## Electronic Supplementary Information (ESI)

Picolinamide-assisted ortho-C-H functionalization of pyrenylglycine derivatives using aryl iodides

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## Contents

X-ray structure and brief crystal data of compounds $6 \mathbf{r}$ $=$ pages 2-3

Copies of NMR spectra (proton, carbon and fluorine NMR) of compounds = pages 4-388
Copies of absorption and preliminary emission spectral data, and integrated fluorescence intensity vs absorbance plots = pages 389-410

Experimental Procedures $=$ pages 411-412


The unit cell contains two molecules


Ellipsoid probability $=50 \%$

X-ray structure of compound 6 r
CCDC 2294407



Ellipsoid probability = 50 \%


X-ray structure one of the molecule
The unit cell contains two molecules,

## Brief crystal data of compound $\mathbf{6 r}$

CCDC 2294407



## checkCIF/PLATON report

Structure factors have been supplied for datablock(s) exp_603
THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report
Datablock: exp_603

| Bond precision: | $\mathrm{C}-\mathrm{C}=0.0056 \mathrm{~A}$ | Wavelength=0.71073 |
| :---: | :---: | :---: |
| Cell: | $\mathrm{a}=9.6137$ (19) | $\mathrm{b}=18.354$ (3) $\mathrm{c}=18.491$ (3) |
|  | alpha=61.325(17) | beta=89.722(15) gamma=89.850(15) |
| Temperature: | 293 K |  |
|  | Calculated | Reported |
| Volume | 2862.5(10) | 2862.5(10) |
| Space group | P -1 | P -1 |
| Hall group | -P 1 | -P 1 |
| Moiety formula | C34 H28 Br N3 O2 | C34 H28 Br N3 O2 |
| Sum formula | C34 H28 Br N3 O2 | C34 H28 Br N3 O2 |
| Mr | 590.49 | 590.50 |
| Dx,g cm-3 | 1.370 | 1.370 |
| Z | 4 | 4 |
| Mu (mm-1) | 1.472 | 1.472 |
| F000 | 1216.0 | 1216.0 |
| F000' | 1215.28 |  |
| h, k, lmax | 14,27,28 | 14,26,27 |
| Nref | 21108 | 17050 |
| Tmin, Tmax | 0.484,0.555 | 0.579,1.000 |
| Tmin' | 0.474 |  |
| ```Correction method= # Reported T Limits: Tmin=0.579 Tmax=1.000 AbsCorr = MULTI-SCAN``` |  |  |
| Data completene | ss $=0.808$ | Theta $(\max )=32.723$ |

$R($ reflections $)=0.0597(8340) \quad$ wR2 (reflections $)=$

[^0]wR2 (reflections) $=$ 0.2088(17050)







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SpinWorks 4: AD-2267-A



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| PPM | 160 | 120 | 80 | 40 | 0 |

SpinWorks 4: AD-2267-A



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| PPM | 190 | 180 | 170 | 160 | 150 | 140 | 130 | 120 |




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| PPM | 9.6 | 9.4 | 9.2 | 9.0 | 8.8 | 8.6 | 9 | 8.4 |

SpinWorks 4: AD-1821
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 19



4a


## 




PROTON CDCl3 /opt/topspin3.5pl2/nmrdata nmrsu 19



4a


## SpinWorks 4: AD-1821

PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 19






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| PPM | 148 | 144 | 140 | 136 | 132 | 128 | 124 |







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| PPM | 7.6 | 7.2 | 6.8 | 6.4 | 6.0 |  |

SpinWorks 4: AD 1872
C13CPD256 CDCI3/opt/topspin3.5pl2/nmrdata nmrsu 34




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| PPM | 160 | 120 | 80 | 40 | 18 |





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| PPM | 148 | 144 | 140 | 136 | 132 | 128 | 124 | 120 |  |

SpinWorks 4: AD 1875RE
PROTON CDCl3 /opt/topspin3.5pl2/nmrdata nmrsu 4


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SpinWorks 4: AD-1873 RE
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 47



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SpinWorks 4: AD-1873 RE
C13CPD256 CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 47



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| PPM | 132 | 130 | 128 | 126 | 124 | 122 | 120 | 11827 |



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| PPM | 8.2 | 8.0 |



SpinWorks 4: AD 2267 RE
PROTON CDCl3 /opt/topspin3.5pl2/nmrdata nmrsu 51


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$4 e$






| PPM | 160 | 150 | 140 | 33 | 120 | 130 |
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| PPM | 60 | 56 | 52 | 48 | 44 | 40 | 36 | 32 | 28 | 24 | 34 | 20 |



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PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 55

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SpinWorks 4: SUB 62
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 55





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| PPM | 6.6 | 6.4 | 6.2 | 6.0 | 5.8 | 5.6 | 5.4 |  |

SpinWorks 4: SUB 62
PROTON CDCI3/opt/topspin3.5pl2/nmrdata nmrsu 55


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| PPM | 160 | 120 | 80 | 40 | 0 |

C13CPD CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 55



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| PPM | 134 | 132 | 130 | 128 | 126 | 124 | 122 | 41 |

## SpinWorks 4: SUB 62

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| PPM | 54 | 50 | 46 | 42 | 38 | 34 | 30 | 426 |



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SpinWorks 4: AD-2252
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PROTON CDCl3 /opt/topspin3.5pl2/nmrdata nmrsu 2



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| PPM | 160 | 120 |  | 80 | 40 | 0 |







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## SpinWorks 4: AD 1789

PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 35


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SpinWorks 4：AD 1789
PROTON CDCI3／opt／topspin3．5pl2／nmrdata nmrsu 35




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C13CPD CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 35




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| PPM | 160 | 120 | 80 | 40 | 80 |  |

SpinWorks 4: AD 1789
C13CPD CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 35



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| PPM | 148 | 144 | 140 | 136 | 132 | 128 | 124 | 120 | 116 | 112 |

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SpinWorks 4: AD 1851 RE
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 13



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| PPM | 160 | 120 | 80 | 40 | 4 |


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| PPM | 140 | 136 | 132 | 128 | 124 | 6520 |

SpinWorks 4: AD 1813
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 23


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| PPM | 10 | 8 |  | 6 |  | 4 | 2 |  | 0 |

PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 23

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SpinWorks 4: AD 1813
SpinWorks 4: AD 1813
C13CPD CDCl3 /opt/topspin3.5pl2/nmrdata nmrsu 23



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| PPM | 148 | 144 | 140 | 136 | 132 | 128 | 124 | $120^{\circ}$ |








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| PPM | 162 | 158 | 154 | 150 | 146 | 142 | 138 | 134 | 130 | 126 | 122 | 118 |

## ${ }^{19}$ F NMR $\left(\sim 376 \mathrm{MHz}, \mathrm{CDCl}_{3}\right)$



| PPM | 0 | -40 | -80 | 1 | 1 | 1 | -200 |
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| PPM | 7.78 | 7.76 | 7.74 | 7.72 | 81 | 7.70 |


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|  |  |  |  |  |  | 82 |
| PPM | 7.2 | 6.8 | 6.4 | 6.0 | 5.6 |  |

## SpinWorks 4：SUB－45

C13CPD256 CDCl3／opt／topspin3．5pl2／nmrdata nmrsu 18

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| PPM | 160 | 120 | 80 | 40 |  |





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| PPM | 160 | 150 | 140 | 130 |  | 120 |



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C13CPD256 CDCl3 /opt/topspin3.5pl2/nmrdata nmrsu 58

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| PPM | 160 | 120 | 80 | 40 |  |





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| PPM | 148 | 144 | 140 | 136 | 132 | 128 | 124 | 89 | 120 |



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| PPM | 12 |  |  | 8 |  |  | 4 | 0 |  |

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| PPM | 160 | 120 | 80 | 40 |  |




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| PPM | 148 | 144 | 140 | 136 | 132 | 128 | 124 | 6 |




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| PPM | 12 |  | 8 |  |  | 4 | 0 |  |




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| PPM | 148 | 144 | 140 | 136 | 132 | 128 | 1201 |

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| PPM | 160 | 120 | 80 | 40 |  |

## SpinWorks 4：AD 1899

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SpinWorks 4：AD－1814
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C13CPD CDCl3 /opt/topspin3.5pl2/nmrdata nmrsu 47




6j




6j


|  |  |  |  |  |  |  |  | 109 |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 148 | 144 | 140 | 136 | 132 | 128 | 124 | 120 |




## SpinWorks 4: AD-1957

PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 6





VVVVン, VVvivi Moviصgin



6k


SpinWorks 4: AD-1957
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 6



|  |  | $\stackrel{\stackrel{\rightharpoonup}{\circ}}{\stackrel{8}{\omega}}$ |  |  |  |  | $\begin{aligned} & 0 \\ & 0 \\ & \infty \\ & \infty \end{aligned}$ | 112 |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | 1 |  |  | , |  |  |  |  |
| PPM | 6.6 | 6.4 | 6.2 | 6.0 | 5.8 | 5.6 | 5.4 | 5.2 |




