Introduction

The research grant is used to support three research activities:

(i) 1 Final year project student – Hydrogenation reaction using selected homogeneous catalysts.

(ii) 1 Phd student – synthesis of potential biologically active compounds.

(iii) 1 MSc. student – synthesis of nanocatalysts for organic reactions.

Acknowledgements

I would like to thank Universiti Sains Malaysia for the financial support for the research projects. I am also grateful to the staff (academic and non-academic) in the School of Chemical Sciences for their support and direct or indirect contributions to the projects.

Sincerely,

.....

(DR SHAFIDA ABD HAMID)

Contents

(i) Hydrogenation Reaction of Alkenes using Homogeneous Catalysts Pd(II), Rh(III) and Ru(III)

Abstract

Hydrogenation reactions of unsaturated compounds were conducted to evaluate the reactivity and selectivity of noble metal catalysts. Homogenous metal catalysts used in this study were palladium (II) acetylacetonate, rhodium (III) acetylacetonate and ruthenium (III) acetylacetonate, while the substrates chosen for the reactions were styrene, crotonaldehyde and crotonic acid. The results showed that all the metal catalysts catalysed the the addition of hydrogen only at the double bond of the olefines without interference of the other functional groups. It was found that the reactivity of the catalyst decreased in the order of Pd > Rh > Ru while crotonaldehyde underwent the reaction more easily compared to styrene and crotonic acid. The IR and 1H NMR analysis of each hydrogenation product indicated that the structure was consistent with the structure predicted for each reaction.

Keywords: Palladium, Rhodium, Ruthenium, Catalytic Hydrogenation

(i) Tindak balas Penghidrogenan Alkena Menggunakan Mangkin Homogen Pd (II), Rh (III) dan Ru (III)

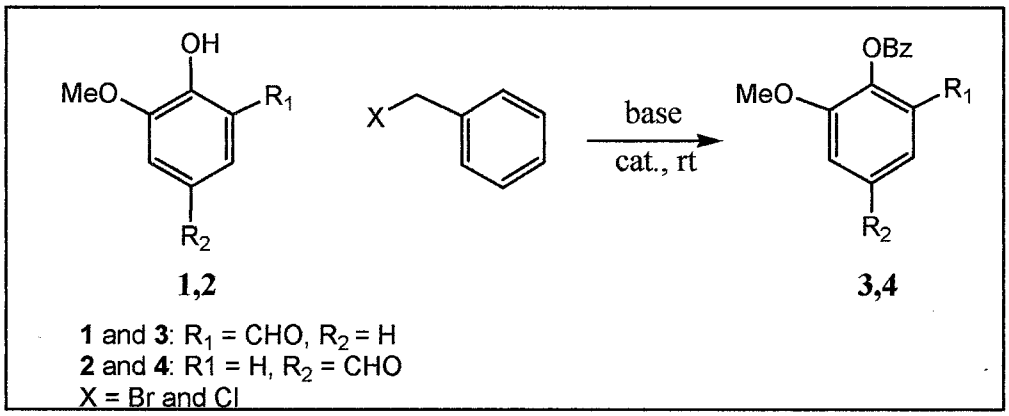
Abstrak

Tindak balas Penghidrogenan bagi sebatian tak tepu dijalankan untuk mengkaji kereaktifan dan keselektifan mangkin logam. Mankin logam homogen digunakan dalam kajian ini ialah paladium (II) asetilasetonat, rodium (III) asetilasetonat dan rutenium (III) asetilasetonat, sementara substrat yang dipilih untuk tindak balas ini ialah stirena, krotonaldehid dan asid krotonik. Hasil penyelidikan menunjukkan bahawa semua mangkin logam dapat memangkinkan penambahan hidrogen hanya pada ikatan dubel pada olefin tanpa mempengaruhi penurunan pada kumpulan berfungsi yang lain. Kereaktifan mangkin jugan didapati menurun mengikut urutan Pd > Rh > Ru sementara krotonaldehid mengalami tindak balas lebih cepat berbanding stirena dan asid krotonik. Analisis IR dan 1H NMR bagi setiap hasil penghidrogenan menunjukkan bahawa struktur yang konsisten dengan yang dijangkakan bagi setiap tindak balas.

