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Synthesis and characterization of dialkylgallium (dialkylindium) complexes of *N*-salicylidene 2-aminopyridine and *N*-salicylidene 2-methoxyaniline: crystal structure of dimethyl[*N*-salicylidene 2-aminopyridine]gallium

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Abstract

Reaction of trimethylgallium (trimethylindium) and triethylgallium (triethylindium) with *N*-salicylidene 2-aminopyridine or *N*-salicylidene 2-methoxyaniline in benzene solution yields seven intramolecular coordinated Group 13 metal Schiff-base complexes. All complexes obtained were characterized by elemental analysis, ^{1}H-NMR, IR and mass spectrometry. The structure of dimethyl[*N*-salicylidene 2-aminopyridine]gallium (**1**) was determined by X-ray single crystal analysis. The Ga atom is four coordinate in the structure. Photoluminescent emission spectra of complexes dimethyl[*N*-salicylidene 2-aminopyridine]gallium (**1**), diethyl[*N*-salicylidene 2-aminopyridine]gallium (**3**), diethyl[*N*-salicylidene 2-aminopyridine]indium (**4**) and dimethyl[*N*-salicylidene 2-methoxylaniline]gallium (**5**) were measured. The maximum emission wavelength is between 503 and 511 nm, respectively on radiation by UV light. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Trialkylgallium; Trimethylindium; *N*-Salicylidene 2-aminopyridine; *N*-Salicylidene 2-methoxyaniline; Electroluminescence

1. Introduction

Organic electroluminescent (OEL) devices have attracted much interest owing to their potential application as large-area light displays since Tang et al. first reported bright organic electroluminescence at low voltages [1,2]. A variety of EL emission materials, including tris(8-hydroxyquinoline)aluminum (Alq$_3$) [2], Schiff-base transition metal complexes [3] and organic polymers [4–6] have been developed, and various emission colors were obtained [1,4–6]. The chemistry of organoaluminum, gallium and indium complexes which contain either Group 15 or Group 16 elements has been under investigation for the last few decades owing to their rich structural chemistry and possible application as precursors of semiconductor materials [7–9] and in organic reactions [10,11]. The application of organoaluminum, gallium or indium complexes as OEL materials has, however, been rarely studied [12,13]. Salicylaldehyde Schiff bases are similar to 8-hydroxyquinoline in structure. They both have one hydroxyl group, a coordination nitrogen atom and a delocalized π-system, although the structure of the Schiff bases is certainly more flexible than that of 8-hydroxyquinoline. Therefore, organometallic complexes of salicylaldehyde Schiff base ligands are expected to show good luminescent properties. As part of our efforts to search for new OEL substances, we wish to report here the synthesis and characterization of alkylgallium and indium complexes. The crystal structure of dimethyl[*N*-salicylidene 2-aminopyridine]gallium (**1**) was determined by X-ray analysis. Photoluminescent emission spectra of the compounds were also measured.

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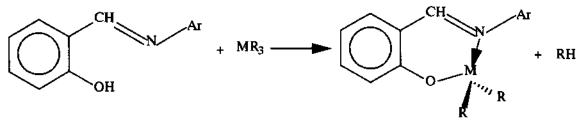
2. Results and discussion

Reaction of gallium and indium alkyls R_3M (M = Ga, In; R = Me, Et) with the Schiff-base ligands of *N*-salicylidene 2-aminopyridine or *N*-salicylidene 2-methoxyaniline proceeded smoothly at room temperature in 1:1 ratio affording the corresponding intramolecular coordinated complexes as shown in Scheme 1.

The complexes were isolated as yellow solids in high yields. Although gallium and indium alkyls are extremely moisture and oxygen sensitive, the complexes obtained are fairly stable on exposure to air. Compounds **1**–**7** could be left at ambient atmosphere for weeks without obvious decomposition. The complexes are nearly insoluble in cold saturated hydrocarbons such as pentane or petroleum and very soluble in unsaturated hydrocarbons such as benzene or toluene. All the products obtained gave satisfactory elemental analysis results and were characterized by IR, ^{1}H-NMR and mass spectrometry respectively.



	Ar	R	M
1	2- Py	Me	Ga
2	2- Py	Me	In
3	2- Py	Et	Ga
4	2- Py	Et	In
5	2-MeOC$_6$H$_4$	Me	Ga
6	2-MeOC$_6$H$_4$	Et	Ga
7	2-MeOC$_6$H$_4$	Et	In

Scheme 1.

