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Redox Properties of Isoxazolo[60]fullerenes with Regard to Substituents and Molecular Structure*

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A series of isoxazolo[60]fullerene derivatives 1(a–k), 5 and 6(l-n) have been synthesized by [2+3]cycloadditions of the corresponding nitriloxides to C_{60}. The phenyl-, 1,4- and 1,2-biphenyl substituents are substituted with electron donor groups in different distances and orientations in order to determine their influence on the redox properties of these fullerene derivatives. The redox-behavior was measured by cyclic voltammetry. In compounds 1b–g, 5l-n and 6l-n no significant shift relative to the reference compound 1a could be found. The insertion of an *o*-phenylene spacer between the isoxazoline ring and the phenyl ring substituted by donor groups in derivatives 1h–k causes a shift of the first reduction potential by 30 mV (1h, 1k) and 60 mV (1i) towards more negative values compared to the reference 1a. Because of the *o*-phenylene spacer the plane of the donor-phenyl ring is forced into a close and parallel orientation to surface of the fullerene suitable for through-space interaction. This geometry was proven by X-ray structure determination of 1k.

Keywords: Fullerenes; Cycloadditions; Donor-acceptor systems; Cyclic voltammetry

INTRODUCTION

Investigation of the unique electrochemical properties of fullerenes has been a challenge for organic and physical chemists since the very beginning of fullerene chemistry. Fullerenes are well known to be electron acceptors [1]. The ability of [60]fullerene to accept up to six electrons reversibly has been confirmed chemically [2] and *via* cyclovoltammetric experiments [3]. Due to the interesting electron acceptor ability fullerenes may be useful for the construction of supramolecular assemblies and new materials. For this purpose fullerene derivatives with electronegative atoms [4] or strong electron withdrawing groups [5] directly connected to the fullerene core in order to enhance the reduction potentials were compared to unsubstituted [60]fullerene. Electron donating substituents connected to the fullerene core *via*

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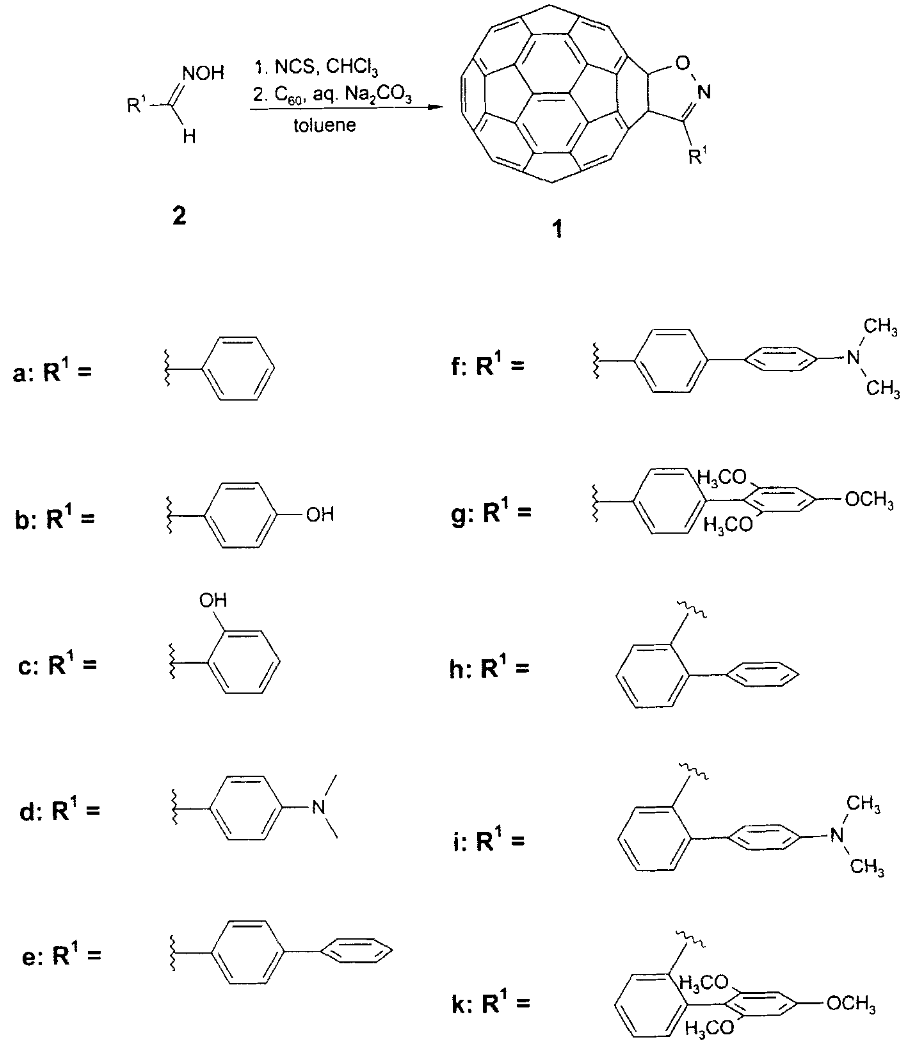
* Dedicated to Professor Fumio Toda on the occasion of his 67th birthday.

† Corresponding author.

pyrolidine rings have also been published [6]. The influence of the electron donating substituents on the electronic behavior of the fullerene system was much smaller than expected. The reasons are due to the cycloaddition reaction which leads inevitably to a fusion position with at least three single bonds connected to each other. The single bonds act as a electronic isolator. For this reason periconjugative effects have been used to improve the electronic interaction of donor and acceptor substituents with the fullerene core [7]. Promising results have recently been published by our group [8] where a new path of electronic interaction *via* a through-space mechanism has been discussed. In this communication we investigated the influence of electron donating substituents in new isoxazolo[60]fullerenes by cyclic voltammetry. Distance, orientation and electron donor properties of the substituents have been varied systematically to get an insight into the influence of electron donors on the redox properties of the cycloadducts **1**, **5** and **6** (Scheme 1 and 3). Furthermore, an X-ray crystal structure of



SCHEME 1

compound **1k** has been determined to examine the orientation of the substituent relative to the fullerene core.

RESULTS AND DISCUSSION

Several [2 + 3] cycloaddition reaction of nitrile oxides [9, 10] or trimethylsilylnitronates [11] to [60] and [70]fullerenes yielding isoxazolofullerenes have been synthesized. **1a** was described in an earlier publication [9a] and is used as a reference compound due to the unsubstituted phenyl ring. The new isoxazolofullerenes **1b – k** were formed *via* a convenient synthetic method (Scheme 1).

The oximes **2** were chlorinated with *N*-chlorosuccinimide (NCS) and subsequently *in situ* dehydrohalogenated forming the reactive nitrile oxides. The 1,3-dipole compounds react in [2 + 3] cycloaddition with [60]fullerene.

Oximes **2b – e** and **2h** have been described in the literature and the new oximes **2f**, **2g**, **2i** and **2k** were obtained from the corresponding aldehydes **3a**, **3b** and **4a**, **4b** respectively (Scheme 2).

The aldehydes were synthesized *via* the Suzuki cross coupling reaction of aromatic bromides with 4-formyl-phenyl-boronic acid and 2-formyl-phenyl-boronic acid respectively.

The [60]fullerene-ester derivatives **5l-n** and **6l-n** were prepared by esterification from the corresponding phenol derivatives **1b** and **1c** (Scheme 3).

The esterification was carried out by a method invented from Steglich *et al.* [12] using dicyclohexylcarbodiimide (DCC) and 4-dimethylaminopyridine (DMAP).

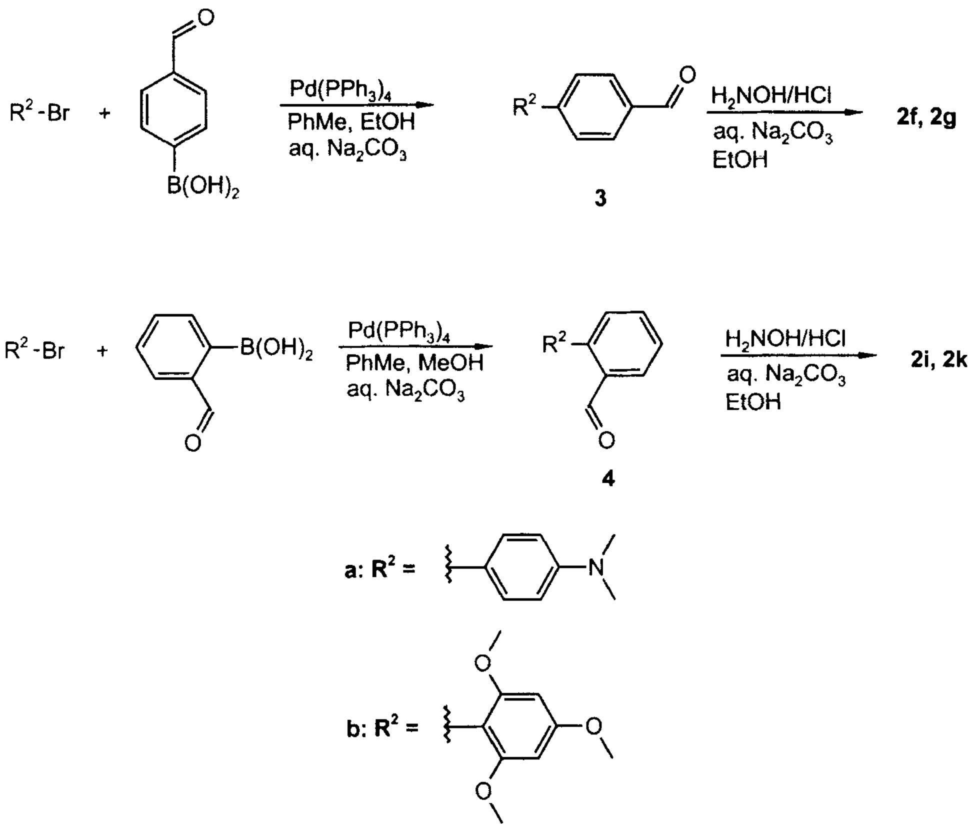
All new compounds have been characterized by IR, UV-VIS, ^{1}H NMR, ^{13}C NMR and high resolution mass spectrometry or elemental analysis respectively. For all cycloadducts **1**, **5** and **6** the expected reduced number of carbon signals in the fullerene region proves mirror symmetry. This leads to the conclusion that the addition of nitrile oxide took place at the [6,6]bond in the [60]fullerene.

Investigations into the redox-behavior of the isoxazolofullerenes **1a – k**, **5l-n** and **6l-n** were carried out by cyclic voltammetry. As discussed earlier [8], irreversible processes will be observed in isoxazolofullerene derivatives if the reduction range is extended to more negative potentials than −1.75 V due to a N – O bond cleavage of the isoxazoline ring. For this reason we use only the first two reduction couples ($E_{1,\mathrm{red.}}$ and $E_{2,\mathrm{red.}}$) and if possible the first oxidation couple ($E_{1,\mathrm{ox.}}$) to discuss the influence of the various donator substituents on the fullerene system (Tab. I).

Investigations by Suzuki *et al.* [13] into the influence of directly attached groups on [60]fullerenes led to the conclusion that saturation of a double bond shifts the reduction potential to more negative values compared with the pristine C_{60}.

In contrast, addition of a nitrile oxide leading to a fused isoxazoline ring at a [6,6] double bond of the [60]fullerene shifts the potentials towards positive values (Tab. I). The redox value for the first reduction of the reference compound **1a** is shifted about 40 mV to higher potentials compared to the naked C_{60} (Diagram 1). Obviously the isoxazoline heterocycle has a strong electron accepting effect on the redox behavior of the fullerene core.

Direct linkage of electron donating substituents to the isoxazoline ring in compounds **1b – d** does not shift the redox potentials significantly. Even the electron-rich dimethyl-amino-phenyl substituent in compound **1d** does not show any effect on the fullerene core in comparison to the reference substance **1a**. Interestingly, in **1d** an additional redox couple could be observed at 0.56 V. Additional reversible oxidation couples of this kind are also observed in compounds **1f** and **1i**. The registered oxidation waves are due to the electrochemical oxidation of the dimethylanilino substituent leading to the reversible formation of a radical cation (Scheme 4).

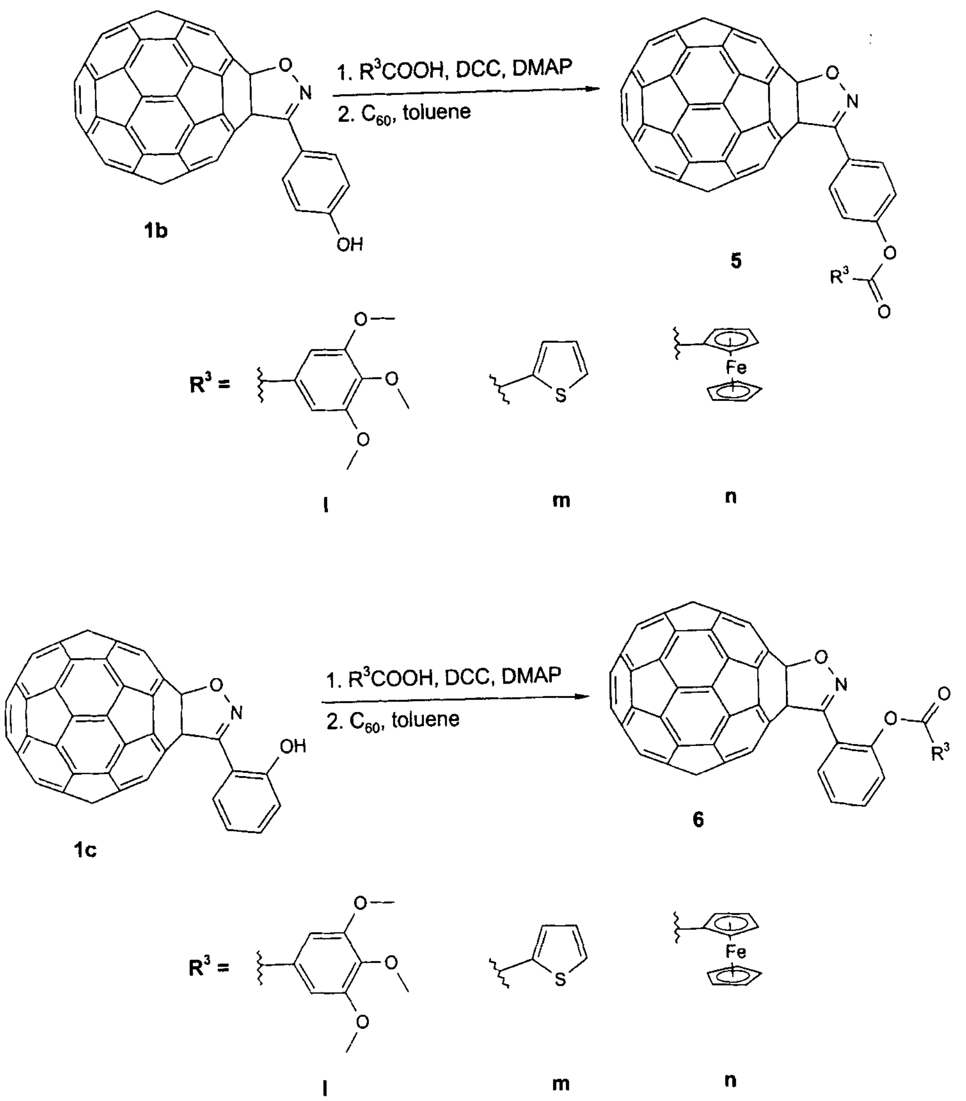


SCHEME 2

Insertion of a *p*-phenylene spacer between the isoxazoline ring and the donor groups in derivatives **1e**–**g** to increase the distance does not change the redox system significantly compared to reference **1a**. Obviously the distance between donor and fullerene core is too large. Observations of the redox behavior of compounds **1b**–**g** with no significant change in the reduction potentials of the fullerene derivatives compared to reference **1a** led to the conclusion that the fusion position act as an electronical isolator. The [2 + 3] cycloaddition of a nitrile oxide at a [6,6] bond in the [60]fullerene unavoidably leads to at least three connected single bonds (see X-ray crystal structure of **1k**). Our search to find a suitable pathway of electronic interaction between donor and fullerene acceptor led us to the synthesis of the derivatives **6l**–**n** and **5l**–**n** where donor groups are connected *via* flexible ester links to *o*-phenylene and *p*-phenylene spacers respectively. Flexibility of the donor groups should allow them to come close to the fullerene surface to evoke a through-space interaction. The electrochemical measurements of derivatives **5l**–**n** and **6l**–**n** indicated no significant change in the redox behavior compared to reference **1a**. The ferrocenyl-ester derivatives **5n** and **6n** show additional oxidation waves due to the formation of a ferrocene/ferrocenium couple. **5n** and **6n** are more difficult to oxidize than the standard unsubstituted ferrocene/ferrocenium couple due to the electron withdrawing ester substituent.