C13CPD CDCl3 /opt/topspin3.5pl2/nmrdata nmrsu 18

24.011
28.483
33.941



|  | , | 1 | 1 | 1 | 114 |
| :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 |  |

C13CPD CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 18



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| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 148 | 144 | 140 | 136 | 132 | 128 | 124 | 115 | 120 |





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61


PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 24





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C13CPD256 CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 8


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| PPM | 160 | 120 | 80 | 40 |  |  |




61

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| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 148 | 144 | 140 | 136 | 132 | 128 | 124 | 120 |


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7.1295


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C13CPD256 CDCI3／opt／topspin3．5pl2／nmrdata nmrsu 18


| VV | $\sigma$ |
| :---: | :---: |
| Vvo | $\omega$ |
| wov | $\square$ |
| $0 \infty$ | $\square$ |
| $\infty$ ¢ | $\omega$ |



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| :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 | 127 |


$148.308-$
$149.721-$
141.054 -




6 m


|  | 1 | I | \| | 1 | 1 |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 150 | 140 | 130 | 120 | 128 |



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|  |  | $\begin{aligned} & \circ \\ & \text { ó } \\ & \text { ob } \end{aligned}$ |  | － |  | $\begin{aligned} & \circ \\ & \stackrel{0}{\infty} \\ & \stackrel{\sim}{\omega} \\ & \hline \end{aligned}$ | $\begin{aligned} & \text { 아 } \\ & \stackrel{\circ}{\circ} \mathrm{O} \\ & 08 \\ & \hline \end{aligned}$ |  | $\begin{aligned} & \stackrel{0}{\sim} \\ & \stackrel{\sim}{u} \\ & \hline \end{aligned}$ |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | 1 |  |  |  |  |  |  |  | － 1 |  |
| PPM | 12 |  |  | 8 |  |  |  | 4 | 0 | 129 |




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$\infty \infty \infty \infty \infty$ NNNべゥ



| VVVVVv | $\checkmark$ | $\checkmark$ | $\checkmark$ | $\checkmark$ | VVVNVV | $\checkmark$ | $\checkmark$ | $v$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| $\cdots \cdots$ | in | － | $\stackrel{\square}{+}$ | － | wiwiwis | N | N | N |
| va virum | $\sigma$ | ＋ | $\infty$ | $\stackrel{\rightharpoonup}{\bullet}$ | －0ソソのけ | $\infty$ | $\omega$ | $\stackrel{+}{\bullet}$ |
| $\omega 6+0$ ¢ю | － | N | $\square$ | － | － $0 \times+$－ | $\omega$ | N | N |
| のज ¢んமo | O | $\omega$ | $\square$ | $\cdots$ | $\checkmark \cup+\checkmark \omega \infty$ | $\omega$ | $\omega$ | $\sigma$ |
| $1111$ |  |  |  |  | 成 |  |  |  |



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C13CPD256 CDCl3 /opt/topspin3.5pl2/nmrdata nmrsu 14



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|  | 1 | 1 | 1 | 1 |  |  |
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| PPM | 160 | 120 | 80 | 40 | 133 | 0 |




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### 6.4332



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PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 22


VVVVNVV VVNVV




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PROTON CDCI3／opt／topspin3．5pl2／nmrdata nmrsu 22

| NVVVVVV | $\checkmark$ | いいいV |
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| － | N | $0^{\circ} 0^{\circ}$ |
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| 以○ャ¢ | $\omega$ | ＋6ソN |
|  | の | の¢ルV |
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|  | \| | 1 | 1 | 1 | 39 |
| :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 | 139 |


| $\begin{array}{cc}\stackrel{\rightharpoonup}{+} & \stackrel{\rightharpoonup}{+} \\ \stackrel{\rightharpoonup}{\circ} & \stackrel{\omega}{\omega} \\ \stackrel{\sim}{*} & \stackrel{0}{\sim}\end{array}$ |  |
| :---: | :---: |
|  | ) |





|  | \| | 1 | 1 | \| | 1 | \| | 1 | 1 | 140 |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 148 | 144 | 140 | 136 | 132 | 128 | 124 | 120 | $116^{140}$ |

## $\because$ $\vdots$ $\vdots$ $\square$



6p


SpinWorks 4：SUB－47 RE
PROTON CDCl3／opt／topspin3．5pl2／nmrdata nmrsu 32

| $\infty \times \infty$ |  | $\infty \times \infty \times \infty$ |
| :---: | :---: | :---: |
| ún | NNNN゙ | $\cdots \mapsto \sim \bigcirc \bigcirc \bigcirc \circ \circ^{\circ}$ |
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6p

| $\begin{aligned} & N \\ & \omega \\ & 0 \end{aligned}$ |  | $\begin{aligned} & \omega \\ & \underset{\sim}{v} \end{aligned}$ | $\begin{aligned} & \circ \\ & \stackrel{\circ}{\circ} \\ & \stackrel{\infty}{\sim} \end{aligned}$ |  |  | $\stackrel{\stackrel{\rightharpoonup}{\omega}}{\underset{y}{u}}$ |  | $\stackrel{\text { N }}{\mathrm{Y}}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | ｜ | T | － | 1 | I | 1 | 1 |  |
| PPM | 8.4 | 8.2 | 8.0 | 7.8 | 7.6 |  | 7.4 | 142 |




6p

|  |  |  |  | 1 |  |
| :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 | 143 |




6p


|  |  |  |  |  |  |  |  | 144 |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 148 | 144 | 140 | 136 | 132 | 128 | 124 |  | 120 |

PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 18


$6 q$




SpinWorks 4: SUB-48RE
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 18



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$6 q$



$6 q$

C13CPD CDCl3/opt/topspin3.5pl2/nmrdata nmrsu 7


$6 q$


FAmpox





|  |  | $\stackrel{\stackrel{-}{-}}{\stackrel{-}{6}}$ | Whrr O vioo inio ow on ancu | $\begin{aligned} & \circ \\ & \stackrel{\circ}{6} \\ & 6 \end{aligned}$ | $\stackrel{\stackrel{\rightharpoonup}{*}}{\stackrel{\sim}{\infty}}$ |  | $\begin{aligned} & \infty \\ & 0 \\ & 0 \\ & \hline 0 \end{aligned}$ |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  |  | , |  |  | 1 | 151 |
| PPM | 12 |  | 8 |  |  | 4 | 0 | 151 |



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| $\infty^{\infty} \infty \infty$ | $\omega 0$ | －NNO＋NOQのम WN |
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SpinWorks 4: AD 2136 RE
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 7



6 r



$6 r$

|  | 1 |  |  |  | 1 |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 200 | 160 | 120 | 80 | 40 | 105 |

SpinWorks 4: AD-2136-RE
C13CPD256 CDCI3/opt/topspin3.5pl2/nmrdata nmrsu 40




6 r

PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 40



6s


| $\infty$ | $\infty$ | $\infty \times \infty$ | ¢ $0 \times \infty \times \infty$ | VソVVVい | $\checkmark$ | VVVV | $\checkmark$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | जن | NNNNN |  | $\cdots$ VVV | in | － | N |
| $\infty \times$ | $\omega \stackrel{ }{ }$ | －WNNO | －ャャのあかんN | ¢Vのソかん | N | $\bigcirc \infty$ | $\infty$ |
| ロம | $\infty$ | ○மNO | リーツのル○の心ソ | －VNON6 | の | Noun | $\omega$ |
| $\stackrel{\rightharpoonup}{\square}$ | $\omega$ | $\omega \rightarrow$ ¢ | のwornのov | $\omega ⿴ 囗 十$ | $\sigma$ | Nown | $\bigcirc$ |
| ， 1 |  | $\frac{111}{11 \mid}$ | 1111111 | $\\|_{\\|}$ |  | ｜｜ |  |



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| PPM | 160 | 120 | 80 | 40 | 0 |



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| :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 |  |


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| $\stackrel{\text {－}}{\bullet}$ |  |





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$6 t$






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PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 56


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SpinWorks 4：AD－2114RE
PROTON CDCI3／opt／topspin3．5pl2／nmrdata nmrsu 56




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SpinWorks 4: AD-2114RE
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 56





6




| $\stackrel{\rightharpoonup}{\bullet}$ | $\mapsto$ | $\stackrel{ }{\bullet}$ | $\stackrel{ }{ }$ |
| :---: | :---: | :---: | :---: |
| $\stackrel{\square}{+}$ | $\stackrel{+}{+}$ | $\stackrel{\rightharpoonup}{\square}$ | $\stackrel{+}{\square}$ |
| 6 | $\infty$ | $\stackrel{\square}{\bullet}$ | $\bigcirc$ |
| $\square$ | $\omega$ | 0 | $\pm$ |
| 6 | $\longmapsto$ | $\checkmark$ | $\bigcirc$ |
| $\checkmark$ | $\mapsto$ | 0 | $\pm$ |




6u





$\infty \infty \infty$ जivicio GNNO
OANN


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SpinWorks 4: AD 2071
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 16




