geq 3)$.[13] The identity of these species was unambiguously proven by ^{119}Sn and ^{125}Te NMR spectroscopy, although all attempts to isolate pure compounds from the mixtures failed. Moreover, reactions between $Te(OH)_6$ and other triorganotin methoxides, R_3SnOMe (R = Me, *c*Hex, Ph), also provided mixtures of products that could not be purified.[14]

The reaction of *mer*-$[(Bu_3SnO)_3Te(OH)_3]$ (**1**) with two equivalents of $(Ph_2SnOH)_2CH_2$ [15] gave, with elimination of water and one equivalent of tributyltin hydroxide (Bu_3SnOH), the spirocyclic tellurastannoxane derivative *trans*-$[(Bu_3SnO)_2\{CH_2(Ph_2SnO)_2\}_2Te]$ (**2**) in good yields [Equation (2)].



1 **2** (2)

Compound **2** is a colorless crystalline material. The ^{119}Sn NMR spectrum ($CDCl_3$) of **2** reveals two signals with an integral ratio of 2:1 belonging to the $(Ph_2SnO)_2CH_2$ moiety and the Bu_3SnO moiety, respectively, at δ = −40.2 $[^2J(^{119}Sn-O-^{117}Sn)$ = 632 Hz; $^2J(^{119}Sn-O-^{125}Te)$ = 353 Hz] and δ = 89.6 $[^2J(^{119}Sn-O-^{125}Te)$ = 393 Hz]. It is interesting to note that the chemical shift for the $(Ph_2SnO)_2CH_2$ moiety at δ = −40.2 is very close to that found for the *cyclo*-stannasiloxane $tBu_2Si(OSnPh_2)_2CH_2$ [$\delta(^{119}Sn)$ = −42.8], which also contains a $(Ph_2SnO)_2CH_2$ fragment incorporated into a six-membered ring.[16] The ^{125}Te NMR spectrum ($CDCl_3$) of compound **2** shows a signal at δ = 726.1 with two $^2J(^{125}Te-O-^{119}Sn)$ couplings of 397 and 355 Hz in the expected integral ratio. The number of signals in the ^{119}Sn NMR spectrum and the observed coupling patterns in both ^{119}Sn and ^{125}Te NMR spectroscopy confirms the coordination geometry of the tin atoms around the tellurium center.

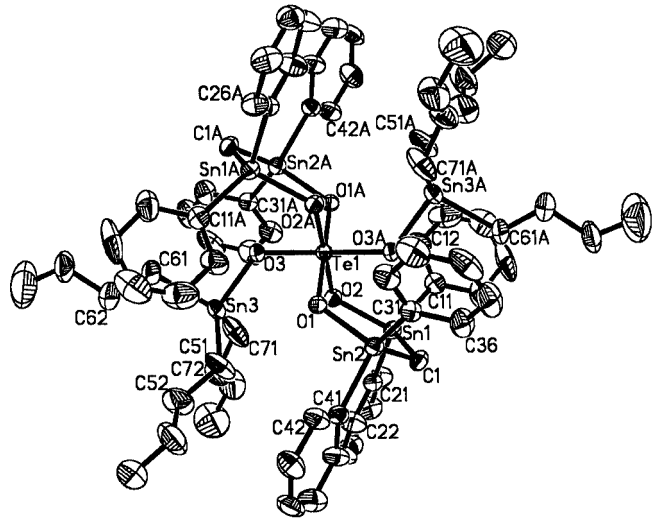


Figure 1. General view (SHELXTL-PLUS) of **2** showing 30% probability displacement ellipsoids and the atom numbering for **2** (symmetry transformations used to generate equivalent atoms: $a = -x + 0.5, -y - 0.5, -z + 1$)

