

## (Supporting Information)

### A New Osmium-Based Reagent for the Dihydroxylation of Alkenes

*Timothy J. Donohoe,<sup>a,\*</sup> Robert M. Harris,<sup>a</sup> Sam Butterworth,<sup>a</sup> Jeremy N. Burrows<sup>b</sup>  
Andrew Cowley,<sup>a</sup> and Jeremy S. Parker<sup>c</sup>*

<sup>a</sup> Department of Chemistry, University of Oxford, Chemistry Research Laboratory, Mansfield Road,  
Oxford, OX1 3TA, UK

<sup>b</sup> AstraZeneca Pharmaceuticals, Mereside, Alderley Park, Macclesfield, Cheshire, SK10 4TG, UK

<sup>c</sup> AstraZeneca, Process R&D Avlon/Charnwood, Avlon Works, Severn Road, Hallen, Bristol,  
BS10 7ZE, UK

[timothy.donohoe@chem.ox.ac.uk](mailto:timothy.donohoe@chem.ox.ac.uk)

#### Table of Contents

S-2 General Details and Procedures

S-2 References

S-3 <sup>1</sup>H and <sup>13</sup>C NMR data for **7**

S-4 <sup>1</sup>H and <sup>13</sup>C NMR data for **8**

S-5 <sup>1</sup>H and <sup>13</sup>C NMR data for **9**

S-6 <sup>1</sup>H and <sup>13</sup>C NMR data for **10**

S-7 <sup>1</sup>H and <sup>13</sup>C NMR data for **13α**

S-8 <sup>1</sup>H and <sup>13</sup>C NMR data for **13β**

S-9 <sup>1</sup>H and <sup>13</sup>C NMR data for **15**

S-10 <sup>1</sup>H and <sup>13</sup>C NMR data for **20**

S-11 <sup>1</sup>H and <sup>13</sup>C NMR data for **21**

S-12 <sup>1</sup>H and <sup>13</sup>C NMR data for **22**

S-13 <sup>1</sup>H and <sup>13</sup>C NMR data for **23**

S-14 <sup>1</sup>H and <sup>13</sup>C NMR data for **26**

## General Details.

Proton nuclear magnetic resonance spectra (NMR) were recorded at 400 MHz.  $^{13}\text{C}$  NMR spectra were recorded at 100 MHz. Infrared spectra (IR) were recorded as evaporated films. Chemical ionisation (CI) was performed using  $\text{NH}_3$ . Electrospray ionisation (ESI) was performed using tetraoctylammonium bromide as the lock mass. All solvents and reagents requiring purification were purified using standard laboratory techniques according to methods published by Perrin,<sup>1</sup> apart from  $\text{CH}_2\text{Cl}_2$ , THF and  $\text{Et}_2\text{O}$  which were dried by filtration through an activated alumina purification column. Petrol refers to petroleum ether in the boiling range 40-60 °C.

## General Experimental Procedures

### General Procedure A. Osmium(VI) intermediate formation with *t*-BuOOH:

A 70% aqueous solution of *tert*-butyl hydroperoxide (12 eq.) was added to a solution of osmium(VI) reagent **7** (1 eq.) and alkene (2 eq.) in  $\text{CHCl}_3$  (50 ml/mmol). The reaction was stirred for 5 min at room temperature, dried ( $\text{MgSO}_4$ ) and evaporated directly onto silica. The product was isolated by flash column chromatography ( $\text{SiO}_2$ , petrol  $\rightarrow$  petrol- $\text{Et}_2\text{O}$ , 80:20).

