

# Dimerization and cyclotrimerization of aldehydes: ruthenium catalyzed formation of esters, 1,3,5-trioxanes, and aldol condensation products from aldehydes

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Dedicated to Professor Jozef J. Ziolkowski on the occasion of his 70th birthday.

## Abstract

[RuCl(SiMe<sub>3</sub>)(CO)(PPh<sub>3</sub>)<sub>2</sub>] (**1**) was found to catalyze dimerization of aldehydes RC(O)H yielding esters RC(O)–OCH<sub>2</sub>R (**3**) in a Tishchenko reaction. Aldehydes with  $\alpha$ -hydrogen atoms (R = Me, Et, *i*Pr, *i*Bu) reacted quantitatively within 2 days (5 mol% catalyst, 70 °C in C<sub>6</sub>D<sub>6</sub>) whereas those without  $\alpha$ -hydrogen atoms (R = *t*Bu, Ph) did not react at all. In the case of benzaldehyde (R = Ph), well shaped crystals of [RuCl(O<sub>2</sub>CPh)(CO)(PPh<sub>3</sub>)<sub>2</sub>] (**4**) precipitated from the reaction mixture. Molecular structures of catalyst **1** and benzoato complex **4** were established by single-crystal X-ray diffraction analyses exhibiting square pyramidal (**1**) and octahedral (**4**) complex, respectively. Using 1:1 mixture of acetaldehyde and propanal a crossed esterification reaction took place forming esters RC(O)–OCH<sub>2</sub>R' (**3**, R/R' = Me/Me, Et/Et; **5**, R/R' = Me/Et, Et/Me). In the case of *t*BuC(O)H a crossed esterification reaction with RC(O)H (R = Me, Et) took place yielding only two products, namely the symmetrical esters RC(O)–OCH<sub>2</sub>R (**3**, R = Me, Et) and the crossed products of the type RC(O)–OCH<sub>2</sub>*t*Bu (**6**, R = Me, Et). As shown with propanal, ester formation is completely suppressed by water. Because ruthenium catalyst **1** was prepared by reaction of [RuCl(H)(CO)(PPh<sub>3</sub>)<sub>3</sub>] (**2**) with H<sub>2</sub>C=CHSiMe<sub>3</sub>, complex **2** was also used as catalyst in the reaction with aldehydes. It proved to be non-active, but in the presence of H<sub>2</sub>C=CHSiMe<sub>3</sub> (H<sub>2</sub>C=CHSiMe<sub>3</sub>:RC(O)H = 1:1) catalytic formation of cyclotrimers of aldehydes (RCHO)<sub>3</sub> (**7**) and aldol condensation products RCH=CR'–C(O)H (**8**, R = Et, *i*Bu; R' = R – CH<sub>2</sub>) was observed.

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**Keywords:** Ruthenium complexes; Dimerization of aldehydes; Tishchenko reaction; 1,3,5-Trioxanes; Aldol condensation

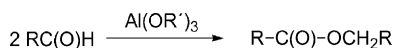
## 1. Introduction

In 1906, Tishchenko [1,2] found that aluminum alkoxides catalyze dimerization of aldehydes yielding esters that occurs by disproportionation of the aldehydes (Scheme 1). In general, more strongly basic catalysts catalyze aldol reaction of aliphatic aldehydes bearing an  $\alpha$ -hydrogen atom. Non-enolizable aldehydes (aromatic aldehydes and aliphatic ones without  $\alpha$ -hydrogen) undergo with stoichiometric amounts of OH<sup>–</sup> disproportionation yielding alcohols and carboxy-

lates (Cannizzaro reaction). Disproportionation of aldehydes – bearing  $\alpha$ -hydrogen or not – into alcohols and carboxylic acids can be catalyzed by platinum group metal complexes under essentially neutral conditions in water [3]. Catalysis of Tishchenko reaction by other platinum group metal complexes, namely by ruthenium [4–8], iridium [9,10], and osmium complexes [11] have also been reported. Overviews both on homogeneous and heterogeneous catalytic systems were given recently [12,13].