Katakunci: Paladium, Rhodium, Rutenium, Penghidrogenan bermangkin

ii) Benzylation of Vanillin - New Improved Time

The conversion of phenols to phenyl ethers is an important transformation in organic synthesis and various methods have been reported to achieve this goal. This reaction is normally used for the protection of hydroxyl group. In this work, *o*-vanillin **1** and *p*-vanillin **2** were reacted with benzyl halides in/without the presence of catalysts at room temperature to produce benzyl *o*-vanillin **3** and benzyl *p*-vanillin **4** respectively (Scheme I).



1,2 **3,4**

1 and **3**: R_1 = CHO, R_2 = H
2 and **4**: R1 = H, R_2 = CHO
X = Br and Cl

Scheme I

The catalyst used in the experiments was Bu_4NI, using DMF or acetone as the solvent. The results are as shown in Table 1 and Table 2.

Table 1 Reaction of **1** and with benzyl halides at room temperature

Entry	X /eq.	Base/eq.	Cat./eq.	solvent	Time/hr	Y%
1	Br [a]	K_2CO_3	-	DMF	24	96
	1.0	5.5				
2	Br	K_2CO_3	Bu_4NI	Acetone	3.5	99
	1.0	5.75	0.1			
3	Cl	K_2CO_3 + KI	Bu_4NI	Acetone	7	60
	1.0	1.05+1.05	0.1			

[a] from J.M Berger, PhD thesis, Virginia polytechnic Institute and State university, 2001

Table 2 Reaction of 2 with benzyl halides at room temperature

Entry	X /eq.	Base/eq.	Cat./eq.	solvent	Time/hr	Y%
4	Br	K_2CO_3	-	DMF	24	93.1
	1.0	5.5				
5	Br	NaH 60%	Bu_4NI	DMF	24	57
	1.0	1.5	0.01			
6	Cl	K_2CO_3	Bu_4NI	Acetone	4	98.5
	1.0	5.5	0.1			

When acetone was used as the solvent (Entry 2), Benzyl o-vanillin produced was easier to be separated (compared with Entry 1, when DMF was used as the solvent) with high yield. The reaction period was also reduced from 24 hours to 3.5 hours when Bu_4NI was used as a catalyst. However, when benzyl chloride was used (Entry 3), the reaction period was also reduced to 7 hours but so did the yield of the reaction.

On the other hand, the reaction time of *p*-vanillin with benzyl bromide using NaH and a base and DMF as a solvent produced only 57% yield eventhough Bu_4NI was added as a catalyst (Entry 5). However, when Benzyl chloride was used, the yield had increased to 98.5 % in 4 hours reaction time (Entry 6).

This synthesis is only a small part of a series of organic syntheses and is still ongoing.

Keywords: Benzylation, vanillin, TBAI

iii) **The Synthesis and Characterisation of Nanogold catalysts**

This project had started since June 2006 and is still an ongoing process.

Gold, as bulk metal is known to be inert towards most molecules and is deemed to be the least reactive metal. Surprisingly, when gold is deposited on selected metal oxides as ultra-fine particles, its chemistry dramatically changes. Hence, many comparative study has been made to investigate the the effect of gold (supported and unsupported) on organic reations. Recently, gold supported catalysts show a remarkable selectivity towards hudrogenation of the conjugated C=O bond is the hydrogenation of α,β-unsaturated aldehydes and ketones. The catalytic behaviour of gold in the hydrogenation of α-β-unsaturated ketones is of a great relevance due to the fact that on classical hydrogenation metal catalysts, the main reaction product is always saturated ketone. Eventhough gold catalysts are not as active, they are more promising than conventional metal hydrogenation catalysts because of their higher selectivity in hydrogenation of α-β-unsaturated aldehydes and ketones, where the challenge lies in hydrogenating the C=O bond preferably over the C=C bond.

(a) Synthesis

Au/TiO_2 catalsyt has been made using deposition-precipitation method with NaOH (DP NaOH) at pH 7. In the preparation step of the catalyst, the molarity of NaOH plays an impotant role and in our work, we chose a medium concentration of ~ 0.5 M to allow better pH control. Initially, the prepared Au/TiO_2 catalyst were synthesised without calcination step, only dried at 100 °C for 24 hours. The prepared Au/TiO_2 is yet to be characterised by TEM, SEM and XRD. However, it has showna positive result in the hydrogenation of α-β-unsaturated aldehyde.

