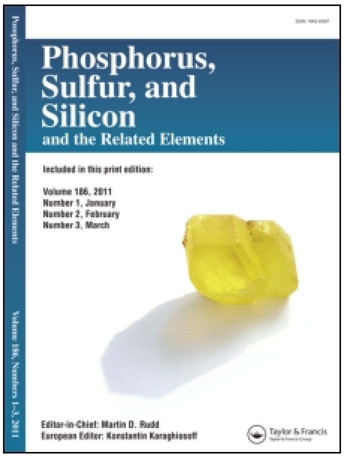

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Preparation and Characterization of Methyl Substituted 2,2'-Dipyridyl Diselenides and -Ditellurides: X-ray Structure of 6,6'-Dimethyl-2,2'-dipyridyl Diselenide

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PREPARATION AND CHARACTERIZATION OF METHYL SUBSTITUTED 2,2′-DIPYRIDYL DISELENIDES AND -DITELLURIDES: X-RAY STRUCTURE OF 6,6′-DIMETHYL-2,2′-DIPYRIDYL DISELENIDE

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A convenient method for the preparation of various methyl substituted 2,2′-dipyridyl diselenides and -ditellurides by the aerial oxidation of lithium 2-pyridylselenolate/tellurolate, prepared from the lithium-halogen exchange between n-butyllithium and 2-bromo methyl substituted pyridines is reported. All the compounds prepared are new and have been characterized by elemental analysis, IR, ^{1}H, ^{13}C, ^{11}Se NMR, and mass spectral studies. Crystal structure of 6,6′-dimethyl-2,2′-dipyridyl diselenide has been determined.

Keywords: Organochalcogens; selenides; selenium; tellurides; tellurium

INTRODUCTION

Organoselenium and tellurium compounds have emerged recently as important reagents and intermediates in organic synthesis,[1,2] accordingly, development of convenient methods for the synthesis of these versatile compounds starting from readily available materials has always remained a subject of current interest.[3] In contrast to alkyl, aryl, and mixed alkylaryl selenium/tellurium compounds, very little is known about the pyridyl selenium/tellurium compounds. In continuation of

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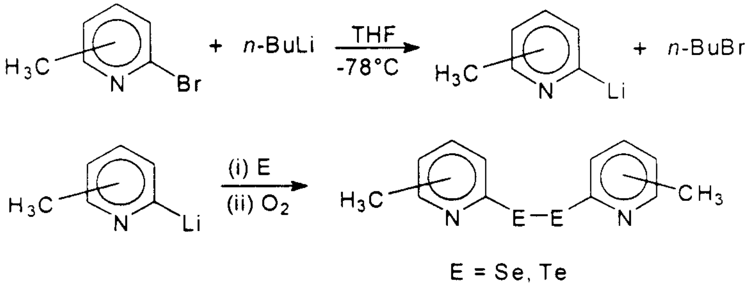
our research[4,5] on the chemistry of organochalcogen compounds, we report in this article on the preparation and characterization of the hitherto unknown stable methyl substituted 2,2′-dipyridyl diselenides and -ditellurides along with the crystal structure of 6,6′-dimethyl-2,2′-dipyridyl diselenide **1**.

RESULTS AND DISCUSSION

Mautner et al.[6] were the first to synthesise 2,2′-dipyridyl diselenide by reacting 2-bromo pyridine with hydrogen selenide (generated by addition of dilute hydrochloric acid to aluminium selenide). This method involves the use of highly poisonous hydrogen selenide gas. A need was, therefore, felt to develop a safe and convenient method for its preparation and subsequently the method was improved upon by reducing elemental selenium with sodium borohydride in various solvents by various workers. All the workers developed different methodologies in the subsequent years[7] and explored variations of the published procedures to improve on the yield of 2,2′-dipyridyl diselenide. We have now developed and optimized the conditions for the preparation of hitherto unknown stable methyl substituted 2,2′-dipyridyl diselenide and -ditelluride by a different route, which avoids the use of toxic gases and afford better yields.

The method involves the bromine exchange of 2-bromo methyl substituted pyridine using *n*-butyllithium in THF at −78°C as depicted below.



The reactions were carried out in the presence of oxygen-free nitrogen. Addition of elemental selenium/tellurium to the lithiated pyridine was made at −78°C and later allowed to come to room temperature. It took nearly 20 min to dissolve elemental selenium whereas it took more than 45 min to dissolve elemental tellurium. This is probably because commercially available tellurium is known to exhibit low chemical reactivity due to its surface oxidation.[8] Aerial oxidation of the above lithium-2-pyridyl selenolate/tellurolate solutions resulted in the formation of the methyl substituted 2,2′-dipyridyl diselenides and ditellurides respectively. The yields obtained in case of diselenides were higher than in case of the ditellurides.

