

Non covalent immobilization of pyrene-tagged ruthenium complexes onto graphene surfaces for recycling in olefin metathesis reactions

Houssein Nasrallah,^a Stéphane Germain,^b Pierre Queval,^b Caroline Bouvier,^a

Marc Mauduit,^b Christophe Crévisy^{b*} and Emmanuelle Schulz^{a*}

^a*Institut de Chimie Moléculaire et des Matériaux d'Orsay, CNRS UMR 8182, Université Paris-Sud,
Université Paris-Saclay, bâtiment 420, 91405 Orsay cedex, France
00 33 1 69 15 73 56, emmanuelle.schulz@u-psud.fr*

^b*Ecole Nationale Supérieure de Chimie de Rennes, CNRS UMR 6226, 11 allée de Beaulieu, CS 50837,
35708 Rennes cedex 7, France
00 33 2 23 23 80 74, christophe.crevisy@ensc-rennes.fr*

CONTENT

Kinetic profile in the homogeneous ene-yne RCM of **S2** and the RCM of **S3** with **C1** and **C2**.

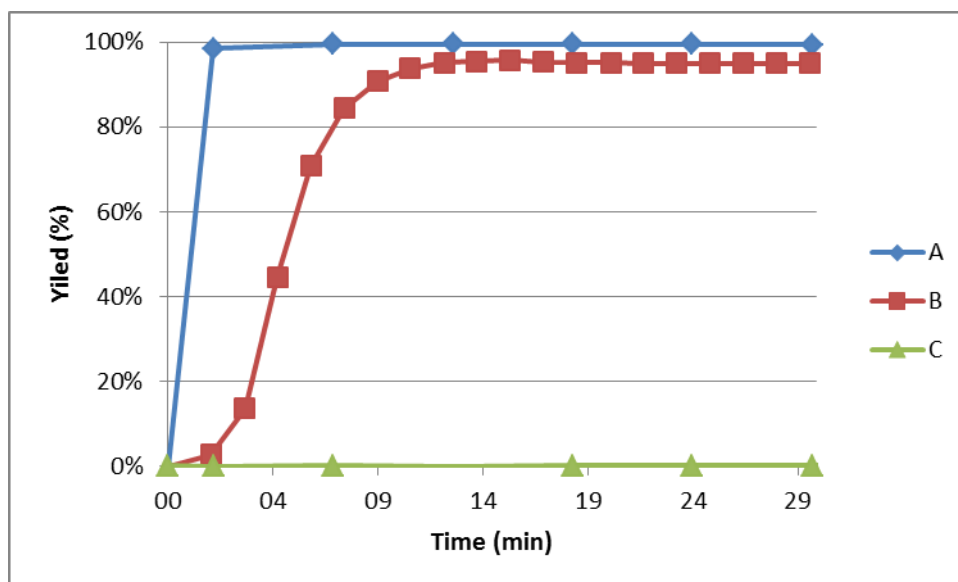
Study of the stability of **C3** in CDCl₃ solution by ¹H-NMR.

UV-Visible spectra for the immobilization of **C1**, **C2**, **C3** and **L**.

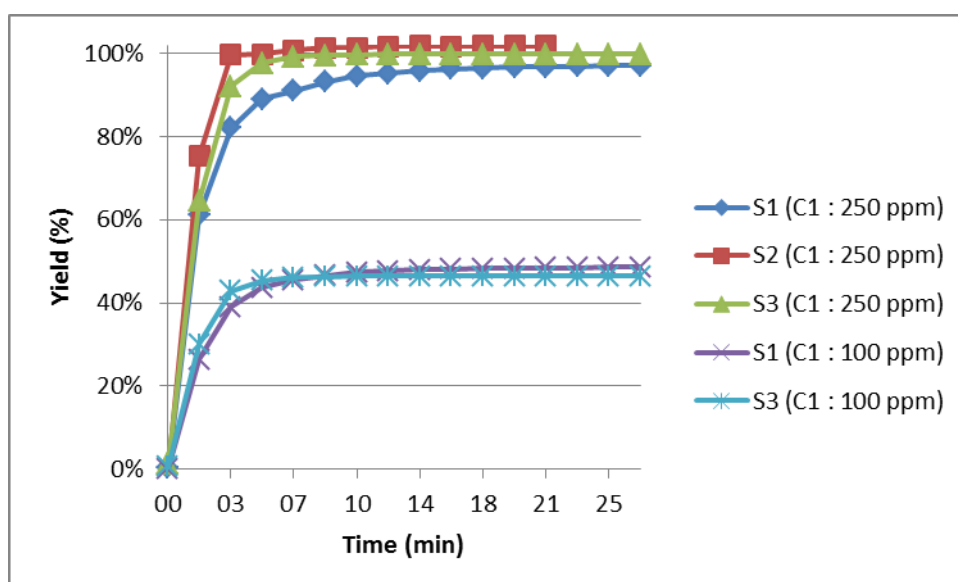
Description of the determination by GC analysis for **S1** (with pentadecane as internal standard), **S2** and **S3**. Copy of representative chromatograms.