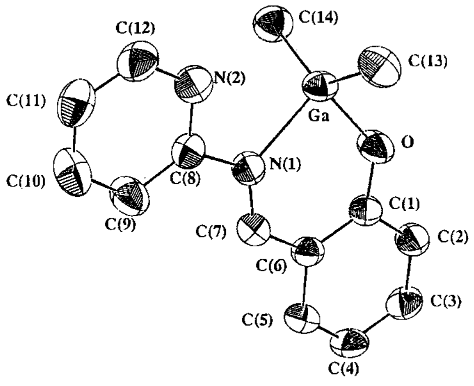


Fig. 1. Molecular structure of dimethyl[*N*-salicylidene 2-aminopyridine]gallium (**1**) showing 50% probability displacement ellipsoids. Hydrogen atoms are omitted for clarity.

Table 1
Selected bond lengths (Å) and angles (°) for $C_{15}H_{16}NOGa$ (**1**)

Ga–O	1.917(2)	C(1)–C(6)	1.422(3)
Ga–N(1)	2.059(2)	C(6)–C(7)	1.421(3)
O–C(1)	1.303(3)	N(1)–C(7)	1.291(3)
N(1)–C(8)	1.414(3)	N(2)–C(8)	1.334(3)
O–Ga–C(14)	105.7(1)	O–Ga–C(13)	105.1(1)
O–Ga–N(1)	90.76(7)	C(14)–Ga–C(13)	131.2(1)
C(14)–Ga–N(1)	105.6(1)	C(13)–Ga–N(1)	110.9(1)
C(7)–C(6)–C(1)	124.1(2)	C(7)–N(1)–Ga	124.2(2)
C(1)–O–Ga	130.7(1)	N(1)–C(7)–C(6)	126.7(2)
N(2)–C(8)–N(1)	112.8(2)	O–C(1)–C(6)	123.4(2)
C(8)–N(1)–Ga	114.9(1)		

In the IR spectra of the complexes, the M–C stretch vibrations are all visible at about 530 cm^{-1}. Comparing the IR spectra of the complexes with those of the free ligands, the appearance of medium to strong C–H stretch vibration bands is distinctive, accompanying the disappearance of the O–H signals. It is very interesting to see, in the ^{1}H-NMR spectra of the complexes, that the signals of the metal bonded methyl or methylene protons in complexes **1**–**4** shift downfield while those in complexes **5**–**7** move upfield compared with the signals of the free metal alkyls. It is believed that the coordination of the 2-methoxyl group to the central metal to form a five coordinate metal center is responsible for the upfield shift of the proton signals. Assumption of coordination of the oxygen in the methoxyl group to the central metal is reasonable as then a stable five membered ring is formed. Coordination of the pyridyl to the central metal should be quite difficult as a four membered ring system is fairly unstable. This was confirmed by the X-ray structure of complex **1**. The molecule ion peaks of the complexes **1**–**7** were all visible in their mass spectra, although the abundances are low for some of the complexes. There is no peak beyond the M$^+$ signal in the spectra. This illustrates the monomeric status of the complexes in the solid state. Fairly strong intensity peaks of the fragments losing one alkyl from the metal are observed for almost all the complexes. The relative intensity of the peaks for the metal containing species agrees well with the isotopic distribution of the metals (^{69}Ga (ca. 60%), ^{71}Ga(ca. 40%) and ^{113}In (ca. 9%), ^{115}In (ca. 91%)).

The molecular structure of dimethyl[*N*-salicylidene 2-aminopyridine]gallium (**1**) was determined by X-ray single crystal analysis. The structure is shown in Fig. 1 and the selected bond lengths and angles are listed in Table 1. In the structure, the N atom of the enamine group coordinates to gallium metal as expected. The geometry of the gallium atom is in a distorted tetrahedron. It is very interesting to see a small angle O–Ga–N(1) (90.76 (7)°) and a large angle C(13)–Ga–C(17) (131.2 (1)°). This cannot be explained by the steric hindrance of the groups on the metal. From the structure of **1**, it is seen that the pyridyl group stays near the gallium atoms. By calculation, the distance between N(2) and the Ga atom is about 2.7 Å. Although the distance is longer than a normal covalent N–Ga bond,