C13CPD CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 16


| VVV | Mu |
| :---: | :---: |
| ソの | -1. |
| - ${ }^{\circ}$ | No |
| $\mapsto \infty$ | -N |
| $0 \times 0$ | $\checkmark 0$ |



$6 v$


|  | 1 | 1 | 1 | 1 | 177 |
| :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 | 0 |




6 v





6w


|  |  | $\begin{aligned} & \circ \\ & \stackrel{\circ}{8} \\ & \stackrel{8}{4} \end{aligned}$ | $\begin{aligned} & \stackrel{N}{\mathrm{o}} \\ & \stackrel{\rightharpoonup}{2} \end{aligned}$ |  |  |  | $\begin{aligned} & \circ \\ & \text { óg } \\ & 0 \end{aligned}$ | $\begin{aligned} & \stackrel{\circ}{\circ} \\ & \stackrel{0}{n} \end{aligned}$ |  |  | $\begin{aligned} & \stackrel{0}{\omega} \\ & \stackrel{\sim}{\circ} \end{aligned}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | 1 |  |  |  |  |  |  |  | 1 | I | 179 |
| PPM | 11 |  |  |  |  | 7 |  |  | 5 | 3 | 1 |




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| $\begin{gathered} \stackrel{N}{\sigma} \\ \underset{\sim}{2} \end{gathered}$ |  | $\begin{aligned} & \stackrel{A}{i n} \\ & \underset{\omega}{n} \end{aligned}$ | $\begin{aligned} & \stackrel{\rightharpoonup}{\dot{\infty}} \\ & \dot{\circ} \end{aligned}$ |  | $\begin{aligned} & \text { on } \\ & \underset{\sim}{\sim} \end{aligned}$ |  | $\stackrel{\sim}{\underset{\sim}{\sim}}$ | $\stackrel{\stackrel{\rightharpoonup}{\sim}}{\sim}$ | $\begin{aligned} & \stackrel{\rightharpoonup}{i} \\ & \stackrel{i}{\sim} \end{aligned}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 8.4 | 8.2 |  | 8.0 | 7.8 | 7.6 |  |  | 4180 |

SpinWorks 4: AD-1858



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SpinWorks 4：AD－1858
C13CPD256 CDCI3／opt／topspin3．5pl2／nmrdata nmrsu 12

| $\mapsto$ | $\stackrel{\rightharpoonup}{\bullet}$ | மคமャ |
| :---: | :---: | :---: |
| $\stackrel{+}{6}$ | $\stackrel{+}{\infty}$ | ＋号号号 |
| $\sigma$ | N | Vのu |
| $\omega$ | $\infty$ | $\stackrel{\text { ®ロの }}{ }$ |
| $\mapsto$ | $\square$ | $\omega \omega \infty$ |









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| :--- |


|  | $\begin{aligned} & \circ \\ & 0 \\ & 0 \\ & i \end{aligned}$ |  |  |  | $\stackrel{N}{N}$ |  | $\begin{aligned} & \omega \\ & \underset{\sim}{\sim} \end{aligned}$ | $\begin{aligned} & \text { u } \\ & \text { N } \end{aligned}$ |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  |  |  |  |  |  | 185 |  |
| PPM | 9.4 | 9.2 | 9.0 | 8.8 | 8.6 | 8.4 | 8.2 |  | 8.0 |



$7.5805-$


$\underset{\substack{\sim \\ \sim \\ \infty \\ \sim \\ \hline}}{ }$



SpinWorks 4: AD-1816
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 28



6x




6x


## SpinWorks 4: AD-1816

C13CPD256 CDCI3/opt/topspin3.5pl2/nmrdata nmrsu 3

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| :---: | :---: |
|  | , |




6x


|  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 150 | 146 | 142 | 138 | 134 | 130 | 126 | 122 |




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VVNVNV ஸ． คャNロルんいに


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|  |  | 1 | 1 | 1 | 193 |
| :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 |  |


122.071





PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 10

4.3860

$6 z$


SpinWorks 4：AD 2081
PROTON CDCI3／opt／topspin3．5pl2／nmrdata nmrsu 10




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SpinWorks 4: AD 2081
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 10


$6 z$

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| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  | 5.6 |  |  | $4.4{ }^{198}$ |
| PPM | 6.4 | 6.0 | 5.6 | 5.2 | 4.8 | 4.4 |



$6 z$


|  |  | \| |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 | 199 |

SpinWorks 4: AD 2081
C13CPD256 CDCI3/opt/topspin3.5pl2/nmrdata nmrsu 10


$6 z$


## SpinWorks 4: AD-1991

PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 28



6za


SpinWorks 4: AD-1991
PROTON CDCl3 /opt/topspin3.5pl2/nmrdata nmrsu 28

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$\qquad$ -


PPM

## 9.6

9.2
8.8


## SpinWorks 4: AD-1991

PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 28



6za


SpinWorks 4: AD-1991
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 28



6za




6za


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| :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 | 0 |

C13CPD256 CDCl3 /opt/topspin3.5pl2/nmrdata nmrsu 28





6za




6zb

.



6zb

|  |  |  | $\stackrel{A}{\infty}$ | $\begin{aligned} & \stackrel{\rightharpoonup}{\sim} \\ & \underset{\sim}{\sim} \end{aligned}$ | $\begin{aligned} & \stackrel{\omega}{\stackrel{\rightharpoonup}{\bullet}} \\ & \stackrel{\sim}{2} \end{aligned}$ |  |  | $\begin{aligned} & \stackrel{A}{+} \\ & \stackrel{+}{N} \\ & \hline \end{aligned}$ |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  |  |  |  |  |  |  |  |  |
| PPM | 8.6 | 8.4 | 8.2 |  | 8.0 | 7.8 | 7.6 | 7.4 | 7.2 | 2087.0 |




6zb



[^1]





6zc


ROTON CDCI3／opt／topspin3．5pl2／nmrdata nmrsu 53



6zc




6zc





6zc

|  |  |  |  | 1 |  | 1 |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 | 14 | 0 |

## SpinWorks 4: AD-2178

C13CPD256 CDCl3 /opt/topspin3.5pl2/nmrdata nmrsu 29







PROTON CDCl3／opt／topspin3．5pl2／nmrdata nmrsu 5

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| :---: | :---: |
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| Vの | $\infty$ の中 |
| WN | の $-\omega$ |
| － | の ルー |

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SpinWorks 4: AD-1976-Re
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 51






| $\begin{aligned} & \text { ㅇ. } \\ & \stackrel{\circ}{\bullet} \end{aligned}$ |  |  |  |  | $\begin{aligned} & \stackrel{\circ}{\circ} \\ & \stackrel{\circ}{4} \end{aligned}$ |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 |
| PPM 3.90 | 3.80 | 3.70 | 3.60 | 3.50 | 3.40 | 3.30 | 3.20 | 3.10 |

PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 51
1.2266
0.9233
0.9413
0.9594




6zd




6zd


SpinWorks 4: AD 1976 RE C13CPD256 CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 60





|  | I | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 221 |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 150 | 146 | 142 | 138 | 134 | 130 | 126 | 122 | 118 |




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SpinWorks 4: AD-1927A
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 13

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| WN | $\checkmark$ N |
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|  | $\stackrel{\stackrel{\rightharpoonup}{+}}{+}$ | $\begin{aligned} & \circ \\ & 0 \\ & 0 \\ & 0 \end{aligned}$ |  |  | $\begin{aligned} & \stackrel{\circ}{\circ} \\ & \stackrel{\circ}{\circ} \end{aligned}$ |  |  | $\begin{aligned} & \omega \\ & \underset{\sim}{e} \end{aligned}$ |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | 1 | 1 | , | 1 |  | - | , |  | 1 | 1 |
| PPM | 6.4 | 6.0 | 5.6 | 5.2 | 4.8 |  | 4.4 | 4.0 | 224 | 3.6 |

C13CPD256 CDCI3／opt／topspin3．5pl2／nmrdata nmrsu 13


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| VVの | WNN |
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SpinWorks 4: AD 1942 A
PROTON CDCl3 /opt/topspin3.5pl2/nmrdata nmrsu 54
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| $\stackrel{\rightharpoonup}{\mathbf{\omega}}$ |
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VVVVVV Vivivi

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VVNVNN w w w w wien wwwn whe No 1 NHO
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| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 152 | 148 | 144 | 140 | 136 | 132 | 128 | 124 | 2320 |


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7.8620


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SpinWorks 4: AD-2258



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7.1524
$-9210^{\circ} L$
$-8166^{\circ} 9$

SpinWorks 4: AD-2258


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| VVソ | טリ | ww | NNN |
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| VVの | $\cdots \infty$ | NN | － |
| wov | $\omega \infty$ | бֹテ | いの－ |
| かのヘ | $\bigcirc 0$ | ツャ | $\stackrel{\square}{\circ}$ |
| $\infty$－ | OG + | $\mapsto \sim$ | $\infty$ |



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## SpinWorks 4: AD-2258

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SpinWorks 4: AD-2258


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609
$6 \varepsilon \varepsilon^{\circ}$


