

Supporting Information

NMR analysis of the enantiomeric purity of chiral diols by a new chiral boron agent

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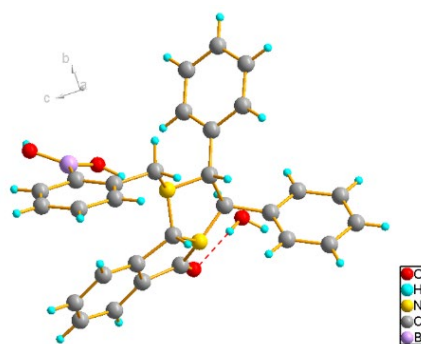
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1. Crystallographic data



The crystal structure of boric acid D

Table S1. Crystal data and structure refinement for boric acid D.

Identification code	2061749
Empirical formula	$C_{29}H_{26}BN_2O_{3.5}$
Formula weight	469.33
Temperature	150.04 K
Crystal system, space group	Orthorhombic, $P2_12_12_1$
$a/\text{\AA}$	7.7796(5)
$b/\text{\AA}$	13.1314(11)
$c/\text{\AA}$	24.231(2)
$\alpha/^\circ$	90
$\beta/^\circ$	90
$\gamma/^\circ$	90
Volume/ \AA^3	2475.4(3)
Z	4
$\rho_{\text{calc}}/\text{cm}^3$	1.259
μ/mm^{-1}	0.082
F (000)	988.0
Crystal size/ mm^3	$0.2 \times 0.15 \times 0.15$
Radiation	MoK α ($\lambda = 0.71073$)
2Θ range for data collection/ $^\circ$	6.206 to 60.058
Index ranges	$-9 \leq h \leq 10, -15 \leq k \leq 18, -34 \leq l \leq 25$
Reflections collected	21320
Independent reflections	7192 [$R_{\text{int}} = 0.0418, R_{\text{sigma}} = 0.0470$]
Data/restraints/parameters	7192/3/339
Goodness-of-fit on F^2	1.081
Final R indexes [$I \geq 2\sigma(I)$]	$R_1 = 0.0451, wR_2 = 0.1076$
Final R indexes [all data]	$R_1 = 0.0499, wR_2 = 0.1108$
Largest diff. peak/hole / $e \text{\AA}^{-3}$	0.42/-0.33
Flack parameter	0.5(10)

2. NMR spectra data

Figure S1: ^1H NMR of boric acid D (400 MHz, CDCl_3)