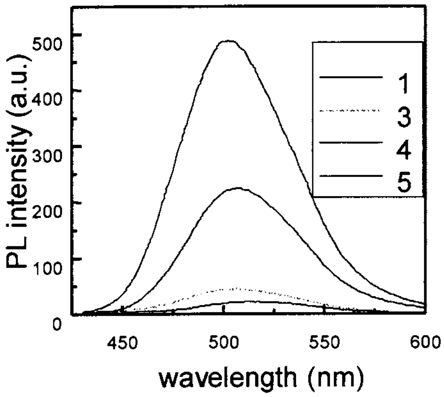


Fig. 2. PL emission spectra of **1**, **3**, **4** and **5**.

there is obviously quite a strong interaction between them. This should be responsible for the distortion of the bond angles around the Ga atom. This implies that there is some interaction between the nitrogen on pyridyl and the gallium atom. The Ga–O bond length (1.917 (2) Å) is comparable to those reported (1.869(2) to 1.874(2) Å in *N*,*N*-ethylenebis(salicylideneiminato)bis[dimethylgallium] [14]; 1.927(3) Å in (2-hydroxy-benzaldehydato)dimethylgallium [15]). The Ga–N(1) distance (2.059 (2) Å) is slightly longer than that in the compound *N*,*N*-ethylenebis(salicylideneiminato)bis[dimethylgallium] (2.026(3) to 2.035(3) Å) [14].

The PL emission spectra of the complexes **1**, **3**, **4** and **5** were measured as shown in Fig. 2. This shows that the emission bands are located in the blue–green region. Their emission maxima are at 506, 506, 511 and 503 nm with intensities of 225, 46.4, 23 and 487 a.u., respectively, for complexes **1**, **3**, **4** and **5**. This shows the alkyl groups on the metal have little influence on the emission maximum. The strength of the emission bands for complexes **1** and **5** is much stronger than that of complexes **3** and **4**, suggesting that the methyl group emissions are superior to the ethyl emissions. This may be attributed to the thermal vibration of different groups. The ethyl group is more free than the methyl group. Increasing the thermal vibration may reduce the emission efficiency by thermal quenching of the photoluminescent emission. Complex **5** exhibits a higher emission intensity than that of complex **1**. This may be attributed to the coordination of the methoxyl group to the metal, which increases the rigidity of the molecule. The OEL properties of the complexes **1**, **3**, **4** and **5** and the other complexes obtained are now under investigation in our group. Their blue–green luminescence feature is desirable.

3. Experimental

All reactions were performed in a glove box under nitrogen. The solvents were refluxed with sodium benzophenone and distilled under nitrogen prior to use. The Schiff-base ligands were prepared by condensation of salicylaldehyde with 2-aminopyridine or 2-methoxyaniline. Trialkylgallium and trialkylindium were provided by the National 863 Program Advanced Material MO Precursors R&D Center of China. ^{1}H-NMR data were collected on a JEOL PMX-60SI or a Bruker ARX-300 spectrometer, with chemical shifts referenced to $SiMe_4$ as internal standard. Infrared spectra were obtained as KBr pellets with a 5DX-FT-2 spectrometer. Mass spectra were measured on a VG-ZAB-HS spectrometer (electron impact ionization). Luminescent spectra were determined with an RF-5000 spectroflurophotometer. Elemental analyses were performed on a Perkin–Elmer 240 C elemental analyzer. Melting points were observed in an electrothermal melting point apparatus in sealed capillaries and were uncorrected.

*3.1. Preparation of dimethyl[N-salicylidene 2-aminopyridine]gallium (**1**)*

A solution of trimethylgallium (0.2 ml, 2 mmol) in 10 ml cyclohexane was added over a period of 10 min dropwise with stirring to a solution of *N*-salicylidene 2-aminopyridine (0.396 g, 2 mmol) in 10 ml cyclohexane and 2 ml benzene. On addition of the solution of trimethylgallium, methane gas was evolved immediately from the mixture and the reaction became exothermic. After the mixture was stirred for an additional 5 min at room temperature, the solvent was removed in vacuo and the yellow powder residue was recrystallized from cyclohexane–benzene solution, giving the pale yellow crystal. Yield 0.54 g (90%). M.p. 112–113°C. IR data (cm^{-1}): 3057.8, 3015.6, 2966.4, 2903.1, 1616.4, 1567.2, 1525, 1440.6, 1384.4, 1335.2, 1293, 1187.5, 1145.3, 786.7, 751.6, 589.8, 533.6. ^{1}H-NMR data (ppm): 9.2 (s, H, –C*H*=N), 8.66(d, 1H, Py–H), 7.33–8.5 (m, 7H, Ar, Py), −0.19 (s, 6H, Ga(CH_3)$_2$). MS data: 297.7 (M$^+$ 0.26%), 296.6 (0.16%), 295.7 (0.33%), 282.7 (68.88%), 281.6 (15.65%), 280.7 (100%), 70.9 (5.3%), 68.9(7.6%). Anal. Calc. for $C_{14}H_{15}N_2OGa$: C, 56.62; H, 5.09; N, 9.43. Found: C, 56.5; H, 4.62; N, 9.54%.

