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Solid state isomerisation reactions of some ruthenium complexes

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Abstract

The isomerisation of *ttt*-$RuCl_2(RNC)_2(PPh_3)_2$ (R = 2,6-xylyl, ^{t}Bu, ^{i}Pr, benzyl, 2-OMe-4-Clphenyl) to *cct*-$RuCl_2(RNC)_2(PPh_3)_2$ has been carried out in the solid state. The reaction is first order and kinetic measurements have yielded activation energies of 210 kJ mol^{-1} (R = ^{t}Bu) and 221 kJ mol^{-1} (R = benzyl) for reactions performed between 160 and 180 °C. XRD analysis of the solid state reaction of *ttt*-$RuCl_2(^tBuNC)_2(PPh_3)_2$ has revealed that the *cct*-isomer produced is a polymorph of that produced by recrystallisation of the pure *cct*-$RuCl_2(^tBuNC)_2(PPh_3)_2$ isomer. A possible mechanism for the isomerisation reaction involving rotation of the two small groups (Cl, RNC) is proposed.

Keywords: Ruthenium; Solid state; Isomerisation; DSC; Isonitrile

1. Introduction

Reactions of inorganic coordination complexes in the solid state have been little studied [1,2]. There are many reasons for the limited exploration of this type of reaction—lack of contact between reactants, decomposition of reactants at the temperatures required for reaction and analytical difficulties. By contrast, the area of organic solid state chemistry has a rich and varied history spanning many decades [3,4].

Some years ago we commenced a study of isomerisation reactions of a range of organometallic complexes in the solid state. The reactions were focussed on pseudo 7-coordinate complexes of the type $CpML_4$ (M = Re, Mo, W; L a range of ligands) [5]. The study revealed that mechanistic pathways not available in a solution phase could occur in the solid state. This led to synthetic strategies to the synthesis of isomers that were not favoured in solution studies.

Studies on 4- and 6-coordinate inorganic systems have also been published in the literature and much of the early work on the study of solid state reactions of classical coordination complexes has been summarised by le May [2]. The summary reveals that in earlier work the emphasis was on the study of 6-coordinate Cr and Co complexes containing OH and H_2O ligands, and indicated the complexities associated with the presence of the OH/H_2O ligands. Thus simpler systems are required to obtain more fundamental information on the complex isomerisation and decomposition of 6-coordinate complexes. Solid state rearrangements are also well known for 4-coordinate Pt complexes of the type L_2PtX_2 (L = neutral donor, X halide) [2,6–11].

In this study, we wish to report our investigation on the solid state isomerisation reaction of a series of 6-coordinate Ru complexes of the type $RuCl_2(RNC)_2(PPh_3)_2$ (R = ^{t}Bu, 2,6-xylyl, benzyl, 2-OMe-4-Clphenyl and ^{i}Pr), complexes that undergo an isomerisation reaction from the *ttt* isomer to the *cct*-isomer (Fig. 1).

A preliminary report on the solid state isomerisation reaction of some related *ttt*-$RuCl_2(RNC)_2(PPh_3)_2$ complexes [13] as well as related *ttt*-$RuCl_2(CO)_2(PR_3)_2$ have been previously documented [14]. No kinetic data were however reported.

Interestingly, we have also observed that the reaction between $RuCl_2(PPh_3)_3$ and RNC to produce *ttt*-$RuCl_2(RNC)_2(PPh_3)_2$ can be carried out in the absence

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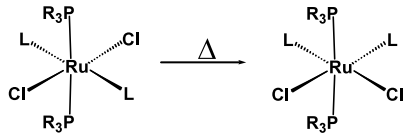

Fig. 1. Conversion of the *ttt* to the *cct*-isomer.

of solvents, and we also report our results on this unexpected finding.