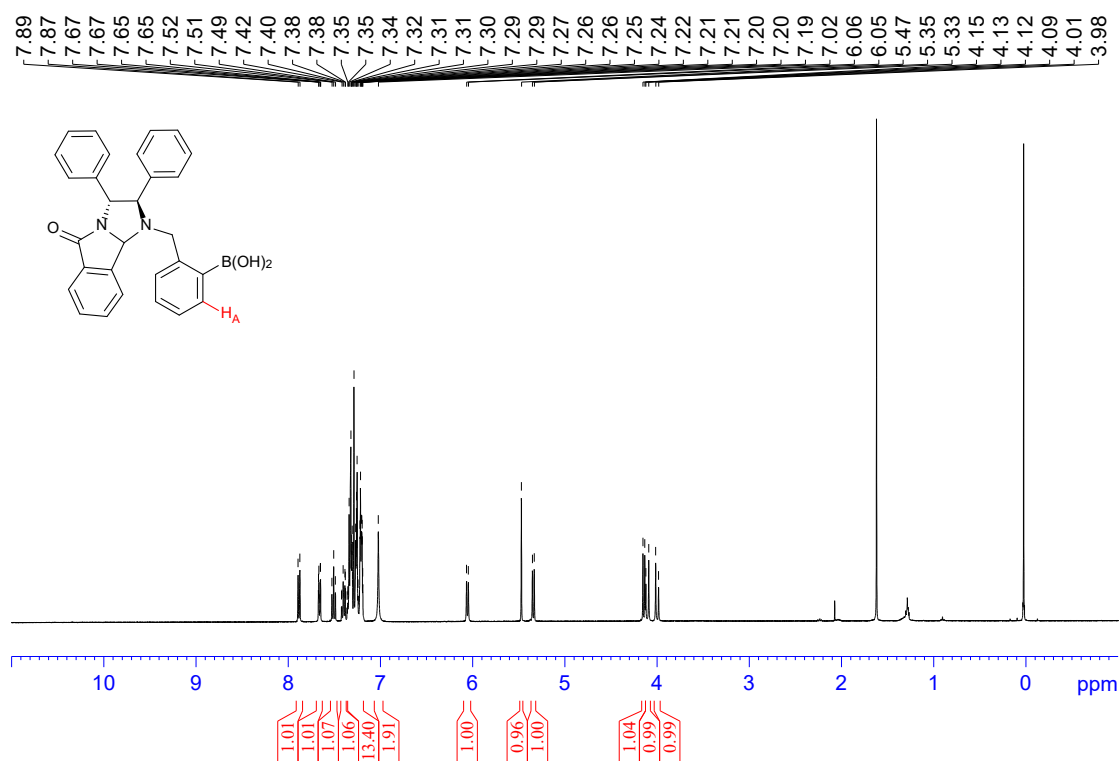


Figure S2: ^{13}C NMR of boric acid D (100 MHz, CDCl_3)

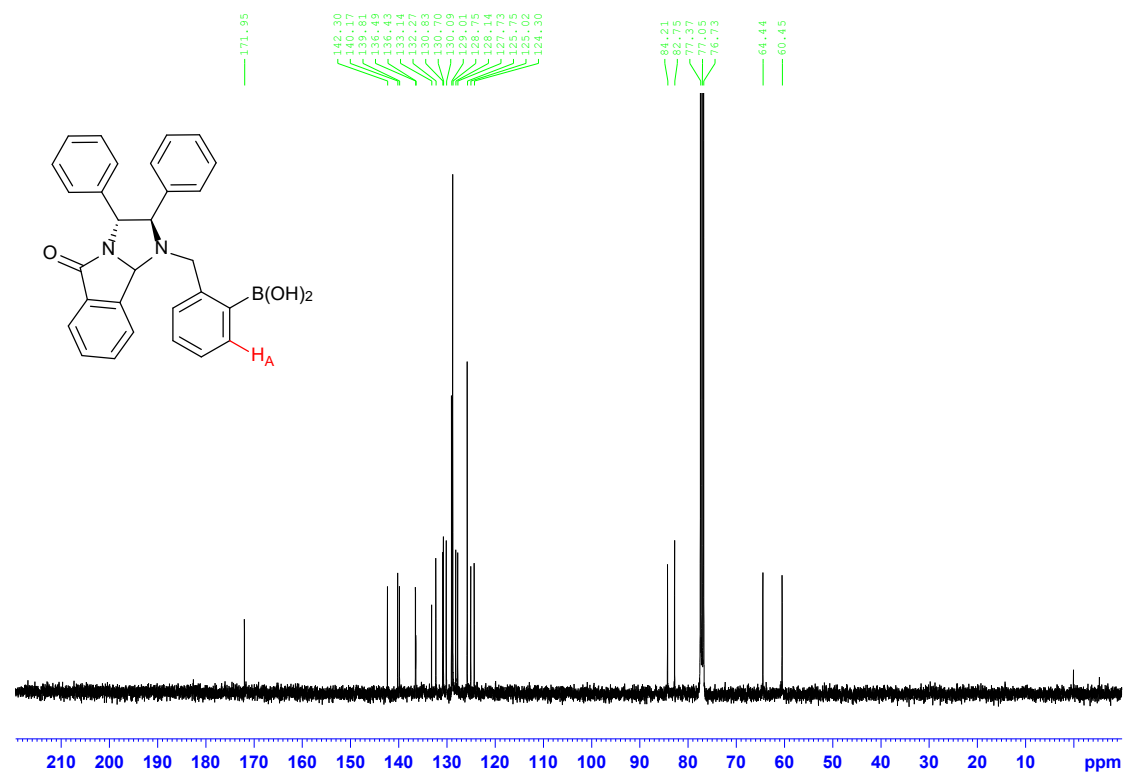


Figure S3: COSY of boric acid D (400 MHz, CD₂Cl₂)