Here we report catalysis of Tishchenko reaction using [RuCl(SiMe<sub>3</sub>)(CO)(PPh<sub>3</sub>)<sub>2</sub>] (**1**) as precatalyst. It has been found a pronounced selectivity such that only aldehydes bearing  $\alpha$ -hydrogen atoms undergo Tishchenko reaction

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Scheme 1.

forming esters. Furthermore,  $[\text{RuCl(H)(CO)(PPh}_3)_3]$  (**2**)/ $\text{H}_2\text{C=CHSiMe}_3$  was found to catalyze cyclotrimerization and aldol condensation of aldehydes yielding  $(\text{RCHO})_3$  (**7**) and  $\text{RCH=CR'-C(O)H}$  (**8**,  $\text{R}' = \text{R} - \text{CH}_2$ ), respectively.

## 2. Results and discussion

$[\text{RuCl(SiMe}_3\text{)(CO)(PPh}_3)_2]$  (**1**) was prepared according to a method described by Wakatsuki from  $[\text{RuCl(H)(CO)(PPh}_3)_3]$  (**2**) and  $\text{H}_2\text{C=CHSiMe}_3$  [14,15]. Crystallization from benzene resulted in formation of well shaped single-crystals suitable for X-ray diffraction analysis. The molecular structure is shown in Fig. 1. The Cl and CO ligands are disordered over two positions with occupancies 0.51/0.49. Coordination geometry at Ru atom is close to a tetragonal pyramid with the strongly  $\sigma$  bound trimethylsilyl ligand in the apical position. Angles between apical and basal ligands ( $L_b$ )  $\text{Si-Ru-L}_b$  are between  $86(1)^\circ$  and  $102.6(2)^\circ$ . Without reporting details,  $[\text{RuCl(SiEt}_3\text{)(CO)(PPh}_3)_2]$  and  $[\text{RuCl}\{\text{Si(OEt)}_3\}\text{(CO)(PPh}_3)_2]$  were stated to have analogous structures [16].

$[\text{RuCl(SiMe}_3\text{)(CO)(PPh}_3)_2]$  (**1**) was found to catalyze dimerization of aldehydes that occurs by disproportionation of the aldehyde in a Tishchenko type reaction (Scheme 2).

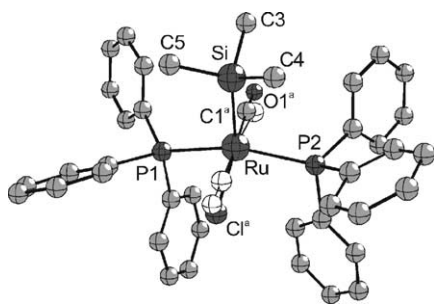
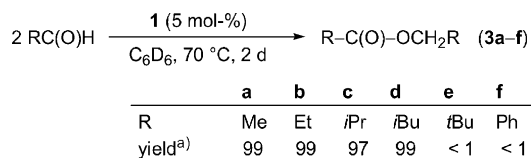


Fig. 1. Molecular structure of  $[\text{RuCl(SiMe}_3\text{)(CO)(PPh}_3)_2]$  (**1**) in the crystal. Atoms with site occupancies 0.51 are marked by exponents "a", the corresponding atoms with site occupancies 0.49 are drawn as colorless spheres. H atoms were omitted for clarity. Selected bond lengths (in Å) and angles (in  $^\circ$ ):  $\text{Ru-C1}$  1.71(2)/1.77(2),  $\text{Ru-Cl}$  2.473(5)/2.464(6),  $\text{Ru-P1}$  2.378(2),  $\text{Ru-P2}$  2.391(2),  $\text{Ru-Si}$  2.381(3),  $\text{C1-O1}$  1.20(3)/1.13(3);  $\text{P1-Ru-P2}$  158.88(9),  $\text{Cl-Ru-C1}$  171(1)/173.2(9),  $\text{Si-Ru-P1}$  102.01(7),  $\text{Si-Ru-P2}$  99.02(8),  $\text{Si-Ru-Cl}$  102.6(2)/99.5(2),  $\text{Si-Ru-C1}$  86(1)/87.3(9).



a) Degree of conversion in mol-% (determined by NMR spectroscopy).

Scheme 2.