Finally, insertion of an *o*-phenylene spacer between the isoxazoline ring and the donor group in derivatives **1h**–**k** led to the desired success. The *o*-phenylene spacer forces a geometry (see X-ray determination of **1k**) in

Scheme 3



SCHEME 3

which the donor substituent comes close to the fullerene surface. The redox potential for the first reduction potential ($E_{1,\text{red.}}$) for **1h** and **1k** are shifted by 30 mV, for **1i** even by 60 mV towards more negative values compared to reference **1a** (Diagram 1).

For the second reduction potential the deviation compared to reference **1a** is even more significant. A difference of 80 mV for **1i** could be detected. Comparison of **1f** with **1i** demonstrates the powerful electronic interaction of the substituent in **1i** with the fullerene core. As discussed before, no significant change in redox potentials for **1f** could be detected. Figure 1 shows the cyclic voltammograms of **1f** and **1i** including the first oxidation wave ($E_{1,\text{ox.}}$).

The electronic interaction of the dimethylamino-phenyl substituent in **1i** leads to a

TABLE I $E_{1/2}$ values (V *vs* Fc/Fc$^+$) of the redox couples of C_{60} and compounds **1a–d**, detected by CV (scan rate 100 mV/s) in 1,2-dichlorobenzene (0.1 mol/L $(n\text{-Bu})_4NPF_6$) at room temperature. Errors are estimated at ± 10 mV. $E_{1/2} = (E^{an}_{peak} + E^{cat}_{peak})/2$

	C_{60}	**1a**	**1b**	**1c**	**1d**	**1e**	**1f**	**1g**	**1h**	**1i**	**1k**	**5l**	**5m**	**5n**	**6l**	**6m**	**6n**
$E_{1,red.}$ [V]	−1.12	−1,08	−1.07	−1.07	−1,08	−1,08	−1,08	−1,08	−1,11	−1.14	−1.11	−1.07	−1,07	−1.08	−1.08	−1,07	−1,08
$E_{2,red}$ [V]	−1,47	−1,43	−1.43	−1.43	−1.42	−1,44	−1,43	−1,45	−1,48	−1.51	−1.47	−1,44	−1,43	−1.44	−1.46	−1.46	−1,45
$E_{1,ox.}$ [V]					0.56		0.39			0.47				0.29			0.28

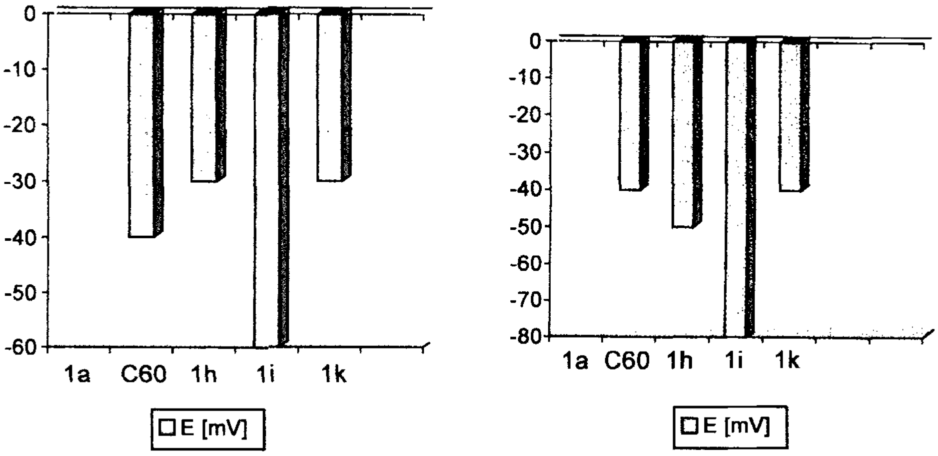
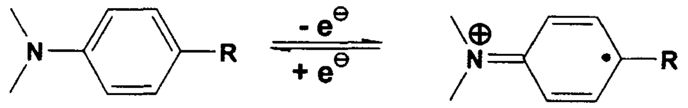


DIAGRAM 1 Deviation of the redox potentials from the derivatives with a *o*-phenylene spacer. Left deviation from the first reduction potential and right from the second reduction potential.



SCHEME 4

decrease of the first reduction potential of 60 mV but on the other hand the redox potential for the first oxidation is increased by 80 mV compared to **1f**. This stresses the electron accepting effect of the fullerene core on the donor substituent in **1i**. Because of this interaction the redox potential range between the first reduction potential and the first oxidation potential is split by 140 mV in **1i** compared to **1f**.

An X-ray crystal structure determination of compound **1k** was carried out showing the arrangement of the trimethoxy-phenyl donor to the [60]fullerene core (Fig. 2).

The crystal was grown in a mixture of 1,2-dichlorobenzene and CS_2 forming a 1 : 1 CS_2-clathrate. The CS_2-molecules fill up the holes within the monoclinic crystal (space group $P2_1/n$) where four molecules are in the unit cell (Fig. 3).

The directly connected phenyl group of the *o*-phenylene spacer in **1k** is pushed away from the fullerene core by repulsive nonbonding contacts (Fig. 4).

The angle (C2–C61–C62) with 124.9(2)° is significantly wider than the complementary angle (N1–C61–C62) with 120.2(2)°. The reasons for the widened angle are the short distances below the van der Waals distance between the biphenyl system and fullerene core. The C ··· C-distances between C62 to C3 and C12

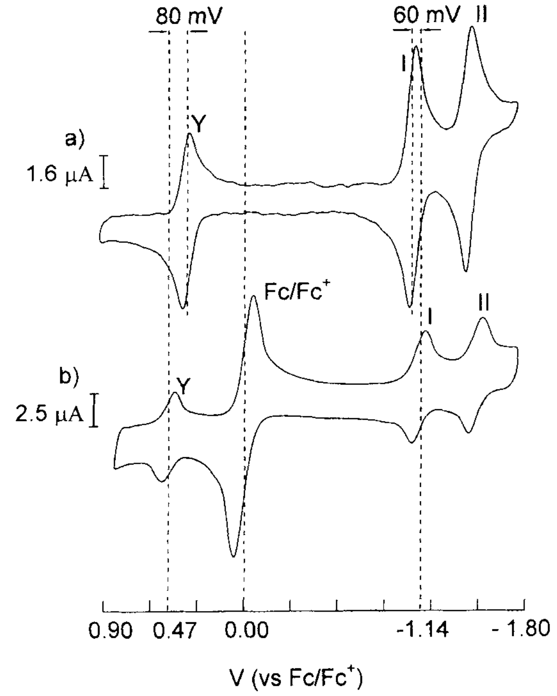


FIGURE 1 Cyclovoltammogram with $v = 100\,\mathrm{mV/s}$: (a) **1f** without ferrocene; (b) **1i** with Fc/Fc^+. I and II are the first and second reduction potentials respectively, *Y* is the first redox potential for the oxidation.

are 3.16 Å and 3.35 Å respectively. There are also attractive interactions between the electron-rich trimethoxy-phenyl-ring and the electrophilic fullerene core (Fig. 5).

The donor-acceptor-contacts are C73 ··· C13 (3.21 Å), C73 ··· C12 (3.28 Å), C68 ··· C12 (3.05 Å), C69 ··· C12 (3.32 Å). The bulky trimethoxy-phenyl substituent in the ortho position of the phenylene spacer also leads to a widening of the angle (C61 - C62 - C63: 123.5(2)°) in comparison with the complementary angle (C61 - C62 - C67: 116.5(2)°). The angle of torsion between the plane of the isoxazoline ring and the directly connected phenyl-ring (C63 - C62 - C61 - C2) in **1k** is 73.3(2)°.

Especially interesting is the short contact below the van der Waals distance between C61 of the imino group within the isoxazoline ring and O2 of the methoxy group approaching the heterocycle from above (Fig. 6). This contact can be considered as a nucleophilic attack of the free electron pair of O2 to the sp^2-hybridized oxime-carbon atom C61. The sp^2-system reacts

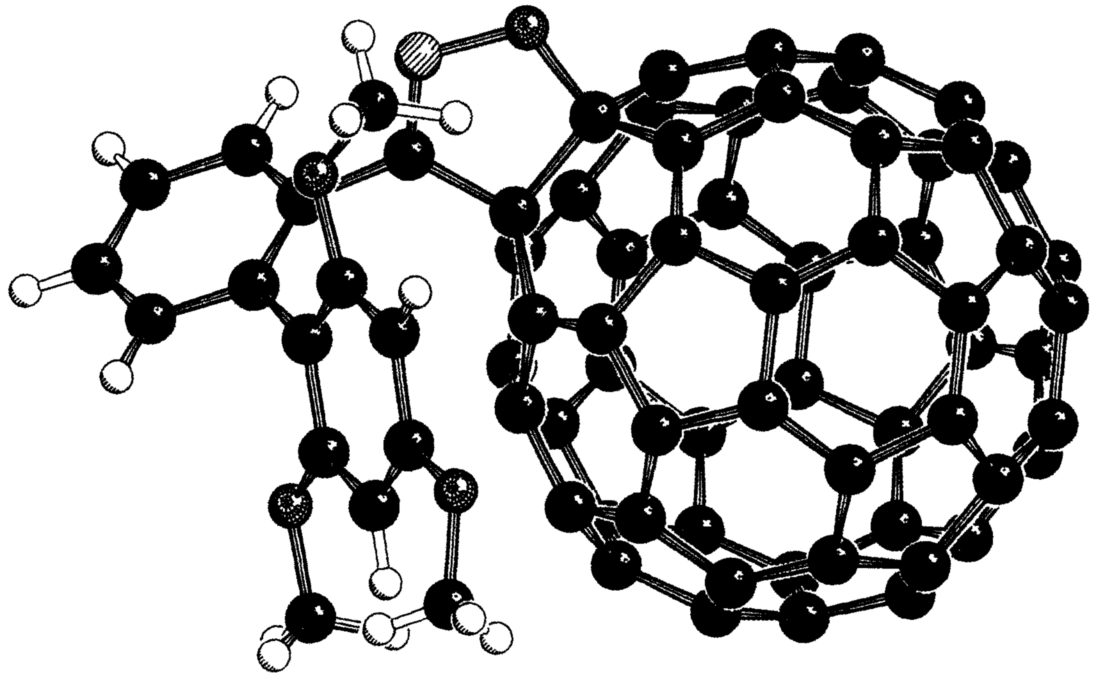


FIGURE 2 Crystal-structure of **1k** (SCHAKAL-plot [15]).

in the way that it deviates from the expected planar arrangement of C2, C62 and N1. C61 is located 0.055(2) Å above this plane towards the direction of the O2. The angle of pyramidalization of C61 is 7.0(3)°.

The planes of the phenyl rings are twisted by 57.9(3)°. Repulsive contacts generate this well-known incline arrangement of the rings for biphenyl systems. The short contacts of C62···C69 (3.21 Å), C64···C73 (3.12 Å), O2···C62 (3.03) and O4···C64 (2.92 Å) are the reason for the twisted arrangement.

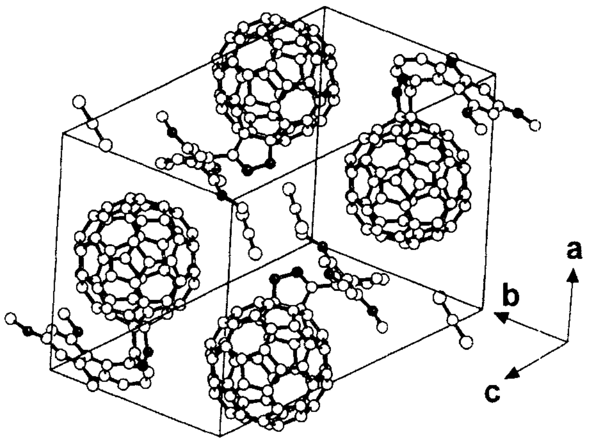


FIGURE 3 Unit cell of **1k**. The hydrogen atoms are omitted.

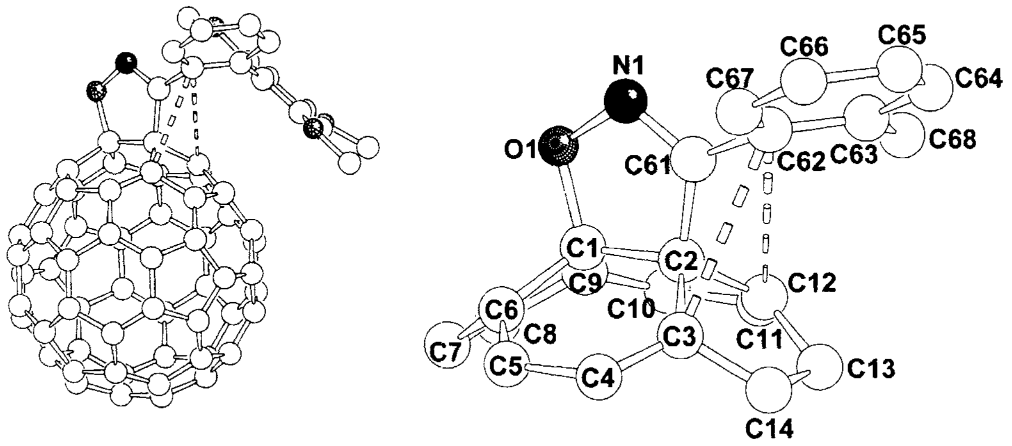


FIGURE 4 Contacts (- - -) below the van der Waals distance in **1k**.

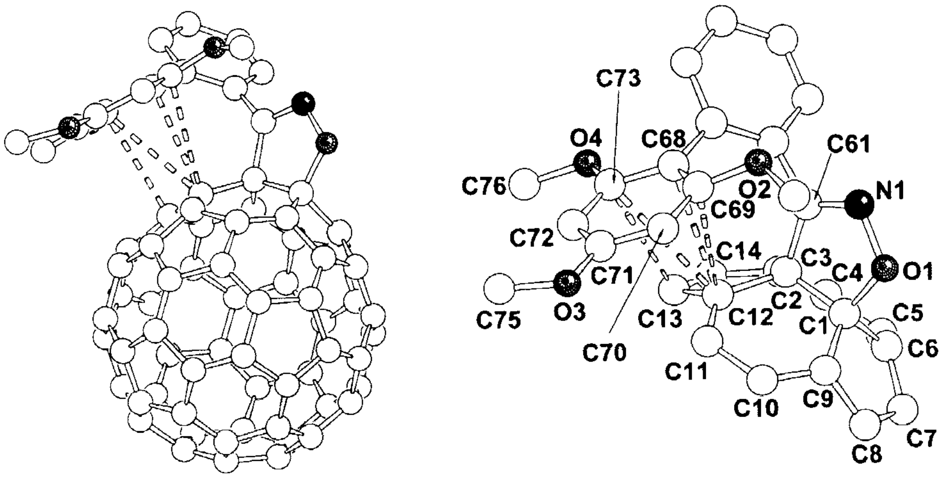


FIGURE 5 Donor-acceptor-contacts (- - -) below the van der Waals distance in **1k**.

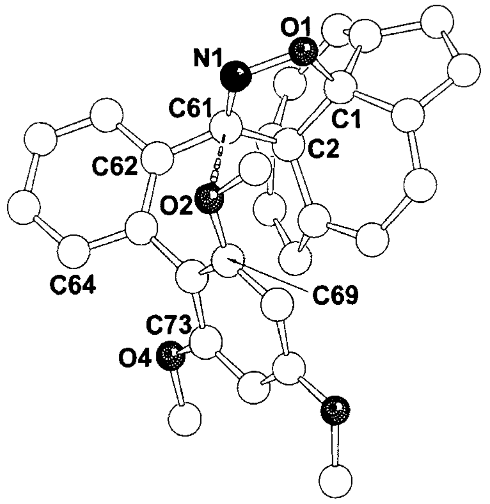


FIGURE 6 Short contact O2···C61 of the methoxy group (O2) to the oxime group (C61–N1–O1) in **1k**.

EXPERIMENTAL

Instrumentation

^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra were recorded on Bruker AC 300 and DRX 300 spectrometers. Chemical shifts are given in parts per million (δ) relative to tetramethylsilane. UV-VIS spectra were taken on a Hewlett Packard HP 8452 A Diode Array spectrophotometer. FT-IR spectra were recorded on a Bruker IFS 66 spectrophotometer and mass spectra were obtained on a VG-ZAB-2F (electron impact), on a JOEL JMS-700 (FAB) and on a Bruker Biflex-(MALDI-TOF; negative mode, matrix: 9-nitroanthracene). Melting points were determined with a Büchi apparatus and are uncorrected. Cyclic voltammograms were taken on a HEKA potentiostat/galvanostat, model 285/IEC using the program HEKA.POT (Vers. 3.01; HEKA Electronic).