Copy of ¹H- and ¹³C-NMR spectra.

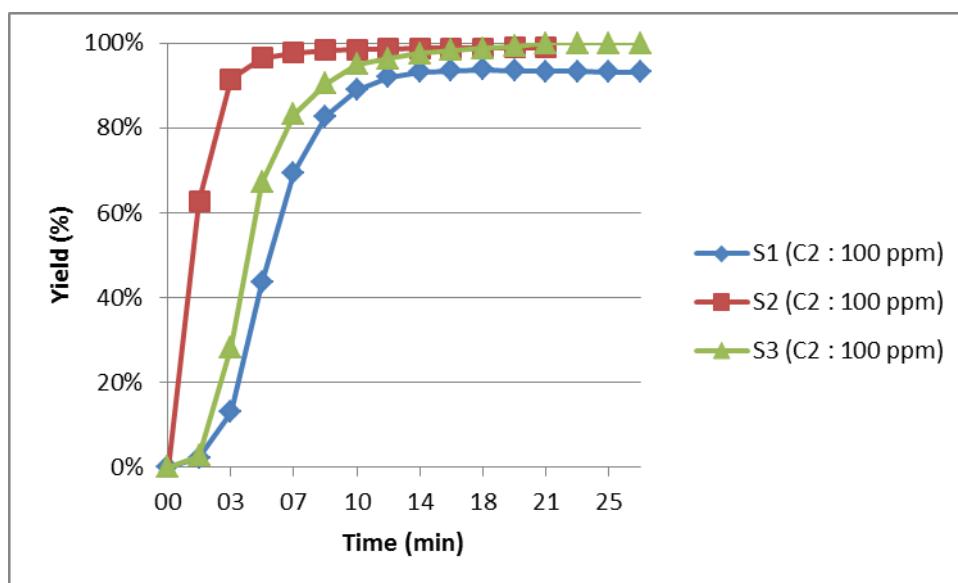
Kinetic profile in the homogeneous ene-yne RCM of **S2** and the RCM of **S3** with **C1** and **C2**.



Kinetic profiles of DEDAM (**S1**) metathesis using **C2** as catalyst. Conditions : [DEDAM]=0.2 M, toluene- D_8 , yield determined by NMR 1H spectroscopy using mesitylene as internal standard. A) [**C2**]=250 ppm, 60 °C, B) [**C2**]=100 ppm, 60 °C, C) [**C2**]=500 ppm, rt.