6zg


|  | 1 | 1 |  |  | 1 | 1 | 1 | 242 |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 52 | 48 | 44 | 40 | 36 | 32 | 28 |  |




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SpinWorks 4: AD-2265 Re
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 55




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SpinWorks 4: AD-2265 Re
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 55



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| $\begin{aligned} & \stackrel{\rightharpoonup}{\sim} \\ & \stackrel{N}{\circ} \end{aligned}$ |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 1 |  |  |  |  |  |  | $247^{1}$ |
| PPM 5.72 | 5.68 | 5.64 | 5.60 | 5.56 | 5.52 | 5.48 |  |



6zh

| $\underset{\sim}{\stackrel{\rightharpoonup}{\omega}}$ |  |  |  |  |  |  | $\begin{aligned} & \omega \\ & \stackrel{\rightharpoonup}{N} \\ & \sim \end{aligned}$ |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  | 1 |  |  |  |  | 248 |  |
| PPM | 3.8 | 3.6 | 3.4 | 3.2 | 3.0 | 2.8 | 2.6 |  | 2.4 |

PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 55


6zh

|  |  | $\begin{aligned} & \omega \\ & \stackrel{\omega}{\omega} \end{aligned}$ |  | $\stackrel{\stackrel{\rightharpoonup}{i}}{\sim}$ |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | 1 |  | 1 | 1 |  | 249 | 1 |
| PPM | 1.90 | 1.80 | 1.70 | 1.60 | 1.50 |  | 1.40 |




6zh


C13CPD256 CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 55


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| :--- | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 150 | 1 | 1 | 130 | 120 |




6zh


|  | T | 1 | + | + | + | 1 | 1 | 1 | 1 | 553 | T |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 56 | 52 | 48 | 44 | 40 | 36 | 32 | 28 | 24 | 20 |  |




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SpinWorks 4: AD-2262 Re
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 54

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SpinWorks 4: AD-2262 Re
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 54




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1.2807



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|  |  |  |

$0.8103-$
$0.8191-$

$0.8404-$
$0.8461-$
$0.8470-$

$0.8704-$
0.8708
0.8746

0.8972
0.9059


6zi




6zi


|  |  |  |  |  |  |
| :--- | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 1 | 1 | 262 |



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| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 150 | 140 | 130 | 3 | 120 |




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|  | 1 | 1 |  | 1 |  | 1 | 1 |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 52 | 48 | 44 | 40 | 36 | 32 | 28 | 26424 |


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vuv -ig Nou ソ®


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SpinWorks 4: AD-2259

5.3737
3.8092


6zj


## SpinWorks 4: AD-2259

$\begin{array}{ll}\stackrel{\circ}{4} & \stackrel{-}{\pi} \\ \stackrel{\infty}{\infty} & \stackrel{\circ}{\circ} \\ \stackrel{\infty}{\infty} & \end{array}$
$1.3144-$
$1.3517-$



6zj


WWNN
NOON
Non NOON MNOV 11


6zj

|  | 1 | 1 | 1 | 1 | 264 |
| :--- | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 | 0 |

SpinWorks 4: AD-2259

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| :---: | :---: |
| 웅 | \% |
| $\stackrel{\sim}{\omega}$ | $\stackrel{ }{+}$ |

$148.2460-$
$149.6998-$


6zj





SpinWorks 4: AD-2259


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|  | 1 | 1 | I |  | 1 | 1 | 1 | 1 | 271 | \| |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 56 | 52 | 48 | 44 | 40 | 36 | 32 | 28 |  | 24 |






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|  | $\begin{aligned} & \circ \\ & 0 \\ & 0 \\ & \hline \end{aligned}$ |  |  |  |  | $\stackrel{\stackrel{\rightharpoonup}{-}}{\substack{\text { - }}}$ | $\begin{aligned} & \stackrel{\rightharpoonup}{\sim} \\ & \stackrel{\sim}{\circ} \end{aligned}$ |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | 1 | I |  | , | 1 | 1 | 273 |  |
| PPM | 9.6 | 9.4 | 9.2 | 9.0 | 8.8 | 8.6 | 273 | 8.4 |



$8.0158-$
$8.0268-$
8.0348
8.0490
8.0621


6zk


7.2830


6zk

| $\stackrel{\stackrel{\rightharpoonup}{i}}{\underset{\sim}{\sim}}$ |  |  | $\underset{\underset{\sim}{\mathrm{N}}}{\mathrm{H}}$ |  | $\begin{aligned} & \stackrel{+}{\sim} \\ & \stackrel{\sim}{\sim} \end{aligned}$ |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | 1 | 1 |  |  |  |  | 275 |  |
| PPM | 7.80 | 7.70 | 7.60 | 7.50 | 7.40 | 7.30 | 275 | 7.20 |




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|  |  |  |  |  |  |  |  |  | 77 |  |
| PPM | 1.8 | 1.6 | 1.4 | 1.2 | 1.0 | 0.8 | 0.6 | 0.4 | 277 | 0.2 |



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| PPM | 160 | 120 | 80 | 40 | 0 |


148.298
149.524




6zk

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| :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 150 | 140 | 130 | 120 |



6zk




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6zl




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SpinWorks 4: AD-2275
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 50



6zI





6zl


PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 50



6zI


SpinWorks 4: AD-2275
C13CPD256 CDCl3 /opt/topspin3.5pl2/nmrdata nmrsu 50



6zl

14.906
28.214
29.001
30.958
31.209 $\downarrow$

|  | 1 | 1 | 1 | 1 |  |
| :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 | $0^{287}$ |

SpinWorks 4: AD-2275
C13CPD256 CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 50



6zI

|  |  |  |  |  |  |  | 288 |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 170 | 160 | 150 | 140 | 130 | 120 |  | 110 |



14.906


6zl


|  | 1 | 1 | 1 | 1 | 289 |  |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| PPM | 50 | 40 | 30 | 20 | 10 |  |



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$\stackrel{+}{N}$





$\begin{array}{ll}v & \\ 0 & \sigma \\ \infty & \sigma \\ + & \omega \\ \square & \infty\end{array}$
$7.4365 —$
7.2834







|  | 1 | \| |  | 1 | 92 |
| :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 | 2 |

123.730 -

121.856 -



|  | \| |  | \| |  |  | 93 |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 140 | 136 | 132 | 128 | 124 | 29 |

SpinWorks 4: AD-1844 PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 47
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SpinWorks 4: AD-1844
PROTON CDCl3 /opt/topspin3.5pl2/nmrdata nmrsu 47





C13CPD256 CDCl3 /opt/topspin3.5pl2/nmrdata nmrsu 47



7b


|  |  | 1 |  |  | 296 |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 |  | 0 |

## SpinWorks 4：AD－1844

C13CPD256 CDCl3／opt／topspin3．5pl2／nmrdata nmrsu 47






|  |  |  |  |  |  |  | 1 |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 170 | 160 | 150 | 140 | 130 | 120 | 97 |

SpinWorks 4: AD-1981
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 7




| VVNV | $\checkmark$ | $\checkmark$ |
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| जن் | $\omega{ }^{\omega}$ | N |
| ソののの | $\checkmark$ | $\infty$ |
| ¢ローN゙号 | ज® | $\stackrel{\omega}{\sim}$ |
| $111$ |  |  |












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| PPM | 140 | 136 | 132 | 128 | 324 |









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| － |  |




|  | 1 | 1 | 1 | 1 | 304 |
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| PPM | 160 | 120 | 80 | 40 | 004 |






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|  |  |  | $\stackrel{+}{\omega}$ $\stackrel{\rightharpoonup}{8}$ | $\begin{aligned} & \stackrel{\circ}{\circ} \\ & \stackrel{+}{\infty} \end{aligned}$ |  | $\begin{aligned} & 0 \\ & \infty \\ & 0 \\ & 0 \end{aligned}$ |  | $\begin{aligned} & \omega \stackrel{\rightharpoonup}{\circ} \\ & \stackrel{\circ}{8} \stackrel{\rightharpoonup}{\sim} \end{aligned}$ |  | $\begin{aligned} & \infty \\ & \underset{\sim}{\circ} \\ & \stackrel{\sim}{n} \end{aligned}$ |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  |  |  |  |  |  |  |  |  | \| |
| PPM | 10 | 8 |  |  | 6 |  | 4 |  | 2 |  | 0 |


| $\infty \times$ | $\infty \times \infty \times \infty$ |
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| $\omega$ | NNமமமம○○○○○○ |
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|  |  | $\begin{aligned} & \omega \\ & \stackrel{\omega}{\bullet} \\ & \stackrel{\infty}{2} \end{aligned}$ | $\begin{aligned} & N \\ & \stackrel{O}{0} \end{aligned}$ | $\begin{aligned} & N \\ & \stackrel{\circ}{\infty} \end{aligned}$ |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | 1 | 1 |  | 1 | 1 | 1 | ， | 1 | 307 |
| PPM | 8.4 | 8.2 |  | 8.0 | 7.8 | 7.6 | 7.4 | 7.2 | 7.0 |



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|  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 | 308 | 0 |