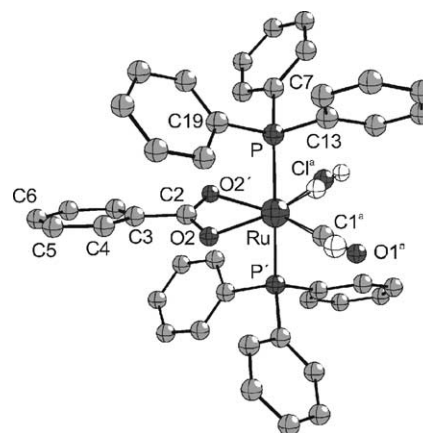
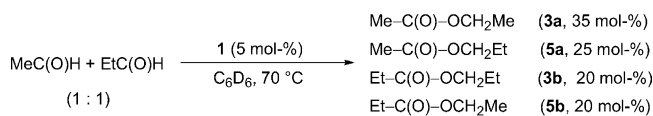


Fig. 2. Molecular structure of  $[\text{RuCl(O}_2\text{CPh)(CO)(PPh}_3)_2]$  (**4**) in the crystal. One set of the atoms with site occupancies 0.5 are marked by exponents "a", the other equivalent set is drawn as colorless spheres. H atoms were omitted for clarity. Selected bond lengths (in Å) and angles (in  $^\circ$ ):  $\text{Ru-C1}^a$  1.772(7),  $\text{Ru-O2}$  2.160(1),  $\text{Ru-P}$  2.4102(8),  $\text{Ru-Cl}^a$  2.372(2),  $\text{C1}^a\text{-O1}^a$  1.179(7);  $\text{P-Ru-P'}$  179.22(2),  $\text{O2-Ru-O2'}$  61.07(7),  $\text{C1}^a\text{-Ru-Cl}^a$  94.2(2),  $\text{C1}^a\text{-Ru-O2'}$  166.0(2),  $\text{Cl}^a\text{-Ru-O2}$  160.79(6),  $\text{O2-C2-O2'}$  118.3(2).

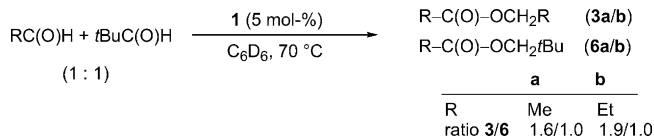
Using 5 mol% ruthenium catalyst **1**, propanal was converted into propyl propylate (**3b**) in benzene at  $70^\circ\text{C}$  to 95% after 1 day and to 99% after 2 days. Acetaldehyde, 2-methylpropanal (isobutyric aldehyde), and 3-methylbutanal (isovaleric aldehyde) behaved similarly yielding the corresponding esters. In contrast to it, 2,2-dimethylpropanal and benzaldehyde were not found to react yielding esters. If any, only traces of esters were formed after a reaction time of 8 days ( $70^\circ\text{C}$ ). Noteworthy, no other catalytic reactions of these two aldehydes were observed.

From reaction solutions using benzaldehyde as reactant well shaped yellowish orange crystals precipitated. By means of single-crystal X-ray diffraction measurement their identity was revealed to be  $[\text{RuCl(O}_2\text{CPh)(CO)(PPh}_3)_2]$  (**4**). The molecular structure is shown in Fig. 2. The carbonyl and chloro ligands are disordered over two equally occupied positions. Overall the (disordered) molecule exhibits crystallographically imposed  $C_2$  symmetry with symmetry axis defined by Ru, C2, C3, and C6 atoms. This disorder seems also to be the reason for the difficulties were encountered with the structure determination of **4** obtained from  $[\text{RuCl}_2(\text{PPh}_3)_3]$  and  $\text{PhC(O)H}$  at  $110^\circ\text{C}$  [17]. Ruthenium atom adopts an octahedral coordination with triphenylphosphine ligands in a mutual *trans* position. The benzoate ligand is symmetrically bidentate coordinated ( $\text{O}_2\text{CPh-}\kappa^2\text{O,O'}$ ). Formation of the coordinatively saturated complex **4** may be responsible for the deactivation of the catalyst. By means of NMR spectroscopy no formation of benzoic acid was detected indicating that complex **4** was formed in a non-catalytic reaction.

Analogous reaction as described in Scheme 2 but using two kinds of aldehydes, namely acetaldehyde and propanal in ratio 1:1 gave evidence for a crossed esterification reaction (Scheme 3). After 3 days (5 mol% **1**,  $70^\circ\text{C}$  in  $\text{C}_6\text{D}_6$ )



Scheme 3.



Scheme 4.