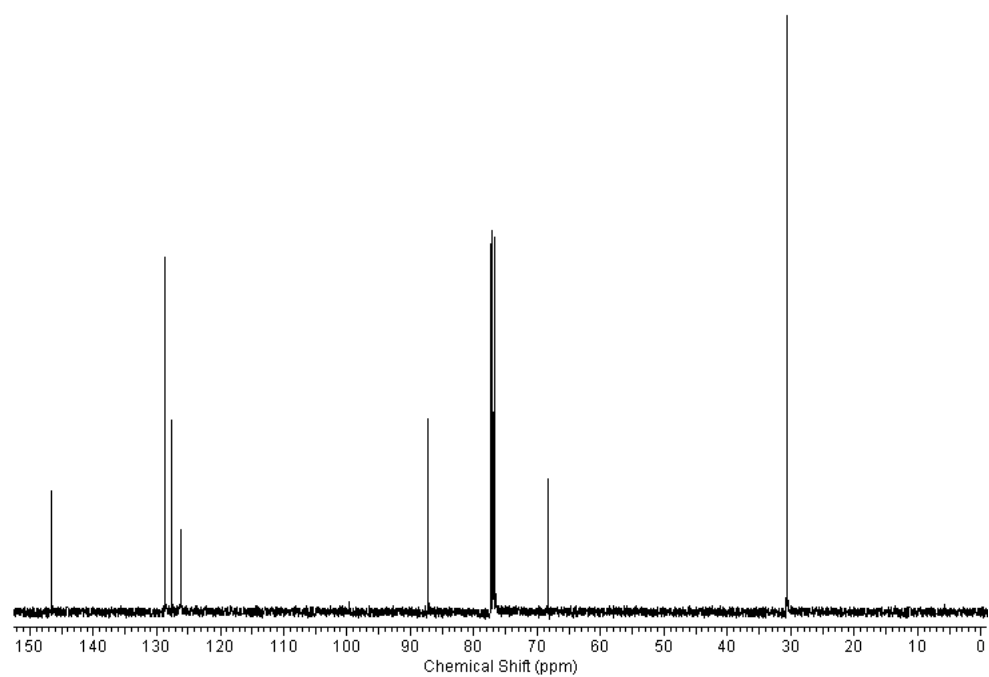
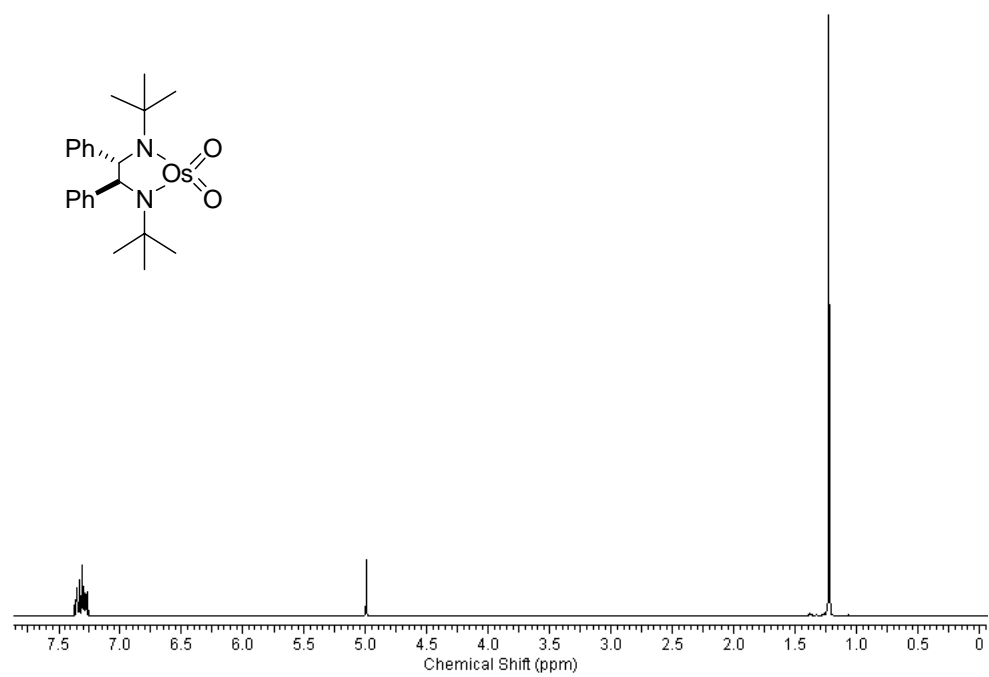
### General Procedure B. Osmium(VI) intermediate formation from 1,2-diamines:

