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Journal of Organometallic Chemistry 531 (1997) 41–46

Journal of Organometallic Chemistry

Reactivity of the maleimidato ligand in CpFe(CO)$_2$(η^1-N-maleimidato) in Diels–Alder reaction with 1,3-diphenylisobenzofuran. The crystal structure of [CpFe(CO)$_2$(η^1-N-1,4-diphenyl-1,4-epoxy-1,4-dihydronaphthalene-2,3-dicarboxyimidato)]

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Received 8 May 1996; revised 25 July 1996

Abstract

The title complex reacts with 1,3-diphenylisobenzofuran to afford the Diels–Alder adduct [CpFe(CO)$_2$(η^1-N-1,4-diphenyl-1,4-epoxy-1,4-dihydronaphthalene-2,3-dicarboxyimidato)] **2** in nearly quantitative yield. The competitive experiment showed that this reaction is around three times slower than the reaction of the parent maleimide with 1,3-diphenylisobenzofuran. The X-ray crystal structure of **2** has been determined. It revealed a scrolled configuration resulting from an *endo* transition state in Diels–Alder reaction and from various hydrogen bonds and π–π interactions.

Keywords: Diels–Alder reaction; Crystal structure; Maleimides

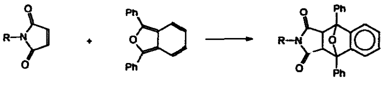
1. Introduction

We have recently reported the synthesis of CpFe(CO)$_2$(η^1-N-maleimidato) **1** (Cp = η^5-cyclopentadienyl) [1]. As maleimides are among the most reactive dienophiles in Diels–Alder reactions [2], we thought that it would be interesting to look at the reactivity of this system complexed to the CpFe(CO)$_2$ moiety. We chose as a diene 1,3-diphenylisobenzofuran.

It is known that this diene reacts with maleimides under mild conditions and affords the products of the [4 + 2] cycloaddition in high yield [2]. We were interested in the comparison of the reactivity towards 1,3-diphenylisobenzofuran of **1** with that of the parent, unsubstituted maleimide by using a competitive reaction approach. Finally, to gain a deeper insight into the stereochemistry of the Diels–Alder adduct formed, we have determined its X-ray crystal structure.

2. Results and discussion

We have found that **1** reacts with 1,3-diphenylisobenzofuran in chloroform solution at room temperature almost instantaneously (Eq. (1)). The adduct **2** [CpFe(CO)$_2$(η^1-N-1,4-diphenyl-1,4-epoxy-1,4-dihydronaphthalene-2,3-dicarboxyimidato)] was isolated in 98% yield and its structure was confirmed by elemental analysis, IR and ^{1}H NMR spectra.



2 R = CpFe(CO)$_2$
3 R = H

(1)

Under the same conditions we reacted maleimide with 1,3-diphenylisobenzofuran and obtained **3** in 99% yield (structure confirmed by IR, ^{1}H NMR and elemental analysis).

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PII S0022-328X(96)06673-9

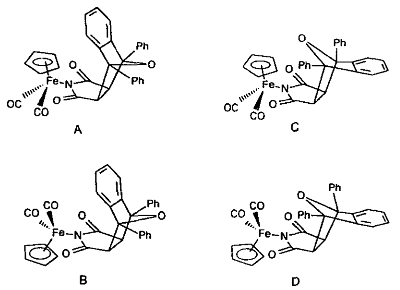


Fig. 1. Possible stereoisomers of **2**.

When an equimolar mixture of **1** and maleimide reacted with 1 equiv. of 1,3-diphenylisobenzofuran, a mixture of **2**, **3** and the starting maleimido compounds was obtained. Analysis of this mixture by means of ^{1}H NMR (at 200 MHz) showed that the ratio of unreacted **1** to maleimide was around 2.7:1. Similarly, the ratio of the products **2** and **3** formed was found to be around 3.0:1. This leads to the conclusion that **1** reacts with 1,3-diphenylisobenzofuran around three times more slowly than the parent maleimide.