$130.4258=$
$130.4841-$
$131.1106-$




|  |  | $\begin{aligned} & \text { How } \\ & \text { Boun } \\ & 060 \end{aligned}$ |  |  | $\stackrel{\div}{8}$ | $\begin{aligned} & \omega \\ & \stackrel{\sim}{\infty} \\ & \underset{\sim}{n} \end{aligned}$ | $$ | $\begin{aligned} & \bullet \\ & \stackrel{\rightharpoonup}{\circ} \\ & \stackrel{\rightharpoonup}{0} \end{aligned}$ |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  |  |  |  |  | 1 |  | 310 | , |
| PPM | 10 | 8 |  | 6 |  | 4 | 2 |  | 310 | 0 |




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7.2834

VNVN
0000 $00^{\circ} 0^{\circ}$ NANN $\infty \times \infty$
$\infty \infty$ ○ー
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SpinWorks 4：SUB－210
C13CPD256 CDCI3／opt／topspin3．5pl2／nmrdata nmrsu


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| :---: | :---: |
| VVor | のベー |
| wov | $\bigcirc 0$ |
| VGr | $\begin{aligned} & \text { बN } \\ & \cup N \end{aligned}$ |
| 1 | $11$ |








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| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 144 | 140 | 136 | 132 | 128 | 124 | 120 | 116 | 11313 |



2.0505
2.4571
$\stackrel{\rightharpoonup}{\omega}$
$\stackrel{\rightharpoonup}{\omega}$
$\stackrel{1}{\omega}$


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10
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|  |  | $\begin{aligned} & \stackrel{\rightharpoonup}{\circ} \\ & \stackrel{-}{\bullet} \\ & \hline \end{aligned}$ | $\begin{aligned} & \text { N } \\ & \text { N } \\ & \text { O} \end{aligned}$ | $\begin{aligned} & \stackrel{\rightharpoonup}{\bullet} \\ & \stackrel{\circ}{\infty} \\ & \hline \end{aligned}$ | $\begin{aligned} & N \\ & \sim \\ & \hline \end{aligned}$ | $\begin{aligned} & N \\ & \stackrel{\rightharpoonup}{0} \\ & N \end{aligned}$ |  | $\begin{aligned} & \underset{\sim}{i} \\ & N \end{aligned}$ | $\begin{aligned} & \circ \\ & 0 \\ & 0 \\ & 0 \\ & \hline \end{aligned}$ |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 8.4 |  |  |  |  | 8.0 | 7.6 | 7.2 |  | 6.815 |








|  | $\mid$ | 1 |  |  |  |  |  |
| :--- | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 140 | 136 | 132 | 128 | 124 |  |  |






1 | | | | | | | 1






|  | 1 | 1 | 1 | 320 |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | 200 | 160 | 120 | 80 | 40 | 0 |





|  |  |  |  |  | 1 |  | 1 | 1 | 321 |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 150 | 146 | 142 | 138 | 134 | 130 | 126 | 122 |  | 118 |



| $\infty$ | $\cdots \infty$ | VVvV | VV |
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| NO | NOCOOVANமOO | －${ }^{\text {a }}$ | Nャト○ |
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| ， |  | $111$ | $111$ |


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| $\stackrel{\infty}{\sim}$ | ज̆ |



SpinWorks 4: AD 1955
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 10




| $\begin{aligned} & \stackrel{\omega}{\omega} \\ & \stackrel{\omega}{\omega} \end{aligned}$ |  |  | $\underset{\sim}{N}$ |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | 1 | 1 | 1 | 1 | 1 | T |
| PPM | 4.00 | 3.90 | 3.80 | 3.70 | 3.60 | 3.50 |






|  |  |  |  |  | 327 |
| :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 | 27 |






|  |  | $\begin{aligned} & \text { ave } \\ & \text { Bion } \\ & \text { Bol } \end{aligned}$ | $$ | $\begin{aligned} & \stackrel{\bullet}{\dot{\sim}} \\ & \stackrel{\sim}{6} \end{aligned}$ | $\begin{array}{ll} \circ & \circ \\ \stackrel{\circ}{0} & \stackrel{i}{\circ} \\ \hline 0 \end{array}$ | $$ |  |  | 329 |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | 1 | 1 |  |  |  |  |  | 1 | , |
| PPM | 12 | 8 |  |  |  | 4 |  | 0 |  |





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| $\sim$$\infty$$\omega$$\sim$ |  |
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$-1090^{\circ} \mathrm{S}$




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| PPM | 160 | 120 | 80 | 40 |  |

SpinWorks 4: AD-2121
C13CPD256 CDCl3/opt/topspin3.5pl2/nmrdata nmrsu 33

| $\begin{aligned} & \stackrel{\rightharpoonup}{+} \\ & \stackrel{+}{\circ} \\ & \stackrel{\rightharpoonup}{v} \end{aligned}$ |
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| PPM | 140 | 136 | 132 | 128 | 124 | 120 | 116 | 112 |  |

SpinWorks 4: AD-2121
C13CPD256 CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 33

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| PPM | 56 | 52 | 48 | 44 | 40 | 36 | 32 | 28 | 3324 |



SpinWorks 4: AD 1982
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 19


SpinWorks 4: AD 1982







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| PPM | 160 | 120 | 80 | 40 |  |






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| PPM | 160 | 120 | 80 | 40 | 0 |


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| PPM | 170 | 160 | 150 | 140 | 130 | 120 | 346 | 110 |



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| PPM | 80 | 70 | 60 | 50 | 40 | 30 |  |







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SpinWorks 4: SUB 216 RE
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 52





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| PPM | 200 | 160 | 120 | 80 | 40 | 0 |



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| PPM | 70 | 60 | 50 | 40 | 30 |  |



the compound exists as diastereomers





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|  | I | , | I | 1 | 356 |
| PPM | 8.30 | 8.20 | 8.10 | 8.00 |  |

SpinWorks 4: AD-2074REP
PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 39


the compound exists as diastereomers
$8 f$


he compound exists as diastereomers
$8 f$



he compound exists as diastereomers
$8 f$



1.2248
1.2703
the compound exists as diastereomers
$8 f$


## SpinWorks 4: AD-2074REP

C13CPD256 CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 39


the compound exists as diastereomers


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the compound exists as diastereomers


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| PPM | 170 | 166 | 162 | 158 | 62 | 154 |


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the compound exists as diastereomers


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| PPM | 132 | 128 | 124 | 120 | 116 | 112 |


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the compound exists as diastereomers


the compound exists as diastereomers


|  |  |  |  |  |  |  | 65 |
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| PPM | 26 | 24 | 22 | 20 | 18 | 16 |  |



the compound exists as diastereomers

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| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  |  |  |  |  |  |  | -6- |
| PPM | 12 | 8 |  |  |  | 4 |  |  |  |



the compound exists as diastereomers

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| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  |  | 8.10 | 8.00 | 367 | 7.90 |
| PPM | 8.40 | 8.30 | 8.20 | 8.10 | 8.00 | 367 | 7.90 |

## SpinWorks 4: AD 2083

PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 42

7.5185

the compound exists as diastereomers


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the compound exists as diastereomers


3.9301
3.7983

the compound exists as diastereomers


the compound exists as diastereomers

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the compound exists as diastereomers


## SpinWorks 4: AD 2083

C13CPD256 CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 42


the compound exists as diastereomers


|  | 1 | 1 | \| | 1 | 1 | 1 |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 170 | 168 | 166 | 164 | 162 | 160 | 37158 |



[^2]
the compound exists as diastereomers




the compound exists as diastereomers


|  | 1 | 1 | 1 | 375 | 1 |
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| PPM | 50 | 40 | 30 | 20 | 10 |





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PROTON CDCI3 /opt/topspin3.5pl2/nmrdata nmrsu 30



|  | $\begin{aligned} & \stackrel{N}{\sim} \\ & \underset{\sim}{\sim} \end{aligned}$ | $\begin{aligned} & \omega \\ & 0 \\ & \hline 0 \\ & \hline \end{aligned}$ |  |  | $\begin{aligned} & \text { e } \\ & i \\ & + \end{aligned}$ |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | , | 20 | 16 | 12 | 379 |
| PPM |  | 2.4 | 2.0 | 1.6 | 1.2 | 379 |




$\stackrel{\sim}{\sim}$ .593

|  |  |  |  | 1 |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 160 | 120 | 80 | 40 | 0 | 0 |






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| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| PPM | 148 | 144 | 140 | 136 | 132 | 128 | 124 | 120 | 116 |  | 112 |




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|  |  | 1 |  |  |  |  |  |  |  |
| PPM | 12 | 8 |  |  |  | 4 |  | 0 | 382 |








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| PPM | 4.0 | 3.8 | 3.6 | 3.4 | 3.2 | 3.0 | 2.8 | 2.6 | 2.4 |



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| PPM | 170 | 160 | 150 | 140 | 130 | 387 |  |





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| PPM | 54 | 50 | 46 | 42 | 38 | 34 | 30 | 238 |