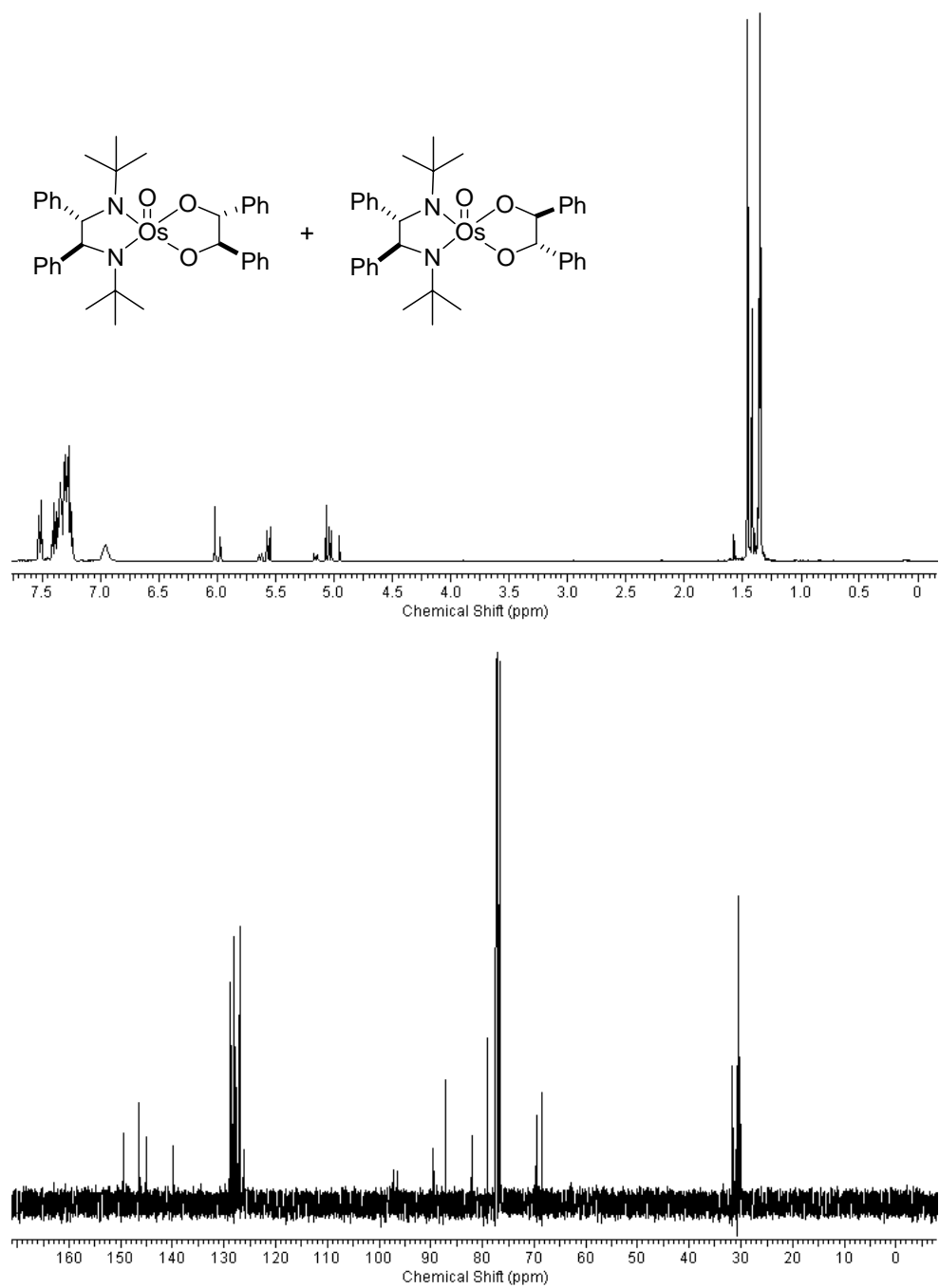
A solution of diamine (1 eq.) and alkene (1.5 eq.) in toluene (50 ml/mmol) was cooled to -78°C and stirred for 15 min. A solution of osmium tetroxide (1.1 eq.) in toluene (10 ml/mmol) was added dropwise, the reaction stirred for 10 min at -78°C and allowed to warm to room temperature. The reaction mixture was evaporated under reduced pressure and the product isolated directly by flash column chromatography ( $\text{SiO}_2$ , petrol  $\rightarrow$  petrol- $\text{Et}_2\text{O}$ , 80:20).