Materials

[60]Fullerene was extracted from fullerene containing carbon soot using a literature procedure [14]. Tetra-*n*-butyl-ammonium hexafluoro-phosphate was purchased from Fluka. 1,2-Dichlorobenzene (spectrophotometric grade, Aldrich) was treated with neutral alumina (dried under vacuum at 250°C) before use. All other reagents were used as purchased from Fluka.

General Procedure for the Synthesis of the Isoxazolo-fullerenes 1b–k

277.5 μmol of the aromatic oxime was solved in 40 ml of chloroform and cooled with an ice bath to 5°C. To this solution 74.2 mg (555.0 μmol; 2 eq.) of *N*-chlorosuccinimide (NCS) and 10 μl of pyridine was added. The reaction mixture was stirred for 3 h at 5°C and afterwards given to a suspension of 200 mg (277.5 μmol) of C_{60} solved in 200 ml of toluene and 23 mg of Na_2CO_3 solved in 5 ml of water. The mixture was stirred for one day and subsequently washed five times with water. The organic solvent was evaporated *in vacuo* and the remaining residue was purified by chromatography (eluant: CS_2).

General Procedure for the Synthesis of the Oximes 2f, 2g, 2i and 2k

1.9 mmol of aromatic aldehyde was dissolved in 50 ml ethanol. The solution was heated under reflux and 1.3 g (19 mmol, 10 eq.) hydroxylammoniumchloride, dissolved in 5 ml water and neutralized with soda to pH = 7, was added. After heating for one hour under reflux, cold water was given to the hot solution until the product started precipitating. The precipitate was washed with water and dried in air.

General Procedure for the Synthesis of the Aldehydes 3a, 3b, 4a and 4b

A solution of 2.04 mmol of aromatic bromide 311 mg (2.04 mmol) of 4-formylphenyl-boronic acid (leading to **3a** and **3b**) or 2-formylphenyl-boronic acid (leading to **4a** and **4b**), 70 mg (60.6 mmol) of tetrakis-(triphenylphosphin)-palladium, 10 ml of ethanol and 20 ml of toluene was stirred under an argon atmosphere. To this solution an aqueous solution of 10 ml of 2 M Na_2CO_3 was added. The suspension was stirred for 24 h at 80°C and afterwards the reaction mixture was washed three times with water. The organic solvent was removed *in vacuo* and the residue was purified by flash chromatography.

General Procedure for the Synthesis of the Ester-derivatives 5l-n and 6l-n

To a solution of 30 mg (35.1 µmol) of **1b** (leading to **5l–n**) or **1c** (leading to **6l–n**) in 100 ml abs. toluene 68.0 mg (351 µmol; 10 eq.) dicyclohexylcarbodiimide (DCC), 351 µmol; 10 eq. carbonic-acid and 1 mg dimethylaminopyridine (DMAP) was given. The mixture was stirred for 16 hours at room temperature and was finally purified *via* chromatography on silica gel.

2′,4′,6′-Trimethoxy-biphenyl-2-carbaldehyde 4b

Yield 717 mg (66%). Mp 114°C. TLC (SiO_2; $CHCl_3$): $R_f = 0.32$. ^{1}H NMR (300 MHz, $CDCl_3$) $\delta = 3.68$ (*s*, 6H, OCH_3.), 3.86 (*s*, 3H, OCH_3), 6.22 (*s*, 2H, arom.), 7.31 (*d*, $^3J_{(H,H)} = 7.0$ Hz, 1H, arom.), 7.38–7.62 (*m*, 2H, arom.) 7.97 (*d*, $^3J_{(H,H)} = 7.2$ Hz, 1H, arom.), 9.72 (*s*, 1H, CHO). ^{13}C-NMR (75 MHz, $CDCl_3$) $\delta = 55.20$ (OCH_3), 55.52 (OCH_3), 90.50 ($C_{arom.}$), 107.26 ($C_{arom.}$, *q*), 126.25 ($C_{arom.}$), 127.06 ($C_{arom.}$), 132.97 ($C_{arom.}$), 133.19($C_{arom.}$), 134.42($C_{arom.}$, *q*), 137.87 ($C_{arom.}$, *q*), 158.28 ($C_{arom.}$, *q*), 161.46 ($C_{arom.}$, *q*), 192.89 (CHO). FT-IR (KBr) cm^{-1} 3064 (*w*, $CH_{arom.}$), 3008 (*m*, $CH_{arom.}$), 2965 (*m*), 2933 (*m*), 2837 (*m*), 1694 (*s*, C=O), 1652 (*m*), 1607 (*s*), 1505 (*m*), 1467 (*s*), 1409 (*s*), 1334 (*s*), 1295 (*w*), 1252 (*s*), 1223 (*s*), 1201 (*s*), 1152 (*s*), 1131 (*s*), 1057 (*s*), 1035 (*s*), 1002 (*m*), 946 (*m*), 919 (*w*), 983 (*w*), 811 (*s*), 775 (*s*), 745 (*m*), 669 (*w*), 642 (*m*), 601 (*w*), 571 (*w*), 538 (*w*). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) 248 (4.28), 274 (3.69), 310 (3.42), 352 (3.10). MS: *m/z* 272 (M^+). HR-MS: *m/z*: 272.106 (M^+, calcd. 272.105). Anal. calcd for $C_{16}H_{16}O_4$ (272.30): C 70.59; H 5.88. Found: C 70.15; H 5.84.

2′,4′,6′-Trimethoxy-biphenyl-2-carbaldoxime 2k

Yield 530 mg (92.2%). TLC (SiO_2; ethyl acetate): $R_f = 0.54$. Mp 125°C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.67 (*s*, 6H, OCH_3.), 3.86 (*s*, 3H, OCH_3), 6.20 (*s*, 2H, arom.), 7.18–7.39 (*m*, 3H, arom.), 7.85 (*s*, 1H, HC=N), 7.88 (*d*, $^3J_{(H,H)} = 7.8$ Hz, 1H, arom.), 8.11 (*s*, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.20 (OCH_3), 55.59 (OCH_3), 90.61 ($C_{arom.}$), 108.98 ($C_{arom.}$, *q*), 124.72 ($C_{arom.}$), 127.02 ($C_{arom.}$), 129.09 ($C_{arom.}$), 131.11 ($C_{arom.}$, *q*), 131.91 ($C_{arom.}$), 134.49 ($C_{arom.}$, *q*), 149.85 (C=N), 158.18 ($C_{arom.}$, *q*), 161.01 ($C_{arom.}$, *q*). FT-IR (KBr) cm^{-1} 3413 (OH, *s*), 3058 (*w*, $CH_{arom.}$), 3000 (*w*, $CH_{arom.}$), 2962 (*w*.), 2939 (*w*), 2839 (*w*), 1602 (*s*, C=N), 1584 (*s*), 1506 (*w*), 1466 (*m*), 1455 (*m*), 1436 (*m*), 1414 (*m*), 1335 (*w*), 1293 (*w*), 1225 (*s*), 1204 (*s*), 1184 (*w*), 1157 (*s*), 1126 (*s*), 1064 (*w*), 1035 (*w*), 1003 (*w*), 947 (*m*), 869 (*w*), 814 (*w*), 771 (*w*), 756 (*w*), 638 (*w*), 573 (*w*), 520 (*w*). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) 248 (3.74), 396 (2.85). MS: *m/z* 287 (M^+), 270 (M^+ –OH), 256 (M^+ –NOH). HR-MS: *m/z*: 287.121 (M^+, calcd. 287.115). Anal. calcd. for $C_{16}H_{17}NO_4$ (287.31): C 66.90; H 5.92; N 4.88. Found: C 66.62; H 5.93; N 4.59.

3′-(2′,4′,6′-Trimethoxy-biphenyl-2-yl)-isoxazolo[4′,5′: 1,2][60]fullerene 1k

Yield 71 mg (25.3%). TLC (SiO_2; toluene): $R_f = 0.30$. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.68 (*s*, 6H, OCH_3.), 3.83 (*s*, 3H, OCH_3), 6.07 (*s*, 2H, arom.), 7.45–7.58 (*m*, 3H, arom.), 7.79 (*d*, $^3J_{(H,H)} = 7.6$ Hz, 1H, arom.). ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.20 (OCH_3), 55.52 (OCH_3), 78.89 ($1C_{fullerene}$, sp^3), 90.64 ($C_{arom.}$), 101.73 ($1C_{fullerene}$, sp^3), 127.26 ($C_{arom.}$), 129.40 ($C_{arom.}$, *q*), 129.85 ($C_{arom.}$), 131.14 ($C_{arom.}$), 133.46 ($C_{arom.}$), 134.02 ($C_{arom.}$, *q*), 134.69 ($C_{arom.}$, *q*), 135.07 (2C), 136.71 (2C), 139.62 (2C), 139.95 (2C), 141.13 (2C), 141.37 (2C), 141.50 (2C), 141.97 (2C), 142.22 (2C), 142.25

(2C), 142.28 (2C), 142.41 (2C), 142.65 (2C), 142.69 (2C), 142.83 (2C), 144.10 (2C), 144.17 (2C), 144.48 (2C), 144.49 (2C), 144.83 (2C), 144.98 (2C), 145.09 (2C), 145.34 (2C), 145.80 (2C), 145.86 (2C), 146.08 (2C), 146.11 (2C), 146.16 (2C), 147.17 (1C), 148.39 (1C), 155.43 (CN), 158.69 ($C_{arom.}$, *q*), 162.48 ($C_{arom.}$, *q*). FT-IR (KBr) cm^{-1} 3054 (*w*, $CH_{arom.}$), 2994 (*w*), 2952 (*w*), 2929 (*w*), 2832 (*w*), 1607 (*m*, C=N), 1582 (*m*), 1540 (*w*), 1506 (*w*), 1494 (*w*), 1477 (*w*), 1455 (*w*), 1432 (*m*, C_{60}), 1413 (*w*), 1334 (*w*), 1297 (*w*), 1261 (*w*), 1224 (*m*), 1205 (*m*), 1187 (*w*, C_{60}), 1157 (*m*), 1129 (*m*), 1114 (*w*), 1068 (*w*), 1032 (*w*), 1002 (*w*), 970 (*w*), 950 (*m*), 922 (*w*), 895 (*w*), 855 (*w*), 837 (*w*), 819 (*w*), 810 (*w*), 789 (*w*), 767 (*w*), 758 (*w*), 747 (*w*), 729 (*w*), 695 (*w*), 665 (*w*), 651 (*w*), 633 (*w*), 604 (*w*), 586 (*w*), 572 (*w*, C_{60}), 564 (*w*), 555 (*w*), 546 (*w*), 527 (*m*, C_{60}), 480 (*w*), 464 (*w*), 444 (*w*). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) 232 (4.92), 256 (5.04), 316 (4.49). MS (MALDI-TOF, negative mode): *m/z*:1006 (M^-), 720 (C_{60}^-). HR-MS: *m/z* 1005.103 (M^+, calcd. 1005.100).

4′-N,N-Dimethylamino-biphenyl-2-carbaldehyde 4a

Yield 743 mg (82.5%). TLC (SiO_2; $CHCl_3$): $R_f = 0.42$. ^1H NMR (300 MHz, $CDCl_3$) δ 3.01 (*s*, 6H, $N[CH_3]_2$.), 6.81 (*d*, $^3J_{(H,H)} = 8.8$ Hz, 2H, arom.), 7.27 (*d*, $^3J_{(H,H)} = 8.8$ Hz, 2H, arom.), 7.38–7.47 (*m*, 2H, arom.), 7.56–7.62 (*m*, 1H, arom.), 8.02 (*dd*, $^3J_{(H,H)} = 8.1$ Hz, $^4J_{(H,H)} = 1.1$ Hz, 1H, arom.), 10.03 (*s*, 1H, CHO). ^{13}C NMR (75 MHz, $CDCl_3$) δ 40.36 ($N(CH_3)_2$), 112.15 ($C_{arom.}$), 125.22 ($C_{arom.}$), 126.68 ($C_{arom.}$), 127.57 ($C_{arom.}$), 130.68 ($C_{arom.}$), 131.13 ($C_{arom.}$), 133.43 ($C_{arom.}$), 133.69 ($C_{arom.}$), 146.34 ($C_{arom.}$), 150.32 ($C_{arom.}$), 192.98 (CHO). FT-IR(KBr) cm^{-1} 2887 (*w*), 1688 (*m*, C=O), 1611 (*m*), 1526 (*m*), 1479 (*w*), 1446 (*w*), 1357 (*m*), 1256 (*w*), 1156 (*m*), 947 (*w*), 825 (*m*), 767 (*m*). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) 230 (4.08), 254 (4.29), 292 (4.17), 356 (3.63), 368 (3.65). MS: *m/z* 225 (M^+), 196 (M^+ −CHO). HR-MS: *m/z*: 225.115 (M^+, calcd. 225.115). Anal. calcd. for $C_{15}H_{15}NO$ (225.29): C 79.97; H 6.71; N, 6.21. Found: C 79.79; H 6.72; N, 6.18.

4′-N,N-Dimethylaminobiphenyl-2-carbaldoxime 2i

Yield 389 mg (81.0%). Mp 230°C. ^1H NMR (300 MHz, $CDCl_3$) δ 3.02 (*s*, 6H, $N[CH_3]_2$.), 6.82 (*d*, $^3J_{(H,H)} = 8.8$ Hz, 2H, arom.), 7.23 (*d*, $^3J_{(H,H)} = 8.8$ Hz, 2H, arom.), 7.33–7.43 (*m*, 3H, arom.), 7.73 (*s*, br., 1H, OH), 7.87 (*d*, $^3J_{(H,H)} = 6.6$ Hz, 1H, arom.), 8.20 (*s*, 1H, CHN). ^{13}C NMR (75 MHz, $CDCl_3$) δ 40.45 ($N(CH_3)_2$), 112.14 ($C_{arom.}$), 126.09 ($C_{arom.}$), 126.65 ($C_{arom.}$), 127.19 ($C_{arom.}$), 129.42 ($C_{arom.}$), 129.59 ($C_{arom.}$), 130.17 ($C_{arom.}$), 130.48 ($C_{arom.}$), 142.42 ($C_{arom.}$), 149.87 ($C_{arom.}$), 150.26 (CHN). FT-IR (KBr) cm^{-1} 3251 (*s*, OH), 3061 (*w*), 2889 (*w*), 2808 (*w*), 1692 (*w*), 1611 (*s*), 1527 (*m*), 1482 (*m*), 1443 (*m*), 1361 (*m*), 1309 (*w*), 1270 (*w*), 1230 (*w*), 1200 (*m*), 1172 (*w*), 1128 (*w*), 1103 (*w*), 1065 (*w*), 966 (*m*), 954 (*m*), 869 (*w*), 823 (*m*), 758 (*m*), 723 (*w*), 697 (*w*), 644 (*w*), 632 (*w*), 563 (*w*), 510 (*w*), 488 (*w*). MS: 240 (M^+), 223 (M^+ −OH), 222 ($M^+ - H_2O$). HR-MS: *m/z* 240.125 (M^+, calcd. 240.126). Anal. calcd. for $C_{15}H_{16}N_2O$ (240.30): C 74.98; H 6.71; N 11.65. Found: C 74.76; H 6.69; N 11.45.

3′-(4′-N,N-Dimethylamino-biphenyl-2-yl)-isoxazolo[4′,5′:1,2][60]fullerene 1i

Yield 65 mg (24.5%). TLC (SiO_2; toluene): $R_f = 0.37$. ^1H NMR (300 MHz, $CDCl_3$: CS_2[1 : 1]) $\delta = 3.04$ (*s*, 6H, 2 × CH_3.), 6.71 (*d*, $^3J_{(H,H)} = 8.9$ Hz, 2H, arom.), 7.38 (*d*, $^3J_{(H,H)} = 8.9$ Hz, 2H, arom.), 7.47–7.63 (*m*, 3H, arom.), 7.76 (*d*, $^3J_{(H,H)} = 7.5$ Hz, 1H, arom.). ^{13}C NMR (75 MHz, $CDCl_3$: CS_2[1 : 1]) $\delta = 40.56$ (2 × CH_3), 80.95 (lC, sp^3), 90.60 (1C, sp^3), 112.71 ($C_{arom.}$), 126.78 ($C_{arom.}$), 126.86 ($C_{arom.}$), 127.76 ($C_{arom.}$), 129.91 ($C_{arom.}$), 130.77 ($C_{arom.}$), 130.95 ($C_{arom.}$), 131.36 ($C_{arom.}$), 135.06 (2C), 137.18 (2C), 139.95 (2C), 139.99 (2C), 141.71 (2C), 141.86 (2C), 142.25 (2C), 142.29 (2C), 142.33 (2C), 142.46 ($C_{arom.}$), 142.57 (2C), 142.70 (2C), 142.72 (2C), 142.86 (2C), 143.71 (2C), 144.05 (2C), 144.17 (2C), 144.82 (2C), 144.86 (2C), 144.93 (2C), 144.96 (2C), 145.13 (2C), 145.19 (2C), 145.74 (2C), 145.76 (2C), 145.88 (2C), 146.07 (2C), 146.11 (2C), 146.22 (2C), 147.05 (1C), 147.73 (1C), 150.69 ($C_{arom.}$), 155.62 (C=N). FT-IR(KBr) cm^{-1} 2923 (*w*), 1608 (*s*, C=N), 1524 (*m*), 1479 (*m*), 1439 (*m*, C_{60}), 1358 (*m*), 1295 (*w*), 1263 (*w*), 1231 (*w*), 1192 (*w*, C_{60}), 1166 (*w*), 1104 (*w*), 1063 (*w*), 1032 (*w*), 970 (*w*), 950 (*w*), 902 (*m*), 855 (*m*), 812 (*m*), 761 (*m*), 730 (*s*), 649 (*w*), 605 (*w*), 571 (*w*, C_{60}), 556 (*w*), 526 (*m*, C_{60}), 478 (*w*). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) 230 (4.68), 256 (4.78), 314 (4.35), 362 (3.83), 374 (3.66). MS (MALDI-TOF, negative mode): *m/z* 959 [M], 720 [C_{60}]. HR-MS: *m/z* 958.112 [M^+, calcd. 958.111].