The absorption spectra and $\lambda_{\text {max }}$ of compounds $4 \mathbf{a}-\mathrm{d}$ (concentration $=0.02 \mathrm{~g} / 100 \mathrm{~mL}$ in $\mathrm{CHCl}_{3}$ )
$\lambda_{\text {max (absorption) }}$ (the absorption band appearing at the longest wavelength) of compounds



4a: $\lambda_{\text {max }}=347 \mathrm{~nm}$

$4 \mathrm{c}: \lambda_{\text {max }}=347 \mathrm{~nm}$


$$
4 b: \lambda_{\max }=347 \mathrm{~nm}
$$



The absorption spectra and $\lambda_{\text {max }}$ of compounds 6a-j
(concentration $=0.02 \mathrm{~g} / 100$ mL in $\mathrm{CHCl}_{3}$ )
$\lambda_{\text {max (absorption) }}$ (the absorption band appearing at the longest wavelength) of compounds


$6 \mathrm{~g}: \lambda_{\text {max }}=351 \mathrm{~nm}$


6a: $\lambda_{\text {max }}=351 \mathrm{~nm}$

$6 \mathrm{~d}: \lambda_{\max }=350 \mathrm{~nm}$

$6 \mathrm{~h}: \lambda_{\text {max }}=351 \mathrm{~nm}$

$6 \mathrm{~b}: \lambda_{\text {max }}=350 \mathrm{~nm}$

$6 \mathrm{e}: \lambda_{\text {max }}=351 \mathrm{~nm}$

$6 i: \lambda_{\text {max }}=350 \mathrm{~nm}$



6c: $\lambda_{\text {max }}=351 \mathrm{~nm}$

$6 \mathrm{f}: \lambda_{\text {max }}=351 \mathrm{~nm}$


$6 \mathrm{j}: \lambda_{\text {max }}=350 \mathrm{~nm}$

The absorption spectra and $\lambda_{\text {max }}$ of compounds 6 k -t
(concentration $=0.02 \mathrm{~g} / 100$ mL in $\mathrm{CHCl}_{3}$ )
$\lambda_{\text {max (absorption) }}$ (the absorption band appearing at the longest wavelength) of compounds

$6 \mathrm{k}: \lambda_{\text {max }}=351 \mathrm{~nm}$

$61: \lambda_{\text {max }}=351 \mathrm{~nm}$

$6 \mathrm{~m}: \lambda_{\text {max }}=351 \mathrm{~nm}$

$6 \mathrm{n}: \lambda_{\text {max }}=351 \mathrm{~nm}$

$60: \lambda_{\text {max }}=351 \mathrm{~nm}$

$6 \mathrm{p}: \lambda_{\text {max }}=351 \mathrm{~nm}$

$6 q: \lambda_{\text {max }}=353 \mathrm{~nm}$

$6 \mathrm{r}: \lambda_{\text {max }}=351 \mathrm{~nm}$


6s: $\lambda_{\text {max }}=351 \mathrm{~nm}$

$6 t: \lambda_{\text {max }}=350 \mathrm{~nm}$

The absorption spectra and
$\lambda_{\text {max }}$ of compounds $6 \mathrm{u}-\mathrm{z}, 6 \mathrm{za}, 6 \mathrm{zb}, 6 \mathrm{zc}$
(concentration $=0.02 \mathrm{~g} / 100$ mL in $\mathrm{CHCl}_{3}$ )
$\lambda_{\text {max (absorption) }}$ (the absorption band appearing at the longest wavelength) of compounds


$6 \mathrm{u}: \lambda_{\text {max }}=351 \mathrm{~nm}$

$6 \mathrm{x}: \lambda_{\text {max }}=351 \mathrm{~nm}$


6za: $\lambda_{\text {max }}=351 \mathrm{~nm}$


6zb $\quad \lambda_{\text {max }}=351 \mathrm{~nm}$


6zc : $\lambda_{\text {max }}=350 \mathrm{~nm}$

The absorption spectra and $\lambda_{\text {max }}$ of compounds $6 \mathbf{z e}, 6 \mathrm{zf}, \mathbf{6 z g}$

## (concentration $=0.02 \mathrm{~g} / 100$

 mL in $\mathrm{CHCl}_{3}$ )$\lambda_{\text {max (absorption) }}$ (the absorption band appearing at the longest wavelength) of compounds



6ze : $\lambda_{\text {max }}=351 \mathrm{~nm}$



6zd : $\lambda_{\text {max }}=349 \mathrm{~nm}$

The absorption spectra and $\lambda_{\text {max }}$ of compounds 7a-h
(concentration $=0.02 \mathrm{~g} / 100$ mL in $\mathrm{CHCl}_{3}$ )
$\lambda_{\max \text { (absorption) }}$ (the absorption band appearing at the longest wavelength) of compounds


$7 \mathrm{a}: \lambda_{\text {max }}=353 \mathrm{~nm}$

$7 \mathrm{~d}: \lambda_{\text {max }}=353 \mathrm{~nm}$

$7 \mathrm{~b}: \lambda_{\text {max }}=350 \mathrm{~nm}$

$7 \mathrm{e}: \lambda_{\text {max }}=350 \mathrm{~nm}$

$7 \mathrm{c}: \lambda_{\text {max }}=349 \mathrm{~nm}$





7h
$\lambda_{\text {max }}=349$ nm 394

The absorption spectra and $\lambda_{\text {max }}$ of compounds 8a-h, 8f2-(D)
(concentration $=0.02 \mathrm{~g} / 100$ mL in $\mathrm{CHCl}_{3}$ )
$\lambda_{\text {max (absorption) }}$ (the absorption
band appearing at the longest



8f: $\lambda_{\text {max }}=350 \mathrm{~nm}$


unds






8a: $\lambda_{\text {max }}=350 \mathrm{~nm}$

$8 \mathrm{~b}: \lambda_{\max }=350 \mathrm{~nm}$


8d : $\lambda_{\text {max }}=350 \mathrm{~nm}$

$\lambda_{\text {max }}=\mathbf{3 5 0} \mathrm{nm}$
$8 g$





8 h
$\min _{2 x}=350 \mathrm{~nm}$

The absorption spectra (using UV-Vis spectroscopy, concentration $=0.02 \mathrm{~g} / 100 \mathrm{~mL}$ in $\mathrm{CHCl}_{3}$ ) were recorded in $\mathrm{CHCl}_{3}$ and $\lambda_{\max \text { (absorption) }}$ (the absorption band appearing at the longest wavelength) of compounds $\mathbf{6 a}, \mathbf{6 j}, \mathbf{6 z}, \mathbf{6 e}, \mathbf{6 y}$

$\lambda_{\max \text { (absorption) }}(\mathrm{nm})=$ Pyrene: 337, 6a: 349, 6j: 349, 6z: 349, 6e: 349, 6y: 350.


Pyrene: $\lambda_{\text {max }}=337 \mathrm{~nm}$


6j: $\lambda_{\text {max }}=349 \mathrm{~nm}$


6e: $\lambda_{\text {max }}=349 \mathrm{~nm}$


6a: $\lambda_{\text {max }}=349 \mathrm{~nm}$

$6 \mathrm{z}: \lambda_{\text {max }}=349 \mathrm{~nm}$


6y: $\lambda_{\text {max }}=349 \mathrm{~nm}$

Preliminary analysis: Emission spectra of compounds $\mathbf{6 a}, \mathbf{6 j}, \mathbf{6 z}$, $\mathbf{6 e}$ and $\mathbf{6 y}$ in $\mathrm{CHCl}_{3}$ (concentration $=3.8 \mu \mathrm{M}$ ) at the excitation wavelength of 350 nm .

$\lambda_{\text {max (emission) }}(\mathrm{nm})$ at the
excitation wavelength of $350 \mathrm{~nm}=$
Pyrene: 393, 6a: 395, 6j: 390, 6z: 396, 6e:
404, 6y: 392.


Pyrene


6j

$6 e$


$6 z$

$6 y$

The absorption spectra (using UV-Vis spectroscopy, concentration $=0.02 \mathrm{~g} / 100 \mathrm{~mL}$ in THF) were recorded in THF and $\lambda_{\max (\text { absorption) }}$ (the absorption band appearing at the longest wavelength) of compounds $\mathbf{6 a}, \mathbf{6 j}, \mathbf{6 z}, \mathbf{6 e}, \mathbf{6 y}$

$6 z$


6a


6


6j

$6 y$
$\lambda_{\max (\text { absorption })}(\mathrm{nm})=$ Pyrene: 336, 6a: 349,
6j: 349, 6z: 350, 6e: 349, 6y: 349.

Preliminary analysis: Emission spectra of compounds $\mathbf{6 a}, \mathbf{6 j}, \mathbf{6 z}$, $\mathbf{6 e}$ and $6 \mathbf{y}$ in THF (concentration $=3.8 \mu \mathrm{M}$ ) at the excitation wavelength of 350 nm .

$\lambda_{\text {max (emission) }}(\mathrm{nm})$ at the
excitation wavelength of $350 \mathrm{~nm}=$
Pyrene: 392, 6a: 395, 6j: 391, 6z: 396, 6e:
405, 6y: 392.