The diselenides and -ditellurides thus prepared are stable enough to be purified by column chromatography (silica gel using hexane-ethylacetate) on a laboratory bench. The diselenides are light yellow colored crystalline compounds whereas the corresponding ditellurides are deep red except 3,3′-dimethyl-2,2′-dipyridyl ditelluride which is extremely deep blue in color. All compounds are soluble in conventional organic solvents. The diselenides and -ditellurides have a shelf life of several months without any sign of decomposition even at room temperature.

^{1}H NMR of the diselenides and ditellurides display two sets of protons, one corresponding to the methyl group attached to the ring and the other corresponding to three types of protons attached to the ring itself. Methyl protons in case of diselenides appear as a singlet in the range 2.0–2.05 (δ ppm) whereas in case of ditellurides in the range 2.27–2.48 (δ ppm). The protons attached to the pyridine ring appear in the range 6.63–8.29 (δ ppm) in these compounds. ^{13}C NMR shows a set of six signals one corresponding to the methyl carbon and five corresponding to the carbon of the pyridine ring. Selenium atom is very sensitive to extremely small electronic and steric changes. Therefore its chemical shifts are suitable tool to probe its chemical environment within the molecule. ^{77}Se (completely decoupled) NMR of methyl substituted 2,2′-dipyridyldiselenides (**1**, **2** and **4**) were recorded in $CDCl_3$ employing selenic acid as an external reference resonating at 0 (δ ppm). In 6,6′-dimethyl-2,2′-dipyridyldiselenide, the selenium signal was observed at −576.767 (δ ppm) whereas in 5,5′-dimethyl-2,2′-dipyridyldiselenide, it resonated at −584.469 (δ ppm) and in 3,3′-dimethyl-2,2′-dipyridyldiselenide, it was observed at −594.362 (δ ppm) relative to selenic acid. Thus it appears that the presence of the methyl group in 3 position (ortho to selenium) reduces the interaction between the selenium lone pairs and the π-electron system of the pyridine ring, which results in the shielding of the selenium nucleus. On the other hand the presence of the methyl group at 6 position increases the interaction between the selenium lone pairs and the π-electron system of the pyridine ring and thus deshields the selenium with respect to 5,5′-dimethyl-2,2′-dipyridyldiselenide. The fragmentation pattern of all the diselenides and ditellurides show molecular-ion peak (M^+) at m/e **344** and **444** respectively. The base-ion peak in case of diselenides was observed at 183 whereas in case of ditellurides it was observed at 92.

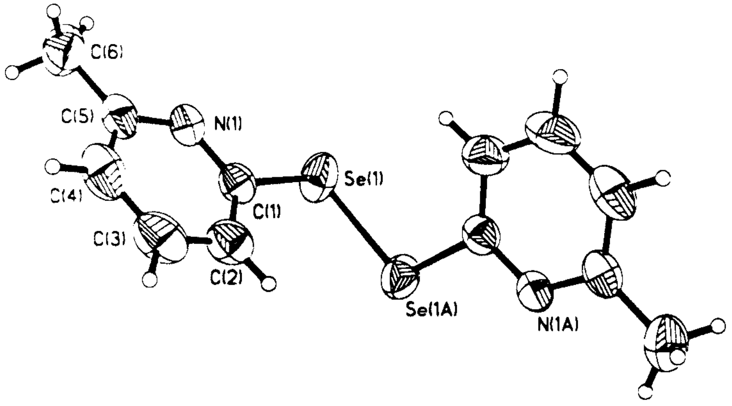


FIGURE 1 A perspective view of structure of **1** with atom numbering scheme (thermal ellipsoids are at 40% probability level).

Solid State Structural Feature of 6,6′-Dimethyl-2,2′-dipyridyldiselenide (1)

The crystals of **1** were obtained by the slow evaporation of mixed solvent of hexane-ethylacetate. In order to understand structural details, single crystal x-ray diffraction study was carried out. A perspective view of structure of **1** with atom numbering scheme is given in Figure 1 with bond lengths and angles in Table I.