(b) Hydrogenation of crotonaldehyde has been conducted using Au/TiO_2 as catalyst in liquid phase in EtOH as the solvent. The aim of this reaction is to see its effectiveness in obtaining unsaturated alcohol (UA) as the main product. 1H NMR analysis show the presence of crotyl alcohol (unsaturated alcohol), butanal and a trace of starting material; crotonaldehyde. All of the above products will be confirmed by GC-MS and the percentage of conversion and selectivity will be determined.

Keywords: Nanocatalyst, gold, hydrogenation

Project Output

(i) One undergraduate student completed her final year project.

(ii) Support one MSc. and one PhD students in their research.

(iii) 2 Publications in international journals (Appendix 1 & 2)

Balance at 31 December 2006 = 1,665.28 (Appendix 3)

31037 Appendix 1

Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368
Editors: **W. Clegg and D. G. Watson**

2-(2-Benzyloxy-3-methoxyphenyl)-1*H*-benzimidazole

Mohammed H. Al-Douh, Shafida A. Hamid, Hasnah Osman, Shea-Lin Ng and Hoong-Kun Fun

Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(2-Benzyloxy-3-methoxyphenyl)-1*H*-benzimidazole

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.001 Å
R factor = 0.050
wR factor = 0.137
Data-to-parameter ratio = 34.6

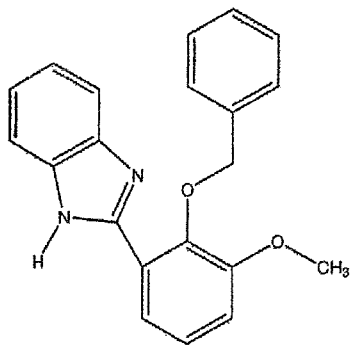
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 31 July 2006
Accepted 15 August 2006

In the title molecule, $C_{21}H_{18}N_2O_2$, all bond lengths and angles are normal. Weak intermolecular N—H···N hydrogen bonds link the molecules into chains along the c axis. The crystal packing is further stabilized by van der Waals forces.

Comment

Benzimidazole and its derivatives are widely used in biological systems (Craigo *et al.*, 1999; Gudmundsson *et al.*, 2000; Trivedi *et al.*, 2006). They are often used in an experimental synthetic search for new drugs (Townsend & Revankav, 1970; Trivedi *et al.*, 2006). Some derivatives of benzimidazole are used as topoisomerase I inhibitors (Kim *et al.*, 1996), and as antitumor (Craigo *et al.*, 1999), antiviral (Gudmundsson *et al.*, 2000) and antibacterial (Khalafi-Nezhad *et al.*, 2005) agents. The title compound, (I), is a new benzimidazole derivative. We present here its crystal structure.



(I)

Bond lengths and angles in (I) show normal values (Allen *et al.*, 1987) and are comparable with those reported for the related structures (Beauchamp *et al.*, 1987). The methoxy group at C12 is almost coplanar with the attached ring [C11—C12—O2—C21 = −3.25 (11)°], while the benzyloxy substituent is twisted away from the attached ring, with a C13—O1—C14—C15 torsion angle of −135.66 (7)°. Intramolecular N1—H1*A*···O1 hydrogen bonds (Fig. 1 and Table 1) generate S(6) ring motifs (Bernstein *et al.*, 1995).

Weak intermolecular N—H···N hydrogen bonds (Table 1) link the molecules into chains extending along the c axis. The crystal packing (Fig. 2) is further stabilized by van der Waals forces.