3′-(2-Biphenyl)-isoxazolo[4′,5′:1,2] [60]fullerene 1h

Yield 24 mg (9.4%). TLC (SiO_2, toluene): $R_f = 0.75$. ^1H NMR (300 MHz, $CDCl_3$) $\delta = 7.07$–7.81 (*m*, 9H, arom.). ^{13}C NMR (75 MHz, $CDCl_3$) $\delta = 81.35$ (1C, sp^3), 102.72 (1C, sp^3), 126.96 ($C_{arom.}$), 127.82 ($C_{arom.}$), 128.25 ($C_{arom.}$), 128.64 ($C_{arom.}$), 130.10 ($C_{arom.}$), 130.53 ($C_{arom.}$), 130.82 ($C_{arom.}$), 131.33 ($C_{arom.}$), 135.59 (2C), 136.77 (2C), 139.35 ($C_{arom.}$), 139.95 (2C), 140.10 (2C), 140.32 (2C), 141.64 (2C), 141.92 (2C), 142.17 (2C), 142.27 (2C), 142.29 (2C), 142.36 (2C), 142.67 (2C), 142.69 (2C), 142.78 ($C_{arom.}$), 143.64 (2C), 144.01 (2C), 144.15 (2C), 144.19 (2C), 144.73 (2C), 145.04 (2C), 145.09 (2C), 145.10 (2C), 145.14 (2C), 145.31 (2C), 145.77 (2C), 145.88 (2C), 146.12 (2C), 146.14 (4C), 147.16 (1C), 147.73 (1C), 155.59 (CNO). FT-IR(KBr) cm^{-1} 3054 (*w*, $CH_{arom.}$), 3020 (*w*), 1612 (*w*, C=N), 1561 (*w*), 1540 (*w*), 1476 (*m*), 1462 (*w*), 1448 (*m*), 1432 (*m*), 1298 (*m*,), 1243 (*w*), 1218 (*w*), 1181 (*w*, C_{60}), 1168 (*w*), 1158 (*w*), 1105 (*w*), 1064 (*w*), 1033 (*m*), 1008 (*w*), 970 (*w*), 903 (*m*), 855 (*m*), 795 (*m*), 773 (*m*), 761 (*m*), 740 (*s*), 731 (*s*), 699 (*s*), 668 (*m*), 652 (*m*), 612 (*w*), 603 (*w*), 586 (*w*), 574 (*m*, C_{60}), 564 (*m*), 552 (*m*), 540 (*m*), 526 (*s*, C_{60}), 480 (*m*), 471 (*w*), 448 (*w*).). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) 232 (4.98), 256 (5.05), 316 (4.58). MS (MALDI-TOF, negative-mode): *m/z* 915 (50) [M^-], 720 (100) [C_{60}^-]. HR-MS: *m/z* 915.072 [M^+, calcd. 915,068].

2′,4′,6′-Trimethoxy-biphenyl-4-carbaldehyde 3b

Yield 223 mg (41.0%). TLC (SiO_2; $CHCl_3$): $R_f = 0.24$. Mp: 82°C. ^1H NMR (300 MHz, $CDCl_3$) δ 3.74 (s, 6H, OCH_3), 3.89 (*s*, 3H, OCH_3), 6.25 (*s*, 2H, arom.), 7.53 (*d*, $^3J_{(H,H)} = 8.1$ Hz, 2H, arom.), 7.90 (*d*, $^3J_{(H,H)} = 8.1$ Hz, 2H, arom.), 10.04 (*s*, 1H, CHO). ^{13}C NMR (75 MHz, $CDC1_3$) δ 55.41 (OCH_3), 55.84 (OCH_3), 90.97 ($C_{arom.}$), 111.17 ($C_{arom.}$, *q*), 128.98 ($C_{arom.}$), 132.04 ($C_{arom.}$), 134.51 ($C_{arom.}$, *q*), 141.34 ($C_{arom.}$, *q*), 158.23 ($C_{arom.}$, *q*), 161.27 ($C_{arom.}$, *q*), 192.19 (CHO). FT-IR(KBr) cm^{-1} 3065 (*w*, $CH_{arom.}$), 3003 (*w*, $CH_{arom.}$), 2968 (*m*), 2943 (*m*), 2837 (*m*), 2735 (*w*), 1695 (*s*, C=O), 1586 (*s*), 1466 (*s*), 1414 (*s*), 1347 (*s*), 1309 (*m*), 1230 (*s*), 1209 (*s*), 1160 (*s*), 1128 (*s*), 1070 (*m*), 1028 (*s*), 1004 (*m*), 948 (*m*), 916 (*w*), 806 (*s*), 782 (*m*), 712 (*w*), 665 (*w*), 633 (*m*), 578 (*w*), 541 (*w*), 499 (*w*), 466 (*w*). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) 232 (4.13), 302 (4.13). MS : *m/z* 272 [M^+]. HR-MS: *m/z* 272.102 [M^+, calc. 272.105]. Anal. calcd for $C_{16}H_{16}O_4$ (272.30): C 70.59; H 5.88. Found: C 70.86; H 5.94.

2′,4′,6′-Trimethoxy-biphenyl-4-carbaldoxime 2g

Yield 428 mg (78.0%). Mp 198°C. ^1H NMR (300 MHz, $CDCl_3$) δ 3.74 (*s*, 6H, OCH_3), 3.89 (*s*, 3H, OCH_3), 6.25 (*s*, 2H, arom.), 7.39 (*d*, $^3J_{(H,H)} =$ 8.1 Hz, 2H, arom.), 7.61 (*d*, $^3J_{(H,H)} = 8.1$ Hz, 2H, arom.), 8.20 (*s*, 1H, HC=N), 8.38 (*s*, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.43 (OCH_3), 55.90 (OCH_3), 91.07 ($C_{arom.}$), 111.82 ($C_{arom.}$, *q*), 126.32 ($C_{arom.}$), 130.00 ($C_{arom.}$, *q*), 131.70 ($C_{arom.}$), 136.28 ($C_{arom.}$, *q*), 150.47 (C=N), 158.33 ($C_{arom.}$, *q*), 160.83 ($C_{arom.}$, *q*). FT-IR(KBr) cm^{-1} 3252 (OH, *s*, br), 2999 (*w*, $CH_{arom.}$), 2944 (*w*.), 2838 (*w*), 1757 (*m*), 1641 (*m*, C=N), 1608 (*s*), 1588 (*s*), 1493 (*m*), 1466 (*m*), 1416 (*m*), 1375 (*w*), 1331 (*w*), 1305 (*w*), 1225 (*s*), 1205 (*m*), 1182 (*w*), 1128 (*s*), 1067 (*w*), 1030 (*w*), 1005 (*w*), 972 (*s*), 870 (*m*), 836 (*m*), 787 (*w*), 723 (*w*), 548 (*w*), 477 (*w*). UV-V1S ($CHCl_3$) λ_{max} nm (lg ε) 230 (4.22), 302 (4.37). MS:

m/z 287 (M^+), 270 (M^+—OH). HR-MS: *m/z* 287.114 (M^+, calc. 287.115). Anal. calcd. for $C_{16}H_{17}NO_4$ (287.31): C 66.90; H 5.92; N 4.88. Found: C 67.09; H 6.06; N 4.80.

3′-(2′,4′,6′-Trimethoxy-biphenyl-4-yl)-isoxazolo[4′,5′: 1,2][60]fullerene 1g

Yield 38 mg (13.7%). TLC (SiO_2; Toluol): $R_f = 0.70$. ^1H NMR (300 MHz, $CDCl_3$) δ 3.74 (*s*, 6H, OCH_3), 3.87 (*s*, 3H, OCH_3), 6.19 (*s*, 2H, arom.), 7.47 (*d*, $^3J_{(H,H)} = 8.2$ Hz, 2H, arom.), 8.21 (*d*, $^3J_{(H,H)} = 8.7$ Hz, 2H, arom.). ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.24 (OCH_3), 55.70 (OCH_3), 91.03 ($C_{arom.}$), 92.79 (1C, sp^3), 101.73 (1C, sp^3), 111.38 ($C_{arom.}$), 126.78 ($C_{arom.}$), 127.94 ($C_{arom.}$), 132.14 ($C_{arom.}$), 136.78 (2C), 137.01 ($C_{arom.}$), 137.12 (2C), 140.34 (2C), 141.76 (2C), 142.17 (2C), 142.38 (2C), 142.44 (2C), 142.51 (2C), 142.53 (2C), 142.88 (2C), 142.90 (2C), 143.02 (2C), 144.16 (2C), 144.17 (2C), 144.46 (2C), 144.80 (2C), 144.89 (2C), 145.14 (2C), 145.19 (2C), 145.26 (2C), 145.52 (2C), 145.68 (2C), 145.98 (2C), 146.02 (2C), 146.16 (2C), 146.27 (2C), 146.32 (2C), 146.43 (2C), 147.30 (1C), 147.80 (1C), 152.15 (CN), 158.28 ($C_{arom.}$, *q*), 161.00 ($C_{arom.}$, *q*). FT-IR(KBr) cm^{-1} 1604 (*s*, C=N), 1487 (*w*), 1452 (*s*), 1433 (*m*, C_{60}), 1400 (*m*), 1331 (*m*), 1303 (*m*), 1261 (*w*), 1223 (*s*), 1203 (*s*), 1157 (*w*, C_{60}), 1130 (*s*), 1115 (*s*), 1068 (*m*), 1037 (*m*), 1002 (*w*), 981 (*w*), 950 (*w*), 906 (*w*), 865 (*m*), 845 (*m*), 818 (*s*), 770 (*w*), 747 (*w*), 725 (*w*), 696 (*w*), 602 (*w*), 586 (*w*), 572 (*w*, C_{60}), 567 (*w*), 554 (*w*), 527 (*s*, C_{60}), 475 (*w*). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) 256 (5.00), 316 (4.60), 360 (4.00). MS (MALDI-TOF, negative-mode): *m/z*: 1006 (M^-), 720 (C_{60}^-). HR-MS: *m/z* 1005.102 (M^+, calc. 1005.100).

4′-N,N-Dimethylamino-biphenyl-4-carbaldehyde 3a

Yield: 304 mg (67.5%). TLC (SiO_2; $CHCl_3$): $R_f = 0.28$. Mp 195°C. ^1H NMR (300 MHz, $CDCl_3$) δ 3.04 (*s*, 6H, $N[CH_3]_2$), 6.82 (*d*, $^3J_{(H,H)} = 9.1$ Hz, 2H, arom.), 7.59 (*d*, $^3J_{(H,H)} = 9.1$ Hz, 2H, arom.), 7.73 (*d*, $^3J_{(H,H)} = 8.2$ Hz, 2H, arom.), 7.90 (*d*, $^3J_{(H,H)} = 8.2$ Hz, 2H, arom.), 10.01 (*s*, 1H, CHO). ^{13}C NMR (75 MHz, $CDCl_3$) δ 40.32 ($N(CH_3)_2$), 112.55 ($C_{arom.}$), 126.22 ($C_{arom.}$), 126.95 ($C_{arom.}$, *q*), 128.03 ($C_{arom.}$), 130.32 ($C_{arom.}$), 134.02 ($C_{arom.}$, *q*), 147.21 ($C_{arom.}$, *q*), 150.71 ($C_{arom.}$, *q*), 191.82 (CHO). FT-IR(KBr) cm^{-1} 2813 (*w*), 1696 (*m*, C=O), 1597 (*s*), 1568 (*w*), 1541 (*w*), 1502 (*w*), 1445 (*w*), 1366 (*m*), 1310 (*w*), 1289 (*w*), 1230 (*w*), 1204 (*m*), 1169 (*m*), 1064 (*w*), 947 (*w*), 843 (*w*), 809 (*s*), 768 (*w*), 689 (*w*), 500 (*w*). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) 252 (4.20), 370 (4.37). MS *m/z* 225 (M^+). HR-MS *m/z*: 225.116 (M^+, calc. 225.115). Anal. calcd. for $C_{15}H_{15}NO$ (225.29): C 79.97; H 6.71; N, 6.21. Found: C 79.75; H 6.63; N 6.10.

4′-N,N-Dimethylamino-biphenyl-4-carbaldoxime 2f

Yield 211 mg (87.9%). Mp 228°C. ^1H NMR (300 MHz, $CDCl_3$) δ 2.99 (*s*, 6H, $N[CH_3]_2$), 6.79 (*d*, $^3J_{(H,H)} = 9.1$ Hz, 2H, arom.), 7.32 (*s*, br., 1H, OH), 7.51 (*d*, $^3J_{(H,H)} = 9.1$ Hz, 2H, arom.), 7.57 (*s*, 4H, arom.), 8.14 (*s*, 1H, CHN). ^{13}C NMR (75 MHz, $CDCl_3$) δ 40.28 ($N(CH_3)_2$), 112.50 ($C_{arom.}$), 125.73 ($C_{arom.}$), 126.11 ($C_{arom.}$), 127.20 ($C_{arom.}$), 127.42 ($C_{arom.}$), 129.37 ($C_{arom.}$), 131.21 ($C_{arom.}$), 142.57 ($C_{arom.}$), 150.20 (CHN). FT-IR(KBr) cm^{-1} 3285 (*s*, br., OH), 2979 (*w*), 2892 (*w*), 2800 (*w*),1598 (*s*), 1538 (*m*), 1504 (*s*), 1444 (*w*), 1356 (*m*), 1308 (*w*), 1287 (*w*), 1227 (*m*), 1204 (*m*), 1170 (*w*), 1125 (*w*), 1062 (*w*), 958 (*s*), 875 (*m*), 842 (*w*), 813 (*s*), 774 (*w*), 715 (*w*), 693 (*w*), 517 (*m*). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) 234 (4.08), 254 (4.14), 336 (4.41). MS *m/z* 240 (M^+), 222 (M^--H_2O). HR-MS *m/z* 240.126 (M^+, calc. 240.126). Anal. calcd. for $C_{15}H_{16}N_2O$ (240.30): C 74.98; H 6.71; N 11.65. Found: C 74.81, H 6.72; N 11.68.

3′-(4′-N,N-Dimethylamino-biphenyl-4-yl)-isoxazolo[4′,5′: 1,2][60]fullerene 1f

Yield 67 mg (25.3%). TLC (SiO_2; toluene): $R_f = 0.55$. ^1H NMR (300 MHz, $CDCl_3$) δ 3.03 (*s*, 6H, $N[CH_3]_2$), 6.82 (*d*, $^3J_{(H,H)} = 9.0$ Hz, 2H, arom.), 7.59 (*d*, $^3J_{(H,H)} = 9.0$ Hz, 2H, arom.), 7.75

(*d*, $^{3}J_{(H,H)} = 8.2$ Hz, 2H, arom.), 8.25 (*d*, $^{3}J_{(H,H)} = 8.2$ Hz, arom.). ^{13}C NMR (125 MHz, $CDCl_3$) δ 40.24 (2 × CH_3), 79.30 (1C, sp^3), 101.73 (1C, sp^3), 112.73 ($C_{arom.}$), 126.34 ($C_{arom.}$) 126.53 ($C_{arom.}$), 127.51 ($C_{arom.}$), 127.80 ($C_{arom.}$), 129.26 ($C_{arom.}$), 133.96 ($C_{arom.}$), 136.78 (2C), 137.07 (2C), 140.33 (2C), 140.37 (2C), 141.76 (2C), 142.14 (2C), 142.36 (2C), 142.40 (2C), 142.51 (2C), 142.52 (2C), 142.89 (2C), 143.03 (2C), 143.13 (2C), 143.48 (2C), 144.16 (2C), 144.46 (2C), 144.70 (2C), 144.87 (2C), 144.99 (2C), 145.19 (2C), 145.27 (2C), 145.48 (2C), 145.67 (2C), 145.98 (2C), 146.03 (4C), 146.28 (2C), 146.32 (2C), 147.30 (1C), 147.81 (1C), 150.31 ($C_{arom.}$), 153.39 (CN). FT-IR(KBr) cm^{-1} 1598 (*w*), 1533 (*w*), 1504 (*w*), 1428 (*w*, C_{60}), 1359 (*w*), 1303 (*w*), 1211 (*w*), 1062 (*w*), 978 (*w*), 946 (*w*), 903 (*m*), 865 (*m*), 810 (*s*), 769 (*w*), 730 (*s*), 571 (*w*), 526 (*w*, C_{60}). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) 230 (4.62), 256 (4.74), 320 (4.45), 406 (3.51), 424 (3.19), 462 (3.12). MS (MALDI-TOF, negative-mode) *m/z* 959 (M^-), 720 (C_{60}^-). HR-MS *m/z*: 958.112 (M^+, calc. 958.110).