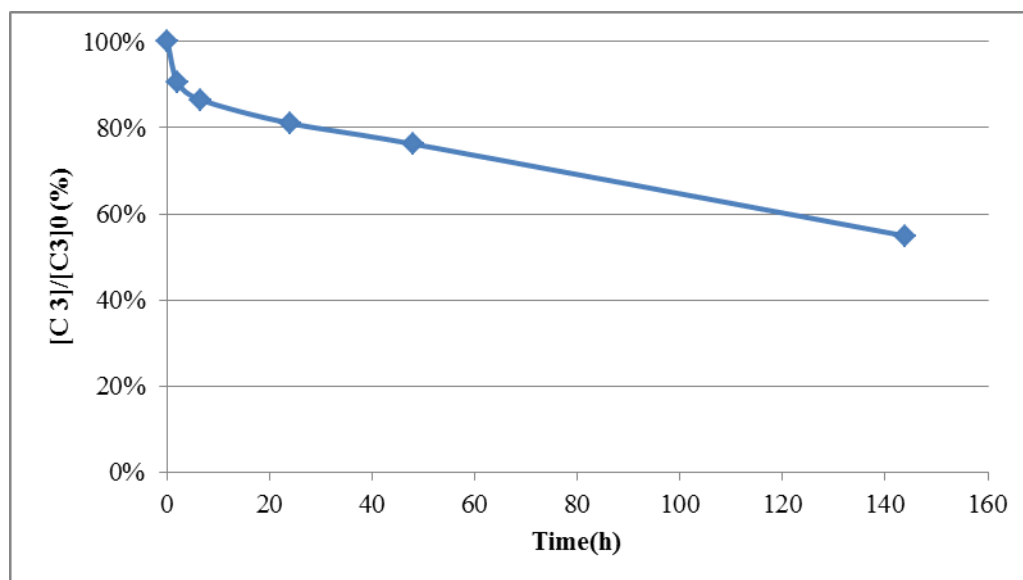


Kinetic profiles of **S1**, **S2** and **S3** metathesis using **C1** as catalyst. Conditions : [**Sx**]=0.2 M, toluene- D_8 , 60 °C, yield determined by NMR 1H spectroscopy using mesitylene as internal standard.



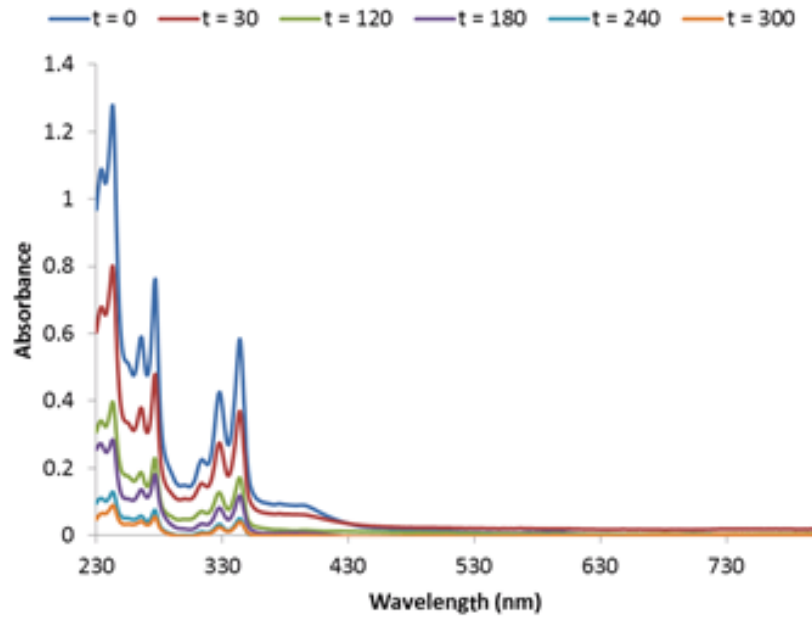
Kinetic profiles of **S1**, **S2** and **S3** metathesis using **C2** as catalyst. Conditions : $[S_x]=0.2$ M, toluene- D_8 , 60 °C, yield determined by NMR 1H spectroscopy using mesitylene as internal standard.