2. Experimental

The RNC ligands (R = ^{t}Bu, 2,6-xylyl, benzyl, and ^{i}Pr) were obtained from Strem Chemicals. 2-OMe-4-ClphenylNC (MeClPhNC) was obtained as a gift from Dr M Layh of the School of Chemistry. The dichloromethane and Et_2O were distilled over $LiAlH_4$ and Na/bezophenone, respectively. The known complexes *ttt*-$RuCl_2(RNC)_2(PPh_3)_2$ (R = ^{t}Bu, 2,6-xylyl) were prepared by modification of the literature method [15]. The corresponding *cct*-isomers were prepared by heating the *ttt* isomers at high temperature in the solid state.

Thermal analysis was carried out on ~10 mg samples under flowing nitrogen at a constant heating rate of 10 °C min^{-1} with a Du Pont Instruments 910 Differential Scanning Calorimeter. Solution IR spectra were measured in CH_2Cl_2 on a Brucker VECTOR 22 FTIR spectrometer. The ^{1}H-NMR spectra were measured on a Brucker AVANCE 300 NMR spectrometer in $CDCl_3$ with Me_4Si as the reference.

2.1. Solution synthesis of *ttt*-$RuCl_2(^tBuNC)_2(PPh_3)_2$

A mixture of $RuCl_2(PPh_3)_2$ (1.0 g, 1.04 mmol) and ^{t}BuNC (0.189 g, 2.28 mmol) was dissolved in 30 ml freshly distilled CH_2Cl_2. The mixture was stirred for 1 h. The solvent was removed in vacuo and the orange residue was washed with 3 × 25 ml portions of Et_2O. The solid was dried under vacuum overnight. Yield = 0.716 g (80%). The analogous procedure was followed for the synthesis of *ttt*-$RuCl_2$(2,6-xylylNC)$_2(PPh_3)_2$ (85%), *ttt*-$RuCl_2$(benzylNC)$_2(PPh_3)_2$ (67%), *ttt*-$RuCl_2$(MeClPhNC)$_2(PPh_3)_2$ (20%) and *ttt*-$RuCl_2(^iPrNC)_2(PPh_3)_2$ (78%). IR and NMR data for the complexes are recorded in Tables 1 and 2, respectively and the DSC data in Table 3. Elemental analysis data (for the new complexes): $RuCl_2(MeClPhNC)_2(PPh_3)_2$ Anal. Found: C, 59.93; N, 2.99; H, 3.99. Calc.: C, 60.53; N, 2.72; H, 4.10%. *ttt*-$RuCl_2(^iPrNC)_2(PPh_3)_2$ Anal. Found: C, 63.12; N, 3.33; H, 5.34. Calc.: C, 63.31; N, 3.36; H, 5.31%.

Table 1
IR spectral positions of the CN bands of *ttt*- and *cct*-$RuCl_2(RNC)_2(PPh_3)_2$ complexes [a]

R	*ttt*-isomer/cm^{-1}	*cct*-isomer/cm^{-1}
^{t}Bu	2126	2112, 2154
Xylyl	2095	2094, 2141
Benzyl	2134	2131, 2176
^{i}Propyl	2131	2119, 2166
MeClPhNC [b]	2082, 2105	2082, 2144, 2105(sh)

[a] Recorded in CH_2Cl_2.
[b] Extra peaks may be associated with restricted rotation of the RNC ligand.

2.2. Solid state synthesis of *ttt*-$RuCl_2(xylylNC)_2(PPh_3)_2$

$RuCl_2(PPh_3)_2$ (0.093 g, 0.097 mmol) and 2,6-xylylNC (0.028 g, 0.21 mmol) were mixed together gently with a spatula and placed in an NMR tube. The NMR tube was evacuated and then purged with nitrogen. The sample mixture was then heated at 100 °C for less than 1 min. The product was left to cool down to room temperature (r.t.). It was then washed with Et_2O to remove the displaced triphenylphosphine ligand (yield: 0.080 g, 90%). Reaction with solid MeClPhNC gave a similar result (55% yield).

Similar reactions were performed (at 50 °C) with the other liquid isonitriles, but here the reactions occurred in the solution state. The liquid phase reaction also occurred slowly at r.t. (colour change seen with time).