3′-(4-Biphenyl)-isoxazolo[4′,5′ :1,2][60]fullerene 1e

Yield 48 mg (18.7%). HPLC: τ_R (flow rate: 13 ml/min; toluene, silica gel) : 8 min. ^{1}H NMR (300 MHz, $CDCl_3$:CS_2 (1:9)) δ 7.31–7.48 (*m*, 3H, arom.), 7.61 (*d*, $^{3}J_{(H,H)} = 6.9$ Hz, 2H, arom.), 7.73 (*d*, $^{3}J_{(H,H)} = 8.3$ Hz, 2H, arom.), 8.27 (*d*, $^{3}J_{(H,H)} = 8.5$ Hz, 2H, arom.). ^{13}C NMR (75 MHz, $CDCl_3$:CS_2; (1:9)) δ 78.91 (1C, sp^3), 97.51 (1C, sp^3), 127.28 ($C_{arom.}$), 127.75 ($C_{arom.}$), 128.18 ($C_{arom.}$), 129.12 ($C_{arom.}$), 129.43 ($C_{arom.}$), 131.76 ($C_{arom.}$), 136.71 (2C), 137.17 (2C), 139.97 (2C), 140.42 (2C), 140.44 ($C_{arom.}$); 141.82 (2C), 142.23 (2C), 142.39 (2C), 142.46 (2C), 142.58 (2C), 142.60 ($C_{arom.}$), 142.95 (2C), 143.09 (2C), 143.18 (2C), 143.48 (2C), 144.17 (2C), 144.52 (2C), 144.76 (2C), 144.88 (2C), 144.93 (2C), 145.22 (2C), 145.31 (2C), 145.50 (2C), 145.72 (2C), 146.00 (2C), 146.01 (2C), 146.07 (2C), 146.32 (2C), 146.36 (2C), 146.48 (2C), 147.32 (1C), 147.81 (1C), 152.57 (C=N). FT-IR(KBr) cm^{-1} 3027 (*w*, $CH_{arom.}$), 2919 (*w*), 1606 (*m*, C=N), 1531 (*w*), 1487 (*w*), 1464 (*w*) 1429 (*w*, C_{60}), 1404 (*w*), 1343 (*w*), 1303 (*m*), 1268 (*w*), 1217 (*w*), 1181 (*w*, C_{60}), 1168 (*w*), 1156 (*w*), 1105 (*w*), 1053 (*w*), 1037 (*w*), 1020 (*w*), 1007 (*w*), 982 (*w*), 932 (*w*), 903 (*m*), 864 (*m*), 843 (*m*), 831 (*w*), 771 (*w*), 762 (*m*), 748 (*w*), 728 (*m*), 696 (*m*), 645 (*w*), 622 (*w*), 604 (*w*), 586 (*w*), 573 (*w*, C_{60}), 565 (*w*), 553 (*w*), 541 (*w*), 527 (*s*, C_{60}), 481 (*w*), 465 (*w*), 424 (*w*). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) 232 (5.05), 256 (5.21), 316 (4.77). MS (MALDI-TOF, negative mode) *m/z* 915 (M^-), 720 (C_{60}^-). HR-MS *m/z*: 915.072 (M^+, calc. 915.068).

3′-(4-Hydroxyphenyl)-isoxazolol[4′,5′:1,2][60]fullerene 1b

Yield 62 mg (25.9%). TLC (SiO_2; toluene): $R_f = 0.10$. ^{1}H NMR (300 MHz, 1,2-dichlorobenzene-d_4) δ 5.57 (*s*, 1H, OH), 7.06 (*d*, $^{3}J_{(H,H)} = 8.6$ Hz, 2H, arom.), 8.32 (*d*, $^{3}J_{(H,H)} = 8.6$ Hz, 2H, arom.). ^{13}C NMR (75 MHz, 1,2-dichlorobenzene-d_4) δ 79.48 (1C), 101.73 (1C), 116.27 ($C_{arom.}$), 121.98 ($C_{arom.}$) 130.84 ($C_{arom.}$), 136.69 (2C), 136.98 (2C), 140.19 (2C), 140.32 (2C), 141.60 (2C), 142.01 (2C), 142.16 (2C), 142.23 (2C), 142.32 (2C), 142.35 (2C), 142.74 (4C), 142.89 (2C), 143.99 (2C), 144.29 (2C), 144.61 (2C), 144.67 (2C), 144.93 (2C), 145.04 (2C), 145.14 (2C), 145.26 (2C), 145.54 (2C), 145.80 (2C), 145.85 (2C), 145.89 (2C), 146.12 (2C), 146.18 (2C), 146.28 (2C), 147.17 (1C), 147.70 (1C), 153.34 (CN), 158.22 ($C_{arom.}$). FT-IR (KBr) cm^{-1} 3448 (*s*, br., OH), 2921 (*w*), 1607 (*w*), 1514 (*w*), 1431 (*w*, C_{60}), 1427 (*w*), 1306 (*w*), 1274 (*w*), 1173 (*w*, C_{60}), 1107 (*w*), 978 (*w*), 867 (*w*), 833 (*w*), 769 (*w*), 624 (*w*), 526 (*w*, C_{60}). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) 256 (4.09), 272 (3.92), 314 (3.54), 346 (3.14), 360 (2.93). MS (MALDI-TOF, negative mode) *m/z* 855 (M^-). HR-MS *m/z*: 855.035 (M^+, calc. 855.031).

3,4,5-Trimethoxybenzoic acid 4-(isoxazolo[4′,5′: 1,2][60]fulleren-3′-yl)-phenyl ester 5l

Yield 36 mg (97.8%). TLC (SiO_2; toluene): $R_f = 0.10$. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.96 (*s*, 9H, 3 × CH_3), 7.43 (*d*, $^{3}J_{(H,H)} = 8.8$ Hz, 2H, arom.), 7.47 (*s*, 2H, arom.), 8.30 (*d*, $^{3}J_{(H,H)} = 8.5$ Hz, 2H, arom.). ^{13}C NMR (125 MHz, $CDCl_3$) δ 56.38 (2 × CH_3), 61.00 (1 × CH_3), 79.10 (1C), 101.73 (1C), 107.59 ($C_{arom.}$), 122.60 ($C_{arom.}$) 123.89 ($C_{arom.}$), 126.66 ($C_{arom.}$), 130.22 ($C_{arom.}$), 133.96 ($C_{arom.}$), 136.78 (2C), 137.12 (2C), 140.31 (2C), 140.44 (2C), 141.74 (2C), 142.05 (2C), 142.31 (4C), 142.48 (2C), 142.49 (2C), 142.87 (2C), 143.02 (2C), 143.18 (2C), 144.12 (2C), 144.28 (2C), 144.42 (2C), 144.48 (2C), 144.76 (2C), 145.19 (2C), 145.28 (2C), 145.42 (2C), 145.68 (2C), 145.73 (2C), 145.99 (2C), 146.04 (2C), 146.30 (2C), 146.33 (2C), 146.43 (2C), 147.32 (1C), 147.83 (1C), 152.84 (CN), 153.14 ($C_{arom.}$), 153.22 ($C_{arom.}$), 164.43 (C=O). FT-IR(KBr) cm^{-1} 1734 (*w*, C=O), 1588 (*w*), 1503 (*w*), 1459 (*w*), 1413 (*w*, C_{60}), 1335 (*w*), 1201 (*m*), 1167 (*m*, C_{60}), 1127 (*m*), 1002 (*w*), 932 (*w*), 905 (*w*), 864 (*m*), 768 (*w*), 730 (*s*), 569 (*w*), 548 (*w*), 526 (*w*, C_{60}). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) = 230 (4.98), 256 (5.11), 316 (4.66), 376 (3.95), 412 (3.57), 424 (3.43), 458 (3.35), 486 (3.34), 582 (3.19). MS (MALDI-TOF, negative mode) *m/z* 1050 (M^-). HR-MS *m/z* 1049.095 (M^+, calc. 1049.090).

Thiophene-2-carboxylic-acid 4-(isoxazolo[4′,5′: 1,2][60]fulleren-3′-yl)-phenyl ester 5m

Yield 29 mg (85.6%). TLC (SiO_2; toluene): $R_f = 0.25$. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.21 (*dd*, $^{3}J_{(H,H)} = 4.8$ Hz, $^{3}J_{(H,H)} = 3.7$ Hz, 1H), 7.45 (*d*, $^{3}J_{(H,H)} = 8.9$ Hz, 2H, arom.), 7.71 (*dd*, $^{3}J_{(H,H)} = 4.8$ Hz, $^{4}J_{(H,H)} = 1.1$ Hz, 1H), 8.02 (*dd*, $^{3}J_{(H,H)} = 3.7$ Hz, $^{4}J_{(H,H)} = 1.1$ Hz 1H), 8.29 (*d*, $^{3}J_{(H,H)} = 8.9$ Hz, 2H, arom.). ^{13}C NMR (75 MHz, $CDCl_3$) δ 79.09 (1C), 101.73(1C), 122.46 ($C_{arom.}$), 126.70 ($C_{arom.}$) 128.16 ($C_{arom.}$), 130.18 ($C_{arom.}$), 132.40 ($C_{arom.}$), 133.96 ($C_{arom.}$), 135.07 ($C_{arom.}$), 136.79 (2C), 137.12 (2C), 140.31 (2C), 140.46 (2C), 141.75 (2C), 142.06 (2C), 142.31 (2C), 142.33 (2C), 142.48 (4C), 142.87 (2C), 142.88 (2C), 143.01 (2C), 144.13 (2C), 144.28 (2C), 144.41 (2C), 144.49 (2C), 144.76 (2C), 145.19 (2C), 145.28 (2C), 145.43 (2C), 145.69 (2C), 145.73 (2C), 145.99 (2C), 146.04 (2C), 146.29 (2C), 146.33 (2C), 146.43 (2C), 147.32 (1C), 147.84 (1C), 152.42 (CN), 153.19 ($C_{arom.}$), 160.08 (C=O). FT-IR(KBr) cm^{-1} 2922 (*w*), 1731 (*s*, C=O), 1504 (*w*), 1412 (*w*, C_{60}), 1356 (*w*), 1304 (*w*), 1266 (*w*), 1202 (*m*), 1165 (*m*, C_{60}), 1081 (*w*), 1058 (*m*), 1016 (*w*), 980 (*w*), 904 (*w*), 865 (*m*), 770 (*w*), 731 (*s*), 569 (*w*), 526 (*w*, C_{60}). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) = 230 (4.94), 256 (5.10), 316 (4.60). MS (MALDI-TOF, negative mode) *m/z* 966 (M^-). HR-MS *m/z* 965.017 (M^+, calc. 965.015).

Ferrocene-carboxylic-acid 4-(isoxazolo[4′,5′:1,2][60]fulleren-3′-yl)-phenyl ester 5n

Yield 35 mg (93.5%). TLC (SiO_2; toluene): $R_f = 0,40$. ^{1}H NMR (300 MHz, 1,2-dichlorobenzene-d_4) δ 4.21 (*s*, 5H, Cp), 4.40 (*s*, 2H, Cp), 4.97 (*s*, 2H, Cp), 7.46 (*d*, $^{3}J_{(H,H)} = 8.7$ Hz, 2H, arom.), 8.39 (*d*, $^{3}J_{(H,H)} = 8.7$ Hz, 2H, arom.). ^{13}C NMR (75 MHz, 1,2-dichlorobenzene-d_4) δ 69.86 (1C, Cp), 70.05 (5C, Cp), 70.77 (2C, Cp), 72.20 (2C, Cp), 78.91 (1C), 99.90 (1C), 122.62 ($C_{arom.}$), 130.01 ($C_{arom.}$), 131.94 ($C_{arom.}$), 136.48 (2C), 136.84 (2C), 139.98 (2C), 140.16 (2C), 141.36 (2C), 141.76 (2C), 141.91 (2C), 142.08 (2C), 142.11 (2C), 142.50 (4C), 142.65 (2C), 143.73 (2C), 144.04 (2C), 144.16 (2C), 144.32 (2C), 144.34 (2C), 144.80 (2C), 144.91 (2C), 144.98 (2C), 145.01 (2C), 145.35 (2C), 145.39 (2C), 145.62 (2C), 145.65 (2C), 145.89 (2C), 145.95 (2C), 146.05 (2C), 146.93 (1C), 147.46 (1C), 152.82 (CN), 153.15 ($C_{arom.}$), 156.30 (C=O). FT-IR(KBr) cm^{-1} 1733 (*s*, C=O), 1506 (*w*), 1451 (*w*), 1357 (*w*), 1304 (*w*), 1271 (*m*), 1203 (*m*), 1167 (*m*, C_{60}), 1094 (*s*), 1050 (*w*), 1016 (*w*), 910 (*w*), 866 (*w*), 824 (*w*), 527 (*w*, C_{60}). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) = 230 (4.09), 256 (5.06), 316 (4.55). MS (MALDI-TOF, negative mode) *m/z* 1068 (M^-). HR-MS *m/z* 1067.021 (M^+, calc. 1067.024).

3′-[4-(N,N-Dimethylamino-phenyl)]-isoxazolo[4′,5′: 1,2][60]fullerene 1d

Yield 70 mg (28.7%). TLC (SiO_2; toluene): $R_f = 0.50$. ^{1}H NMR (300 MHz, CS_2 : $CDCl_3$ (1:1)

δ 3.05 (*s*, 6H, 2 × CH_3), 6.78 (*d*, $^{3}J_{(H,H)} = 7.9$ Hz, 2H, arom.), 8.10 (*d*, $^{3}J_{(H,H)} = 7.9$ Hz, 2H, arom.). ^{13}C NMR (75 MHz, $CDCl_3$) δ 39.87 ($N[CH_3]_2$), 82.33 (1C), 102.92 (1C), 111.83 ($C_{arom.}$), 116.07 ($C_{arom.}$), 129.85 ($C_{arom.}$), 136.53 (2C), 136.74 (2C), 140.04 (2C), 140.07 (2C), 141.48 (2C), 141.95 (2C), 142.15 (2C), 142.22 (2C), 142.28 (2C), 142.32 (2C), 142.65 (4C), 142.79 (2C), 143.95 (2C), 144.27 (2C), 144.73 (2C), 144.95 (2C), 145.02 (2C), 145.08 (2C), 145.29 (2C), 145.32 (2C), 145.38 (2C), 145.73 (2C), 145.78 (2C), 145.98 (2C), 146.03 (2C), 146.07 (2C), 146.19 (2C), 147.07 (1C), 147.56 (1C), 151.30 (CN), 152.90 ($C_{arom.}$). FT-IR(KBr) cm^{-1} 1607 (*m*, C=N), 1523 (*m*), 1431 (*w*), 1362 (*w*) 1309 (*w*), 1231 (*w*), 1189 (*w*), 1057 (*w*), 974 (*w*), 944 (*m*), 902 (*w*), 864 (*w*), 809 (*w*), 729 (*w*), 569 (*w*, C_{60}), 527 (*m*, C_{60}). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) = 232 (4.85), 256 (5.01), 320 (3.64). HR-MS *m/z* 882.079 (M^{+}, calc. 882,080).