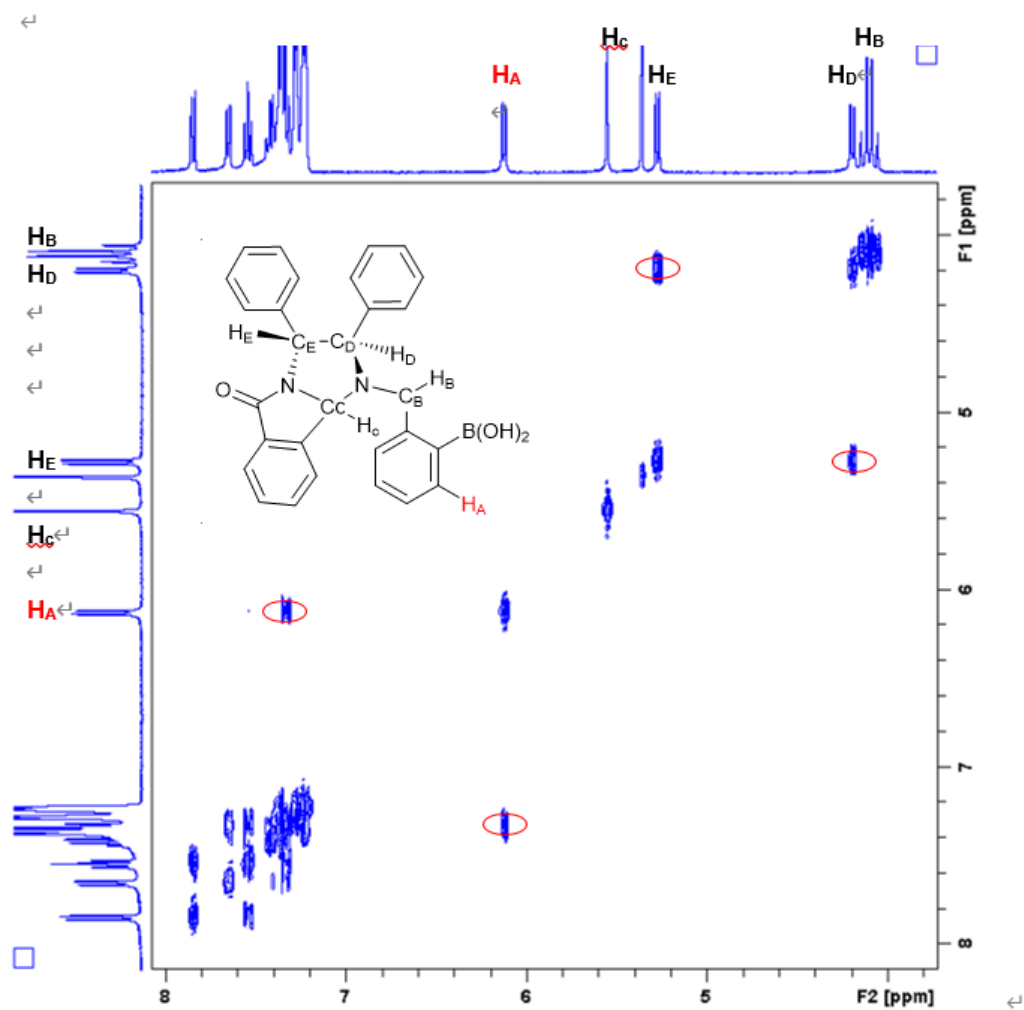


Figure S5. NMR data attesting for the absence of racemization in the derivatization process