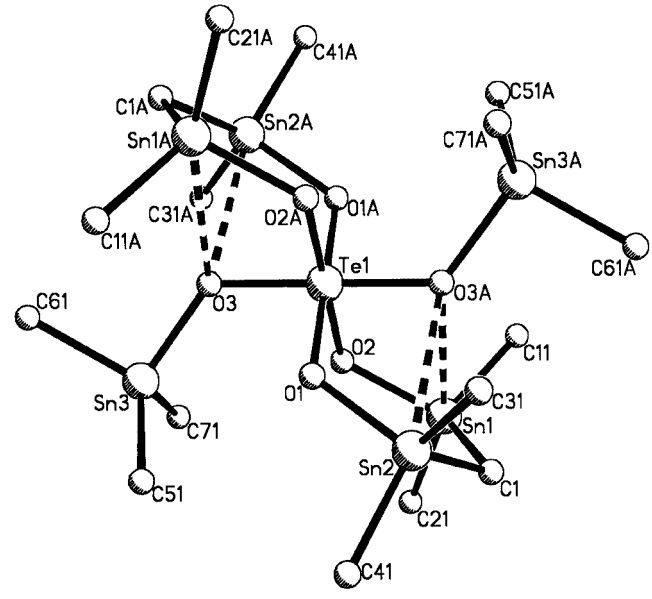


Figure 2. General view (SHELXTL-PLUS) of **2** demonstrating the [4+1] coordination of tin atoms Sn(1) and Sn(2); only primary carbon atoms are shown for clarity (symmetry transformations used to generate equivalent atoms: $a = -x + 0.5, -y - 0.5, -z + 1$)

The molecular structure of **2** is shown in Figure 1 and 2, and selected bond lengths, bond angles and torsion angles are listed in Table 1. The structure contains a crystallographic center of inversion across the tellurium atom Te(1), which coordinated in an octahedral fashion to six oxygen atoms. The mean Te−O bond length amounts to 1.912(3) Å, which is close to the bond length found in $[(Me_2SiO)_2]_3Te$.[4] The central mononuclear TeO_6 core is stannylated at O(3)/O(3a) by two tributylstannyl units and at O(1), O(2)/O(1a), O(2a) by two chelating bis(diphenylstannyl)methyl moieties, giving rise to the formation of a spiroundecane skeleton. The six-membered Te(1)−O(1)−Sn(2)−

Table 1. Selected bond lengths [Å], bond angles, and torsion angles [°] for **2** (symmetry transformations used to generate equivalent atoms: $a = -x + 1/2, -y - 1/2, -z + 1$)

Te(1)−O(1)	1.915(3)	Te(1)−O(2)	1.907(3)
Te(1)−O(3)	1.914(3)	Sn(1)−O(2)	1.986(3)
Sn(1)−O(3a)	2.944(3)	Sn(1)−C(1)	2.123(5)
Sn(1)−C(11)	2.137(6)	Sn(1)−C(21)	2.128(5)
Sn(2)−O(1)	1.982(3)	Sn(2)−O(3a)	3.003(3)
Sn(2)−C(1)	2.128(5)	Sn(2)−C(31)	2.117(5)
Sn(2)−C(41)	2.137(5)	Sn(3)−O(1)	3.518(3)
Sn(3)−O(2)	3.398(3)	Sn(3)−O(3)	2.015(3)
Sn(3)−C(51)	2.124(6)	Sn(3)−C(61)	2.124(6)
Sn(3)−C(71)	2.106(7)		
O(1)−Te(1)−O(2)	89.68(13)	O(1)−Te(1)−O(2a)	90.32(13)
O(1)−Te(1)−O(3)	90.78(13)	O(1)−Te(1)−O(3a)	89.22(13)
O(2)−Te(1)−O(3)	91.00(12)	O(2)−Te(1)−O(3a)	89.00(12)
O(1)−Te(1)−O(1a)	180.00(16)	O(2)−Te(1)−O(2a)	180.00(17)
O(3)−Te(1)−O(3a)	180.000(1)	O(2)−Sn(1)−O(3a)	62.28(10)
O(2)−Sn(1)−C(1)	110.01(15)	O(2)−Sn(1)−C(11)	108.1(2)
O(2)−Sn(1)−C(21)	101.58(16)	O(3a)−Sn(1)−C(1)	73.24(15)
O(3a)−Sn(1)−C(11)	83.08(16)	O(3a)−Sn(1)−C(21)	163.05(16)
C(1)−Sn(1)−C(11)	117.2(2)	C(1)−Sn(1)−C(21)	110.4(2)
C(11)−Sn(1)−C(21)	108.4(2)	O(1)−Sn(2)−O(3a)	61.32(10)
O(1)−Sn(2)−C(1)	112.33(15)	O(1)−Sn(2)−C(31)	108.01(18)
O(1)−Sn(2)−C(41)	101.32(16)	O(3a)−Sn(2)−C(1)	71.89(15)
O(3a)−Sn(2)−C(31)	86.24(10)	O(3a)−Sn(2)−C(41)	160.76(14)
C(1)−Sn(2)−C(31)	114.9(2)	C(1)−Sn(2)−C(41)	111.0(2)
C(31)−Sn(2)−C(41)	108.3(2)	O(3)−Sn(3)−C(61)	97.0(2)
O(3)−Sn(3)−C(51)	110.7(2)	C(51)−Sn(3)−C(61)	114.6(4)
O(3)−Sn(3)−C(71)	109.8(2)	C(61)−Sn(3)−C(71)	110.5(4)
C(51)−Sn(3)−C(71)	113.2(4)	Te(1)−O(1)−Sn(2)	122.17(16)
Te(1)−O(2)−Sn(1)	121.55(14)	Sn(1)−C(1)−Sn(2)	112.9(2)
Te(1)−O(3)−Sn(3)	127.55(15)		
C(1)−Sn(1)−O(2)−Te(1)	48.9(3)	C(1)−Sn(2)−O(1)−Te(1)	−40.1(3)
O(2)−Sn(1)−C(1)−Sn(2)	−1.8(3)	O(2)−Te(1)−O(1)−Sn(2)	71.65(19)
O(1)−Sn(2)−C(1)−Sn(1)	−1.5(3)	O(1)−Te(1)−O(2)−Sn(1)	−77.58(19)