*3.2. Preparation of dimethyl[N-salicylidene 2-aminopyridine]indium (**2**)*

This was prepared and purified in the same manner as described for **1** from *N*-salicylidene 2-aminopyridine (0.396 g, 2 mmol) and trimethylindium (0.32 g, 2 mmol). The compound was isolated as yellow crystal. Yield 0.61 g (89%). M.p. 214–215°C. IR data (cm^{-1}): 3064.8, 3008.6, 2917.2, 1609.4, 1581.2, 1525, 1461.7, 1391.4, 1328.1, 1180.5, 1152.3, 1124.2, 920.3, 857, 751.6, 533.6, 512.5. ^{1}H-NMR data (ppm): 8.6 (s, H, –C*H*=N), 8.33 (d, 1H, Py–*H*), 6.33–8.0 (m, 7H, Ar–*H*, Py–*H*), −0.06 (s, 6H, In (C*H*$_3$)$_2$). MS data: 341.9 (M$^+$ 1.38%), 326.9 (93.08%), 311.9 (28.35%), 233.8 (14.97%), 198 (19.35%), 181 (16.66%), 114.9 (100%). Anal. Calc. for $C_{14}H_{15}N_2OIn$: C, 49.15; H, 4.42; N, 8.19. Found: C, 48.08; H, 4.24; N, 8.98%.

3.3. Preparation of diethyl[N-salicylidene 2-aminopyridine]gallium (3)

The preparation was the same as described for **1**, using *N*-salicylidene 2-aminopyridine (0.396 g, 2 mmol) and triethylgallium (0.32 g, 2 mmol) in 2 ml benzene. The yellow crystals were isolated and purified as described for **1**. Yield 0.57 (88%). M.p. 73–74°C. IR data (cm^{-1}): 3051.6, 2934.7, 2884.6, 2860.1, 2807.8, 1616.4, 1585.9, 1531.6, 1568.4, 1457.9, 1435.5, 1389.8, 1331.2, 1291.8, 1186.5, 1148.1, 998.8, 925.7, 850.5, 797.1, 780.4, 758.9. ^{1}H-NMR data (ppm): 9.11 (s, H, –C*H*=N), 8.59(d, 1H, Py–H), 6.4–8.1 (m, 7H, Ar–*H*, Py–H), 0.57 (q, 4H, Ga(*CH$_2$*CH$_3$)$_2$), 0.92 (t, 6H, Ga(CH$_2$*CH$_3$*)$_2$)). MS data: 325.1 (M$^+$ 0.01%), 298 (14.91%), 297.1 (85.28%), 296 (24.39%), 295.1 (100%), 268.1 (2.93%), 267.1 (4.73%), 241.5 (4.69%), 187.9 (9.20%), 181.0 (7.94%), 79 (49.67%), 78.0 (30.96%), 70.9 (71.95%), 68.9 (88.27%). Anal. Calc. for $C_{16}H_{19}N_2OGa$: C, 59.12; H, 5.89; N, 8.62. Found: C, 59.20; H, 5.91; N, 8.59%.