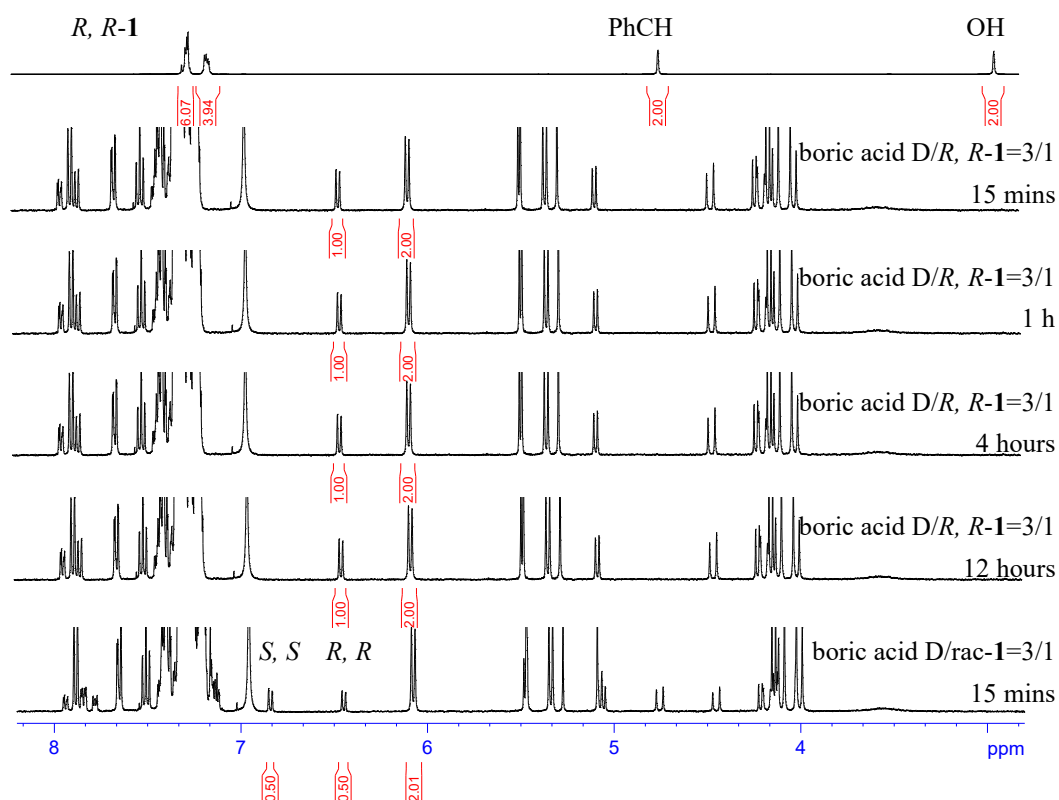


Figure S6. NMR data of optimization of derivatization reaction conditions

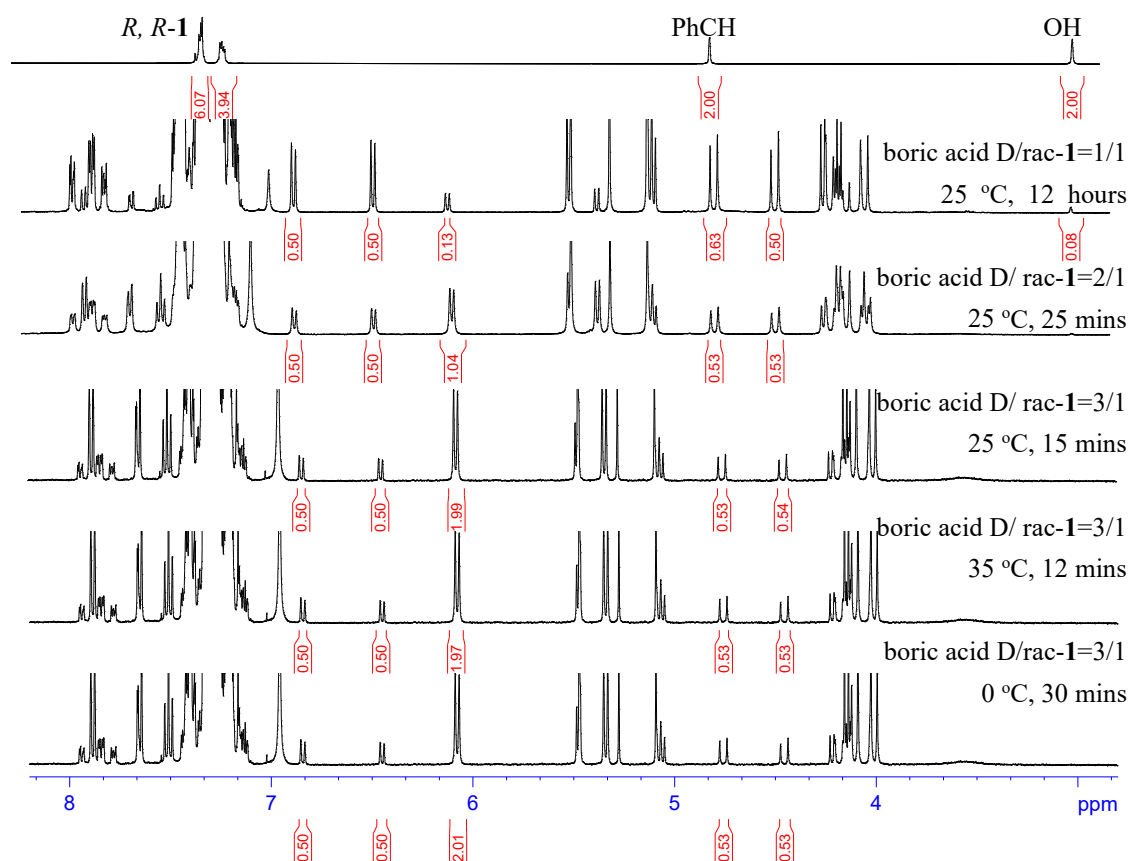