In the crystal lattice the molecule is located on a two fold axis with Se(1)-Se(1)#1 bond length 2.2935(11)Å and angle C(1)-Se(1)-Se(1)#1 104.47°(14). These values are comparable with the values found in other aromatic diselenides.[9] Each pyridine ring in the molecule is substituted

TABLE I Selected Bond Distances (Å) and Angles (°) for Compound **1**

Se(1)-C(1)	1.929(5)	Se(1)-Se(1)#1	2.2935(11)
N(1)-C(1)	1.314(6)	N(1)-C(5)	1.343(6)
C(1)-C(2)	1.380(7)	C(2)-C(3)	1.358(8)
C(3)-C(4)	1.374(9)	C(4)-C(5)	1.378(8)
C(5)-C(6)	1.484(8)		
C(1)-Se(1)-Se(1)#1	104.47(14)	C(1)-N(1)-C(5)	118.9(4)
N(1)-C(1)-C(2)	124.5(4)	N(1)-C(1)-Se(1)	109.8(3)
C(2)-C(1)-Se(1)	125.6(4)	C(3)-C(2)-C(1)	116.5(5)
C(2)-C(3)-C(4)	120.1(5)	C(3)-C(4)-C(5)	120.1(5)
N(1)-C(5)-C(4)	119.8(5)	N(1)-C(5)-C(6)	117.0(4)
C(4)-C(5)-C(6)	123.2(5)		

Symmetry transformations used to generate equivalent atoms: #1 $-x+1, y, -z+1/2$.

at 2 position [Se—C bond length 1.929(5)Å] and at 6 position [H_3C—C bond length 1.484(8)Å]. The molecular geometry of the pyridine ring is normal in terms of bond lengths and angles. It is worth mentioning that Se-Se is coplanar with both the pyridine rings as can be seen from the torsion angle Se(1A)-Se(1)-C(1)-C(2) of −1.8(4). Dihedral angle between two pyridine rings is 90.1°, which are orthogonal to each other. Short contact is observed between Se(1) and H(2)#1 (2.957).

EXPERIMENTAL

All the reactions were carried out in dry oxygen-free nitrogen atmosphere. Solvents were dried and distilled freshly under nitrogen. Elemental selenium (Aldrich), tellurium (Aldrich) and 2-amino methyl substituted pyridines (Lancaster) were used as received. 2-Bromo methyl substituted pyridines were prepared by an adapation of the Craig's method.[10] The melting points reported are uncorrected. NMR spectra were recorded in $CDCl_3$ using TMS as an internal standard on Bruker AC-300F, 300 MHz spectrometer. ^{77}Se-BCM (single pulsed with complete decoupling) NMR spectra of the compounds (**1**, **2**, and **4**) were recorded on JNM-AL400, 400 MHz spectrometer with TH5AT/FG2 probe. The mass spectra (EI, 70 eV) were obtained on a VG-Analytical 11-2505-70 SMS Mass spectrometer. Infrared spectra were recorded between KBr pellets on a Perkin-Elmer model 1430 ratio recording spectrometer. Carbon, hydrogen, and nitrogen were estimated miroanalytically using Perkin-Elmer 2400 CHN elemental analyzer. Selenium/tellurium was estimated by standard method.[11]

Synthesis of 2-bromo methyl substituted pyridines: To a mechanically stirred solution of 2-amino methyl substituted pyridine (54 g, 0.5 mol) in 48% HBr (190 ml), bromine (75 ml, 1.5 mol) was added dropwise in an ice-bath followed by the slow addition of aqueous solution of sodium nitrite (87.1 g, 1.5 mol) in 120 ml water. The temperature must be maintained below 0°C throughout the entire procedure. The mixture was neutralized with aqueous sodium hydroxide, extracted with diethyl ether, dried over anhydrous sodium sulphate, and concentrated in vacuum to give oil, which was distilled.

2-Bromo-6-methyl pyridine: B.p. 60–62°C/4 mm (Lit.,[12] 62–65°C/25 mm Hg), yield 67.6 g (89%). ^{1}H NMR: δ, 2.50 (s, 3H), 7.09 (d, 1H), 7.25 (d, 1H), 7.42 (t, 1H). ^{13}C NMR: δ, 23.5, 121.5, 124.3, 138.0, 140.5, 159.1.

2-Bromo-5-methyl pyridine: M.p. 41–42°C (Lit.,[13] m.p. 49–50°C), yield 69 g (91%). ^{1}H NMR: δ, 2.27 (s, 3H), 7.33 (s, 2H), 8.16 (s, 1H). ^{13}C NMR: δ, 17.6, 127.3, 132.3, 138.7, 139.1, 150.2.