3′-(2-Hydroxyphenyl)-isoxazolo[4′,5′:1,2][60]fullerene 1c

Yield 50 mg (21.0%). TLC(SiO_2; toluene / acetonitrile (9:1) $R_f = 0.55$. ^{1}H NMR (300 MHz, $CDCl_3:CS_2[1:1]$) δ 6.95–7.00 (*m*, 1H, arom.), 7.14 (*d*, $^{3}J_{(H,H)} = 7.0$ Hz, 1H, arom.), 7.40–7.46 (*m*, 1H, arom.), 8.64 (*d*, $^{3}J_{(H,H)} = 8.0$ Hz, 1H, arom.), 10.22 (*s*, 1H, OH). ^{13}C NMR (125 MHz, $CDCl_3:CS_2[1:1]$) δ 78.71 (1C), 98.88 (1C), 114.12 ($C_{arom.}$), 118.20 ($C_{arom.}$), 119.78 ($C_{arom.}$), 128.06 ($C_{arom.}$), 132.24 ($C_{arom.}$), 136.75 (2C), 137.42 (2C), 140.07 (2C), 140.52 (2C), 141.68 (2C), 141.97 (2C), 142.39 (2C), 142.49 (2C), 142.58 (2C), 142.61 (2C), 143.01 (4C), 143.10 (2C), 144.17 (2C), 144.28 (2C), 144.58 (4C), 144.70 (2C), 145.24 (2C), 145.37 (2C), 145.44 (2C), 145.87 (2C), 145.90 (2C), 146.09 (2C), 146.10 (2C), 146.36 (2C), 146.46 (2C), 146.56 (2C), 147.42 (1C), 147.87 (1C), 152.59 (CN), 158.41 ($C_{arom.}$). FT-IR(KBr) cm^{-1} 3445 (*w*, br., OH), 2921 (*w*), 2849 (*w*), 1615 (*w*), 1587 (*w*), 1490 (*w*), 1457 (*w*, C_{60}), 1427 (*w*), 1388 (*w*), 1318 (*w*), 1302 (*w*), 1253 (*w*), 1223 (*w*), 1185 (*w*, C_{60}), 1168 (*w*), 1158 (*w*), 993 (*w*), 936 (*w*), 923 (*w*), 903 (*w*), 873 (*m*), 823 (*w*), 769 (*w*), 748 (*w*), 729 (*w*), 681 (*w*), 665 (*w*), 653 (*m*), 621 (*w*), 604 (*w*), 87 (*w*), 574 (*w*, C_{60}), 564 (*w*), 554 (*w*), 543 (*w*), 527 (*s*, C_{60}), 481 (*w*). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) = 230 nm (4.96), 256 (5.09), 318 (4.65), 354 (4.20), 362 (4.11), 372 (4.00). MS (MALDI-TOF, negative mode) *m/z* 855 (M^{-}). HR-MS *m/z*: 855.034 (M^{+}, calc. 855.032).

3,4,5-Trimethoxybenzoic-acid 2-(isoxazolo[4′,5′:1,2][60]fulleren-3′-yl)-phenyl ester 6l

Yield 17 mg (45.6%). TLC (SiO_2; toluene / acetonitrile (95:5) $R_f = 0.78$. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.92 (*s*, 3H, 1 × CH_3), 4.01 (*s*, 6H, 2 × CH_3), 7.44–7.50 (*m*, 1H, arom.), 7.57–7.67 (*m*, 2H, arom.), 7.60 (*s*, 2H, arom.), 7.86 (*dd*, $^{3}J_{(H,H)} = 7.7$ Hz, $^{4}J_{(H,H)} = 1.5$ Hz, 1H, arom.). ^{13}C NMR (75 MHz, $CDCl_3$) δ 56.30 (2 × CH_3), 60.77 (1 × CH_3), 81.16 (1C), 95.59 (1C), 108.05 ($C_{arom.}$), 108.08 ($C_{arom.}$), 121.29 ($C_{arom.}$), 124.07 ($C_{arom.}$), 124.27 ($C_{arom.}$), 126.12 ($C_{arom.}$), 130.78 ($C_{arom.}$), 131.73 ($C_{arom.}$), 136.63 (2C), 136.74 (2C), 140.13 (2C), 140.60 (2C), 141.76 (2C), 141.94 (2C), 141.97 (2C), 142.20 (2C), 142.34 (2C), 142.35 (2C), 142.76 (2C), 142.78 (2C), 142.94 (2C), 143.58 (2C), 143.70 (2C), 144.04 (2C), 144.24 (2C), 144.65 (2C), 145.11 (2C), 145.14 (2C), 145.17 (2C), 145.49 (2C), 145.53 (2C), 145.91 (2C), 145.98 (2C), 146.25 (2C), 146.28 (2C), 146.32 (2C), 147.25 (1C), 147.82 (1C), 149.70 ($C_{arom.}$), 151.34 (CN), 153.22 ($C_{arom.}$), 164.14 (C=O). FT-IR(KBr) cm ^{1} 3438 (*s*, br., OH), 2998 (*w*), 2932 (*w*), 1736 (*s*, C=O), 1698 (*w*), 1626 (*w*), 1587 (*s*), 1541 (*w*), 1501 (*m*), 1459 (*m*, C_{60}), 1414 (*m*), 1335 (*s*), 1305 (*w*), 1231 (*w*), 1196 (*s*), 1168 (*s*, C_{60}), 1128 (*s*), 1104 (*m*), 1049 (*w*), 1030 (*w*), 1003 (*w*), 972 (*w*), 938 (*w*), 858 (*w*), 834 (*w*), 771 (*w*), 751 (*w*), 728 (*w*), 715 (*w*), 666 (*w*), 650 (*w*), 604 (*w*), 588 (*w*), 574 (*w*), 565 (*w*), 555 (*w*), 527 (*s*, C_{60}), 480 (*w*). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) = 232 (4.92), 256 (5.02), 316 (4.57). MS (MALDI-TOF, negative mode) *m/z* 1050 (M^{-}). HR-MS *m/z* 1049.092 (M^{+}, calc. 1049.090).

Thiophene-2-carboxylic-acid 2-(isoxazolo[4′,5′:1,2][60]fulleren-3′-yl)-phenyl ester 6m

Yield 21 mg (62.0%). TLC (SiO_2; toluene) $R_f = 0.62$. ^1H NMR (300 MHz, $CDCl_3$) δ 7.24 (*dd*, $^3J_{(H,H)} = 4.8$ Hz, $^3J_{(H,H)} = 3.7$ Hz, 1H), 7.44–7.68 (*m*, 3H, arom.), 7.74 (*dd*, $^3J_{(H,H)} = 4.8$ Hz, $^4J_{(H,H)} = 1.1$ Hz, 1H), 7.85 (*dd*, $^3J_{(H,H)} = 7.3$ Hz, $^4J_{(H,H)} = 1.4$ Hz, 1H, arom.), 8.11(*dd*, $^3J_{(H,H)} = 3.7$ Hz, $^4J_{(H,H)} = 1.1$ Hz, 1H). $^{13}CNMR$ (75 MHz, $CDCl_3$)$\delta = 80.86$ ($1C_{Fulleren}$, sp^3), 97.66 ($1C_{Fulleren}$, sp^3), 121.33 ($C_{arom.}$), 124.00 ($C_{arom.}$), 126.25 ($C_{arom.}$), 128.21 ($C_{arom.}$), 130.98 ($C_{arom.}$), 131.71 ($C_{arom.}$), 132.52 ($C_{arom.}$), 134.30 ($C_{arom.}$), 135.59 ($2C_{Fulleren}$, $1C_{arom.}$), 136.75 (2C), 140.09 (2C), 140.63 (2C), 141.79 (2C), 142.06 (2C), 142.07 (2C), 142.23 (2C), 142.34 (2C), 142.36 (2C), 142.75 (2C), 142.77 (2C), 142.89 (2C), 143.75 (2C), 143.90 (2C), 144.11 (2C), 144.22 (2C), 144.76 (2C), 145.13 (2C), 145.19 (2C), 145.27 (2C), 145.52 (2C), 145.60 (2C), 145.91 (2C), 145.97 (2C), 146.25 (4C), 146.30 (2C), 147.22 (1C), 147.84 (1C), 149.30 ($C_{arom.}$), 151.28 (CN), 159.97 (C=O). FT-IR(KBr) cm^{-1} 1728 (*s*, C=O), 1601 (*w*), 1520 (*w*), 1488 (*w*), 1413 (*m*, C_{60}), 1357 (*w*), 1304 (*w*), 1253 (*m*), 1220 (*w*), 1194 (*s*, C_{60}), 1117 (*w*), 1082 (*w*), 1055 (*s*), 1029 (*w*), 972 (*w*), 906 (*m*), 858 (*m*), 734 (*s*), 669 (*w*), 650 (*w*), 604 (*w*), 571 (*w*), 559 (*w*), 526 (*s*, C_{60}), 483 (*w*). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) = 232 (5.11), 256 (5.21), 314 (4.78), 350 (4.37), 362 (4.26), 372 (4.11). MS (MALDI-TOF, negative mode) *m*/*z* 966 (M^-). HR-MS *m*/*z* 965.010 (M^+, calc. 965.015).

Ferrocene-carboxylic-acid 2-(isoxazolo[4′,5′:1,2][60]fulleren-3′-yl)-phenyl ester 6n

Yield 21 mg (56.1%). TLC (SiO_2; toluene) $R_f = 0.36$. ^1H NMR (300 MHz, $CDCl_3 : CS_2$ [1:1]) δ 4.41 (*s*, 5H, Cp), 4.51 (*d*, $^3J_{(H,H)} = 1.8$ Hz, 2H, Cp), 5.00 (*d*, $^3J_{(H,H)} = 1.8$ Hz, 2H, Cp), 7.38–7.61 (*m*, 3H, arom.), 7.82 (*d*, $^3J_{(H,H)} = 7.7$ Hz, 1H, arom.). ^{13}C NMR (75 MHz, $CDCl_3$) $\delta = 69.91$ (1C, Cp), 70.19 (5C, Cp), 70.88 (2C, Cp), 72.18 (2C, Cp), 85.75 (1C), 100.20 (1C), 120.98 ($C_{arom.}$), 123.94 ($C_{arom.}$), 125.64 ($C_{arom.}$), 130.86 ($C_{arom.}$), 131.54 ($C_{arom.}$), 136.72 (2C), 136.80 (2C), 140.12 (2C), 140.64 (2C), 141.77 (2C), 142.10 (2C), 142.16 (2C), 142.23 (2C), 142.36 (2C), 142.38 (2C), 142.76 (2C), 142.78 (2C), 142.91 (2C), 143.86 (2C), 144.13 (2C), 144.16 (2C), 144.23 (2C), 144.80 (2C), 145.14 (2C), 145.19 (2C), 145.32 (2C), 145.56 (2C), 145.92 (2C), 145.98 (4C), 146.27 (2C), 146.28 (2C), 146.31 (2C), 147.23 (1C), 147.84 (1C), 149.73 ($C_{arom.}$), 151.39 (CN), 169.84 (C=O). FT-IR(KBr) cm^{-1} 2922 (*w*), 1733 (*s*, C=O), 1633 (*w*, br.), 1440 (*w*, C_{60}), 1474 (*w*), 1306 (*w*), 1264 (*m*), 1200 (*m*), 1091 (*m*), 1051 (*w*), 910 (*w*), 823 (*w*), 754 (*w*), 527 (*w*, C_{60}). UV-VIS ($CHCl_3$) λ_{max} nm (lg ε) = 230 (4.83), 256 (4.94), 316 (4.46). MS (MALDI-TOF, negative mode) *m*/*z* 1068 (M^-). HR-MS *m*/*z*: 1067.023 (M^+, calc. 1067.024).

Crystal Structure Analysis of 1k

Table II contains the crystallographic data and details of the refinement procedure. The reflections were collected with a Nonius-Kappa-CCD-diffractometer (Mo $K\alpha$-radiation, graphite monochromator). The structure was solved by Direct Methods (SHELXS-97[16]). Refinement

TABLE II Crystallographic data and parameters of measurement of **1k**

Compound	**1k**
Empirical formula	$C_{76}H_{15}NO_4{}^*CS_2$
Molecular mass [g/mol]	1082,02
Crystal size [mm]	0.5 × 0.45 × 0.25
Crystal color	black
Crystal shape	irregular
Space group	$P2_1/n$
a [Å]	13.8367 (2)
b [Å]	17.0477 (3)
c [Å]	18.9991 (2)
β [°]	107.555 (1)
V [$Å^3$]	4272.9 (1)
D_{calc} [Mg/m^3]	1.68
Z	4

TABLE II (Continued)

Compound	**1k**
$F(000)$	2192
Temperature [K]	113
h_{min}/h_{max}	−18/18
k_{min}/k_{max}	−22/22
l_{min}/l_{max}	−25/25
Θ range [°]	1.6−28.3
μ [mm^{-1}]	0.20
Refl. collected	40540
Refl. unique	10598
Refl. observed [$I > 2\sigma(I)$]	7601
Variables	817
$(\Delta/\sigma)_{max}$	< 0.001
R	0.050
R_w	0.131
S(Gof)	1.06
$(\Delta\rho)_{max}$ [e Å^{-3}]	0.40
$(\Delta\rho)_{min}$ [e Å^{-3}]	−0.68

was carried out with SHELXL-97. [17] The structural parameters of the non-hydrogen atoms were refined anisotropically according to a full-matrix least-squares technique (F^2). All hydrogen atoms were refined isotropic. One CS_2 unit was included per host molecule. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Center as supplementary publication no. CCDC-141814. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [fax: int. code +44(1223)336-033, e-mail: deposit@ccdc.cam.ac.uk].

Supporting Materials

TABLE III Atomic coordinates and equivalent isotropic displacement parameters (A^2) for **1k**. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor

	x	y	z	U(eq)
S1	0.11700 (5)	0.03870 (4)	0.14396 (4)	0.04771 (18)
S2	−0.10573 (5)	−0.00188 (4)	0.05803 (4)	0.04656 (18)
C100	0.00639 (19)	0.01890 (12)	0.10145 (12)	0.0375 (5)
O1	0.06327 (9)	0.70575 (7)	−0.17992 (7)	0.0202 (3)
O2	0.03838 (9)	0.72323 (7)	0.01522 (7)	0.0192 (3)
O3	0.28275 (10)	0.89891 (7)	0.16355 (7)	0.0236 (3)
O4	0.35317 (9)	0.62218 (7)	0.15445 (7)	0.0191 (3)
N1	0.04847 (11)	0.65375 (9)	−0.12643 (8)	0.0192 (3)
C1	0.17212 (12)	0.71695 (10)	−0.16893 (9)	0.0159 (3)
C2	0.22614 (12)	0.67004 (10)	−0.09619 (9)	0.0144 (3)
C3	0.29911 (13)	0.60568 (10)	−0.10526 (9)	0.0148 (3)
C4	0.30412 (13)	0.58012 (10)	−0.17238 (9)	0.0162 (3)
C5	0.25249 (13)	0.62406 (10)	−0.24074 (9)	0.0178 (4)
C6	0.19822 (13)	0.69080 (10)	−0.23074 (9)	0.0166 (4)
C7	0.20869 (13)	0.75833 (10)	−0.28005 (9)	0.0181 (4)
C8	0.20711 (13)	0.82815 (11)	−0.23667 (10)	0.0190 (4)
C9	0.19680 (13)	0.80429 (10)	−0.16678 (10)	0.0164 (4)
C10	0.24961 (13)	0.84287 (10)	−0.10371 (10)	0.0176 (4)
C11	0.30107 (13)	0.79928 (10)	−0.03557 (9)	0.0165 (4)
C12	0.29718 (13)	0.71860 (10)	−0.03427 (9)	0.0146 (3)
C13	0.38758 (13)	0.67481 (10)	0.00058 (9)	0.0149 (3)
C14	0.38865 (13)	0.60552 (10)	−0.04271 (9)	0.0154 (3)
C15	0.48034 (13)	0.57619 (10)	−0.04866 (10)	0.0181 (4)
C16	0.48523 (14)	0.54863 (10)	−0.11891 (10)	0.0194 (4)
C17	0.39884 (14)	0.55124 (10)	−0.17999 (10)	0.0190 (4)
C18	0.40672 (14)	0.57598 (10)	−0.25109 (10)	0.0201 (4)
C19	0.31685 (14)	0.62087 (11)	−0.28828 (9)	0.0195 (4)
C20	0.32402 (14)	0.68543 (11)	−0.33118 (9)	0.0200 (4)