Diels–Alder reactions are usually controlled by interactions of frontier molecular orbitals of both partners [2]. The decrease in reaction rate on going from maleimide to **1** can be explained by assuming that the $CpFe(CO)_2$ group behaves as an electon-donating substituent and raises the energy of LUMO of the ethylenic bond in the maleimidato ligand. In 'normal electron demand' Diels–Alder reactions (i.e. reactions controlled by HOMO (diene)–LUMO (dienophile) interactions), this should slow down the reaction. An indication that the $CpFe(CO)_2$ group really donates electron density strongly towards the maleimidato ligand is provided by comparison of the IR stretching frequencies of imide carbonyls in maleimide (1730 cm^{-1}) and in **1** (1655 cm^{-1}) (both values measured in chloroform solution).

One can consider four different stereoisomers of **2** resulting from different transition states of the Diels–Alder reaction (*exo* vs. *endo*) and from the rotation around the iron–nitrogen bonds. They are shown in Fig. 1. If one assumes that the investigated reaction proceeds, similarly to other Diels–Alder reactions studied up to now, in the *endo* manner, one can reduce the number of possible structures to two (**A** and **B**). However, the isolated product showed a ^{1}H NMR spectrum consistent with the presence of only one stereoisomer or with fast rotation around the Fe–N axis (single resonances for Cp and for the bridgehead protons 1,4). Interestingly, the signal of the Cp protons in **2** (4.57 ppm) is significantly shifted upfield in comparison with the same signals in **1** (5.06 ppm) [1] and $CpFe(CO)_2(\eta^1$-N-succinimidato) (5.02 ppm) [3]. This might suggest the influence of the aromatic ring current effect as in structure **A**, in which the Cp ring lies in the ring current zone of the benzo ring. Consequently, the likely conformation of **2** in solution seems to be **A**.

2.1. X-ray crystal structure of 2

Unambiguous evidence for coordination of the $CpFe(CO)_2$ moiety to deprotonated N(1) is provided by the X-ray crystal structure of **2**. A perspective view of this complex is shown in Fig. 2, together with the atomic numbering. Crystal data are listed in Table 1, final atomic coordinates in Table 2, and selected bond lengths and angles in Table 3.

The coordination of the iron atom gives rise to a 'three-legged piano stool' configuration. According to Davies and coworkers [4], such complexes can most often be described in terms of a pseudo-octahedral arrangement of the ligands around the iron atom. The crucial structural features of pseudo-octahedral coordination are the three bond angles between M–(piano leg) bonds, close to 90°, and the three angles between M–(piano leg) bonds and the line M–Cp^{cent} (where Cp^{cent} denotes the centroid of a cyclopentadienyl group), close to 125°. In **2** these angles range from 91.4(1) to 95.4(2)° and from 121.6(4) to 124.4(4)° respectively. The geometry of the $CpFe(CO)_2$ moiety is similar to that observed in other related complexes [9,10]. The cyclopentadienyl ring is nearly planar (within a 3σ range) and the Fe–Cp^{cent} plane distance of 1.725(2) Å is only slightly longer than the reported average value of 1.71 Å [5]. The heterocyclic ligand in **2** is η^1-N(1)-coordinated to the iron atom, with an Fe–N(1) bond of 1.970(2) Å.

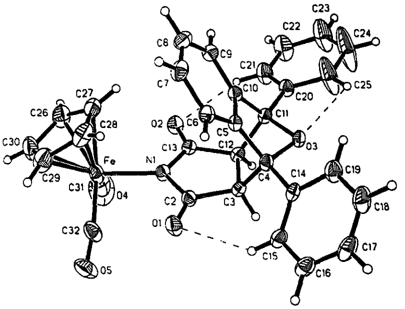


Fig. 2. The thermal ellipsoid plot of the molecule of **2** and intramolecular hydrogen bonds. Atomic displacement parameters are shown at the 30% probability level.