Experimental

A 100 ml three-necked round-bottomed flask was equipped with a nitrogen inlet adapter, rubber septum, glass stopper and magnetic

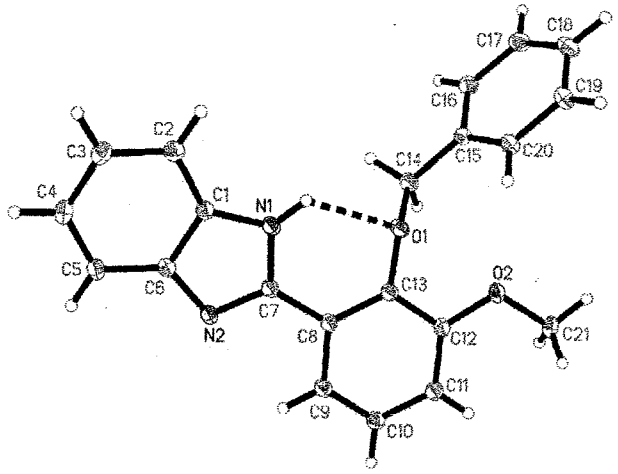


Figure 1
View of (I), showing 50% probability displacement ellipsoids and the atomic numbering. The dashed line indicates an intramolecular hydrogen bond.

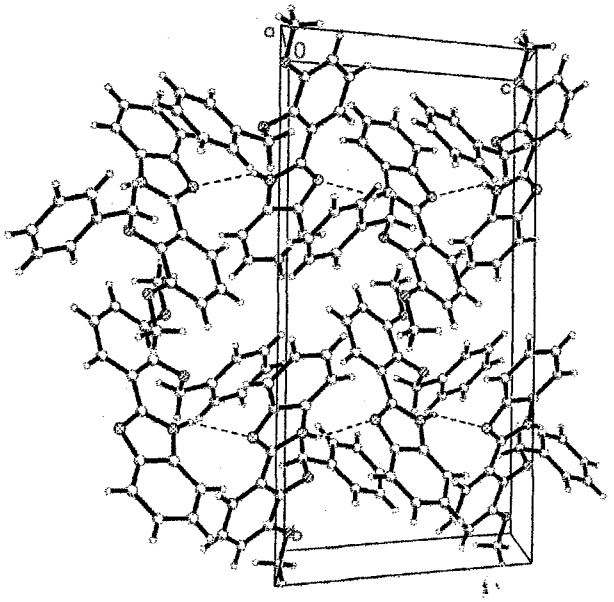


Figure 2
The crystal packing of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

stirring bar. The flask was charged with 5 ml of dichloromethane and benzyl-*o*-vanillin (484.6 mg, 2 mmol) and was cooled in an ice–water bath while a solution of *o*-phenylenediamine (216.3 mg, 2 mmol) in 5 ml dichloromethane was added dropwise *via* a syringe over 15 min. After 30 min, 10 mg anhydrous magnesium sulfate was added in one portion. The ice–water bath was removed, and the reaction mixture was stirred at room temperature for 2 h. The resulting mixture was then filtered through a sintered glass funnel with the aid of two 10 ml portions of dichloromethane; the filtrate was concentrated at reduced pressure by rotary evaporation at room temperature, affording a yellowish brown syrup. This material was dissolved in 150 ml of ethanol heated in an 353 K water bath while 270 ml of hot water was added with stirring. The resulting solution was allowed to cool to room temperature and was then cooled in an ice–water bath for 2 h. Filtration provided a light yellow powder of (I). The product was then purified by column chromatography with 30% ethanol in diethyl ether. Single crystals suitable for X-ray diffraction were obtained from ethanol–acetone (99:1 *v*/*v*).

Crystal data

$C_{21}H_{18}N_2O_2$
$M_r = 330.37$
Monoclinic, $P2_1/c$
$a = 9.5417\ (1)$ Å
$b = 18.4590\ (3)$ Å
$c = 11.0653\ (2)$ Å
$\beta = 123.814\ (1)^\circ$
$V = 1619.27\ (4)$ Å^3
$Z = 4$
$D_x = 1.355$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 100.0\ (1)$ K
Block, yellow
0.61 × 0.28 × 0.22 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.893$, $T_{max} = 0.981$
54869 measured reflections
8446 independent reflections
6691 reflections with $I > 2\sigma(I)$
$R_{int} = 0.054$
$\theta_{max} = 37.5^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.137$
$S = 1.06$
8446 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0705P)^2 + 0.2664P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.61$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1	0.86	2.14	2.693 (1)	122
N1—H1A···N2^{i}	0.86	2.57	3.313 (1)	145

Symmetry code: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$.