2.3. Kinetic studies

Kinetic studies were performed at constant temperature (in the range 160–180 °C) by heating ca. 10 mg of the solid sample in a sealed NMR tube for various time intervals. The NMR tubes containing the solid samples were evacuated and purged with nitrogen prior to heating. After heating, the samples were cooled to r.t. and then dissolved in $CDCl_3$ and taken for ^{1}H-NMR measurement. The % *ttt* isomer versus time curves and the Arrhenius plot for each individual isomerisation process was constructed from the data. Typical data are shown in Figs. 2 and 3.

2.4. XRD data collection

Samples were ground with an agate pestle and mortar to a fine powder in preparation for powder X-ray diffraction analysis. A Philips PW 1710 diffractometer fitted with a Philips PW 1820 goniometer (with secondary beam monochromator and zero-background sample holder) was used to collect the diffractograms. Copper K-alpha radiation (λ = 1.5418 Å) was employed, with generator tension and current being 40 kV and 20 mA,

Table 2
^{1}H-NMR data for $RuCl_2(RNC)_2(PPh_3)_2$ complexes

R	CH_x region/ppm	Phenyl region/ppm
^{t}Bu-*ttt*	1.0 (s)	7.26–7.33 (m), 7.80–7.87 (m)
^{t}Bu-*cct*	0.72 (s)	7.31–7.34 (m), 7.86–7.90 (m)
2,6-Xylyl-*ttt*	2.05 (s)	6.91 (d), 6.94 (t), 7.10–7.14 (m), 7.78–7.85 (m)
2,6-Xylyl-*cct*	1.81 (s)	6.81 (d), 6.92 (t), 7.05–7.09 (m), 7.88–7.94 (m)
Benzyl-*ttt*	4.32 (s)	6.92–7.02 (m), 7.13–7.28 (m), 7.71–7.80 (m)
Benzyl-*cct*	3.76 (s)	6.60–6.63 (m), 7.13–7.30 (m), 7.90–8.00 (m)
^{i}Pr-*ttt*	0.92–0.95 (CH_3 d), 3.4–3.5 (CH, m)	7.30–7.32 (m), 7.79–7.82 (m)
^{i}Pr-*cct*	0.59–0.62 (CH_3 d), 3.4–3.6 (CH, m)	7.26–7.34 (m), 7.90–8.00 (m)
MeClPh-*ttt*	3.49 (s)	6.31–6.32 (d), 6.66–6.69 (d), 7.10–7.149 (dd), 7.24–7.26 (m), 7.84–7.9 (m)
MeClPh-*cct*	3.48 (d)	6.28, 6.29 (d); 6.56, 6.60 (d); 7.02, 7.03 (d); 7.05, 7.06 (d); 7.13–7.32 (m), 7.97–8.03 (m)

Recorded in $CDCl_3$.

Table 3
DSC of the *ttt*- and *cct*-$RuCl_2(RNC)_2(PPh_3)_2$ complexes

R	Exothermic peak/°C	Endothermic peak/°C
ttt-^{t}Bu	210	253
cct-isomer	–	254
ttt-Xylyl	230	317
cct-isomer	–	318
ttt-Benzyl	211	258–266
cct-isomer	–	254
ttt-^{i}Propyl	228	288
cct-isomer	–	290
ttt-MeClPhNC	208	240, 257 [a]
cct-isomer	–	240, 261 [a]

[a] Peak associated with decomposition; confirmed by TGA analysis.

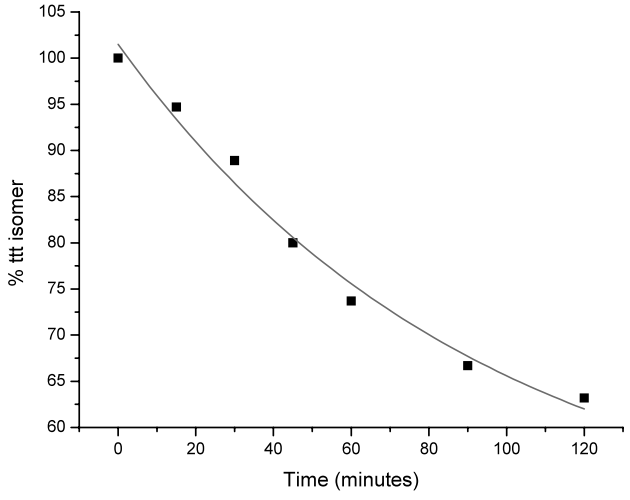

Fig. 2. Typical plot of % *ttt* isomer versus time plot for *ttt*-$RuCl_2(benzylNC)_2(PPh_3)_3$ (160 °C).