2-Bromo-4-methyl pyridine: B.p. 68–70°C/4 mm Hg (Lit.,[13] b.p. 223–224°C/760 mm Hg), yield 64.6 g (85%). ^{1}H NMR: δ, 2.31 (s, 3H), 7.07–7.05 (d, 1H), 7.29 (s, 1H), 8.20–8.18 (d, 1H). ^{13}C NMR: δ, 20.48, 123.68, 128.42, 142.0, 149.4, 150.0.

2-Bromo-3-methyl pyridine: B.p. 212°C/760mm Hg (Lit.,[13] b.p. 218–219°C/760 mm Hg), yield 66.1 g (80%). ^{1}H NMR: δ, 2.32 (s, 3H), 7.18–7.14 (t, 1H), 7.51–7.48 (d, 1H), 8.16–8.14 (s, 1H). ^{13}C NMR: δ, 21.19, 122.1, 138.0, 143.7, 146.4.

General Procedure for the Preparation of Methyl Substituted 2,2′-Dipyridyl Diselenides

In a clean, flame dried two-necked round bottom flask was placed 3.49 g (20.2 mmol) of 2-bromo methyl substituted pyridine in tetrahydrofuran. The solution was cooled to −78°C to which 24.3 mmol of *n*-BuLi was added dropwise. The solution turned from colorless to red. The solution was stirred for 20 minutes and 1.9 g (24.2 mmol) of elemental selenium was added. The reaction mixture was slowly brought to room temperature till all the elemental selenium dissolves. The supply of nitrogen was discontinued and the reaction mixture was exposed to aerial oxidation for another 30 min. When clear yellow solution was obtained, the solvent was stripped off on the rota-evaporator and the product was washed with water and then extracted with diethyl ether. The ether extract was dried over anhydrous sodium sulphate and the diethyl ether removed on a rota-evaporator. The crude product was subjected to column chromatography on silica gel using hexane-ethyl acetate (10:1) as eluent affording pure methyl substituted 2,2′-dipyridyl diselenide as dark yellow needles.

6,6′-Dimethyl-2,2′-dipyridyl diselenide (1): Yield, 6.86 g (62%) m.p. 61–62°C. ^{1}H NMR: δ, 2.52 (s, 6H), 6.92–6.89 (d, 2H), 7.43–7.36 (t, 2H), 7.61–7.58 (d, 2H). ^{13}C NMR: δ, 24.2, 120.5, 136.0, 137.5, 153.6, 158.5. ^{77}Se NMR: δ, −576.767. IR (KBr, cm^{-1}): 3060, 2960, 2920, 1580, 1540, 1430, 1120, 1090, 1020, 840, 780, 660, 540. MS(EI): 344 $[M]^+$ (27.1); 264 $[M\text{-}Se]^+$ (43.3); 183 $[M\text{-}Se_2H]$ (81); 92 $[M{-}CH_3PySe_2]^+$ (100). Anal. Calcd. for: $C_{12}H_{12}N_2Se_2$; C, 41.86, H, 3.48, N, 8.13. Found: C, 41.08, H, 3.28, N, 8.42.

5,5′-Dimethyl-2,2′-dipyridyl diselenide (2): Yield, 7.75 g (70%) m.p. 67–68°C. ^{1}H NMR: δ, 2.27 (s, 6H), 7.35–7.32 (d, 2H), 7.68–7.66 (d, 2H), 8.27 (s, 2H). ^{13}C NMR: δ, 17.8, 123.3, 130.7, 136.1, 149.8, 150.7. ^{77}Se NMR: δ, −584.469. IR (KBr, cm^{-1}): 3030, 2983, 2914, 1579, 1559, 1446, 668, 1219, 1081, 1040, 910, 822, 723, 592, 479. ME(EI): 344 $[M]^+$ (34.3); 263 $[M\text{-}SeH]^+$ (6.2); 183 $[M\text{-}Se_2H]$ (100); 92 $[M{-}CH_3PySe_2]^+$

(60.5). Anal. Calcd. for: $C_{12}H_{12}N_2Se_2$; C, 41.86, H, 3.48, N, 8.13. Found: C, 41.12, H, 3.35, N, 8.32.