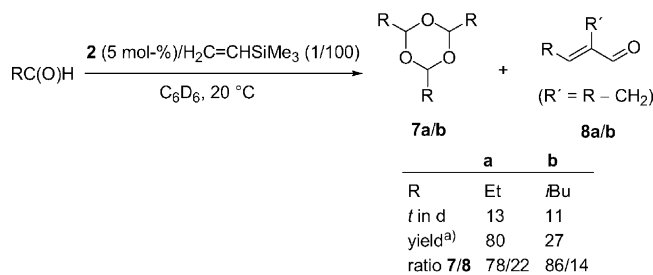
degree of conversion was about 70%. All four possible esters – the two symmetrical (**3a** and **3b**) and the two crossed products (**5a** and **5b**) – were formed. The slight excess (60:40) of acetic over propionic esters (**3a/5a** versus **3b/5b**) indicates that acetaldehyde was more readily oxidized in this reaction.

Although 2,2-dimethylpropanal was found not to dimerize yielding an ester (vide supra), a crossed esterification reaction with acetaldehyde and propanal took place. But only two products were formed, namely one symmetrical ester **3a/b** and one crossed ester **6a/b** (Scheme 4). Thus, 2,2-dimethylpropanal was reduced but not oxidized in the Tishchenko reaction catalyzed by ruthenium complex **1**. The observation that after two days (5 mol% **1**, 70 °C in C<sub>6</sub>D<sub>6</sub>) MeC(O)H and EtC(O)H, respectively, were completely converted into esters but not *t*BuC(O)H indicates the relatively low reactivity of 2,2-dimethylpropanal.

As shown with propanal as example, the reaction is sensitive to the presence of water. Addition of 2–10 equivalents of water with respect to **1** suppressed nearly completely ester formation. After 8 days, degree of conversion of the aldehyde was only 6 mol%, yielding cyclic trimer (EtCHO)<sub>3</sub> (**7a**) and propyl propylate (**3b**) in about 5:1 ratio.

By reaction of [RuCl(SiMe<sub>3</sub>)(CO)(PPh<sub>3</sub>)<sub>2</sub>] (**1**) with equimolar amounts of AgBF<sub>4</sub> or TlPF<sub>6</sub> in benzene the chloro ligand was substituted by an only weakly coordinating BF<sub>4</sub><sup>−</sup> and PF<sub>6</sub><sup>−</sup> ligand, respectively. But, these catalysts did not catalyze ester formation as shown with propanal as example. Instead of that, cyclotrimerization forming (EtCHO)<sub>3</sub> (**7a**) took place in the case of **1**/AgBF<sub>4</sub> (5 mol%, 60 °C, degree of conversion: ca. 20%) whereas with **1**/TlPF<sub>6</sub> only traces of the cyclotrimer **7a** were formed.

Trimethylvinylsilane is described to undergo with **1** a metathesis via insertion into the Ru–Si bond [18]. Here we found that addition of trimethylvinylsilane (H<sub>2</sub>C=CHSiMe<sub>3</sub>:RC(O)H = 1:1; R = Me, Et, *i*Bu) did not activate catalyst but resulted in lowering of reaction rate. After 3 days degree of conversion yielding esters **3** was about 70% (**3a**), 95% (**3b**) and 40% (**3d**). In the latter case also cyclotrimerization forming (*i*BuCHO)<sub>3</sub> (**7b**, about 5%) was observed. Absence of ethylene in reaction mixtures indicated that also metathesis of the vinylsilane was suppressed.



a) Degree of conversion in mol-% (determined by NMR spectroscopy).

Scheme 5.

Because ruthenium catalyst **1** was prepared by reaction of [RuCl(H)(CO)(PPh<sub>3</sub>)<sub>3</sub>] (**2**) with H<sub>2</sub>C=CHSiMe<sub>3</sub> [14,15], complex **2** was also used as catalyst in the reaction with aldehydes. But at 20 °C and even at 70 °C it proved to be non-active. On the other hand, in the presence of H<sub>2</sub>C=CHSiMe<sub>3</sub> (H<sub>2</sub>C=CHSiMe<sub>3</sub>:RC(O)H = 1:1) formation of cyclotrimers of aldehydes (**7**) and aldol condensation products (**8**) was catalyzed (Scheme 5). As shown with propanal as example the ratio cyclotrimer/aldol condensation product is dependent on reaction temperature such that the higher the temperature the lower the overall activity (Table 1). Most likely due to the reversibility of cyclotrimerization reactions of aldehydes, the ratio **7a/8a** is shifted in favor of **8a** with longer reaction times. Thus, methylrhenium trioxide was found to catalyze cyclotrimerization of aldehydes selectively and also the reverse reaction [19].