The absorption spectra (using UV-Vis spectroscopy, concentration $=0.01 \mathrm{~g} / 100 \mathrm{~mL}$ in $\mathrm{CHCl}_{3}$ ) were recorded in $\mathrm{CHCl}_{3}$ and $\lambda_{\text {max (absorption) }}$ (the absorption band appearing at the longest wavelength) of compounds $\mathbf{7 b}, \mathbf{7 c}, \mathbf{7 d}, \mathbf{7 e}, \mathbf{7 f}, \mathbf{7 g}, \mathbf{7 h}, \mathbf{8 a}, \mathbf{8 b}, \mathbf{8 c}, \mathbf{8 e}, \mathbf{8 f}, \mathbf{8 g}, \mathbf{8 h}$.
For all the compounds $\lambda_{\text {max }}=350 \mathrm{~nm}$.







The absorption spectra (using UV-Vis spectroscopy, concentration $=0.01 \mathrm{~g} / 100 \mathrm{~mL}$ in $\mathrm{CHCl}_{3}$ ) were recorded in $\mathrm{CHCl}_{3}$ and $\lambda_{\text {max (absorption) }}$ (the absorption band appearing at the longest wavelength) of compounds $\mathbf{6 z g}, \mathbf{6 z h}, \mathbf{6 z i}, \mathbf{6 z j}, \mathbf{6 z k}, \mathbf{6 z l}$.
For all the compounds $\lambda_{\max }=350 \mathrm{~nm}$.



6zg: $\mathrm{R}=\mathrm{OMe}$


6zj: $\mathrm{R}=\mathrm{OMe}$


6zh: $\mathrm{R}=\mathrm{Me}$ 6zi: $\mathrm{R}=\mathrm{Ac}$


6zk: $\mathrm{R}=\mathrm{Cl}$
6zI: R = OEt

Preliminary analysis: emission spectra of $\mathbf{7 d}, \mathbf{7 e}, \mathbf{7 g}, \mathbf{7 h}, \mathbf{8 a}, \mathbf{8 b}, \mathbf{8 c}, \mathbf{8 e}, \mathbf{8 g}$ in $\mathrm{CHCl}_{3}$ at 350 nm . The concentration of all the sample solutions is $6.06 \mu \mathrm{M}$.


$7 \mathrm{~g}:$

7e: R=Me


$\lambda_{\text {max }}$ (emission) (nm) at the excitation wavelength of $350 \mathrm{~nm}=7 \mathrm{~d}: 392,7 \mathrm{e}: 392,7 \mathrm{~g}: 392$, 7h: 392.

$\lambda_{\text {max }}$ (emission) (nm) at the excitation wavelength of $350 \mathrm{~nm}=\mathbf{8 a}: 396, \mathbf{8 b}: 396, \mathbf{8 c}$ : 390, 8e: 390, 8g: 390.


Optical absorption (1A) and emission (1B) spectra of representative compounds. (1A): The absorption spectra of $\mathbf{6 a}, \mathbf{6 j}, \mathbf{6 z}, \mathbf{6 e} \mathbf{a n d} \mathbf{6 y}$ (concentration $=0.02 \mathrm{~g} / 100$ mL in $\mathrm{CHCl}_{3}$ ) in $\mathrm{CHCl}_{3}, \lambda_{\text {max (absorption) }}(\mathrm{nm})=$ Pyrene: 337, 6a: 349, $6 \mathrm{j}: 349,6 \mathrm{z}: 349,6 \mathrm{e}: 349,6 \mathrm{y}: 350$. (1B): Emission spectra of $6 \mathbf{a}, 6 \mathrm{j}, 6 \mathrm{z}, 6 \mathrm{e}$ and 6 y in $\mathrm{CHCl}_{3}$ (concentration $=3.8 \mu \mathrm{M}$ ) at the excitation wavelength of $350 \mathrm{~nm} \lambda_{\text {max (emission) }}(\mathrm{nm})=$ Pyrene: $393, \mathbf{6 a}: 395,6 \mathrm{j}: 390,6 \mathrm{z}: 396,6 \mathbf{e}: 404,6 \mathrm{y}: 392$.

Figure 1 (in the manuscript)


Optical absorption (1C) and emission (1D) spectra of representative compounds. (1C): The absorption spectra of $\mathbf{6 a}, \mathbf{6 j}, \mathbf{6 z}, \mathbf{6 e}$ and $\mathbf{6 y}$ (concentration $=0.02 \mathrm{~g} / \mathbf{1 0 0}$
 THF) at the excitation wavelength of $350 \mathrm{~nm} \lambda_{\text {max (emission) }}(\mathrm{nm})=$ Pyrene: 392, 6a: 395, 6j: 391, 6z: 396, 6e: 405, 6y: 392

Figure 1 (in the manuscript)


Optical absorption (1G) and emission (1E), (1F) spectra of representative compounds. (1G): The absorption spectra of $\mathbf{6 z g}, \mathbf{6 z h}, \mathbf{6 z i} \mathbf{6} \mathbf{6 z j} \mathbf{6} \mathbf{6 z} \mathbf{~ a n d ~} \mathbf{6 z l}$ (concentration $=0.01 \mathrm{~g} / 100 \mathrm{~mL}$ in $\mathrm{CHCl}_{\mathbf{3}}$ ), $\lambda_{\text {max (absorption) }}(\mathrm{nm})=\mathbf{6 z g}, \mathbf{6 z h}, \mathbf{6 x}, \mathbf{6 z j}, \mathbf{6 z k}$ and $\mathbf{6 z l}=350 \mathrm{~nm}$. (1E) and (1F): Emission spectra of $\mathbf{7 d} \mathbf{d} \mathbf{7 e}, \mathbf{7 g}, \mathbf{7 h}, \mathbf{8 a}, \mathbf{8 b}, \mathbf{8 c}, 8 \mathbf{8}$ and $\mathbf{8 g}$ (concentration $=6.06 \mu \mathrm{M}$ in $\mathrm{CHCl}_{3}$ ) at the $\mathbf{e x c i t a t i o n ~}$ wavelength of $350 \mathrm{~nm} \lambda_{\text {max (emission) }}(\mathrm{nm})=7 \mathrm{dd}: 392,7 \mathrm{e}: 392,7 \mathrm{~g}: 392,7 \mathrm{~h}: 392, \mathbf{8 a}: 396, \mathbf{8 b}: 396, \mathbf{8 c}: 390,8 \mathrm{8e}: 390, \mathbf{8 g}: 390$.

Figure 1 (in the manuscript)

## Fluorescence Quantum yield ( $\Phi$ ) calculation.

The quantum yield of $\mathbf{6 j}, \mathbf{6 y}, \mathbf{6 z}, \mathbf{7 h}, \mathbf{8 a}$ was calculated using anthracene ( $\Phi=0.27$ ) as reference (ref. A. Srivastava, S. Grewal, S. Singh, Rajani and S. Venkataramani, ChemPhotoChem, 2023, 7, e202300029). For the calculation of quantum yield different concentration of each compound were made in ethanol $(\eta=1.36)$ with the absorbance less than 0.1 nm at 355 nm . Fluorescence spectra were recorded for all the solutions at same of excited $\lambda_{\max }$. Then by comparing the integrated photoluminescence intensity and absorbency values of all the sample solutions with the reference anthracene quantum yields were calculated. The quantum yield was calculated following the given equation below.
$\Phi_{\mathrm{X}}=\Phi_{\mathrm{ST}}\left(\mathrm{m}_{\mathrm{X}} / \mathrm{m}_{\mathrm{ST}}\right)\left(\eta_{\mathrm{x}}^{2} / \eta_{\mathrm{sT}}^{2}\right)$
Where $\Phi$ is the quantum yield, $\eta$ is the refractive index of solvent, $m$ is slope, $X$ is the sample and ST is the standard.


Figure S1. Integrated Fluorescence Intensity vs Absorbance plot for the anthracene.


Figure S2. Integrated Fluorescence Intensity vs Absorbance plot for the compound $\mathbf{6 y}$.


Figure S3. Integrated Fluorescence Intensity vs Absorbance plot for the compound $\mathbf{6 a}$.


Figure S4. Integrated Fluorescence Intensity vs Absorbance plot for the compound $\mathbf{6 j}$.


Figure S5. Integrated Fluorescence Intensity vs Absorbance plot for the compound $\mathbf{6 z}$.


Figure S6. Integrated Fluorescence Intensity vs Absorbance plot for the compound 7h.


Figure S7. Integrated Fluorescence Intensity vs Absorbance plot for the compound 8a.