## References

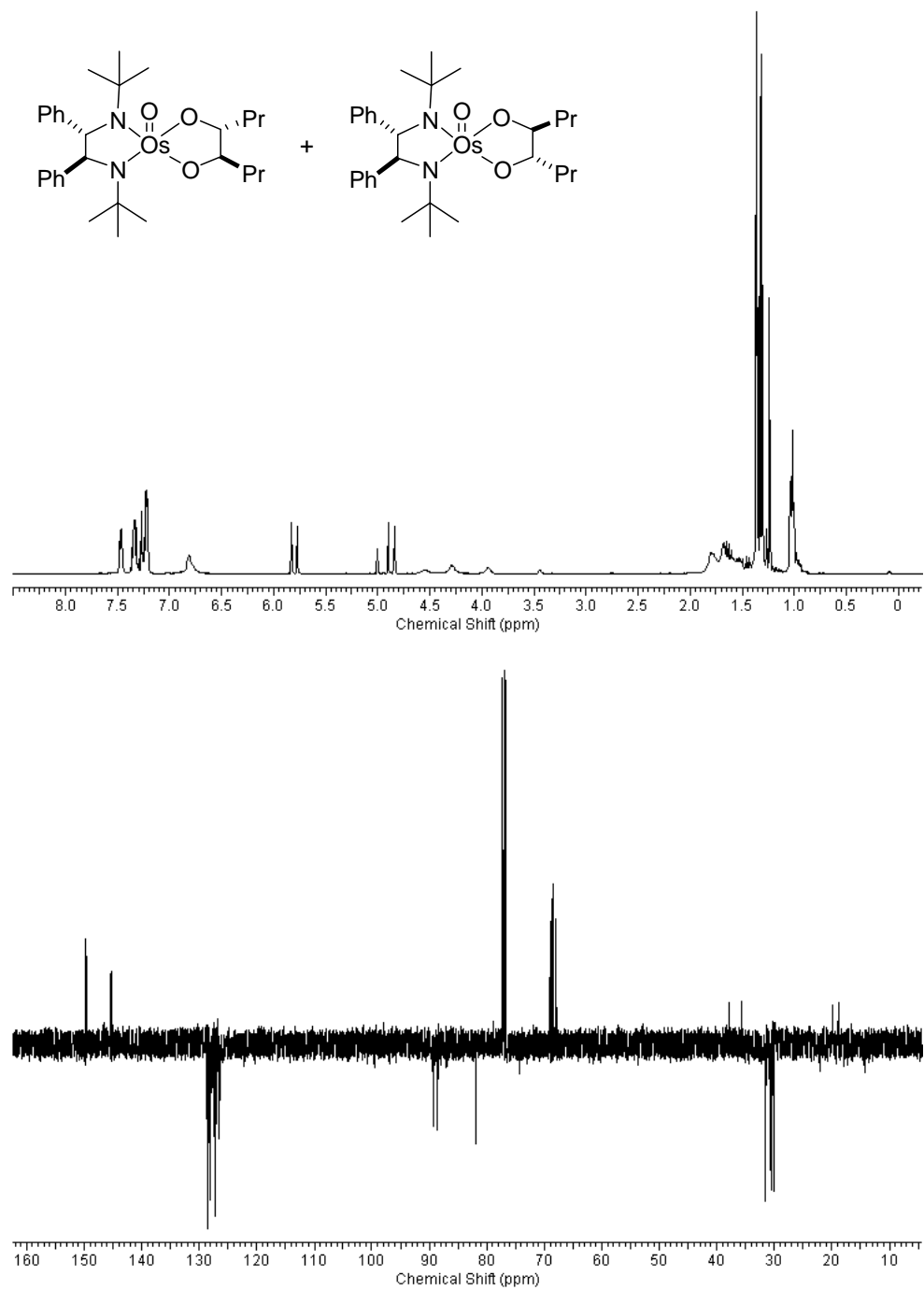
1. Armarego, W. L. F.; Perrin, D. O. *Purification of Laboratory Chemicals*, Butterworth and Heinemann, Oxford, 1996 (4<sup>th</sup> Edition).