respectively. Samples were run as powders on rotating silicon wafers using silicone grease. Diffractograms were recorded in step-scan mode, from start angle (2θ) 3.000° to end angle (2θ) 70.000°, with a step size of (2θ) 0.020° and time per step of 25 s. Diffraction data was recollected after the initial X-ray exposure and found

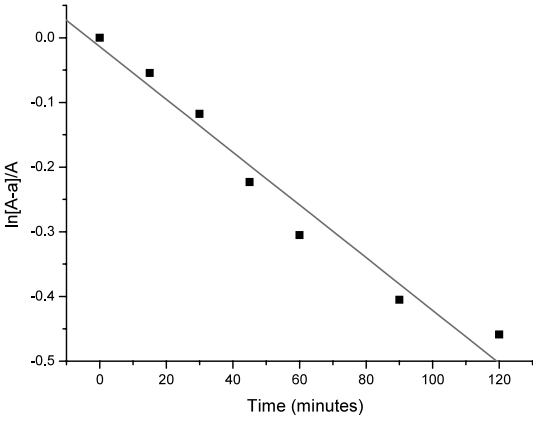

Fig. 3. Typical plot of $\ln \alpha$ versus time plot for $RuCl_2(benzylNC)_2(PPh_3)_2$ (160 °C).

to be essentially unchanged—hence samples showed no significant evidence of degradation within the X-ray beam. Diffractograms for RNC = ^{t}BuNC are presented in Fig. 4.

3. Results and discussion

3.1. Synthesis and characterisation of $RuCl_2(RNC)_2(PPh_3)_2$

$RuCl_2(PPh_3)_3$ and an appropriate amount of RNC were stirred together in CH_2Cl_2 at room temperature to give the yellow complexes of *ttt*-$RuCl_2(RNC)_2(PPh_3)_2$ with yields between 67 and 85%. Attempts to prepare these complexes in refluxing solvent, as described in the literature [15], yielded a mixture of a black and yellow solid. Separation of the mixture by column chromatography led to the disappearance of much of the yellow complex on the column resulting in a very low yield of the product. The Ru complexes are soluble in most of the polar organic solvents and insoluble in non-polar solvents.