C(1)−Sn(1)−O(2) ring exhibits an envelope conformation with Te(1) lying 1.133(3) Å above the plane of fitted atoms O(1), O(2), Sn(1), C(1), Sn(2). The maximal deviation of fitted atoms from the plane amounts to 0.027 Å. The Sn(1)−C(1)−Sn(2) angle of 112.9(2)° is close to the corresponding angle in the six-membered stannasiloxane ring *cyclo*-*t*$Bu_2Si(OSnPh_2)_2CH_2$ [113.9(4)°],[16] and related open-chain derivatives $H_2C(SnPh_2Cl)_2$ [113.4(10)°,[17] and $H_2C(SnPh_2Cl)_2$·HMPA (114.0°)].[18] A remarkable feature in the structure of compound **2** is the intramolecular Sn(1)−O(3a)/Sn(2)−O(3a) distances of 2.944(3)/3.003(3) Å, which are shorter than the sum of the van der Waals radii[19] of tin (2.20 Å) and oxygen (1.50 Å). As a consequence, the coordination geometry at Sn(1) and Sn(2) can best be described as a monocapped tetrahedron {[4+1]-coordination, geometrical goodness[20−22] Δ(Σ): 14.93° for Sn(1) and 14.62° for Sn(2)} with Sn(1) deviating 0.610(3) Å in the direction of C(21) from the plane defined by C(1), C(11), O(2), and Sn(2) deviating 0.609(3) Å in the direction of C(41) from the plane defined by C(1), C(31), O(1). Most interestingly, and caused by the ring constraint, the oxygen atom O(3a) approaches the Sn(1) and Sn(2) atoms through the faces defined by C(1), C(11), O(2) and C(1), C(31), O(1), and forces the phenyl rings containing C(21) and C(41), respectively, into the axial positions of the trigonal bipyramid, whereas O(2), C(1), C(11) and O(1), C(1), C(31) become equatorial. This is a rather unusual situation for pentacoordinated triorganotin compounds as the axial positions are usually occupied by the more electronegative substituents and the equatorial positions by the three organic substituents. Another consequence of the intramolecular Sn(1)−O(3a)/Sn(2)−O(3a) distances mentioned above is the increase of the O(3a)−Sn(3a) bond length [2.015(3) Å] with respect to the Sn(1)−O(2)/Sn(2)−O(1) bond lengths of 1.986(3)/1.982(3) Å, and the decrease of the Te(1)−O(3a)−Sn(3a) angle [127.55(15)°] in comparison with related Te−O−Si angles in $[(Me_3SiO)_8Te_2O_2]$ (**II**) (135.1−141.9°).[4] The intramolecular Sn(3)−O(1)/Sn(3)−O(2) distances of 3.518(3)/3.398(3) Å are also shorter than the sum of the van der Waals radii of the corresponding atoms but apparently they are not short enough to significantly disturb the tetrahedral geometry of Sn(3) (mean angle 109.3°).