This bond is nearly coplanar with the N(1)–C(2) and N(1)–C(13) bonds. The complexes $CpFe(CO)_2L$, where L is a nitrogen donor cyclic ligand, have not been investigated much. A literature search revealed only five structures of this type. The reported Fe–N distances are 1.979(7) Å [6], 1.970(7) Å [7], 1.98(3) Å [8], 1.956(3) Å [9] and 1.991(4) Å [10] for L = 1,2,3-triazole, benzimidazole, pyridine, phthalimide anion and ftorafur anion respectively. The Fe–N(1) bond of 1.970(2) Å in **2** is consistent with the average value of these bonds observed for the first four cited complexes. As we can see from Fig. 2, the polycyclic η^1-N ligand adopts a scrolled conformation with the interplanar angles given in Fig. 3. Such a conformation results undoubtedly from the *endo* transition state in the Diels–Alder reaction. The succinimide ring II is ideally planar. The torsion angles of tetrahydrofuran III and dihydrofuran IV rings are given in Fig. 4. Both rings have the envelope conformations and, as would be expected, the tetrahydrofuran ring is more puckered ($|\varphi|_{av} = 37.6°$) than the dihydrofuran ring ($|\varphi|_{av} = 33.8°$). The observed scrolled shape of the ligand gives an example of the distinct influence of the weak hydrogen bonds on the molecular conformation and configuration. Two C–H · · · O hydrogen bonds bind the phenyl ring VIII with oxygen atom O3 of the furan and O2 of the phtalimide ring. The phenyl ring VII forms only one intramolecular hydrogen bond C15–H · · · O1. The geometry of these bonds is given in Table 3. In the investigated molecule **2** the cyclopentadienyl ring I and the benzo ring VI adopt the *cis* configuration with respect to the succin-

Table 1
Crystal data and structure refinement [a]

(a) Crystal parameters	
Empirical formula	$C_{31}H_{21}FeNO_5$
Formula weight	543.34
Crystal system	monoclinic
Crystal color	red
Crystal size (mm^3)	0.15 × 0.2 × 0.2
Space group	$P2_1/n$
a (Å)	13.037(2)
b (Å)	11.926(2)
c (Å)	16.409(1)
β (°)	99.54(1)
V (Å^3)	2516.0(6)
Z	4
D_c ($Mg\,m^{-3}$)	1.434
$F(000)$	1120
μ(Mo Kα) (mm^{-1})	0.643
T (K)	293
(b) Data collection and refinement	
Theta range for data collection	$1.85° < \theta < 27.56°$
Data collected (h, k, l)	$-16 \le h \le 16, 0 \le k \le 15, 0 \le l \le 21$
Reflections collected	6209
Independent observed reflections	5819
Refinement method	Full-matrix least-squares on F^2
Extinction coefficient	0.0021(4)
Final R [b]	0.0468
Δ/σ (max)	0.05
$\Delta\rho_{min}$ (e Å^{-3})	−0.38
$\Delta\rho_{max}$ (e Å^{-3})	0.334
N_o/N_v	16.9
Goodness-of-fit [c]	0.969
Weighting scheme	$w^{-1} = \sigma^2(F_o) + (0.0605P)^2 + 0.64P$ $P = (\max(F_o^2, 0) + 2F_c^2)/3$

[a] Siemens P3 diffractometer; graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å).

[b] $R = \Sigma|F_o - F_c|/\Sigma F_o$.

[c] Goodness-of-fit $= [\Sigma w|F_o^2 - F_c^2|^2/(N_o - N_v)]^{1/2}$, where N_o is the number of observations and N_v the number of variables.