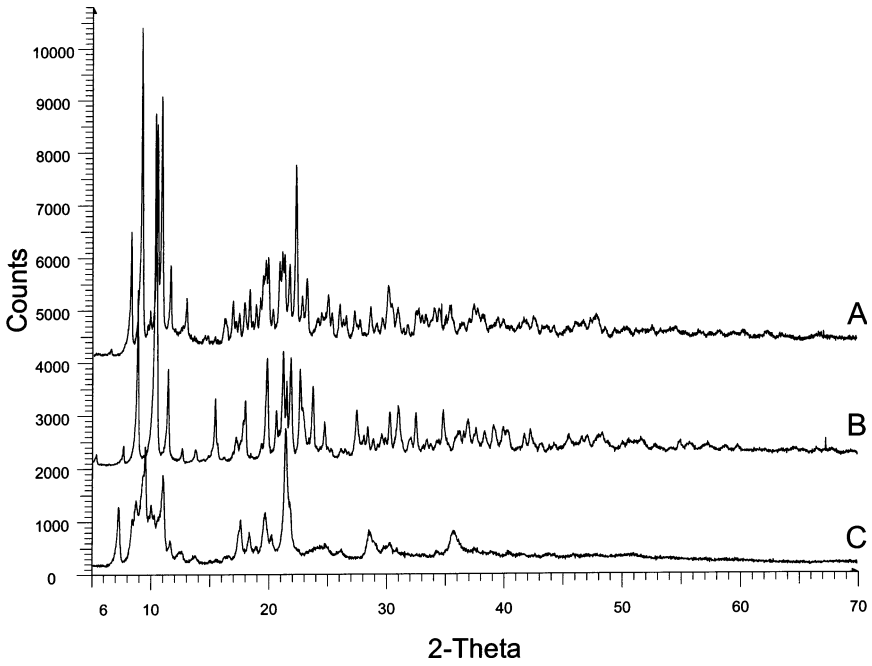

Fig. 4. XRD plots. (A) *ttt*-$RuCl_2(^tBuNC)_2(PPh_3)_2$; (B) *cct*-$RuCl_2(^tBuNC)_2(PPh_3)_2$ (obtained after complete solid state conversion from the *ttt* isomer); (C) *cct*-$RuCl_2(^tBuNC)_2(PPh_3)_2$ (obtained after recrystallisation from dichloromethane solution).

Heating a mixture of $RuCl_2(PPh_3)_3$ and 2,6-xylylisocyanide in the solid state between room temperature and 60 °C did not reveal any visual reaction (10 min) but at 100 °C rapid formation of a yellow complex occurred (< 1 min). A white deposit also formed on the complex that was identified as the triphenylphosphine ligand. Washing the solid with diethyl ether left behind a bright yellow solid. Since the melting point of the isonitrile is ca. 175 °C this suggests that the reaction occurred in the solid phase or between a solid and a vapour (xylylisonitrile has a high vapour pressure at room temperature). Either way, the Ru complex remained in the solid state throughout the reaction. A similar result was obtained with solid 2-OMe-4-ClphenylNC, showing the generality of the reaction.

Reaction between $RuCl_2(PPh_3)_3$ and the liquid isonitriles occurred without the addition of solvent at room temperature to slowly (or more rapidly at 50 °C) give the required complexes in > 85% yield. Thus very clean reactions are possible in the absence of solvent [16,17].

Characterisation of the isonitrile complexes by ^{1}H-NMR and IR spectroscopy gave data consistent with the structure of *ttt*-$RuCl_2(RNC)_2(PPh_3)_2$. When *ttt*-$RuCl_2(RNC)_2(PPh_3)_2$ was heated in the solid state pale yellow to near white complexes of *cct*-$RuCl_2(RNC)_2(PPh_3)_2$ (R = ^{t}Bu, 2,6-xylyl, ^{i}Pr, benzyl, MeClPh) were produced in quantitative yield. The complexes with R = ^{t}Bu, benzyl and 2,6-xylyl have been prepared before while the other complexes are new. The solid state isomerisation reaction of the complexes $RuCl_2(RNC)_2(PPh_3)_2$ was monitored by IR, DSC, ^{1}H-NMR and XRD techniques.

3.1.1. IR studies

Solution IR spectra of the *ttt*-isomers display one strong ν(CN) band. Two bands were noted for the MeClPh complex; this could arise from two different arrangements of the bulky OMe groups attached to the ligand. Analogous *ttt*-$RuCl_2(EtNC)_2(EPh_3)_2$ (where E = P, As or Sb and X = Cl or Br) complexes were also reported to show one absorption band, both in the solid state and in solution [12]. The ν(CN) band position increases in the order 2,6-xylyl < MeClPh < ^{t}Bu < ^{i}Pr ≤ benzyl, suggestive of a correlation with the electron donating capacity of the RNC ligand.

When the complexes *ttt*-$RuCl_2(RNC)_2(PPh_3)_2$ are heated in the solid state two ν(CN) IR bands, with positions different from that of the original samples, were detected. These are associated with the cis arrangement of the isonitrile ligands and the band positions again vary with the electron donating capacity of the RNC ligand.

3.1.2. Characterization by ^{1}H-NMR spectroscopy

^{1}H-NMR spectral data for the samples are given in Table 2. The NMR data are consistent with the proposed molecular structures.