Study of the stability of **C3** in $CDCl_3$ solution by 1H -NMR

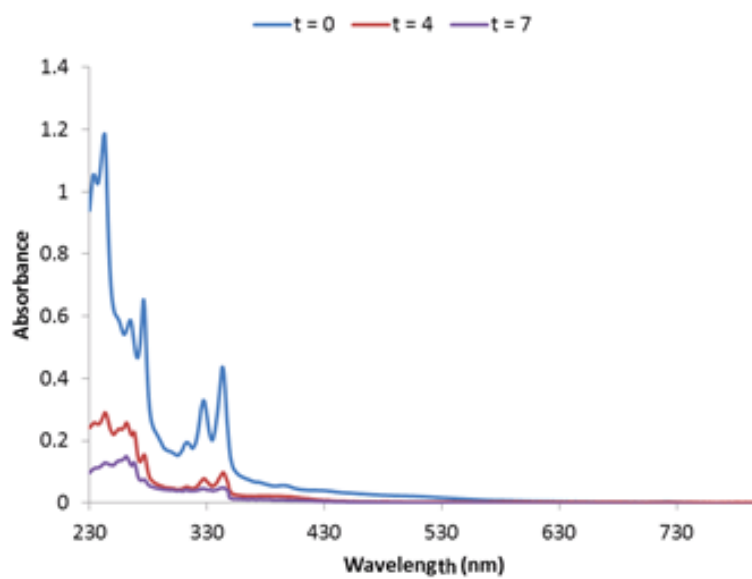


Study of the stability of **C3** in $CDCl_3$ solution by 1H -NMR spectroscopy. Conditions : $[C3]_0 = 32$ mM, 1,1,2,2-tetrachloroethane (internal standart) 16 mM, $CDCl_3$ 500 μ L, 60 °C. Degradation mesured by integrating Ru=CH signal against internal standard.

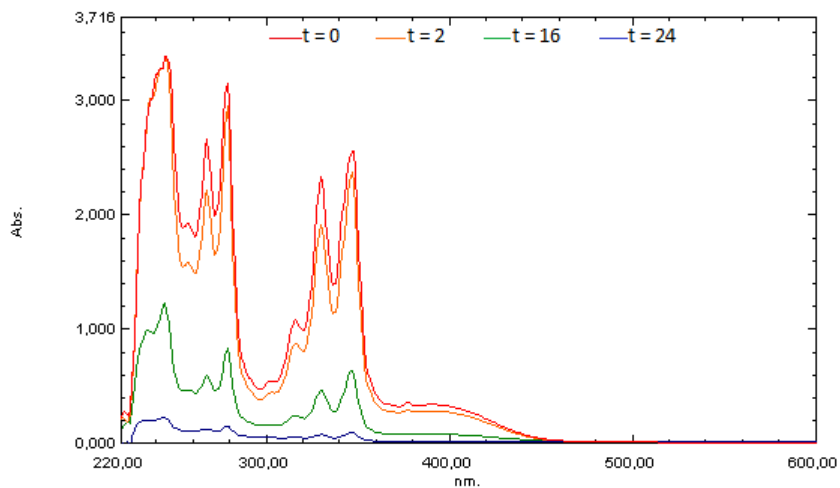
UV-Visible spectra for the immobilization of C1, C2, C3 and L



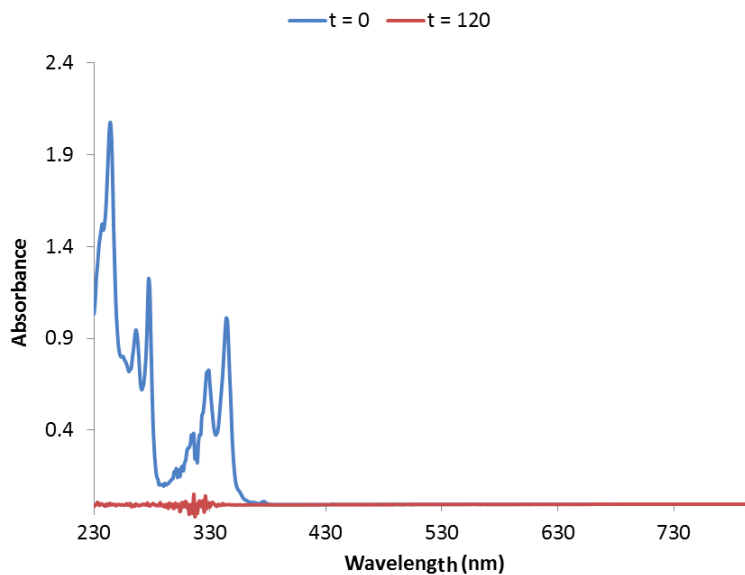
UV-Vis Spectra for the immobilization of **C1** on rGO, in DCM, room temperature, **C1**/rGO = 1/20, t(min)



UV-Vis Spectra for the immobilization of **C2** on rGO, in DCM, room temperature, **C2**/rGO = 1/20, t(h).