Table 2
Atomic coordinates for Fe, N, O, C and U_{eq}

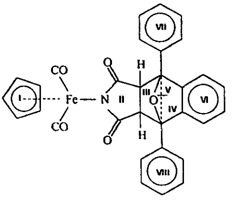
Atom	x	y	z	U_{eq}
Fe	0.41021(3)	0.24855(3)	0.47984(3)	0.0552(2)
N(1)	0.2700(2)	0.1825(2)	0.46747(14)	0.0423(5)
O(1)	0.31773(15)	0.0050(2)	0.51242(14)	0.0640(6)
O(2)	0.17429(15)	0.3369(2)	0.41750(14)	0.0574(5)
O(3)	−0.00572(12)	0.01292(14)	0.36704(11)	0.0427(4)
O(4)	0.3508(3)	0.4371(3)	0.5750(3)	0.1416(15)
O(5)	0.5018(2)	0.1139(2)	0.6218(2)	0.0936(9)
C(2)	0.2506(2)	0.0731(2)	0.4861(2)	0.0415(6)
C(3)	0.1352(2)	0.0490(2)	0.4686(2)	0.0383(6)
C(4)	0.0984(2)	−0.0324(2)	0.3951(2)	0.0382(6)
C(5)	0.1527(2)	0.0027(2)	0.3239(2)	0.0382(6)
C(6)	0.2346(2)	−0.0407(2)	0.2914(2)	0.0453(7)
C(7)	0.2658(2)	0.0161(3)	0.2245(2)	0.0535(7)
C(8)	0.2176(2)	0.1136(3)	0.1952(2)	0.0561(8)
C(9)	0.1368(2)	0.1596(2)	0.2301(2)	0.0473(7)
C(10)	0.1050(2)	0.1027(2)	0.2940(2)	0.0388(6)
C(11)	0.0248(2)	0.1267(2)	0.3494(2)	0.0403(6)
C(12)	0.0862(2)	0.1606(2)	0.4362(2)	0.0398(6)
C(13)	0.1791(2)	0.2389(2)	0.4379(2)	0.0410(6)
C(14)	0.0943(2)	−0.1561(2)	0.4128(2)	0.0412(6)
C(15)	0.1568(2)	−0.2046(2)	0.4795(2)	0.0493(7)
C(16)	0.1525(2)	−0.3196(2)	0.4929(2)	0.0577(8)
C(17)	0.0865(3)	−0.3860(3)	0.4413(3)	0.0676(10)
C(18)	0.0245(3)	−0.3389(3)	0.3738(3)	0.0723(10)
C(19)	0.0291(2)	−0.2246(2)	0.3590(2)	0.0589(8)
C(20)	−0.0678(2)	0.1963(2)	0.3140(2)	0.0491(7)
C(21)	−0.0637(2)	0.3101(3)	0.3115(2)	0.0686(10)
C(22)	−0.1481(3)	0.3741(3)	0.2772(3)	0.0922(13)
C(23)	−0.2369(3)	0.3257(4)	0.2443(4)	0.134(2)
C(24)	−0.2438(4)	0.2131(4)	0.2478(6)	0.214(4)
C(25)	−0.1592(3)	0.1474(3)	0.2827(4)	0.155(3)
C(26)	0.4479(3)	0.3580(3)	0.3890(3)	0.0835(12)
C(27)	0.3957(3)	0.2668(3)	0.3515(2)	0.0735(10)
C(28)	0.4521(3)	0.1708(3)	0.3760(3)	0.0826(11)
C(29)	0.5415(3)	0.2020(4)	0.4293(3)	0.0973(15)
C(30)	0.5394(3)	0.3194(3)	0.4400(3)	0.0948(14)
C(31)	0.3725(3)	0.3617(3)	0.5384(3)	0.0895(13)
C(32)	0.4604(3)	0.1645(3)	0.5665(2)	0.0705(10)
Calculated coordination of the center of the C_5H_5 ring				
Cp^{cent}	0.4753	0.2634	0.3972	

Table 3
Selected bond lengths (Å) and angles (°)

(a) Fe coordination sphere					
Fe–N(1)	1.970(2)	C(31)–Fe–N(1)	91.4(1)	Fe–N(1)–C(13)	124.7(2)
Fe–C(31)	1.773(4)	C(32)–Fe–N(1)	94.0(1)	Cp^{cent}–Fe–N(1)	122.0(4)
Fe–C32	1.774(4)	C(31)–Fe–C(32)	95.4(2)	Cp^{cent}–Fe–C(31)	124.4(4)
Fe–Cp^{cent}	1.725(2)	Fe–N(1)–C(2)	124.0(2)	Cp^{cent}–Fe–C(32)	121.6(4)
(b) (η^1-N-$C_{24}H_{16}NO_3$) ligand					
N(1)–C(2)	1.373(3)	C(2)–N(1)–C(13)	111.3(2)	O(3)–C(11)–C(20)	111.3(2)
N(1)–C(13)	1.379(3)	C(4)–O(3)–C(11)	97.8(2)	O(3)–C(11)–C(10)	100.4(2)
O(1)–C(2)	1.219(3)	O(1)–C(2)–N(1)	124.3(2)	C(20)–C(11)–C(10)	117.5(2)
O(2)–C(13)	1.215(3)	O(1)–C(2)–C(3)	125.0(2)	O(3)–C(11)–C(12)	100.0(2)
O(3)–C(4)	1.463(3)	N(1)–C(2)–C(3)	110.6(2)	C(20)–C(11)–C(12)	117.8(2)
O(3)–C(11)	1.457(3)	C(2)–C(3)–C(12)	104.2(2)	C(10)–C(11)–C(12)	107.1(2)
O(4)–C(31)	1.142(4)	C(2)–C(3)–C(4)	115.7(2)	C(13)–C(12)–C(3)	104.0(2)
O(5)–C(32)	1.147(4)	C(12)–C(3)–C(4)	102.4(2)	C(13)–C(12)–C(11)	117.2(2)
C(2)–C(3)	1.512(3)	O(3)–C(4)–C(14)	111.2(2)	C(3)–C(12)–C(11)	102.2(2)
C(3)–C(12)	1.532(3)	O(3)–C(4)–C(5)	100.5(2)	O(2)–C(13)–N(1)	124.7(2)
C(3)–C(4)	1.560(4)	C(14)–C(4)–C(5)	117.0(2)	C(26)–C(27)–C(28)	109.0(3)
C(4)–C(14)	1.506(3)	O(3)–C(4)–C(3)	99.4(2)	N(1)–C(13)–C(12)	109.9(2)
C(4)–C(5)	1.521(3)	C(14)–C(4)–C(3)	118.5(2)		
C(10)–C(11)	1.521(3)	C(5)–C(4)–C(3)	107.4(2)		
C(11)–C(12)	1.567(4)	C(10)–C(5)–C(4)	105.6(2)		
C(12)–C(13)	1.527(3)	C(5)–C(10)–C(11)	105.1(2)		
(c) Carbonyl groups					
C(31)–O(4)	1.142(4)	Fe–C(31)–O(4)	177.4(4)		
C(32)–O(5)	1.147(4)	Fe–C(32)–O(5)	173.6(3)		
(d) Hydrogen bonds					
D–H···A	H···A	D···A	D–H···A		
C(15)–H(15)···O(1)	2.49	3.250(4)	139		
C(21)–H(21)···O(2)	2.48	3.309(5)	146		
C(25)–H(25)···O(3)	2.40	2.753(6)	103		
C(29)–H(29)···O(1)^{i}	2.24	3.132(6)	160		