To get coordinatively non-saturated catalyst complexes, AgBF<sub>4</sub> and TlPF<sub>6</sub> was added to [RuCl(H)(CO)(PPh<sub>3</sub>)<sub>3</sub>] (**2**) in benzene resulting in precipitation of AgCl and TlCl, respectively. Using the supernatant as catalyst, with propanal/H<sub>2</sub>C=CHSiMe<sub>3</sub> as example, at room temperature only cyclotrimerization took place yielding (EtCHO)<sub>3</sub> (**7a**, yield, 2/9 d: 33/56%, AgBF<sub>4</sub>; 50/71%, TlPF<sub>6</sub>). Traces of aldol condensation product **8a** were only found with TlPF<sub>6</sub> as cocatalyst. Overall, as expected [20,21] catalytic formation of trioxanes **7** and aldol condensation products **8** is markedly dependent on Lewis acidity of the catalyst complex but the role of the vinylsilane is as yet unknown.

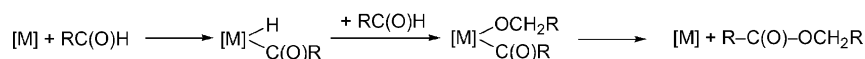
It is premature to discuss the reaction mechanism for ester formation reactions catalyzed by ruthenium complex **1**. Other transition metal catalyzed Tishchenko

Table 1

Temperature dependence of degree of conversion (in mol%) of the reaction of EtC(O)H yielding (EtCHO)<sub>3</sub> (**7a**) and Et-CH=C(Me)-C(O)H (**8a**) catalyzed by [RuCl(H)(CO)(PPh<sub>3</sub>)<sub>3</sub>] (**2**) (5 mol% in C<sub>6</sub>D<sub>6</sub>)

t	T (°C)		
	20–25 <sup>a</sup>	40 <sup>a</sup>	70 <sup>a</sup>
1/2 h	29 (100/0)	ca. 1 (100/0)	5 (100/0)
5 h	62 (100/0)	33 (80/20)	11 (100/0)
1 d	75 (90/10)	44 (80/20)	
13 d	80 (78/22)	50 (50/50)	23 (99/1)

<sup>a</sup> In parentheses the ratio **7a/8a** is given.



Scheme 6.

type reactions are discussed mainly in terms of oxidative C–H addition of the first aldehyde, insertion of the second one into the M–H bond followed by reductive elimination with C–O bond formation (Scheme 6). Ruthenium precatalysts like  $[\text{RuH}_2(\text{PPh}_3)_4]$  [4,5] and  $[\{\text{Ru}(\text{CO})_2\}_2(\mu\text{-H})(\mu\text{-C}_4\text{Ph}_4\text{COHOC}_4\text{Ph}_4)]/\text{HCOOH}$  [7] exhibited a higher activity than complex **1**, but in contrast to **1**, all these catalysts – so far investigated – catalyze both dimerization of aldehydes with and without  $\alpha$ -hydrogen atoms. Provided that sterical requirements do not markedly affect reaction, pronounced chemoselectivity of complex **1** may give rise to speculate on vinylalcohol or ketene like intermediate complexes.

### 3. Experimental

All reactions and manipulations were carried out under purified argon using standard Schlenk techniques. Solvents were dried and distilled under argon according to standard methods.  $\text{C}_6\text{D}_6$  was distilled from Na/K alloy.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on Varian Unity 500, VXR 400, and Gemini 200 spectrometers. Protio impurities and  $^{13}\text{C}$  resonances of the solvents were used as internal standards. GC/MS analyses were carried out using Hewlett Packard gas chromatograph (GC 5890 Series II) and mass spectrometer (MS 5972 Series) equipped with mass selective detector (70 eV).  $[\text{RuCl}(\text{SiMe}_3)(\text{CO})(\text{PPh}_3)_2]$  (**1**) [14,15] and  $[\text{RuCl}(\text{H})(\text{CO})(\text{PPh}_3)_3]$  (**2**) [22] were obtained by published methods. The other chemicals were commercially available. Prior to use aldehydes were distilled from  $\text{Na}_2\text{CO}_3$ . Trimethylvinylsilane contained traces of tetrahydrofuran, which were removed by stirring over  $\text{ZrCl}_4$  followed by vaporization.