H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å and N—H = 0.86 Å. The H atoms were refined as riding and the U_{iso} values were freely refined.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

MHAD and SAH thank the Malaysian Government and Universiti Sains Malaysia for IRPA short term grant (No. 304/PKIMIA/636108).

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Appendix 2

S1037

electronic reprint

Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Editors: **W. Clegg and D. G. Watson**

2-Benzyloxy-3-methoxybenzaldehyde (benzyl-*o*-vanillin)

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2-Benzyloxy-3-methoxybenzaldehyde (benzyl-*o*-vanillin)

Mohammed H. Al-Douh,[a] Shafida A. Hamid,[a]‡ Hasnah Osman,[a] Shea-Lin Ng[b] and Hoong-Kun Fun[b]*

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Correspondence e-mail: hkfun@usm.my

Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.051
wR factor = 0.187
Data-to-parameter ratio = 23.8

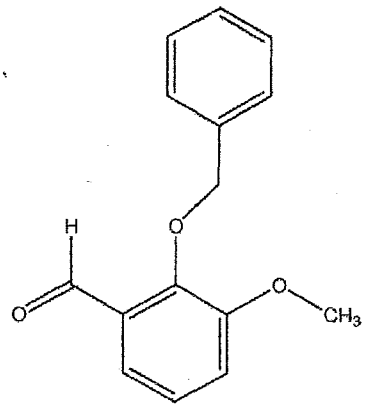
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 22 September 2006
Accepted 25 September 2006

In the title compound, $C_{15}H_{14}O_3$, the dihedral angle between the two benzene rings is 23.33 (6)°. Molecules are linked into a chain along the b axis by intermolecular C—H···O interactions.

Comment

The Williamson ether synthesis (McOmif, 1963; Feuer & Hooz, 1967; Benedict *et al.*, 1979; Spivey & Srikaran, 2001) is a common method employed in the benzylation of phenols and hydroxybenzaldehydes. These compounds are used in the synthesis of a variety of heteroatomic functional groups. The benzylation provides a protecting group for alcohols. This process is important in producing new materials such as antioxidants, plastic, rubber and petroleum products (Devassy *et al.*, 2005; Sawant *et al.*, 2005). *o*-Vanillin and 2-hydroxybenzaldehyde have been extensively used as the first step to produce coumarin derivatives (Scott & Raston, 2000) and neolignan derivatives (Juhasz *et al.*, 2000), which have high levels of biological activity. It is also used to produce new azo Schiff base dyes (Jarrahpour & Zarei, 2004). In addition, vanillin, the *p*-hydroxy isomer of *o*-vanillin, is used as a DNA–PK inhibitor and has been found tosensitize cells to *cis*-platin (Durant & Karran, 2003). The title compound, (I), was used as a key for synthesizing new anticancer drugs (Cotterill *et al.*, 1994), and was prepared earlier with melting points 318–319 K (Proffh, 1957) and 318–320 K (Cotterill *et al.*, 1994), and as a golden liquid (Berger, 2001). However, its crystal structure was never presented. In view of its importance, we present here its crystal structure.



(I)

Bond lengths and angles in (I) have normal values (Allen *et al.*, 1987) and agree well with those found in 4-benzyloxy-3-methoxybenzaldehyde (vanillin benzyl ether) (Gerkin, 1999).

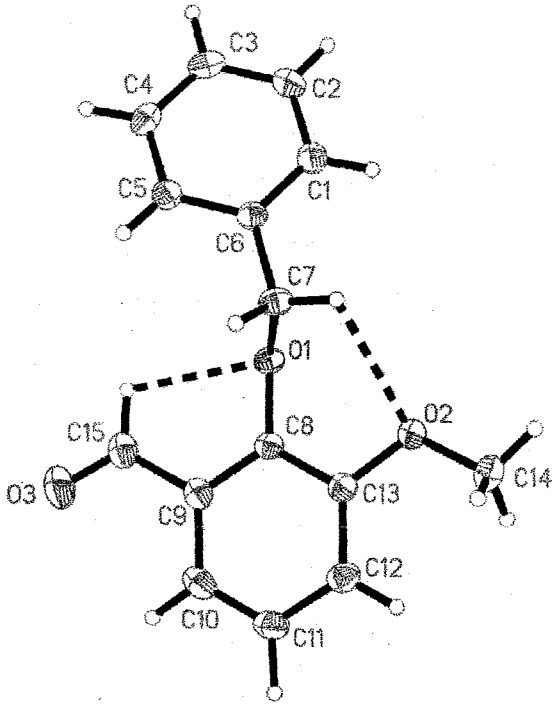


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. The dashed lines indicate intramolecular hydrogen bonds.