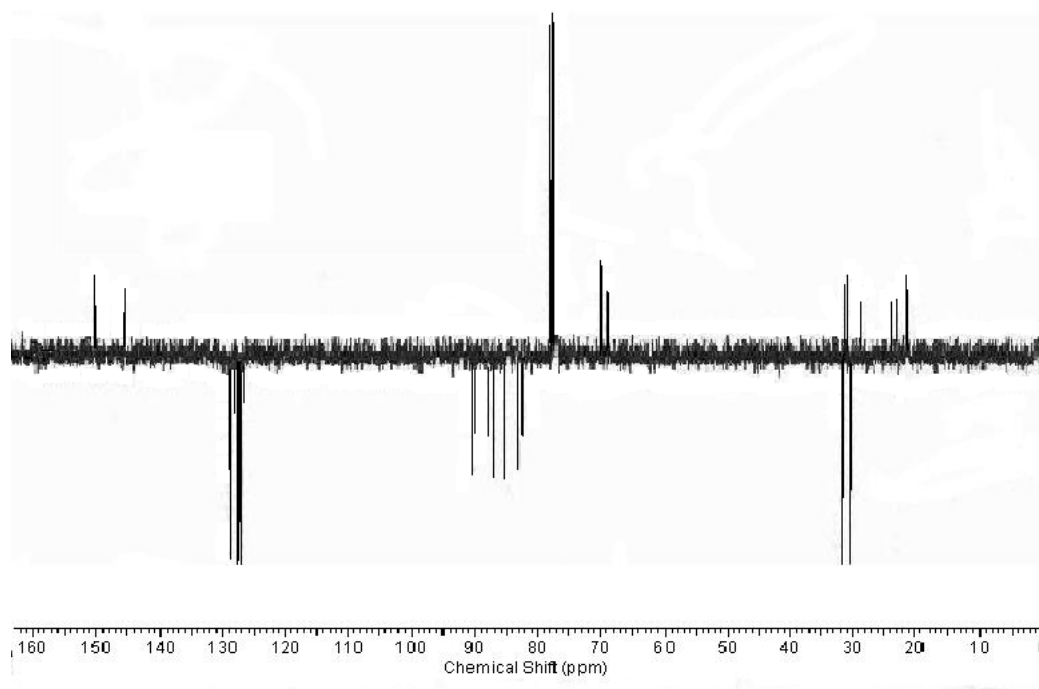
7



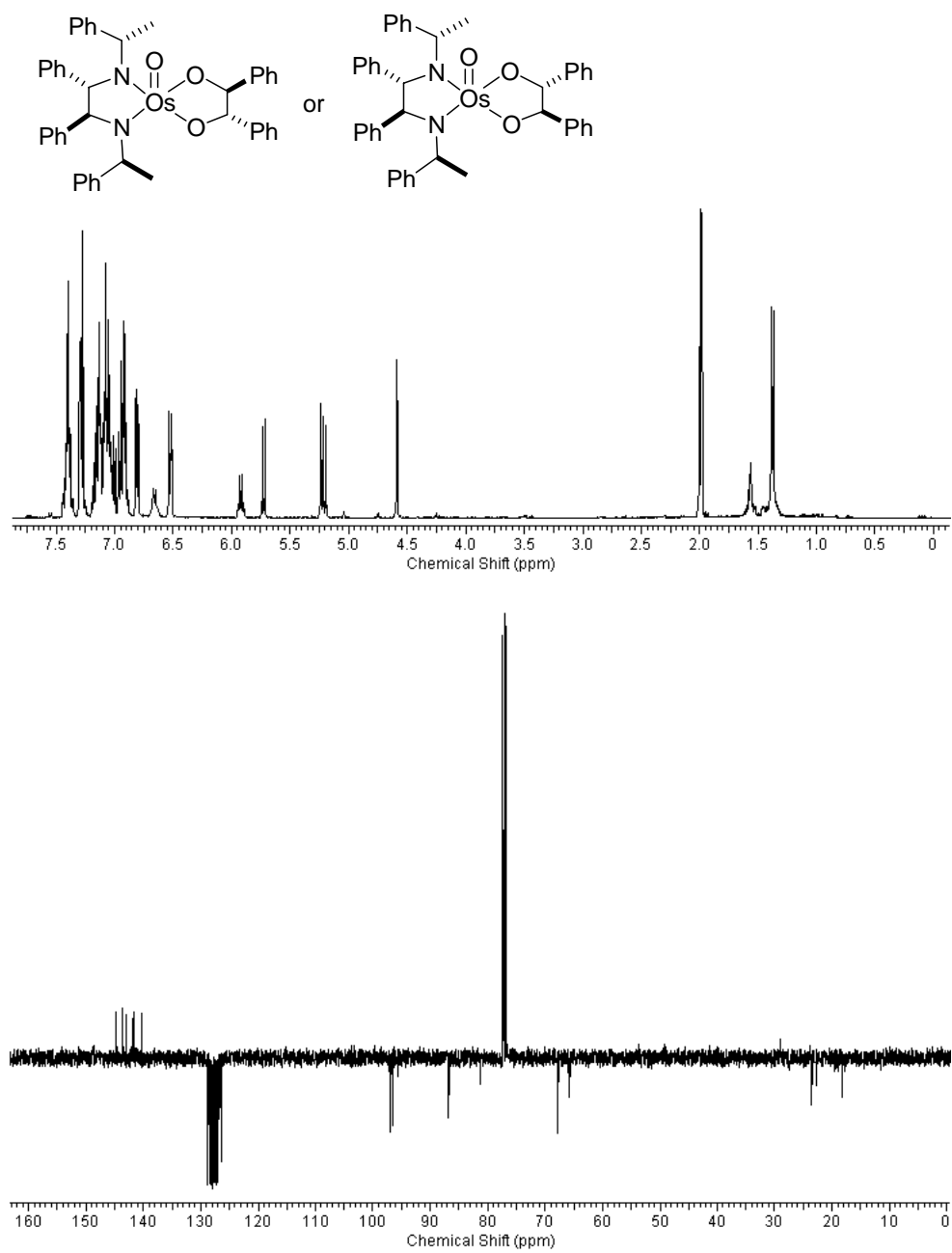