The *ttt*-$RuCl_2(^tBuNC)_2(PPh_3)_2$ isomer undergoes 100% conversion to the *cct* complex on heating. Similar results were found for the other complexes. The CH_3/CH_2 resonance of the RNC ligand of the *ttt* isomer always shifted downfield as the *cct*-isomer formed. These NMR absorptions were thus chosen to monitor the extent of the isomerisation reaction as a function of time (see below).

3.1.3. DSC studies

The results of a DSC study for the different RNC complexes are shown in Table 3. The DSC profiles of all the *ttt* complexes exhibit the same pattern, with an exotherm (isomerisation peak) appearing before the endotherm (melting peak). The endotherm corresponds to the melting point of the *cct*-isomer and ranges between 250 and 320 °C. The exothermic peaks range between 210 and 230 °C. A DSC profile for a typical isomer pair viz. *ttt*- and *cct*-$RuCl_2(^iPrNC)_2(PPh_3)_2$ is shown in Fig. 5.

It will be noted that the profiles for the two isomers differ. Both isomers exhibit the same endotherm (melting peak; 288 °C) but only the *ttt* isomer exhibits an exotherm (isomerisation peak; 228 °C) (Fig. 2). The DSC profile reveals that the isomerisation of the *ttt* complexes takes place in the solid state. This was confirmed by heating the *ttt* isomer to above the exotherm and then cooling the sample. Part of the sample was then used to record a NMR spectrum that revealed only the *cct*-isomer while the second part was used to re-record the DSC that indicated no exotherm in the rerun.

The DSC profiles of R = 2,6-xylyl, and ^{t}Bu have been reported previously [13] and minor changes in the positions of the endothermic/exothermic peaks between our data and the literature data were noted. The differences are associated with different operating conditions and the different instruments used.

The DSC studies on the *ttt* complexes show that the complexes undergo similar rearrangement processes irrespective of the RNC ligand used.

3.1.4. XRD studies

A preliminary XRD study of the complexes was attempted and data for $RuCl_2(^tBuNC)_2(PPh_3)_2$ are shown in Fig. 4. Fig. 4A gives the XRD pattern for the *ttt* isomer. The conversion of this *ttt* isomer to the *cct*-isomer was established by powder XRD. The XRD pattern of the fully isomerised material produced in the solid state reaction i.e. *cct*-$RuCl_2(^tBuNC)_2(PPh_3)_2$ (degree of reaction established by solution NMR spectroscopy) is shown in Fig. 4B. The two isomers have quite distinctive XRD patterns. The *cct*-isomer was then recrystallised (CH_2Cl_2–hexane) and the XRD powder pattern recorded (Fig. 4C). The patterns of the two *cct* samples are quite different, suggesting that the *cct*-isomer can exist as a number of polymorphs. This issue is currently under investigation.

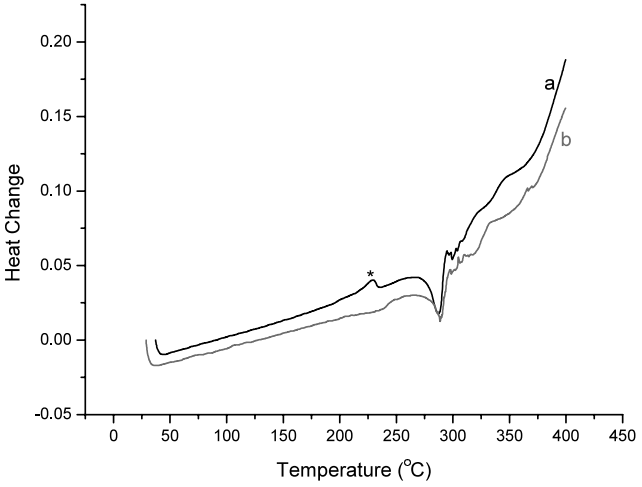

Fig. 5. DSC profile of (a) *ttt*- and (b) *cct*-$RuCl_2(^iPrNC)_2(PPh_3)_2$ recorded at a scan rate of 10 °C min^{-1}.