Symmetry code: ^{i} $1-x, -y, 1-z$.

imide ring plane II. This gives occasion to a nearly perpendicular localization of the cyclopentadienyl ring I and the C5–C10 benzo ring VI (dihedral angle I–VI = 84.3°) with close van der Waals contacts between H27 and C8/C9 atoms (H27–C8 = 2.94, H27–C9 = 3.00 Å). The distance of H27 to the mean plane of VI is 2.88 Å, and to the center of VI 3.20 Å. This means that H27 is located more closely to the π-cloud at the periphery of the benzo ring than to its center. This type of hydrogen bonding interaction with the π-electrons of aromatic systems has already been observed, and seems to be as common as hydrogen bondings directed to the center of the aromatic ring [11].



I - II	32.9(1)	III - V	59.0(2)
II - III	58.2(1)	IV - V	53.0(2)
II - IV	68.0(1)	IV - VI	2.3(1)
		VII - VIII	29.6(1)

Fig. 3. The numbering of the selected planes and the observed interplanar angles (°) in 2.

The C27–H27 bond is inclined at 35° to the plane of VI, so the observed interaction is intermediate between offset π–π interaction and edge–face interaction [12,13].

Fig. 5 shows the intermolecular hydrogen bonds and mutual orientation of aromatic rings.

In conclusion, both ^{1}H NMR solution study and X-ray crystallographic results lead to the conlusion that the preferred conformation of 2 is the scrolled conformation A. This conformation corroborates the *endo* transition state of Diels–Alder reaction and is stabilized

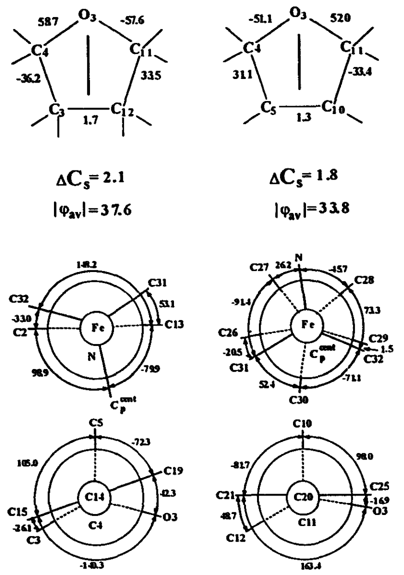


Fig. 4. The conformation of tetrahydrofuran (III) and dihydrofuran (IV) rings and selected Newman projections in 2.