Experimental Section

General Remarks: All manipulations were performed under an inert atmosphere of argon using standard Schlenk and vacuum line tech-

niques. Solvents were distilled from the appropriate desiccants prior to use. Literature procedures were used to prepare Bu_3SnOMe[23] and $(Ph_2SnOH)_2CH_2$.[15] The $Te(OH)_6$ was purchased from Fluka. NMR spectra were recorded in $CDCl_3$ using a Jeol GX 270 and a Varian 300 Unity Plus spectrometer at 85.39 (^{125}Te), 111.86 (^{119}Sn), 75.44 (^{13}C) and 299.98 (^{1}H) MHz. Chemical shifts (δ) are given in ppm and are referenced to Me_2Te (^{125}Te), Me_4Sn (^{119}Sn) and Me_4Si (^{13}C, ^{1}H). The elemental analyses were performed on an instrument from Carlo Erba Strumentazione (Modell 1106). The IR spectra were recorded on a Bruker FTIR IFS 113v spectrometer. The molecular weight measurement was carried out using a Knaur osmometer.

Synthesis of *mer*-[$(Bu_3SnO)_3Te(OH)_3$] (1): A mixture of Bu_3SnOMe (7.71 g, 24.0 mmol) and $Te(OH)_6$ (1.84 g, 8.0 mmol) was heated in toluene (10 mL) at 85 °C until the $Te(OH)_6$ was completely dissolved (approx. 4 h) and then for 2 h at 110 °C. Volatile components were removed in vacuum affording a colorless waxy residue of **1** (8.78 g, 8.0 mmol, 100%, m.p. 42 °C). ^{1}H NMR: δ = 0.92 (9 H), 1.24 (6 H), 1.34 (6 H), 1.61 (6 H). ^{13}C NMR δ = 13.8, 13.7 (β-*C*), 17.5 [$^1J(^1C-^{119}Sn)$ = 369 Hz; α-*C*], 17.8 [$^1J(^1C-^{119}Sn)$ = 366 Hz; α-*C*], 27.4, 27.5 (γ-*C*), 28.3 (δ-*C*). ^{119}Sn NMR: δ = 81.9 [1Sn, $^2J(^{119}Sn-O-^{125}Te)$ = 434 Hz], 93.6 [2 Sn, $^2J(^{119}Sn-O-^{125}Te)$ = 504 Hz]. ^{125}Te NMR: δ = 721.5 [$^2J(^{125}Te-O-^{119}Sn)$ = 501, $^2J(^{125}Te-O-^{119}Sn)$ = 432 Hz]. IR (KBr): $\tilde{\nu}$ = 3372 br. (ν_{OH}), 2956 vs, 2924 vs, 2871 vs, 2853.7 vs, 1463 s, 1377 m, 1074 m, 876 m, 734 s, 668 m, 621 s, 474 m cm^{-1}. $C_{36}H_{84}O_6Sn_3Te$ (1096.89): calcd. C 39.42, H 7.72; found C 40.25, H 8.20. MW ($CHCl_3$, 20 mg mL^{-1}): 1071.