TABLE III (Continued)

	x	*y*	*z*	*U*(eq)
C21	0.26884 (14)	0.75590 (11)	−0.32683 (9)	0.0202 (4)
C22	0.33108 (14)	0.82323 (11)	−0.33209 (9)	0.0208 (4)
C23	0.32946 (14)	0.88975 (11)	−0.29084 (10)	0.0211 (4)
C24	0.26614 (14)	0.89305 (11)	−0.24220 (10)	0.0211 (4)
C25	0.32024 (14)	0.93474 (10)	−0.17596 (11)	0.0215 (4)
C26	0.31288 (13)	0.90943 (10)	−0.10811 (10)	0.0193 (4)
C27	0.40226 (14)	0.90685 (10)	−0.04403 (10)	0.0204 (4)
C28	0.39485 (13)	0.83962 (10)	0.00026 (9)	0.0178 (4)
C29	0.48192 (14)	0.79757 (11)	0.03666 (9)	0.0194 (4)
C30	0.47821 (14)	0.71310 (11)	0.03690 (9)	0.0174 (4)
C31	0.57308 (13)	0.68328 (11)	0.02967 (9)	0.0184 (4)
C32	0.57422 (13)	0.61627 (10)	−0.01160 (9)	0.0188 (4)
C33	0.63796 (13)	0.61346 (11)	−0.06009 (10)	0.0204 (4)
C34	0.58315 (14)	0.57158 (10)	−0.12612 (11)	0.0212 (4)
C35	0.59039 (14)	0.59553 (10)	−0.19438 (10)	0.0215 (4)
C36	0.50026 (14)	0.59779 (11)	−0.25819 (10)	0.0217 (4)
C37	0.50757 (14)	0.66536 (11)	−0.30296 (10)	0.0209 (4)
C38	0.42181 (14)	0.70820 (11)	−0.33871 (9)	0.0211 (4)
C39	0.42587 (14)	0.79359 (11)	−0.33938 (9)	0.0209 (4)
C40	0.51614 (14)	0.83197 (11)	−0.30396 (9)	0.0198 (4)
C41	0.51499 (14)	0.90127 (11)	−0.26025 (10)	0.0203 (4)
C42	0.42374 (14)	0.93042 (10)	−0.25384 (10)	0.0210 (4)
C43	0.41810 (14)	0.95782 (10)	−0.18280 (11)	0.0212 (4)
C44	0.50392 (14)	0.95566 (10)	−0.12163 (10)	0.0214 (4)
C45	0.49559 (14)	0.93007 (10)	−0.05053 (10)	0.0214 (4)
C46	0.58612 (14)	0.88581 (11)	−0.01317 (10)	0.0204 (4)
C47	0.57899 (13)	0.82097 (11)	0.02922 (9)	0.0198 (4)
C48	0.63533 (13)	0.75022 (10)	0.02473 (10)	0.0196 (4)
C49	0.69674 (13)	0.74775 (10)	−0.02126 (10)	0.0195 (4)
C50	0.69767 (13)	0.67803 (11)	−0.06449 (10)	0.0205 (4)
C51	0.70539 (13)	0.70253 (11)	−0.13589 (10)	0.0200 (4)
C52	0.65305 (13)	0.66233 (11)	−0.19927 (10)	0.0203 (4)
C53	0.60184 (14)	0.70520 (11)	−0.26631 (10)	0.0204 (4)
C54	0.60618 (13)	0.78692 (11)	−0.26673 (9)	0.0196 (4)
C55	0.66068 (13)	0.82877 (11)	−0.20042 (10)	0.0190 (4)
C56	0.70949 (13)	0.78766 (11)	−0.13626 (10)	0.0191 (4)
C57	0.70415 (13)	0.81560 (11)	−0.06515 (9)	0.0196 (4)
C58	0.65018 (14)	0.88364 (11)	−0.06118 (10)	0.0200 (4)
C59	0.59893 (14)	0.92614 (10)	−0.12827 (10)	0.0198 (4)
C60	0.60412 (13)	0.89942 (10)	−0.19632 (10)	0.0192 (4)
C61	0.13292 (13)	0.63510 (10)	−0.07995 (9)	0.0152 (3)
C62	0.13641 (12)	0.57626 (10)	−0.02179 (9)	0.0146 (3)
C63	0.16595 (12)	0.59391 (10)	0.05394 (9)	0.0147 (3)
C64	0.16425 (13)	0.53336 (10)	0.10332 (10)	0.0170 (4)
C65	0.13342 (13)	0.45813 (10)	0.07871 (10)	0.0181 (4)
C66	0.10489 (13)	0.44107 (10)	0.00366 (10)	0.0186 (4)
C67	0.10693 (13)	0.49950 (10)	−0.04592 (10)	0.0170 (4)
C68	0.19718 (12)	0.67400 (10)	0.08267 (9)	0.0147 (3)
C69	0.13359 (13)	0.73978 (10)	0.06114 (9)	0.0153 (3)
C70	0.16420 (14)	0.81464 (10)	0.08616 (10)	0.0177 (4)
C71	0.25942 (14)	0.82426 (10)	0.13755 (9)	0.0174 (4)
C72	0.32463 (14)	0.76120 (10)	0.16243 (10)	0.0171 (4)
C73	0.29265 (13)	0.68689 (10)	0.13474 (9)	0.0155 (3)
C74	−0.01898 (16)	0.78700 (12)	−0.02552 (12)	0.0255 (4)
C75	0.36810 (17)	0.90637 (12)	0.22751 (11)	0.0263 (4)
C76	0.45228 (14)	0.63368 (12)	0.20456 (11)	0.0231 (4)

TABLE IV Hydrogen coordinates and isotropic displacement parameters (A^2) for **1k**

	x	*y*	*z*	*U*(eq)
H64	0.1845 (13)	0.5441 (10)	0.1547 (10)	0.008 (4)
H65	0.1300 (14)	0.4181 (11)	0.1131 (10)	0.014 (5)
H66	0.0823 (15)	0.3913 (12)	−0.0132 (11)	0.023 (5)
H67	0.0874 (15)	0.4912 (11)	−0.0964 (11)	0.017 (5)
H70	0.1174 (15)	0.8594 (12)	0.0719 (10)	0.020 (5)
H72	0.3919 (17)	0.7683 (12)	0.1981 (12)	0.027 (6)
H74A	−0.0433 (17)	0.8192 (14)	0.0088 (13)	0.039 (6)
H74B	−0.0703 (19)	0.7638 (13)	−0.0604 (14)	0.033 (6)
H74C	0.0239 (17)	0.8202 (13)	−0.0521 (12)	0.035 (6)
H75A	0.3613 (17)	0.9598 (14)	0.2441 (12)	0.036 (6)
H75B	0.4293 (16)	0.9016 (11)	0.2165 (11)	0.020 (5)
H75C	0.3658 (15)	0.8699 (12)	0.2641 (11)	0.025 (5)
H76A	0.4821 (16)	0.5809 (13)	0.2143 (11)	0.027 (5)
H76B	0.4468 (15)	0.6574 (12)	0.2521 (11)	0.023 (5)
H76C	0.4956 (17)	0.6715 (14)	0.1819 (12)	0.039 (6)

TABLE V Anisotropic displacement parameters (A^2) for **1k**. The anisotropic displacement factor exponent takes the form: $-2pi^2[h^2a^{*2}U11 + \cdots + 2hka^*b^*U12]$

	*U*11	*U*22	*U*33	*U*23	*U*13	*U*12
S1	0.0508 (4)	0.0439 (4)	0.0526 (4)	0.0081 (3)	0.0219 (3)	0.0071 (3)
S2	0.0526 (4)	0.0452 (4)	0.0469 (4)	0.0086 (3)	0.0226 (3)	0.0096 (3)
C100	0.0570 (16)	0.0271 (11)	0.0370 (12)	0.0107 (9)	0.0270 (11)	0.0136 (10)
O1	0.0118 (6)	0.0279 (7)	0.0201 (6)	0.0085 (5)	0.0033 (5)	−0.0012 (5)
O2	0.0134 (6)	0.0177 (6)	0.0237 (7)	0.0017 (5)	0.0015 (5)	0.0008 (5)
O3	0.0278 (7)	0.0141 (6)	0.0241 (7)	−0.0028 (5)	0.0007 (6)	−0.0012 (5)
O4	0.0150 (6)	0.0168 (6)	0.0216 (6)	−0.0019 (5)	−0.0005 (5)	0.0019 (5)
N1	0.0172 (8)	0.0222 (8)	0.0185 (7)	0.0041 (6)	0.0058 (6)	−0.0022 (6)
C1	0.0098 (8)	0.0197 (9)	0.0166 (8)	0.0010 (7)	0.0017 (7)	−0.0009 (6)
C2	0.0133 (8)	0.0167 (8)	0.0128 (8)	0.0009 (7)	0.0031 (6)	−0.0014 (6)
C3	0.0145 (8)	0.0122 (8)	0.0181 (8)	0.0025 (7)	0.0053 (7)	−0.0021 (6)
C4	0.0186 (9)	0.0112 (8)	0.0184 (8)	−0.0019 (7)	0.0050 (7)	−0.0045 (6)
C5	0.0176 (9)	0.0190 (9)	0.0148 (8)	−0.0035 (7)	0.0017 (7)	−0.0075 (7)
C6	0.0123 (8)	0.0225 (9)	0.0124 (8)	0.0005 (7)	−0.0004 (6)	−0.0051 (7)
C7	0.0114 (8)	0.0254 (9)	0.0133 (8)	0.0027 (7)	−0.0024 (7)	−0.0017 (7)
C8	0.0111 (8)	0.0216 (9)	0.0218 (9)	0.0082 (7)	0.0010 (7)	0.0043 (7)
C9	0.0097 (8)	0.0182 (8)	0.0206 (9)	0.0049 (7)	0.0035 (7)	0.0040 (6)
C10	0.0154 (9)	0.0158 (8)	0.0241 (9)	−0.0002 (7)	0.0098 (7)	0.0040 (7)
C11	0.0144 (9)	0.0213 (9)	0.0157 (8)	−0.0045 (7)	0.0073 (7)	0.0018 (7)
C12	0.0137 (8)	0.0193 (8)	0.0126 (8)	−0.0014 (7)	0.0068 (6)	−0.0018 (7)
C13	0.0140 (8)	0.0209 (8)	0.0101 (7)	0.0033 (7)	0.0040 (6)	0.0004 (7)
C14	0.0166 (9)	0.0144 (8)	0.0151 (8)	0.0053 (7)	0.0050 (7)	0.0005 (6)
C15	0.0175 (9)	0.0156 (8)	0.0206 (9)	0.0079 (7)	0.0048 (7)	0.0032 (7)
C16	0.0238 (10)	0.0093 (8)	0.0278 (10)	0.0022 (7)	0.0118 (8)	0.0041 (7)
C17	0.0219 (10)	0.0108 (8)	0.0267 (10)	−0.0034 (7)	0.0110 (8)	−0.0016 (7)
C18	0.0244 (10)	0.0173 (8)	0.0203 (9)	−0.0103 (7)	0.0091 (8)	−0.0044 (7)
C19	0.0209 (9)	0.0224 (9)	0.0149 (8)	−0.0090 (7)	0.0048 (7)	−0.0080 (7)
C20	0.0191 (9)	0.0298 (10)	0.0094 (8)	−0.0041 (7)	0.0016 (7)	−0.0058 (7)
C21	0.0164 (9)	0.0300 (10)	0.0095 (8)	0.0034 (7)	−0.0031 (7)	−0.0044 (7)
C22	0.0178 (9)	0.0307 (10)	0.0111 (8)	0.0075 (7)	0.0000 (7)	−0.0028 (7)
C23	0.0188 (9)	0.0221 (9)	0.0199 (9)	0.0132 (8)	0.0023 (7)	0.0022 (7)
C24	0.0170 (9)	0.0204 (9)	0.0239 (9)	0.0110 (8)	0.0034 (7)	0.0072 (7)

TABLE V (Continued)

	*U*11	*U*22	*U*33	*U*23	*U*13	*U*12
C25	0.0196 (9)	0.0109 (8)	0.0338 (10)	0.0045 (7)	0.0078 (8)	0.0050 (7)
C26	0.0196 (9)	0.0123 (8)	0.0280 (10)	−0.0028 (7)	0.0102 (8)	0.0034 (7)
C27	0.0228 (10)	0.0165 (8)	0.0244 (9)	−0.0090 (7)	0.0108 (8)	−0.0008 (7)
C28	0.0205 (9)	0.0189 (8)	0.0166 (8)	−0.0065 (7)	0.0095 (7)	−0.0026 (7)
C29	0.0202 (9)	0.0275 (9)	0.0105 (8)	−0.0060 (7)	0.0046 (7)	−0.0049 (7)
C30	0.0168 (9)	0.0256 (9)	0.0085 (8)	0.0021 (7)	0.0018 (7)	−0.0017 (7)
C31	0.0132 (9)	0.0280 (10)	0.0113 (8)	0.0056 (7)	−0.0004 (7)	0.0012 (7)
C32	0.0152 (9)	0.0215 (9)	0.0182 (9)	0.0089 (7)	0.0029 (7)	0.0057 (7)
C33	0.0141 (9)	0.0220 (9)	0.0243 (9)	0.0086 (8)	0.0047 (7)	0.0088 (7)
C34	0.0200 (9)	0.0150 (8)	0.0303 (10)	0.0016 (8)	0.0104 (8)	0.0083 (7)
C35	0.0211 (10)	0.0195 (9)	0.0281 (10)	−0.0029 (8)	0.0137 (8)	0.0061 (7)
C36	0.0247 (10)	0.0200 (9)	0.0238 (9)	−0.0091 (8)	0.0126 (8)	−0.0011 (7)
C37	0.0245 (10)	0.0254 (9)	0.0167 (8)	−0.0069 (7)	0.0122 (7)	−0.0032 (8)
C38	0.0236 (10)	0.0305 (10)	0.0100 (8)	−0.0052 (7)	0.0061 (7)	−0.0068 (8)
C39	0.0217 (10)	0.0309 (10)	0.0098 (8)	0.0046 (7)	0.0043 (7)	−0.0039 (8)
C40	0.0203 (9)	0.0269 (9)	0.0137 (8)	0.0066 (7)	0.0075 (7)	−0.0025 (7)
C41	0.0208 (9)	0.0205 (9)	0.0193 (9)	0.0063 (7)	0.0058 (7)	−0.0048 (7)
C42	0.0227 (10)	0.0164 (8)	0.0232 (9)	0.0108 (7)	0.0057 (8)	−0.0007 (7)
C43	0.0236 (10)	0.0091 (8)	0.0318 (10)	0.0038 (7)	0.0095 (8)	0.0008 (7)
C44	0.0253 (10)	0.0112 (8)	0.0282 (10)	−0.0044 (7)	0.0087 (8)	−0.0057 (7)
C45	0.0264 (10)	0.0154 (8)	0.0231 (9)	−0.0094 (7)	0.0085 (8)	−0.0060 (7)
C46	0.0190 (9)	0.0237 (9)	0.0171 (9)	−0.0097 (7)	0.0033 (7)	−0.0090 (7)
C47	0.0178 (9)	0.0283 (10)	0.0110 (8)	−0.0053 (7)	0.0008 (7)	−0.0064 (7)
C48	0.0133 (9)	0.0295 (10)	0.0123 (8)	0.0003 (7)	−0.0017 (7)	−0.0037 (7)
C49	0.0091 (8)	0.0292 (10)	0.0161 (9)	0.0028 (7)	−0.0026 (7)	−0.0019 (7)
C50	0.0096 (8)	0.0285 (10)	0.0214 (9)	0.0062 (8)	0.0018 (7)	0.0061 (7)
C51	0.0111 (9)	0.0264 (9)	0.0238 (9)	0.0035 (8)	0.0072 (7)	0.0052 (7)
C52	0.0170 (9)	0.0234 (9)	0.0240 (9)	−0.0004 (8)	0.0115 (7)	0.0059 (7)
C53	0.0191 (9)	0.0278 (10)	0.0188 (9)	−0.0027 (8)	0.0125 (7)	0.0003 (7)
C54	0.0168 (9)	0.0279 (10)	0.0168 (9)	0.0005 (8)	0.0092 (7)	−0.0025 (7)
C55	0.0144 (9)	0.0254 (9)	0.0184 (9)	0.0017 (7)	0.0068 (7)	−0.0049 (7)
C56	0.0104 (8)	0.0270 (10)	0.0205 (9)	0.0010 (8)	0.0055 (7)	−0.0027 (7)
C57	0.0105 (8)	0.0304 (10)	0.0163 (8)	−0.0006 (8)	0.0015 (7)	−0.0063 (7)
C58	0.0171 (9)	0.0231 (9)	0.0174 (9)	−0.0041 (7)	0.0018 (7)	−0.0089 (7)
C59	0.0184 (9)	0.0169 (8)	0.0237 (9)	−0.0020 (7)	0.0059 (7)	−0.0084 (7)
C60	0.0171 (9)	0.0189 (9)	0.0217 (9)	0.0039 (7)	0.0061 (7)	−0.0061 (7)
C61	0.0134 (8)	0.0159 (8)	0.0161 (8)	−0.0031 (7)	0.0040 (7)	−0.0028 (6)
C62	0.0096 (8)	0.0169 (8)	0.0171 (8)	0.0009 (7)	0.0036 (6)	−0.0004 (6)
C63	0.0097 (8)	0.0166 (8)	0.0177 (8)	0.0013 (7)	0.0041 (6)	−0.0003 (6)
C64	0.0131 (8)	0.0214 (9)	0.0166 (9)	0.0013 (7)	0.0046 (7)	0.0010 (7)
C65	0.0145 (9)	0.0166 (8)	0.0243 (9)	0.0056 (8)	0.0076 (7)	0.0029 (7)
C66	0.0128 (9)	0.0142 (8)	0.0288 (10)	−0.0029 (7)	0.0060 (7)	−0.0009 (7)
C67	0.0129 (9)	0.0204 (9)	0.0175 (9)	−0.0028 (7)	0.0043 (7)	−0.0007 (7)
C68	0.0142 (8)	0.0173 (8)	0.0139 (8)	−0.0007 (7)	0.0061 (7)	−0.0019 (6)
C69	0.0147 (8)	0.0186 (8)	0.0143 (8)	0.0008 (7)	0.0067 (7)	−0.0014 (6)
C70	0.0186 (9)	0.0186 (9)	0.0175 (8)	0.0012 (7)	0.0077 (7)	0.0025 (7)
C71	0.0231 (9)	0.0149 (8)	0.0159 (8)	−0.0020 (7)	0.0082 (7)	−0.0025 (7)
C72	0.0153 (9)	0.0203 (9)	0.0148 (8)	−0.0007 (7)	0.0034 (7)	−0.0009 (7)
C73	0.0167 (9)	0.0168 (8)	0.0137 (8)	0.0013 (7)	0.0054 (7)	0.0007 (7)
C74	0.0184 (10)	0.0234 (10)	0.0291 (11)	0.0028 (9)	−0.0016 (9)	0.0042 (8)
C75	0.0324 (12)	0.0194 (10)	0.0221 (10)	−0.0001 (8)	0.0009 (9)	−0.0027 (8)
C76	0.0182 (10)	0.0226 (10)	0.0235 (10)	−0.0011 (8)	−0.0015 (8)	0.0032 (8)