Table S1. Quantum yield of $\mathbf{6 y}, \mathbf{6 a}, \mathbf{6 j}, \mathbf{6 z}, \mathbf{7 h}, 8 \mathrm{Ba}$.

| Compound | slope $\left(\mathrm{m}_{\mathrm{x}}\right)$ | quantum yield $(\Phi)$ |
| :---: | :---: | :---: |
| $\mathbf{6 y}$ | 151762.60046 | 0.226 |
| $\mathbf{6 a}$ | 93113.22571 | 0.139 |
| $\mathbf{6 j}$ | 46274.97295 | 0.069 |
| $\mathbf{6 z}$ | 112328.07115 | 0.169 |
| $\mathbf{7 h}$ | 60141.06904 | 0.089 |
| $\mathbf{8 a}$ | 2969.97295 | 0.004 |

## Experimental

## General

${ }^{1} \mathrm{H}$ and ${ }^{13} \mathrm{C}\{1 \mathrm{H}\}$ NMR spectra of compounds were recorded (using TMS as an internal standard) in 400 and $\sim 101 \mathrm{MHz}$ spectrometers, respectively. The HRMS analysis data of samples were obtained from the QTOF mass analyzer using the electrospray ionization (ESI) method. FT-IR spectra of samples were recorded as neat or thin films. Column chromatography purification of crude reaction mixtures was carried out on silica gel (100-200 mesh). Reactions were conducted in anhydrous solvents under a nitrogen atm wherever required. Organic layers obtained after the workup were dried using anhydrous $\mathrm{Na}_{2} \mathrm{SO}_{4}$. Thin layer chromatography (TLC) analyses were performed on silica gel or alumina plates and components were visualized by UV light or under iodine vapor. Isolated yields of all the products are reported and yields were not optimized.

General procedure for the synthesis of compounds 4a-h: A solution of aromatic aldehyde ( 1 equiv), carboxylic acid ( 1 equiv), ammonia solution ( 7 N in methanol, $25-50$ equiv), and isocyanide ( 1 equiv) in TFE ( 2 mL ) was stirred at room temperature for 24 h . The reaction mixture was diluted with $\mathrm{CH}_{2} \mathrm{Cl}_{2}$ and washed with water and brine. The organic extracts were combined, and dried over anhydrous $\mathrm{Na}_{2} \mathrm{SO}_{4}$ and the solvent was evaporated. The crude mixture was subjected to column chromatography to afford the compound 4a-h (Procedure adapted from ref. L. A. Polindara-García et al Eur. J. Org. Chem., 2022, e202101517).

General procedure for the synthesis of compounds $\mathbf{6 a - z}, \mathbf{6 z a}-\mathbf{6 z c}, \mathbf{6 z g}-6 \mathrm{zn}$ : A solution of pyrenylglycinamide 4 ( 1 equiv), aryl iodide (4 equiv), KOAc ( 5 equiv), $\mathrm{Pd}(\mathrm{OAc})_{2}(10 \mathrm{~mol} \%)$, and $\mathrm{CuBr}_{2}(10 \mathrm{~mol} \%)$ in dry toluene $(1.5 \mathrm{~mL})$ was heated in a sealed tube vial under conventional heating (oil bath) at $130^{\circ} \mathrm{C}$ for 48 h . The tube was flashed with nitrogen before heating. The reaction mixture was concentrated under reduced pressure. The crude mixture was subjected to column chromatography to afford the corresponding C-H arylated pyrenylglycine.

General procedure for the synthesis of compounds 6ze: A solution of pyreneglycinamide 4a ( 1 equiv), methyl 4-(bromomethyl)benzoate (4 equiv), $\mathrm{Pd}(\mathrm{OAc})_{2}$ ( $10 \mathrm{~mol} \%$ ), $\mathrm{K}_{2} \mathrm{CO}_{3}$ (2 equiv), NaOTf ( 3 equiv) in tert-amylOH ( 1.5 mL ) was heated in a sealed tube vial under conventional heating (oil bath) at $120^{\circ} \mathrm{C}$ for 48 h . The tube was flashed with nitrogen before heating. The reaction mixture was concentrated under reduced pressure. The crude mixture was subjected to column chromatography to afford the compound $\mathbf{6 z e}$.

General procedure for the synthesis of compounds 6zf: A solution of pyreneglycinamide $4 \mathbf{a}$ ( 1 equiv), 1 -iodobutane (4 equiv), $\mathrm{Pd}(\mathrm{OAc})_{2}(10 \mathrm{~mol} \%), \mathrm{Ag}_{2} \mathrm{CO}_{3}$ (2 equiv), $(\mathrm{BnO})_{2} \mathrm{PO}_{2} \mathrm{H}$ ( 0.3 equiv) in tert-amylOH ( 1.5 mL ) was heated in a sealed tube vial under conventional heating (oil bath) at $120^{\circ} \mathrm{C}$ for 48 h . The tube was flashed with nitrogen before heating. The reaction mixture was concentrated under reduced pressure. The crude mixture was subjected to column chromatography to afford the compounds $\mathbf{6 z f}$.

General procedure for the synthesis of compounds 6zd: A solution of pyreneglycinamide 4 ( 1 equiv), 1 -iodopentane ( 4.0 equiv), $\mathrm{Pd}(\mathrm{OAc})_{2}(10 \mathrm{~mol} \%$ ), KOAc ( 2.0 equiv) in dry $1,4-$ dioxane ( 1.5 mL ) was heated in a sealed tube vial under conventional heating (oil bath) at 130 ${ }^{\circ} \mathrm{C}$ for 48 h . The tube was flashed with nitrogen before heating. The reaction mixture was concentrated under reduced pressure. The crude mixture was subjected to column chromatography to afford the compound $\mathbf{6 z d}$.

General procedure for the synthesis of compounds 7a-h: To an appropriate arylated compound 6 ( 1 equiv) dissolved in $\mathrm{H}_{2} \mathrm{O} / \mathrm{THF}(1: 1,2-4 \mathrm{~mL}), 12 \mathrm{~N} \mathrm{HCl}(0.33-0.5 \mathrm{~mL})$ was added. The mixture was stirred at rt for 15 min . Zinc dust ( 15 equiv) was then added in three portions and the mixture was stirred at rt for $18-36 \mathrm{~h}$. The mixture was transferred to a separating funnel with $2 \mathrm{~N} \mathrm{NaOH}(20 \mathrm{~mL})$ and extracted with ethyl acetate. The reaction mixture was concentrated under reduced pressure. The crude mixture was subjected to column chromatography to afford compound $\mathbf{7 a} \mathbf{a}$ (the plicolinamide directing group removal procedure was carried out using the standard reported procedure).

General procedure for the synthesis of compounds 8a-h and 8f2-(D): An appropriate amount of $N$-protected amino acid (1 equiv), $N$-(3-dimethylaminopropyl)- $N^{\prime}$ ethylcarbodiimide hydrochloride ( 1.1 equiv), 1-hydroxybenzotriazole hydrate ( 1.1 equiv) in dry DCM ( 5 mL ) was stirred for 1 h at $0^{\circ} \mathrm{C}$ under a nitrogen atmosphere. Then, an appropriate amount of amine compound 7 ( 1 equiv) was added to the above mixture and stirred for 24-25 h at room temperature. The resulting solution was then subjected to aqueous workup and washed with aqueous $\mathrm{NaHCO}_{3}$ solution (two times). The resulting solution mixture was concentrated and purified on silica gel column chromatography (EtOAc/hexane) to give the corresponding pyrene-based peptides.


General procedure for the synthesis of pyrene-4-carbaldehyde (3d): To a solution of 1,2,3,6,7,8-hexahydropyrene (3da, $208 \mathrm{mg}, 1 \mathrm{mmol}$ ) in $\mathrm{CH}_{2} \mathrm{Cl}_{2}(10 \mathrm{~mL})$ was added stannic chloride ( $140 \mu \mathrm{~L}, 1.2 \mathrm{mmol}$ ), and the mixture was cooled to $0^{\circ} \mathrm{C}$ under a nitrogen atmosphere. Then, $1,1^{\prime}$-dichlorodimethyl ether ( $136 \mu \mathrm{~L}, 1.5 \mathrm{mmol}$ ) was added by a syringe, and the mixture was stirred at $0^{\circ} \mathrm{C}$ for 5 h . The reaction was monitored by TLC. After completion, the reaction mixture was gradually warmed to rt and then quenched with ice water, acidified by dilute hydrochloric acid, and extracted with DCM. The organic phase was dried over anhydrous $\mathrm{Na}_{2} \mathrm{SO}_{4}$, filtrated, and then concentrated to get the crude product 3db which is used for the next step without purification. To a solution of 1,2,3,6,7,8-hexahydropyrene-4-carbaldehyde ( $\mathbf{3 d b}$, 1 mmol ) in dry toluene ( 5 mL ) was added DDQ ( 2 equiv) and the reaction mixture was heated at $100^{\circ} \mathrm{C}$ for 20 h . The reaction was monitored by TLC. The reaction mixture was concentrated under reduced pressure. The crude mixture was subjected to column chromatography to afford the compound 3d ( $86 \%$ yield). (The preparation of $\mathbf{3 d}$ was carried out using the reported procedure: (a) P.-F. Li and C.-F. Chen, J. Org. Chem., 2012, 77, 9250. (b) K. W. Bair, C. W. Andrews, R. L. Tuttle, V. C. Knick, M. Cory and D. D. McKee, J. Med. Chem., 1991, 34, 1983).


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