Synthesis of *trans*-{$(Bu_3SnO)_2[CH_2(Ph_2SnO)_2]_2Te$]} (2): A mixture of **1** (1.10 g, 1 mmol) and $(Ph_2SnOH)_2CH_2$ (1.19 g, 2 mmol) in toluene (10 mL) was heated for 4 h at 80 °C. The precipitate formed (Bu_3SnOH) was separated by filtration and the volatile components were removed under vacuum. The solid residue was recrystallised from hexane/dichloromethane (3:1) affording **2** (1.57 g, 0.82 mmol, 82%, m.p. 173 °C). ^{1}H NMR: δ = 0.90 (6 H), 1.22 [4 H, $^2J(^1H-^{119}Sn)$ = 120 Hz; SnCH_2Sn], 1.23 (4 H), 1.34 (4 H), 1.62 (4 H), 7.0−8.1 (40 H, *Ph*). ^{13}C NMR: δ = −2.1 [$^1J(^{13}C-^{119}Sn)$ = 378 Hz; SnCH_2Sn], 13.6 (β-*C*), 17.4 [$^1J(^{13}C-^{119}Sn)$ = 360 Hz; α-*C*], 27.1 (γ-*C*), 28.0 (δ-*C*), 128.1 (*p*-*C*), 145.3 [$^1J(^{13}C-^{119}Sn)$ = 526 Hz; *i*-*C*], 128.3 (*p*-*C*), 128.8 (*m*-*C*), 129.0 (*m*-*C*), 136.0 (*o*-*C*) 136.1 (*o*-*C*), 143.5 [$^1J(^{13}C-^{119}Sn)$ = 684 Hz; *i*-*C*]. ^{119}Sn NMR: δ = −40.2 [4 Sn, $^2J(^{119}Sn-O-^{117}Sn)$ = 632, $^2J(^{119}Sn-O-^{125}Te)$ = 353 Hz; *Sn*CH_2Sn], 89.6 [2 Sn, $^3J(^{119}Sn-O-^{125}Te)$ = 393 Hz; *Sn*Bu_3]. ^{125}Te NMR: δ = 726.1 [$^2J(^{125}Te-O-^{119}Sn)$ = 397, $^2J(^{125}Te-O-^{119}Sn)$ = 355 Hz]. IR (KBr): $\tilde{\nu}$ = 3062 s, 3009 s, 2953 vs, 2918 vs, 2868 s, 1480 s, 1462 s, 1428 vs, 1374 m, 1345 m, 1297 m, 1188 m, 1076 vs, 1022 m, 997 m, 958 m, 867 m, 711 vs, 608 vs, 527 vs, 466 vs, 447 s, 343 s, 319s cm^{-1}. $C_{74}H_{98}O_6Sn_6Te$ (1923.6): calcd. C 46.21, H 5.14; found C 45.99, H 5.20.

X-ray Crystallographic Study. Crystal Data and Structure Solution of 2: $C_{74}H_{98}O_6Sn_6Te$, M_r = 1923.26, *monoclinic*, *C*2/*c*, *a* = 20.571(1), *b* = 14.150(1), *c* = 27.974(1) Å, β = 101.239(1)°, *V* = 7986.5(7) Å^3, *Z* = 4, $D_{calcd.}$ = 1.600 g·cm^{-3}, λ(Mo-K_α) = 0.71069 Å, *F*(000) = 3768, μ = 2.250 mm^{-1}, *T* = 291(1) K. The data collection covered almost the whole sphere of reciprocal space with 360 frames via ω-rotation (Δ/ω = 1°) at 2 × 10 s per frame on a Nonius KappaCCD diffractometer with a completeness of 93.2% in the θ range of 4.07 to 24.99°. The structure was solved by direct methods SHELXS-97[24] and refined by full-matrix least-squares calculations using SHELXL-97.[25] All non-H atoms were refined anisotropically. The H-atoms were placed in geometrically calculated positions using a riding model (including free rotation around C−C). The aromatic H atoms were refined with a common isotropic temperature factor [C−H_{aryl} 0.93 Å, U_{iso} 0.143(6) Å^2], while for the aliphatic H atoms the atomic displacement parameters were fixed to 1.5 times those of the C atoms. R_1 = 0.0317 for 3961 reflections with $I > 2\sigma(I)$ and wR_2 = 0.0698 for 6551 independent reflections with 395 refined parameters. The max./min. residual electron densities were 0.362 and −0.369 e Å^{-3}.

CCDC-173037 contains the supplementary crystallographic data for this paper (excluding structure factors). These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; Fax: (internat.) +44-1223/336-033; E-mail: deposit@ccdc.cam.ac.uk].

Acknowledgments

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