UV-Vis Spectra for the immobilization of **C3** on rGO, in DCM, room temperature, **C3**/rGO = 1/40, t(h)

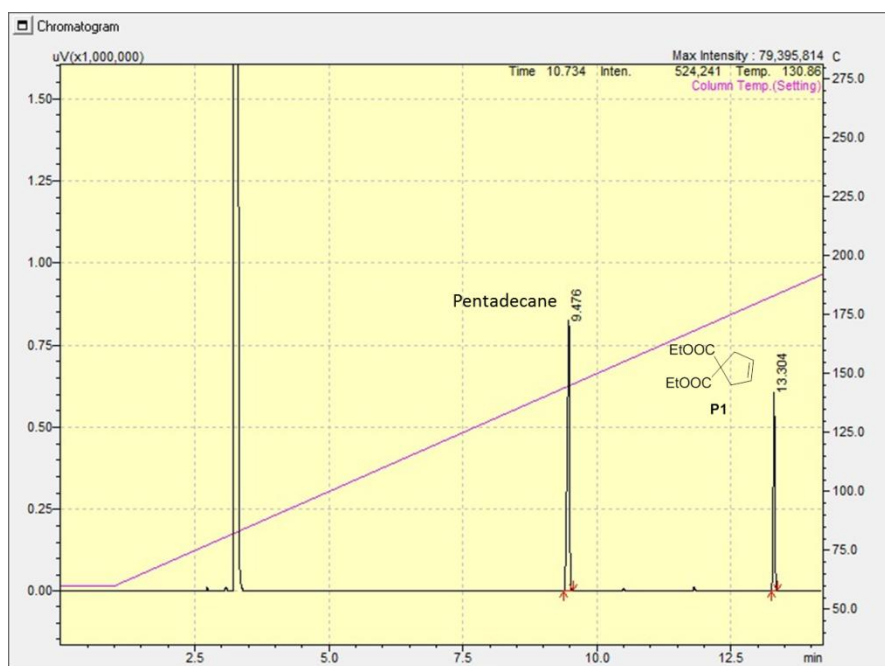
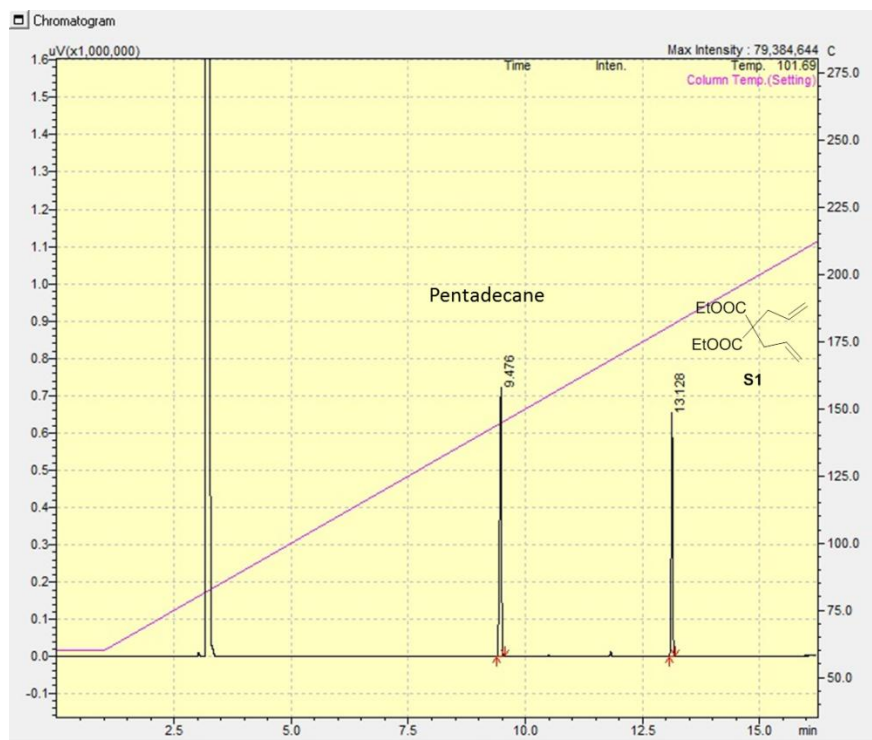