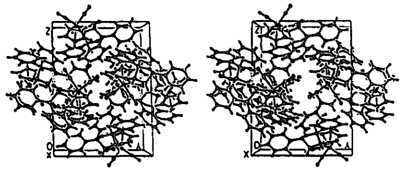


Fig. 5. Molecular packing of 2.

by several types of hydrogen bond and π–π interaction.

3. Experimental

All reactions with organoiron complexes were carried out under argon. Chloroform was distilled over calcium hydride thoroughly before use. The starting complex **1** was prepared according to the earlier published procedure [1]. Other chemicals were purchased from Aldrich and used as received. ^{1}H NMR spectra were taken on a Varian Gemini 200 BB spectrometer at 200 MHz in chloroform solution containing TMS as internal standard.

IR spectra were run on a Specord 75 IR spectrometer.

3.1. Diels–Alder reation of 1 with 1,3-diphenylisobenzofuran

CpFe(CO)$_2$(η^1-N-maleimidato) (**1**) (82 mg, 0.3 mmol) and 1,3-diphenylisobenzofuran (81 mg, 0.3 mmol) were dissolved in chloroform (5 ml). TLC showed instantaneous formation of the product. The reaction mixture was concentrated and chromatographed on silica gel 60 (Merck, 230–400 mesh ASTM), using chloroform as eluent to afford **2** as yellow crystals. Yield 159 mg (98%). IR (CHCl$_3$, ν [cm^{-1}]): 2040, 2000 (Fe–CO), 1640 (imide CO). ^{1}H NMR (CDCl$_3$, δ [ppm]): 4.10 (s, 2H) bridgehead H, 4.57 (s, 5H) cyclopentadienyl, 7.05 (m, 4H) and 7.45 (m, 6H) aromatic H, 8.10 (m, 4H) aromatic *ortho* H. Anal. Found: C, 68.54; H, 4.21; N, 2.59. $C_{31}H_{21}FeNO_5$ Calc.: C, 68.53; H, 3.94; N, 2.58%.

3.2. Diels–Alder reaction of maleimide with 1,3-diphenylisobenzofuran

This reaction was carried out analogously to that described above to afford **3** as colorless crystals from chloroform–hexane in 99% yield. As shown by elemental analysis and ^{1}H NMR spectrum in dmso-d_6, this material retained residual chloroform (0.2 molecule). IR (CHCl$_3$, ν [cm^{-1}]): 1720 (CO imide). ^{1}H NMR (CDCl$_3$, δ [ppm]): 4.15 (s, 2H) 1,4-bridgehead H, 7.1 (m, AA′BB′, 4H) benzo ring H, 7.53 (m, 6H) *meta*- and *para*-H of Ph rings, 8.0 (m, 5H) *ortho*-H of Ph rings. Anal. Found: C, 74.10; H, 4.67; N, 3.49; Cl, 5.19. $C_{24}H_{17}NO_3 \cdot 0.2CHCl_3$ Calc.: C, 74.29; H, 4.43; N, 3.58; Cl, 5.44%.

3.3. Competitive reaction of 1 and maleimide with 1,3-diphenylisobenzofuran

1 (196 mg, 0.72 mmol) and maleimide (69 mg, 0.72 mmol) were dissolved in chloroform (4 ml). To this solution a solution of 1,3-diphenylisobenzofuran (197 mg, 0.72 mmol) was added with vigorous stirring. TLC showed the presence of **2** and **3** together with unreacted **1** and maleimide. The solvent was evaporated, a part of the residue dissolved in CDCl$_3$ and analyzed by ^{1}H NMR.

4. Crystal structure analysis

Crystals of **2** suitable for X-ray study were grown from layered chloroform–hexane during two days. Crystal data and details of data collection and refinement are listed in Table 1. The intensities were corrected for Lorentz and polarization effects. An empirical correction was made for absorption [14] using a Ψ scan around the diffraction vectors of eight selected reflections. The structure was solved by direct methods, and refined by the full-matrix least-squares method using the SHELXL programs [15,16]. All non-hydrogen atoms were refined anisotropically. All H atoms were located in the difference map at $R = 0.05$ but, since their isotropic refinement was not successful, they were placed in calculated positions and refined isotropically by the 'riding mode'. A complete table of atomic coordinates, anisotropic displacement parameters, bond lengths and angles, observed and calculated structure factors is available from the authors.

Acknowledgements

This work was supported by Grant 477 from the University of Łódź and Grant I17/Dz.St./49 from the Technical University of Łódź.

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