3.2. Kinetic studies

The kinetic studies of the isomerisation reactions were followed by heating solid samples for periods of time and then dissolving the material and evaluating the degree of reaction by ^{1}H-NMR spectroscopy. The peaks that appeared in the CH_3/CH_2 region of the NMR spectrum were used to monitor these reactions. For example, in the ^{t}Bu complex, the methyl peak at 1.00 ppm gradually disappears and is replaced by the peak at 0.70 ppm corresponding to the newly formed *cct*-isomer. By measuring the disappearance of the *ttt* complex, as a function of time, it is therefore possible to evaluate the advance of the solid state isomerisation reaction.

Kinetic studies were performed on ruthenium complexes with R = ^{t}Bu and benzyl. When complexes with R = 2,6-xylyl or ^{i}Pr were used, the reaction progression was difficult to follow as the corresponding *cct*-isomers were poorly soluble in the solvents used, and hence precipitate out of, $CDCl_3$ or C_6D_6.

The percent conversion (α) at a given time t was calculated from the expression

$$\alpha_{(ttt)} = \{I_{(ttt)}/[(I_{(ttt)} + I_{(cct)}]\} \times 100$$

where $I_{(ttt)}$ is the intensity of the *ttt*-isomer and $I_{(cct)}$ is the intensity of the *cct*-isomer, the intensity data being obtained from the solution NMR spectra of the samples after reaction. The α versus time curves for the isomerisation process for the two *ttt* complexes show a similar sigmoid shape (see Fig. 3). The plots of ln α versus time yielded a straight line that is expected of a first order reaction (see Fig. 4).

From these plots, the rate constant values for ^{t}Bu complexes were found to be $0.0044 \pm 6.2 \times 10^{-4}$, $0.0025 \pm 3.2 \times 10^{-4}$, $8.58 \times 10^{-4} \pm 4.3 \times 10^{-4}$ at 185, 180 and 175 °C, respectively. The activation energy, Ea, derived from the least-squares analysis of the best fit from the ln K versus $1/T$ plot was found to be 210 kJ mol^{-1}.

For the benzyl complex, the samples were heated at 160, 170 and 180 °C and the rate constants were found to be $0.0041 \pm 3 \times 10^{-4}$, $0.0057 \pm 4.0 \times 10^{-4}$ and

$0.029 \pm 0.004\ s^{-1}$, respectively. The activation energy Ea was found to be 221 kJ mol^{-1}.

3.2.1. Mechanism

The conversion of the *ttt* isomer to the *cct*-isomer in the solid state is unidirectional, and no *ccc* isomer was detected [18]. The reaction is unimolecular and first order. It is assumed that the isomerisation reaction is an intramolecular process involving no loss of ligands from the Ru during the ligand exchange.

Three possible mechanisms for solid state reactions of 6-coordinate metal complexes have been proposed and these involve rotation of two or three ligands relative to the other ligands [19]. A consideration of the complexes synthesised in this study (see Fig. 1) suggests that only one pair of ligands (Cl, RNC) needs to exchange to bring about the isomerisation reaction. These are the small ligands. Movement of the large PPh_3 ligands in the solid state is less likely although flexing of these and the other ligands to assist with the process is expected. The reaction could thus entail a direct Cl/RNC exchange via ligand rotation of 180° around an axis through Ru—i.e. via a bicapped tetrahedral intermediate followed by a 180° rotation around the tetrahedral edge [20]. This would be a high-energy process, consistent with the activation energy data generated (> 200 kJ mol^{-1}).

4. Conclusion

The isomerisation reaction of a series of *ttt*-$RuCl_2(RNC)_2(PPh_3)_2$ (R = 2,6-xylyl, ^{t}Bu, ^{i}Pr, benzyl, MeClPh) complexes to their *cct*-$RuCl_2(RNC)_2(PPh_3)_2$ isomers has been studied in the solid state. Kinetic data for the unidirectional reaction were obtained and are consistent with a first order reaction. A mechanism in which only two groups (Cl, RNC) interchange was proposed, similar to that proposed recently for some 7-coordinate Re complexes [5].

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