9

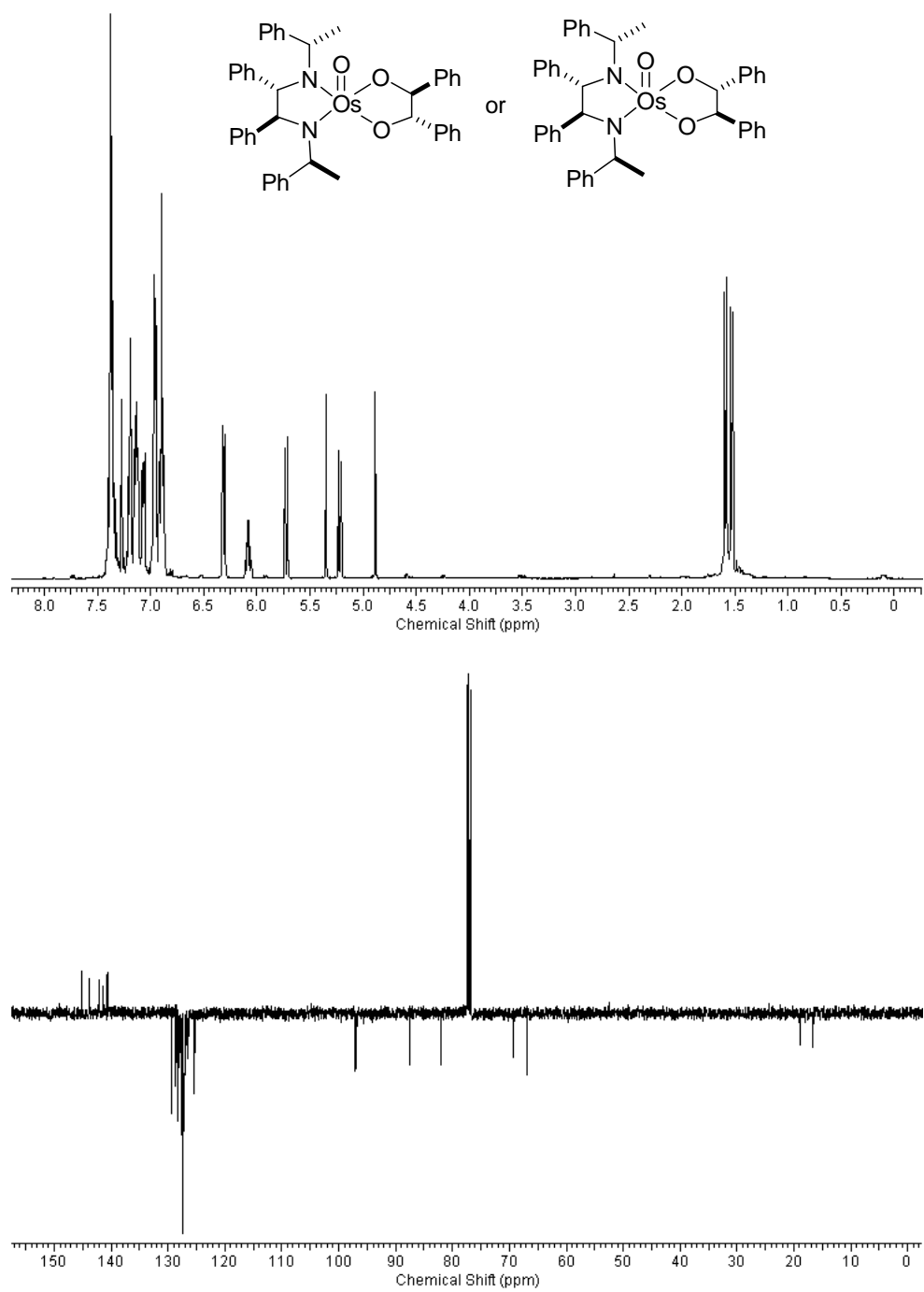