UV-Vis Spectra for the immobilization of **L** on rGO, in DCM, room temperature, **L**/rGO = 1/10, t(min)

Description of the determination by GC analysis for S1 (with pentadecane as internal standard), S2 and S3. Copy of representative chromatograms

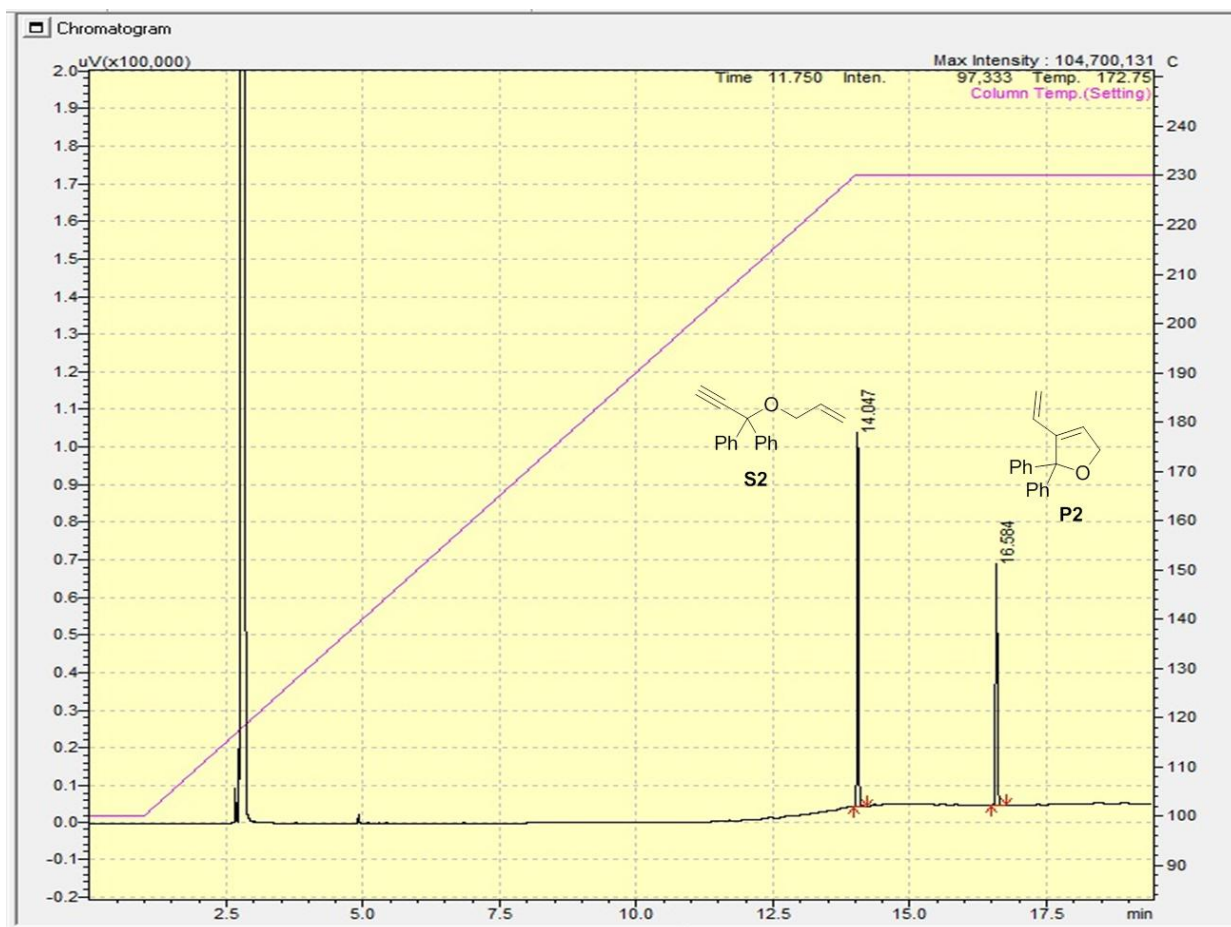
For substrate **S1** : Column type: DB-23, 60m × 0.25mm, 0.25µm.

Method: 60 °C (1 min), 10 °C/min to 250 °C (5 min).