4,4′-Dimethyl-2,2′-dipyridyl diselenide (3): Yield, 6.64 g (60%) m.p. 96–98°C. ^1H NMR: δ, 2.20 (s, 6H), 6.69–6.00 (d, 2H), 7.60 (s, 2H), 8.30–8.29 (d, 2H). ^{13}C NMR: δ, 21.0, 123.4, 123.9, 148.8, 149.0, 154.0. IR (KBr, cm^{-1}): 3060, 2960, 2920, 1580, 1540, 1460, 1270, 1120, 1080, 840, 700, 500. ME(EI): 344 $[M]^+$ (36); 263 $[M\text{-}SeH]^+$ (10.1); 183 $[M\text{-}Se_2H]$ (100); 92 $[M{-}CH_3PySe_2]^+$ (63). Anal. Calcd. for: $C_{12}H_{12}N_2Se_2$; C, 41.86, H, 3.48, N, 8.13. Found: C, 41.72, H, 3.93, N, 8.42.

3,3′-Dimethyl-2,2′-dipyridyl diselenide (4): Yield, 7.97 g (72%) m.p. 142–144°C. ^1H NMR: δ, 2.42 (s, 6H), 6.99–6.96 (d, 2H), 7.30–7.27 (d, 2H), 8.26 (s, 2H). ^{13}C NMR: δ, 20.5, 121.7, 133.5, 136.6, 147.7, 153.0. ^{77}Se NMR: δ, −594.362. IR (KBr, cm^{-1}): 3030, 2960, 2920, 1664, 1570, 1543, 1460, 1277, 1060, 785, 635, 575, 470. ME(EI): 344 $[M]^+$ (19.6); 263 $[M\text{-}SeH]^+$ (24.4); 183 $[M\text{-}Se_2H]$ (100); 92 $[M{-}CH_3PySe_2]^+$ (62.2). Anal. Calcd. for: $C_{12}H_{12}N_2Se_2$; C, 41.86, H, 3.48, N, 8.13. Found: C, 41.45, H, 3.62, N, 8.34.

General Procedure for Preparation of Methyl Substituted 2,2′-Dipyridyl Ditellurides

Methyl substituted 2,2′-dipyridyl ditellurides were prepared by the same method as applied for the preparation of corresponding diselenides.

6,6′-Dimethyl-2,2′-dipyridyl ditelluride (5): Yield, 3.36 g (20%) m.p. 55–56°C. ^1H NMR: δ, 2.48 (s, 6H), 6.63–6.60 (d, 2H), 7.24–7.19 (t, 2H), 7.63–7.61 (d, 2H). ^{13}C NMR: δ, 24.1, 121.1, 127.9, 134.9, 158.6, 136.9. IR (KBr, cm^{-1}): 3042, 2916, 1571, 1429, 1376, 1242, 1163, 1083, 988, 826, 781, 655, 545. ME(EI): 444 $[M]^+$ (5.5); 314 $[M\text{-}Te]^+$ (5.5); 314 $[M\text{-}Te]^+$ (6.5); 183 $[M{-}Te_2H]^+$ (30.9); 92 $[M{-}CH_3PyTe_2]^+$ (100). Anal. Calcd. for: $C_{12}H_{12}N_2Te_2$; C, 32.43, H, 2.70, N, 6.30. Found: C, 32.21, H, 2.52, N, 6.18.

5,5′-Dimethyl-2,2′-dipyridyl ditelluride (6): Yield, 4 g (25%) m.p. 110–112°C. ^1H NMR: δ, 1.97 (s, 6H), 6.63–6.60 (d, 2H), 7.24–7.19 (t, 2H), 7.63–7.61 (d, 2H). ^{13}C NMR: δ, 24.1, 121.1, 127.9, 134.9, 136.9, 158.6. IR (KBr, cm^{-1}): 3033, 2969, 2917, 1582, 1544, 1526, 1368, 1272, 1073, 983, 835, 694, 520. ME(EI): 444 $[M]^+$ (8.2); 314 $[M\text{-}Te]^+$ (12.1); 183 $[M{-}Te_2H]^+$ (67.3); 92 $[M{-}CH_3PyTe_2]^+$ (100). Anal. Calcd. for: $C_{12}H_{12}N_2Te_2$; C, 32.43, H, 2.70, N, 6.30. Found: C, 32.83, H, 2.12, N, 6.19.