3.4. Preparation of diethyl[N-salicylidene 2-aminopyridine]indium (4)

The preparation was the same as described for **1**, using *N*-salicylidene 2-aminopyridine (0.396 g, 2 mmol) and triethylindium (0.404 g, 2 mmol) in 2 ml benzene. The yellow crystals were isolated and purified as described for **1**. Yield 0.65 g (88%). M.p. 77°C. IR data (cm^{-1}): 3054.7, 3013.8, 2928.2, 2903.9, 2852.2, 1616.1, 1583.1, 1564.1, 1527.8, 1474.8, 1456.0, 1429.3, 1388.5, 1349.2, 1326.4, 1290.1, 1231.7, 1174.6, 1149.9, 1034.2, 918.0, 861.0, 780.6, 730.9, 694.2. ^{1}H-NMR data (ppm): 8.60 (s, H, –C*H*=N), 8.30 (d, 1H, Py–*H*), 6.33–7.90 (m, 7H, Ar–*H*, Py–*H*), 0.80 (q, 4H, In (*CH$_2$*CH$_3$)$_2$), 1.10 (t, 6H, In(CH$_2$*CH$_3$*)$_2$)). MS data: 369.9 (M$^+$ 0.05%), 341.7 (18.67%), 340.7 (85.73%), 312.6 (5.76%), 311.6 (33.78%), 310.6 (3.98%), 233.7 (10.71%), 197.9 (8.46%), 114.8 (100%), 79.0 (37.06%), 77.9 (12.06%), 52.0 (9.10%), 51.0 (10.50%). Anal. Calc. for $C_{16}H_{19}N_2OIn$: C, 51.92; H, 5.17; N, 7.57. Found: C, 52.09; H, 5.31; N, 7.57%.

3.5. Preparation of dimethyl[N-salicylidene 2-methoxylaniline]gallium (5)

This was prepared and purified in the same manner as described for **1** from *N*-salicylidene 2-methoxylaniline (0.422 g, 2 mmol) and trimethylgallium (0.2 ml, 2 mmol). The compound was isolated as yellow crystal. Yield 0.56 g (86%). M.p. 76°C. IR data (cm^{-1}): 3082.1, 3054.5, 3011.8, 2972.1, 2934.1, 2834.8, 1613.8, 1497.1, 1464.7, 1442.7, 1438.3, 1340.5, 1315.2, 1257.0, 1196.0, 1173.3, 1149.1, 1127.4, 1119.9, 1049.8, 1028.0, 985.3, 923.3, 864.2, 810.2, 756.4, 586.3. ^{1}H-NMR data (ppm): 8.26 (s, H, –C*H*=N), 6.70–7.39 (m, 8H, *Ar–H*), 3.86 (s, 3H, *–OCH$_3$*), −0.33 (s, 6H, Ga(C*H*$_3$)$_2$). MS data: 325.9 (M$^+$ 0.11%), 313.0 (11.48%), 312.0 (68.56%), 311.0 (17.62%), 309.9 (100.0%), 296.6 (12.07%), 294.9 (17.84%), 281.9 (25.20%), 280.8 (12.77%), 279.9 (37.28%), 70.9 (47.63%), 68.9 (71.07%),. Anal. Calc. for $C_{16}H_{18}NO_2Ga$: C, 58.94; H, 5.56; N, 4.30. Found: C, 58.85; H, 5.49; N, 4.26%.

3.6. Preparation of diethyl[N–salicylidene 2–methoxylaniline]gallium (6)

This was prepared and purified in the same manner as described for **1** from *N*–salicylidene 2–methoxyaniline (0.422 g, 2 mmol) and trimethylgallium (0.32 g, 2 mmol). The compound was isolated as yellow crystal. Yield 0.60 g (85%). M.p. 47°C. IR data (cm^{-1}): 3056.3, 3016.9, 2954.4, 2933.2, 2858.6, 1609.7, 1583.1, 1537.4, 1497.3, 1463.7, 1442.1, 1384.2, 1340.3, 1317.3, 1259.8, 1251.0, 1224.7, 1186.2, 1193.3, 1147.4, 1126.8, 1118.0, 1023.2, 985.6, 932.9, 917.8, 853.3, 810.3, 749.4. ^{1}H-NMR data (ppm): 8.16 (s, H, –C*H*=N), 6.67–7.30 (m, 8H, Ar–*H*), 3.80 (s, 3H, *–OCH$_3$*), 0.43 (q, 4H, Ga (*CH$_2$*CH$_3$)$_2$), 0.97 (t, 6H, Ga (CH$_2$*CH$_3$*)$_2$)). MS data: 353.8 (M$^+$ 0.02%), 326.9 (21.11%), 326.0 (87.71%), 325.1 (31.98%), 324.2 (100.0%), 311.8 (3.58%), 310.8 (2.51%), 309.8 (5.10%), 296.1 (8.10%), 295.1 (3.01%), 284.2 (7.72%), 283.1 (46.79%), 282.1 (12.24%), 281.1 (68.72%), 70.9 (58.08%), 68.9 (81.50%). Anal. Calc. for $C_{18}H_{22}NO_2Ga$: C, 61.05; H, 6.26; N, 3.96. Found: C, 61.16; H, 6.10; N, 3.76%.