TABLE VI Bond lengths [A] and angles [deg] for **1k**

S1-C100	1.537 (3)
S2-C100	1.562 (3)
O1-N1	1.4092 (18)
O1-C1	1.469 (2)
O2-C69	1.372 (2)
O2-C74	1.428 (2)
O3-C71	1.368 (2)
O3-C75	1.422 (2)
O4-C73	1.367 (2)
O4-C76	1.428 (2)
N1-C61	1.274 (2)
C1-C6	1.524 (2)
C1-C9	1.525 (2)
C1-C2	1.576 (2)
C2-C12	1.529 (2)
C2-C61	1.534 (2)
C2-C3	1.536 (2)
C3-C4	1.369 (2)
C3-C14	1.435 (2)
C4-C17	1.448 (2)
C4-C5	1.483 (2)
C5-C6	1.374 (3)
C5-C19	1.448 (2)
C6-C7	1.436 (2)
C7-C21	1.390 (3)
C7-C8	1.452 (3)
C8-C24	1.398 (3)
C8-C9	1.436 (2)
C9-C10	1.369 (2)
C10-C26	1.451 (2)
C10-C11	1.476 (2)
C11-C12	1.377 (2)
C11-C28	1.445 (2)
C12-C13	1.435 (2)
C13-C30	1.397 (2)
C13-C14	1.442 (2)
C14-C15	1.400 (2)
C15-C16	1.436 (3)
C15-C32	1.449 (3)
C16-C17	1.393 (3)
C16-C34	1.456 (3)
C17-C18	1.450 (3)
C18-C36	1.392 (3)
C18-C19	1.449 (3)
C19-C20	1.391 (3)
C20-C21	1.439 (3)
C20-C38	1.456 (3)
C21-C22	1.456 (2)
C22-C23	1.382 (3)
C22-C39	1.451 (3)
C23-C24	1.454 (3)
C23-C42	1.457 (3)
C24-C25	1.442 (3)
C25-C26	1.392 (3)
C25-C43	1.453 (3)
C26-C27	1.451 (3)
C27-C45	1.391 (3)
C27-C28	1.444 (3)
C28-C29	1.393 (3)
C29-C30	1.441 (3)
C29-C47	1.448 (2)
C30-C31	1.452 (2)
C31-C32	1.388 (3)
C31-C48	1.450 (2)
C32-C33	1.455 (2)
C33-C50	1.394 (3)
C33-C34	1.444 (3)
C34-C35	1.392 (3)
C35-C52	1.451 (3)
C35-C36	1.454 (3)
C36-C37	1.453 (3)
C37-C38	1.384 (3)
C37-C53	1.449 (3)
C38-C39	1.457 (3)
C39-C40	1.391 (3)
C40-C41	1.447 (3)
C40-C54	1.453 (3)
C41-C42	1.396 (3)
C41-C60	1.447 (2)
C42-C43	1.452 (3)
C43-C44	1.389 (3)
C44-C59	1.448 (3)
C44-C45	1.457 (3)
C45-C46	1.451 (3)
C46-C47	1.389 (3)
C46-C58	1.452 (3)
C47-C48	1.453 (3)
C48-C49	1.391 (3)
C49-C50	1.447 (3)
C49-C57	1.448 (3)
C50-C51	1.454 (2)
C51-C52	1.385 (3)
C51-C56	1.453 (3)
C52-C53	1.454 (3)
C53-C54	1.395 (3)
C54-C55	1.446 (2)
C55-C56	1.392 (2)
C55-C60	1.451 (3)
C56-C57	1.455 (2)
C57-C58	1.394 (3)
C58-C59	1.452 (3)
C59-C60	1.393 (3)
C61-C62	1.482 (2)
C62-C63	1.405 (2)
C62-C67	1.406 (2)
C63-C64	1.400 (2)
C63-C68	1.485 (2)
C64-C65	1.387 (3)
C64-H64	0.949 (18)
C65-C66	1.391 (3)
C65-H65	0.955 (19)
C66-C67	1.377 (3)

TABLE VI (Continued)

C66-H66	0.93 (2)
C67-H67	0.926 (19)
C68-C69	1.408 (2)
C68-C73	1.408 (2)
C69-C70	1.383 (2)
C70-C71	1.393 (3)
C70-H70	0.99 (2)
C71-C72	1.391 (2)
C72-C73	1.392 (2)
C72-H72	0.98 (2)
C74-H74A	0.99 (2)
C74-H74B	0.90 (3)
C74-H74C	1.05 (2)
C75-H75A	0.98 (2)
C75-H75B	0.93 (2)
C75-H75C	0.94 (2)
C76-H76A	0.98 (2)
C76-H76B	1.01 (2)
C76-H76C	1.06 (2)
S1-C100-S2	179.53 (18)
N1-O1-C1	110.18 (12)
C69-O2-C74	117.26 (14)
C71-O3-C75	115.88 (14)
C73-O4-C76	117.24 (14)
C61-N1-O1	110.76 (13)
O1-C1-C6	109.76 (13)
O1-C1-C9	110.02 (13)
C6-C1-C9	101.50 (13)
O1-C1-C2	104.67 (13)
C6-C1-C2	115.55 (14)
C9-C1-C2	115.38 (13)
C12-C2-C61	116.10 (13)
C12-C2-C3	100.45 (13)
C61-C2-C3	111.48 (13)
C12-C2-C1	115.13 (14)
C61-C2-C1	99.68 (13)
C3-C2-C1	114.71 (13)
C4-C3-C14	119.83 (15)
C4-C3-C2	123.48 (15)
C14-C3-C2	109.94 (14)
C3-C4-C17	120.20 (16)
C3-C4-C5	120.59 (15)
C17-C4-C5	107.22 (15)
C6-C5-C19	120.24 (16)
C6-C5-C4	120.46 (15)
C19-C5-C4	107.63 (15)
C5-C6-C7	119.47 (16)
C5-C6-C1	123.48 (15)
C7-C6-C1	109.61 (15)
C21-C7-C6	121.01 (17)
C21-C7-C8	120.13 (16)
C6-C7-C8	108.56 (15)
C24-C8-C9	121.12 (17)
C24-C8-C7	119.81 (16)
C9-C8-C7	108.37 (15)
C10-C9-C8	119.84 (16)

TABLE VI (Continued)

C10-C9-C1	123.25 (15)
C8-C9-C1	109.61 (15)
C9-C10-C26	119.82 (16)
C9-C10-C11	120.98 (15)
C26-C10-C11	107.72 (15)
C12-C11-C28	120.10 (16)
C12-C11-C10	120.49 (15)
C28-C11-C10	107.50 (15)
C11-C12-C13	119.63 (15)
C11-C12-C2	123.13 (15)
C13-C12-C2	109.99 (14)
C30-C13-C12	120.80 (16)
C30-C13-C14	120.15 (15)
C12-C13-C14	108.69 (14)
C15-C14-C3	120.72 (16)
C15-C14-C13	120.28 (15)
C3-C14-C13	108.42 (14)
C14-C15-C16	119.35 (16)
C14-C15-C32	119.61 (16)
C16-C15-C32	108.63 (15)
C17-C16-C15	119.65 (16)
C17-C16-C34	120.14 (17)
C15-C16-C34	107.80 (16)
C16-C17-C4	120.20 (16)
C16-C17-C18	119.89 (16)
C4-C17-C18	108.59 (15)
C36-C18-C19	119.86 (17)
C36-C18-C17	120.10 (17)
C19-C18-C17	108.25 (15)
C20-C19-C5	120.11 (17)
C20-C19-C18	120.08 (16)
C5-C19-C18	108.32 (15)
C19-C20-C21	119.62 (16)
C19-C20-C38	120.08 (17)
C21-C20-C38	107.94 (16)
C7-C21-C20	119.48 (16)
C7-C21-C22	120.07 (17)
C20-C21-C22	108.61 (16)
C23-C22-C39	120.64 (17)
C23-C22-C21	119.77 (17)
C39-C22-C21	107.60 (16)
C22-C23-C24	120.54 (17)
C22-C23-C42	119.90 (17)
C24-C23-C42	107.36 (16)
C8-C24-C25	119.06 (16)
C8-C24-C23	119.67 (17)
C25-C24-C23	108.66 (16)
C26-C25-C24	119.44 (16)
C26-C25-C43	119.81 (17)
C24-C25-C43	107.93 (16)
C25-C26-C10	120.65 (16)
C25-C26-C27	120.30 (16)
C10-C26-C27	107.72 (15)
C45-C27-C28	120.57 (17)
C45-C27-C26	119.74 (17)
C28-C27-C26	108.70 (15)

TABLE VI (Continued)

C29 – C28 – C27	119.67 (16)
C29 – C28 – C11	120.54 (16)
C27 – C28 – C11	108.35 (15)
C28 – C29 – C30	119.31 (15)
C28 – C29 – C47	119.89 (16)
C30 – C29 – C47	108.10 (16)
C13 – C30 – C29	119.52 (16)
C13 – C30 – C31	119.46 (16)
C29 – C30 – C31	108.38 (15)
C32 – C31 – C48	120.19 (16)
C32 – C31 – C30	120.40 (16)
C48 – C31 – C30	107.59 (15)
C31 – C32 – C15	120.09 (16)
C31 – C32 – C33	119.79 (17)
C15 – C32 – C33	107.61 (15)
C50 – C33 – C34	120.25 (16)
C50 – C33 – C32	119.69 (17)
C34 – C33 – C32	107.85 (16)
C35 – C34 – C33	119.87 (17)
C35 – C34 – C16	119.81 (17)
C33 – C34 – C16	108.11 (16)
C34 – C35 – C52	120.15 (17)
C34 – C35 – C36	120.00 (16)
C52 – C35 – C36	107.73 (16)
C18 – C36 – C37	119.95 (17)
C18 – C36 – C35	120.06 (17)
C37 – C36 – C35	108.38 (16)
C38 – C37 – C53	120.18 (17)
C38 – C37 – C36	120.30 (17)
C53 – C37 – C36	107.56 (16)
C37 – C38 – C20	119.73 (17)
C37 – C38 – C39	120.11 (16)
C20 – C38 – C39	107.86 (16)
C40 – C39 – C22	119.73 (17)
C40 – C39 – C38	119.82 (17)
C22 – C39 – C38	107.99 (15)
C39 – C40 – C41	119.88 (17)
C39 – C40 – C54	120.00 (17)
C41 – C40 – C54	107.97 (15)
C42 – C41 – C60	119.92 (17)
C42 – C41 – C40	120.50 (16)
C60 – C41 – C40	108.05 (16)
C41 – C42 – C43	119.94 (17)
C41 – C42 – C23	119.35 (17)
C43 – C42 – C23	108.07 (16)
C44 – C43 – C42	119.79 (17)
C44 – C43 – C25	120.18 (17)
C42 – C43 – C25	107.98 (16)
C43 – C44 – C59	120.38 (17)
C43 – C44 – C45	119.79 (17)
C59 – C44 – C45	107.73 (16)
C27 – C45 – C46	119.74 (17)
C27 – C45 – C44	120.17 (17)
C46 – C45 – C44	108.13 (16)
C47 – C46 – C45	119.72 (17)
C47 – C46 – C58	120.53 (17)

TABLE VI (Continued)

C45 – C46 – C58	107.85 (15)
C46 – C47 – C29	120.40 (17)
C46 – C47 – C48	119.57 (16)
C29 – C47 – C48	107.88 (15)
C49 – C48 – C31	120.28 (16)
C49 – C48 – C47	120.17 (16)
C31 – C48 – C47	108.05 (16)
C48 – C49 – C50	119.55 (16)
C48 – 49 – C57	120.03 (17)
C50 – C49 – C57	108.36 (16)
C33 – C50 – C49	120.50 (16)
C33 – C50 – C51	119.65 (17)
C49 – C50 – C51	108.03 (16)
C52 – C51 – C56	120.08 (16)
C52 – C51 – C50	120.19 (17)
C56 – C51 – C50	107.79 (15)
C51 – C52 – C35	119.90 (16)
C51 – C52 – C53	120.12 (17)
C35 – C52 – C53	107.93 (16)
C54 – C53 – C37	119.97 (16)
C54 – C53 – C52	119.78 (16)
C37 – C53 – C52	108.40 (16)
C53 – C54 – C55	119.99 (16)
C53 – C54 – C40	119.92 (16)
C55 – C54 – C40	107.97 (16)
C56 – C55 – C54	120.16 (17)
C56 – C55 – C60	119.87 (16)
C54 – C55 – C60	107.95 (15)
C55 – C56 – C51	119.87 (16)
C55 – C56 – C57	120.08 (17)
C51 – C56 – C57	108.04 (15)
C58 – C57 – C49	120.11 (16)
C58 – C57 – 56	120.05 (16)
C49 – C57 – C56	107.79 (16)
C57 – C58 – C46	119.59 (16)
C57 – C58 – C59	119.71 (16)
C46 – C58 – C59	108.05 (16)
C60 – C59 – C44	119.79 (16)
C60 – C59 – C58	120.23 (17)
C44 – C59 – C58	108.23 (16)
C59 – C60 – C41	120.16 (17)
C59 – C60 – C55	120.06 (16)
C41 – C60 – C55	108.06 (15)
N1 – C61 – C62	120.16 (15)
N1 – C61 – C2	114.49 (15)
C62 – C61 – C2	124.91 (14)
C63 – C62 – C67	119.97 (15)
C63 – C62 – C61	123.54 (15)
C67 – C62 – C61	116.48 (15)
C64 – C63 – C62	118.03 (15)
C64 – C63 – C68	119.57 (15)
C62 – C63 – C68	122.40 (15)
C65 – C64 – C63	121.43 (16)
C65 – C64 – H64	119.3 (10)
C63 – C64 – H64	119.2 (10)
C64 – C65 – C66	120.14 (17)

TABLE VI (Continued)

C64–C65–H65	120.2 (11)
C66–C65–H65	119.6 (11)
C67–C66–C65	119.48 (16)
C67–C66–H66	119.9 (12)
C65–C66–H66	120.5 (12)
C66–C67–C62	120.93 (17)
C66–C67–H67	122.6 (12)
C62–C67–H67	116.4 (12)
C69–C68–C73	116.82 (15)
C69–C68–C63	122.58 (15)
C73–C68–C63	120.59 (15)
O2–C69–C70	122.96 (15)
O2–C69–C68	114.64 (14)
C70–C69–C68	122.38 (16)
C69–C70–C71	118.45 (16)
C69–C70–H70	120.8 (11)
C71–C70–H70	120.4 (11)
O3–C71–C72	122.54 (16)
O3–C71–C70	115.65 (15)
C72–C71–C70	121.77 (16)
C71–C72–C73	118.37 (16)
C71–C72–H72	121.5 (12)
C73–C72–H72	120.1 (12)
O4–C73–C72	122.15 (15)
O4–C73–C68	115.70 (15)
C72–C73–C68	122.11 (16)
O2–C74–H74A	108.1 (13)
O2–C74–H74B	104.5 (14)
H74A–C74–H74B	112 (2)
O2–C74–H74C	111.6 (12)
H74A–C74–H74C	111.8 (18)
H74B–C74–H74C	108.3 (19)
O3–C75–H75A	102.8 (13)
O3–C75–H75B	112.1 (12)
H75A–C75–H75B	109.5 (18)
O3–C75–H75C	111.6 (12)
H75A–C75–H75C	110.5 (18)
H75B–C75–H75C	110.1 (18)
O4–C76–H76A	105.3 (12)
O4–C76–H76B	109.5 (11)
H76A–C76–H76B	109.7 (16)
O4–C76–H76C	111.1 (12)
H76A–C76–H76C	112.2 (18)
H76B–C76–H76C	108.9 (17)

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