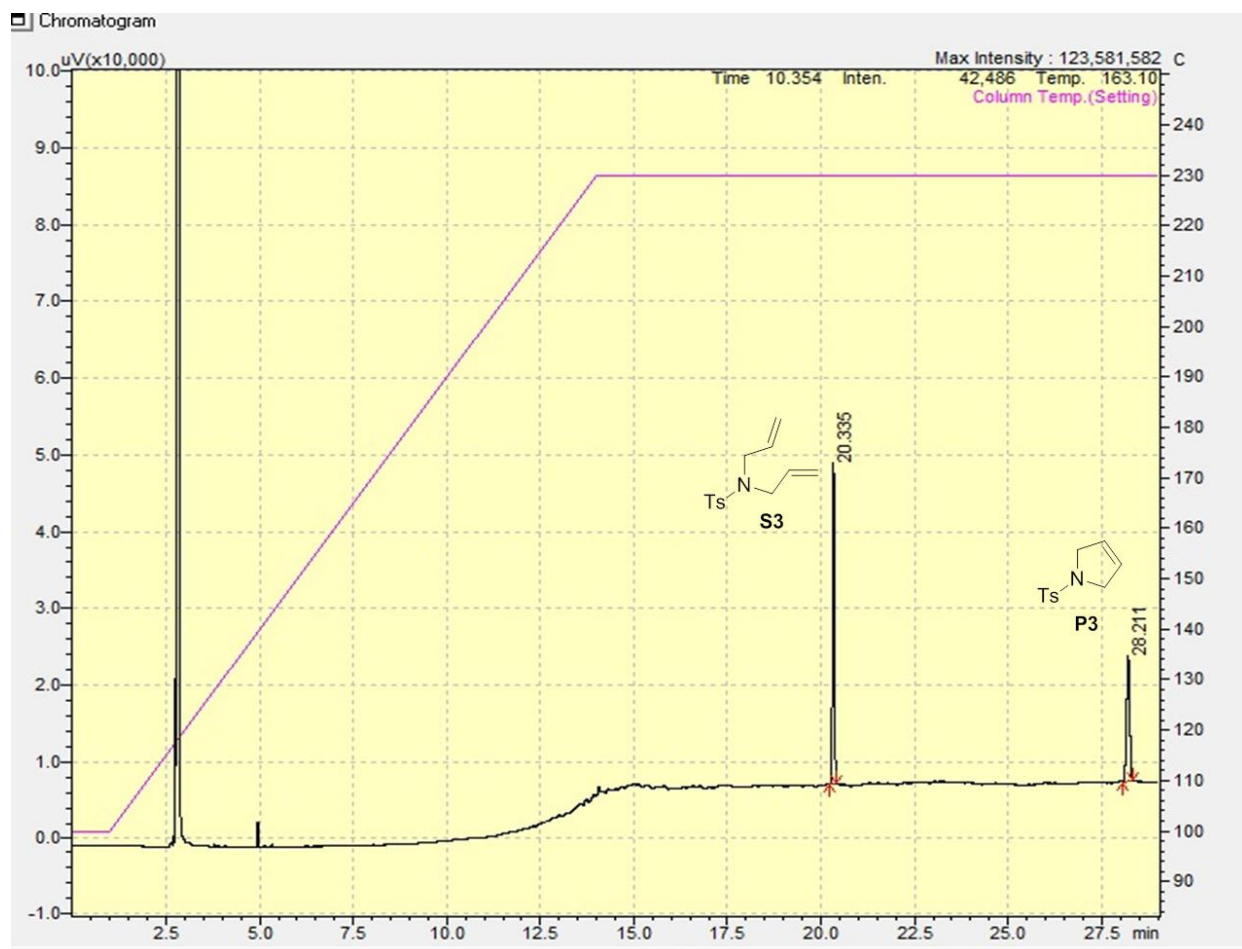
For substrate **S2** : Column type: DB-23, 60m × 0.25mm, 0.25µm.

Method: 100 °C (1 min), 10 °C/min to 250 °C (15 min).



For substrate **S3** : Column type: DB-23, 60m × 0.25mm, 0.25µm.

Method: 100 °C (1 min), 10 °C/min to 250 °C (15 min).



Copy of ¹H- and ¹³C-NMR spectra