4,4′-Dimethyl-2,2′-dipyridyl ditelluride (7): Yield, 2.61 g (22%) Semi solid. ^1H NMR: δ, 2.27 (s, 6H), 6.62–6.61 (d, 2H), 7.85 (s, 2H), 8.29–8.28 (d, 2H). ^{13}C NMR: δ, 20.7, 122.5, 131.5, 135.1, 146.1, 149.3. IR

(KBr, cm^{-1}): 3025, 2963, 2908, 1685, 1573, 1546, 1261, 1097, 1020, 865, 685, 473. ME (EI): 444 $[M]^+$ (1.8); 314 $[M\text{-}Te]^+$ (41.15); 183 $[M{-}Te_2H]^+$ (37.5); 92 $[M{-}CH_3PyTe_2]^+$ (100). Anal. Calcd. for: $C_{12}H_{12}N_2Te_2$; C, 32.43, H, 2.70, N, 6.30. Found: C, 32.74, H, 2.13, N, 6.92.

3,3′-Dimethyl-2,2′-dipyridyl ditelluride (8): Yield, 8.73 g (52%) m.p. 120–125°C. ^1H NMR: δ, 2.41 (s, 6H), 7.14–7.10 (m, 2H), 7.47–7.45 (d, 2H), 8.14–8.12 (d, 2H). ^{13}C NMR: δ, 24.52, 122.5, 134.8, 136.2, 147.1, 154.8. IR (KBr, cm^{-1}): 3060, 2960, 2920, 1580, 1540, 1430, 1120, 1090, 1020, 840, 780, 660, 540. ME(EI): 444 $[M]^+$ (7.33); 314 $[M\text{-}Te]^+$ (14.51); 183 $[M{-}Te_2H]^+$ (76.62); 92 $[M{-}CH_3PyTe_2]^+$ (100). Anal. Calcd. for: $C_{12}H_{12}N_2Te_2$; C, 32.43, H, 2.70, N, 6.30. Found: C, 32.60, H, 2.42, N, 6.18.

X-Ray Crystal Structure Determination of 6,6′-Dimethyl-2,2′-dipyridyl Diselenide (1)

Intensity data were collected on a Siemens P4 single crystal diffractometer equipped with molybdenum sealed tube ($\lambda = 0.71073$ Å) and highly-oriented graphite monochromator using crystals of dimensions 0.34 × 0.28 × 0.25 mm, mounted in Lindmann glass capillaries at 293(2) K. The cell parameters and their standard deviation were

TABLE II Crystal Data and Structure Refinement Details for Compound **1**

Empirical formula	$C_{12}H_{12}N_2Se_2$	
Formula weight	342.16	
Crystal system, space group	Monoclinic, C2/c	
Unit cell dimensions	a = 20.079(2)Å	$\alpha = 90°$
	b = 8.649(1)Å	$\beta = 108.06(1)°$
	c = 7.925(1)Å	$\gamma = 90°$
Volume	1308.5 (3)$Å^3$	
Z, Calculated density	4, 1.737 Mg/m^3	
Absorption coefficient	5.626 mm^{-1}	
F(000)	664	
Max. and min. transmission	1.00, 0.804	
Theta range for data collection	2.13 to 24.50°	
Index ranges	$-23 \leq h \leq 0$, $-10 \leq k \leq 0$, $-8 \leq 1 \leq 9$	
Reflection collected	1118	
Independent reflections	1080[$R_{int} = 0.0268$]	
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	1080/0/74	
Goodness on fit on F^2	1.065	
Data to parameter ratio	14.6:1	
Final R indices, 873 reflections, [I > 2s(I)]	R1 = 0.0400, wR2 = 0.0991	
R indices (all data)	R1 = 0.0530, wR2 = 0.1062	
Largest diff. peak and hole	0.478 and −0.556e.$Å^{-3}$	

obtained by least square to 40 reflections. The 2θ–θ scan mode was used with variable scan speed ranging from 2.0 to 60.0°/min in ω. Three reflections were used to monitor the stability and orientation of the crystal and were remeasured after every 97 reflections. All other relevant information about the data collection and the refinement are presented in Table II.

The crystal structure of **1** was solved by direct method using SHELX-97[14] and also refined on F^2 using the same one. All the nonhydrogen atoms were refined anisotropically. The hydrogen atoms were included in the ideal positions with fixed isotropic U values and were riding with their respective nonhydrogen atoms. A weighting scheme of the form $w = 1/[\sigma^2(F_0^2) + 0.0534P)^2 + 2.34P]$, $P = [\max(F_0^2, 0) + 2^*F_0^2]/3$ was used. The refinement converged to a final R value of 0.0400, (wR2 = 0.0991 for 873 reflections) $[I > 2\sigma(I)]$.*

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*Data deposited at CCDC (No. 159119 for compound 1).