#### 3.1. Catalytic reactions

A NMR tube was charged under argon with 0.4 ml of a 5 mol% solution of the catalyst in  $\text{C}_6\text{D}_6$ . After adding appropriate amounts of aldehyde and – if necessary – trimethylvinylsilane by means of syringes, NMR tubes were closed by melting. Reactions were performed in a shaking machine with automatic heat regulation. In appropriate time intervals  $^1\text{H}$  NMR spectra were recorded to monitor reactions using the intensity ratios of  $\text{RC}(\text{O})\text{H}$ ,  $\text{RC}(\text{O})\text{OCH}_2\text{R}'$  and  $(\text{RCHO})_3$  signals. Upon completion of reaction, identities of products were confirmed by comparison of their  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra with that of authentic samples as well as by GC/MS measurements.

#### 3.2. Reaction of ruthenium catalysts with $\text{AgBF}_4/\text{TIPF}_6$

To a vigorously stirred suspension of  $\text{AgBF}_4$  or  $\text{TIPF}_6$  ( $6 \times 10^{-5}$  mol) in  $\text{C}_6\text{D}_6$  (0.7 ml), a solution of **1** or **2** ( $6 \times 10^{-5}$  mol) in  $\text{C}_6\text{D}_6$  (0.7 ml) was added. The mixture was stirred at room temperature for 1 day in the dark forming a precipitate of  $\text{AgCl}$  and  $\text{TlCl}$ , respectively. After filtration, 0.7 ml of the clear solution were transferred into the NMR tube for catalytic runs.

#### 3.3. Crystallographic studies

Single-crystals which were suitable for X-ray diffraction measurements were obtained from benzene (**1**) and directly from catalytic runs using benzaldehyde as substrate (**4**), respectively. Intensity data were collected on a STOE-IPDS diffractometer with Mo-K $\alpha$  radiation (0.71073 Å, graphite monochromator,  $T=220(2)$  K). A summary of crystallographic data, data collection parameters, and refinement parameters is given in Table 2. Absorption correction for **4** was applied numerically ( $T_{\text{min}}/T_{\text{max}}$  0.800/0.878). The structures were solved by direct methods with SHELXS-97 and refined using full-matrix least-squares routines against  $F^2$  with SHELXL-97 [23]. Non-hydrogen atoms were refined with anisotropic and hydrogen atoms with isotropic displacement parameters. H atoms were added to the model in their calculated positions. In **1** and in **4** Cl, C1 and O

Table 2  
Crystal data and structure refinement for **1** and **4**

	<b>1</b>	<b>4</b>
Empirical formula	$\text{C}_{20}\text{H}_{20}\text{ClO}_2\text{RuSi}$	$\text{C}_{44}\text{H}_{35}\text{ClO}_3\text{P}_2\text{Ru}$
fw	502.91	810.18
Cryst. syst, space group	Monoclinic, $P2_1/c$	Monoclinic, $C2/c$
$a$ (Å)	11.635(4)	17.180(3)
$b$ (Å)	18.824(3)	11.506(3)
$c$ (Å)	16.983(3)	19.761(3)
$\beta$ (°)	95.85(3)	103.070(19)
$V$ (Å <sup>3</sup> )	3700.3(15)	3805.2(12)
$Z$	4	4
$\rho_{\text{calc}}$ (g/cm <sup>3</sup> )	0.903	1.414
$\mu$ (Mo-K $\alpha$ ) (mm <sup>-1</sup> )	0.619	0.606
$F(000)$	1012	1656
Scan range (°)	2.48–25.84	2.15–25.90
No. of reflns. collected	3535	14328
No. of indep reflns.	2816 ( $R_{\text{int}}=0.0390$ )	3619 ( $R_{\text{int}}=0.0388$ )
No. of params. refined	446	316
Goodness-of-fit on $F^2$	0.883	1.074
$R1, wR2$ ( $I > 2\sigma(I)$ )	0.0340, 0.0514	0.0256, 0.0691
$R1, wR2$ (all data)	0.0745, 0.0587	0.0297, 0.0708
Largest diff. peak/hole (e/Å <sup>3</sup> )	0.196/–0.225	0.388/–0.288

atoms are disordered over two positions with site occupancies 0.51(2)/0.49(2) (**1**) and 0.50/0.50 (**4**), respectively. Crystallographic data (excluding structure factors) have been deposited at the Cambridge Crystallographic Data Center as supplementary publication nos. CCDC-252208 (**1**) and CCDC-252209 (**4**). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge, CB2, 1EZ, UK. [Fax: (internat.) +44(0)1223/336-033; e-mail: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk)].

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