3.7. Preparation of diethyl[N-salicylidene 2-methoxylaniline]indium (7)

This was prepared and purified in the same manner as described for **1** from *N*-salicylidene 2-methoxyaniline (0.422 g, 2 mmol) and triethylindium (0.404 g, 2 mmol). The compound was isolated as yellow crystal. Yield 0.67 g (85%). M.p. 101°C. IR data (cm^{-1}): 3067.7, 3036.8, 3009.2, 2953.2, 2925.6, 2898.7, 2856.4, 1605.1, 1578.5, 1529.3, 1494.3, 1459.4, 1438.0, 1388.3, 1350.2, 1325.3, 1301.7, 1243.9, 1219.0, 1186.1, 1167.7, 1144.6, 1126.2, 1116.6, 1051.0, 1010.1, 982.8, 918.9, 848.3, 804.9, 756.7, 638.6. ^{1}H-NMR data (ppm): 8.33 (s, H, –C*H*=N), 6.60–7.30 (m, 8H, *Ar–H*), 3.90 (s, 3H, –*OCH$_3$*), 0.73 (q, 4H, In (*CH$_2$*CH$_3$)$_2$), 1.10 (t, 6H, In(CH$_2$*CH$_3$*)$_2$)). MS data: 399.9 (M$^+$ 0.03%), 370.9 (22.08%), 369.9 (84.42%), 368.9 (5.91%), 341.9 (6.48%), 340.9 (33.13%), 233.9 (8.89%), 114.9 (100.00%), 77.0

(6.01%), 68.9 (1.63%). Anal. Calc. for $C_{18}H_{22}NO_2In$: C, 54.18; H, 5.56; N, 3.51. Found: C, 54.52; H, 4.97; N, 3.47%.

*3.8. Determination of the crystal structure of **1***

A crystal of complex **1** was sealed in a thin walled glass capillary under a nitrogen atmosphere. Data collection was performed with Mo–K_α radiation ($\lambda = 0.71073$ Å) on an Enraf-Nonius CAD-4 diffractometer equipped with graphite monochromator (Table 2). The structure was solved by direct methods using MITHRIL [16] and subsequent Fourier techniques. Hydrogen atoms were added geometrically (before the final amendment). All calculations were performed using the TEXSAN [17] program system. ORTEP II [18] was used to produce the figures and CAD-4 software [19] was used for data collection and cell refinement. Selected bonds and angles of complex **1** are listed in Table 1. Crystallographic data are summarized in Table 2. The overall molecular geometry of complex **1** with the atomic labeling scheme is shown in Fig. 1.

Table 2
Crystal data, collection parameters, and refinements

Empirical formula	$C_{14}H_{15}N_2OGa$
Formula weight	297.01
Space group	$P21/n$ (#14)
Crystal system	Monoclinic
Unit cell parameters	
a (Å)	10.367(2)
b (Å)	10.491(1)
c (Å)	13.104(2)
β (°)	111.51(1)
V (Å^3)	1325.9 (4)
Z	4
Crystal dimensions (mm)	0.55 × 0.30 × 0.25
Crystal colour	Pale yellow
D_{calc} (g cm^{-3})	1.488
$F(000)$	608
μ(Mo–K_α) (cm^{-1})	20.55
Temperature (K)	296
T(max)/T(min)	1/0.7087
Diffractometer	Enraf-Nonius CAD-4
Monochromator	Graphite
Radiation	Mo–K_α
$2\theta_{max}$ (°)	57.9
Index range (°)	$0 \leq h \leq 14$, $-14 \leq k \leq 0$, $-17 \leq l \leq 17$
Reflections collected	3886
Independent reflections	3727
Independent reflections observed ($I > 3.00\sigma(I)$)	3010
R_{int}	4.1
Variables	164
$R(F)$	0.031
wR	0.044
Goodness of fit	1.25
$\Delta\rho_{max}$ (e Å^{-3})	0.40
$\Delta\rho_{min}$ (e Å^{-3})	−0.32
$(\Delta/\sigma)_{max}$	0.009

4. Supplementary material

Full information on the crystal structure has been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 140562. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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