Figure S7. ¹H NMR of boric acid D and racemic 1

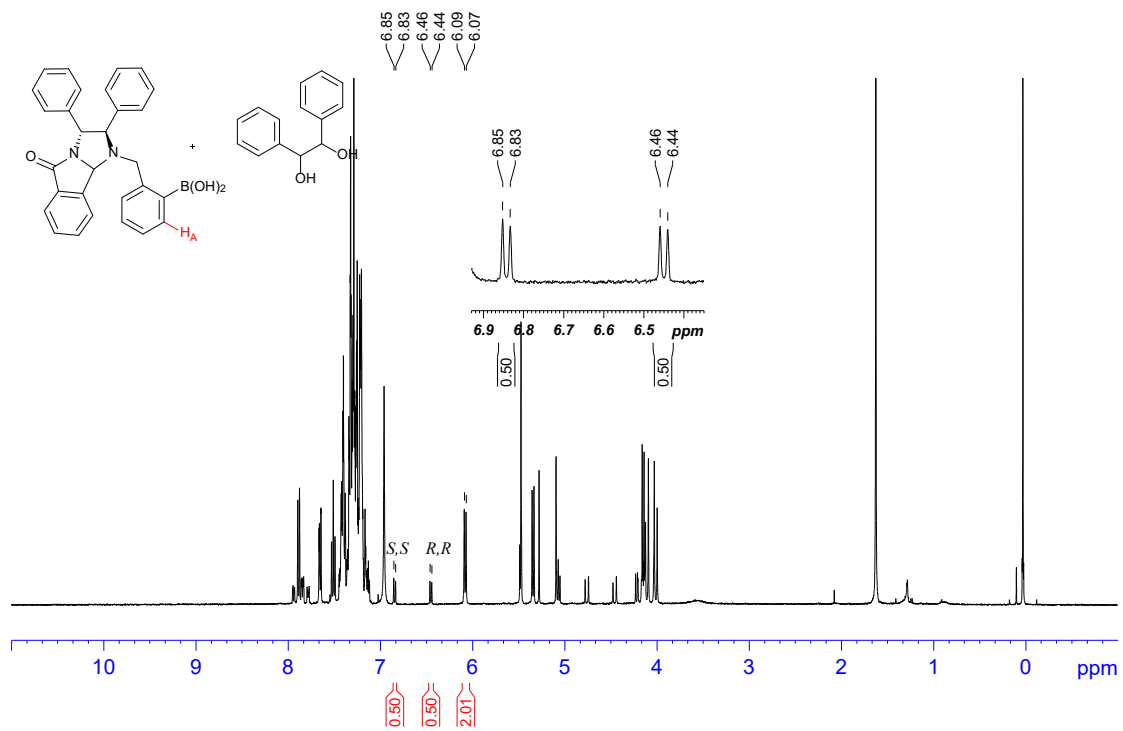


Figure S8. ¹H NMR of boric acid D and racemic 2