**13**  
Steroisomer  $\alpha$ :

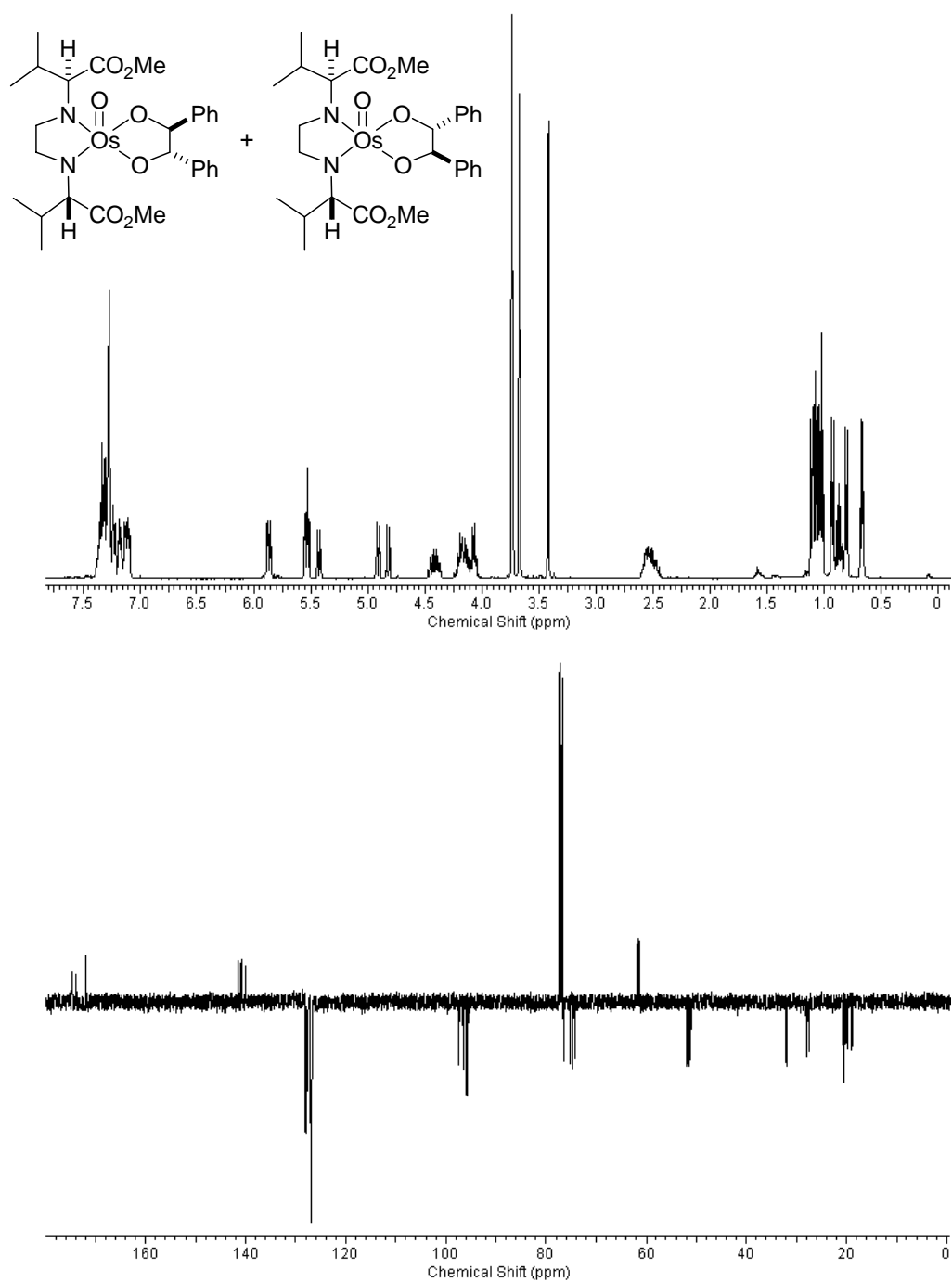