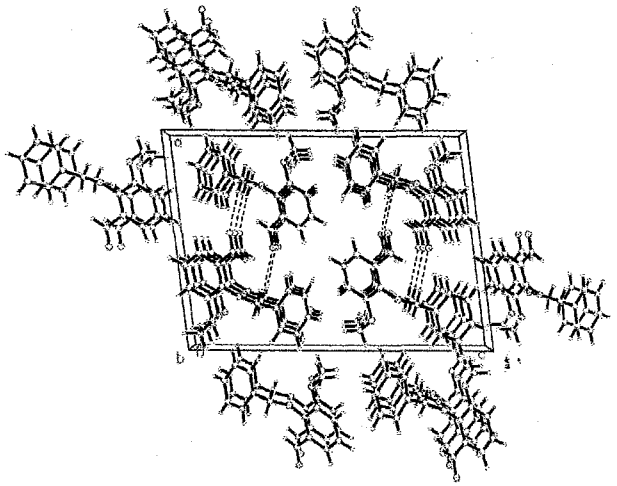


Figure 2
The crystal packing of (I), viewed down the *b* axis. Hydrogen bonds are shown as dashed lines.

The methoxy group at C13 is almost coplanar with the attached ring, with a C14—O2—C13—C12 torsion angle of −11.00 (18)°. The dihedral angle between the two benzene planes is 23.33 (6)° and the relative orientation of these planes is also characterized by a C8—O1—C7—C6 torsion angle of −165.34 (10)°.

In the crystal structure, all O atoms are involved in intra- and intermolecular C—H···O interactions (Table 1). The molecules are stacked along the *b* axis, and the screw-related molecules in adjacent columns are connected by C7—H7*A*···O3^{i} interactions (Table 1).

Experimental

Compound (I) was prepared by adding benzyl bromide (0.6 ml, 5.05 mmol) to a solution of *o*-vanillin (768 mg, 5.05 mmol), K_2CO_3 (4000 mg, 29 mmol) and Bu_4NI (188 mg, 0.51 mmol) in acetone (20 ml) with stirring for 3.5 h. The mixture was filtered and washed with acetone (5 ml); the solvent was then removed by rotary evaporation. Crushed ice (25 g) and $CHCl_3$ (25 ml) were added to the crude solution. The solution was shaken and the organic layer was collected by a separatory funnel. The resulting solution was washed with 10% NaOH (3 × 25 ml) followed by water (3 × 25 ml). The organic layer was dried over $MgSO_4$, filtered and the solvent was removed by rotary evaporation. The crude product was then purified by column chromatography with acetone–$CHCl_3$ (1:9). The product was dissolved in dimethyl sulfoxide, and single crystals suitable for X-ray diffraction were obtained by slow evaporation of the solvent at room temperature.

Crystal data

$C_{15}H_{14}O_3$
M_r = 242.26
Monoclinic, $P2_1/c$
a = 13.7203 (3) Å
b = 4.6599 (1) Å
c = 19.1552 (5) Å
β = 97.736 (1)°
V = 1213.55 (5) Å^3
Z = 4
D_x = 1.326 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100.0 (1) K
Block, colourless
0.48 × 0.28 × 0.12 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.807, T_{max} = 0.989
13932 measured reflections
3905 independent reflections
2949 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 31.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.187
S = 1.04
3905 reflections
164 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1222P)^2 + 0.1169P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7*B*···O2	1.01	2.45	2.974 (2)	112
C15—H15*A*···O1	0.98	2.43	2.815 (2)	102
C7—H7*A*···O3^{i}	1.00	2.57	3.434 (2)	144

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$

H atoms were placed in calculated positions, with C—H distances in the range 0.94–1.04 Å. The U_{iso}(H) values were set equal to 1.5U_{eq}

of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

We thank the Malaysian Government and Universiti Sains Malaysia for the IRPA short term grant 304/PKIMIA/636108.

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