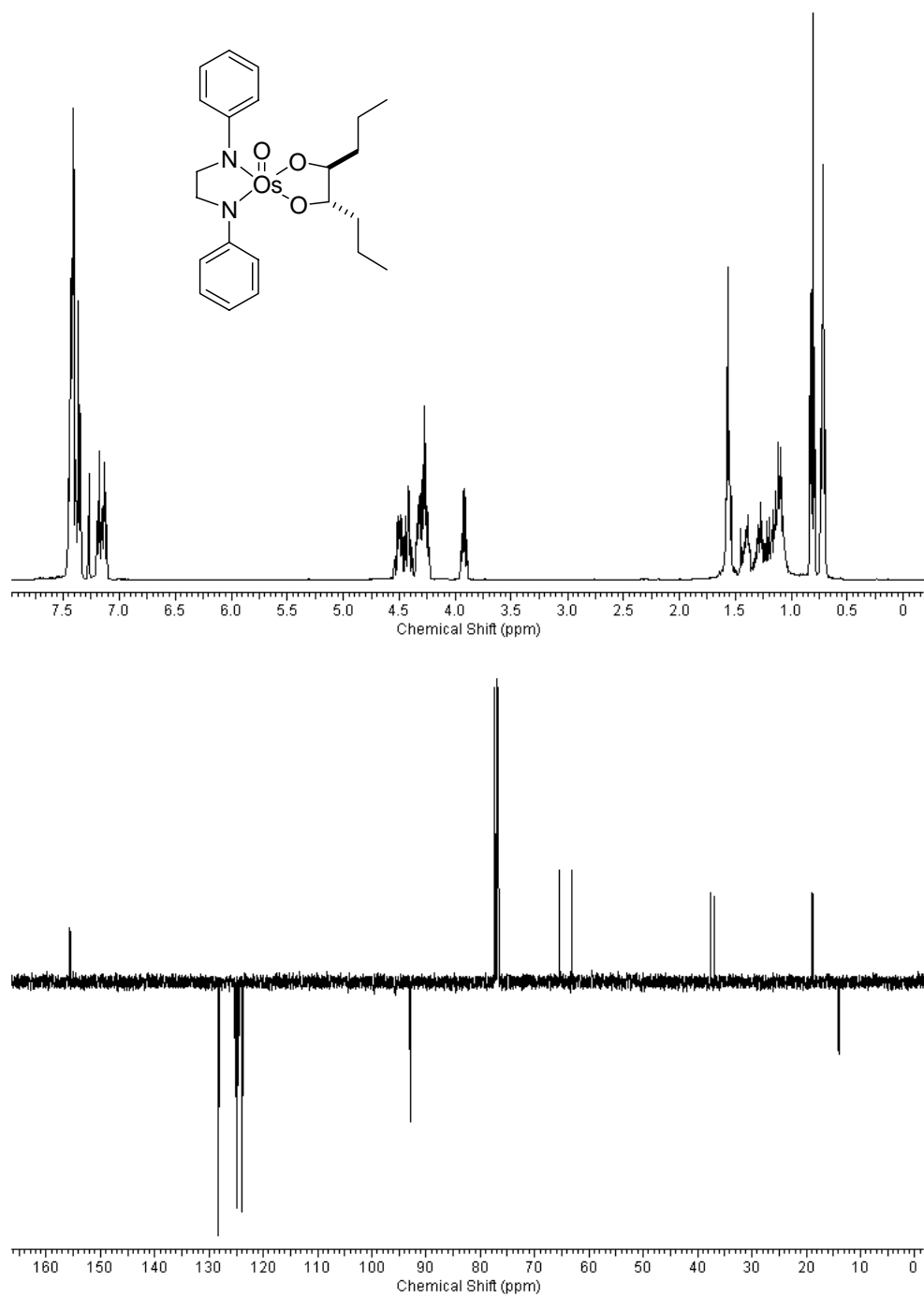
Stereoisomer  $\beta$ :





15





21