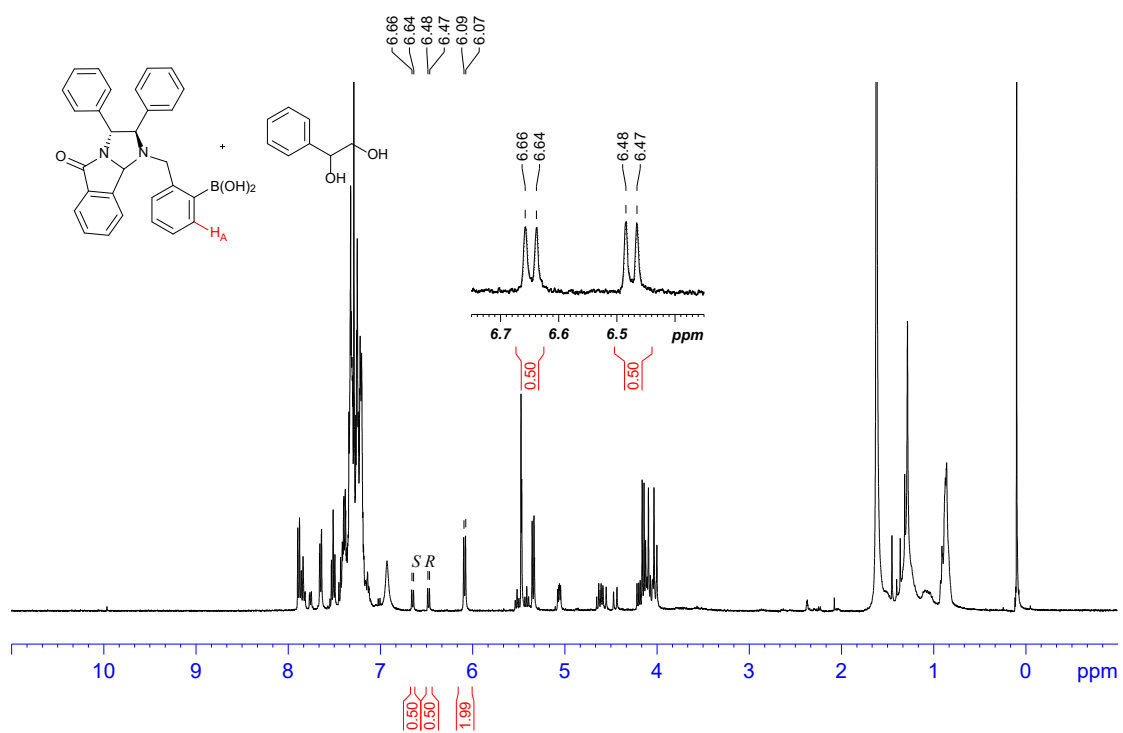


Figure S9. ¹H NMR of boric acid D and R-2

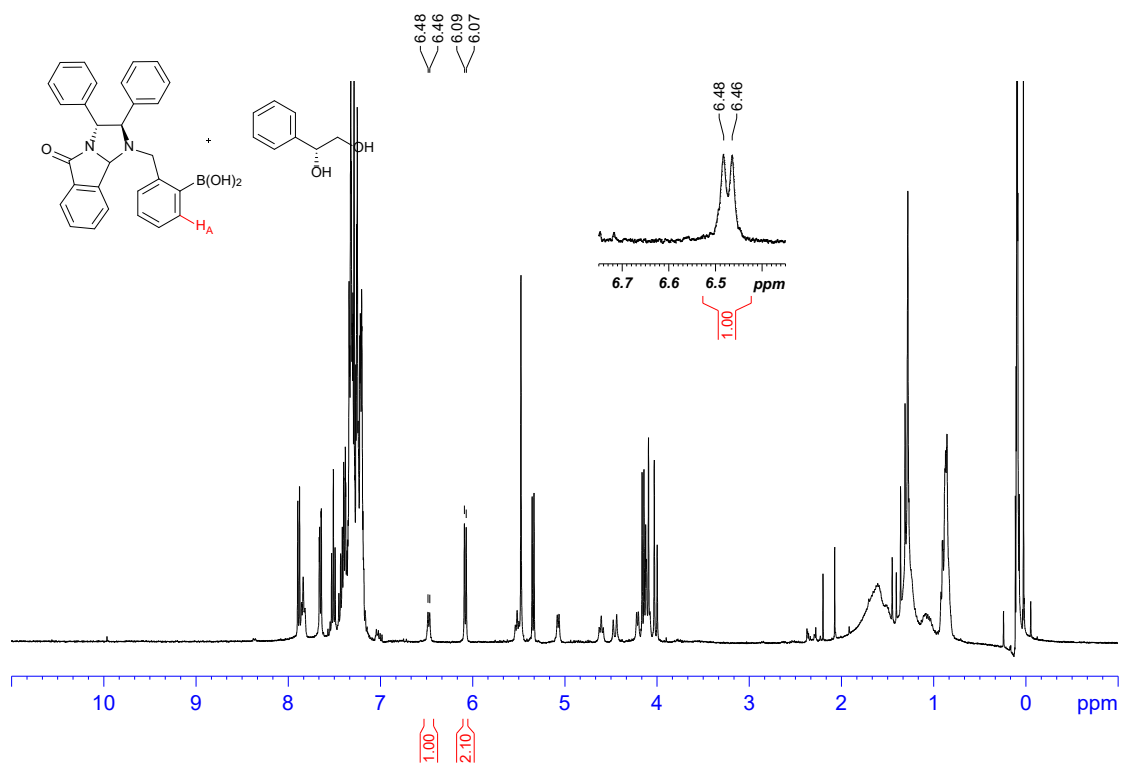


Figure S10. ¹H NMR of boric acid D and racemic 3

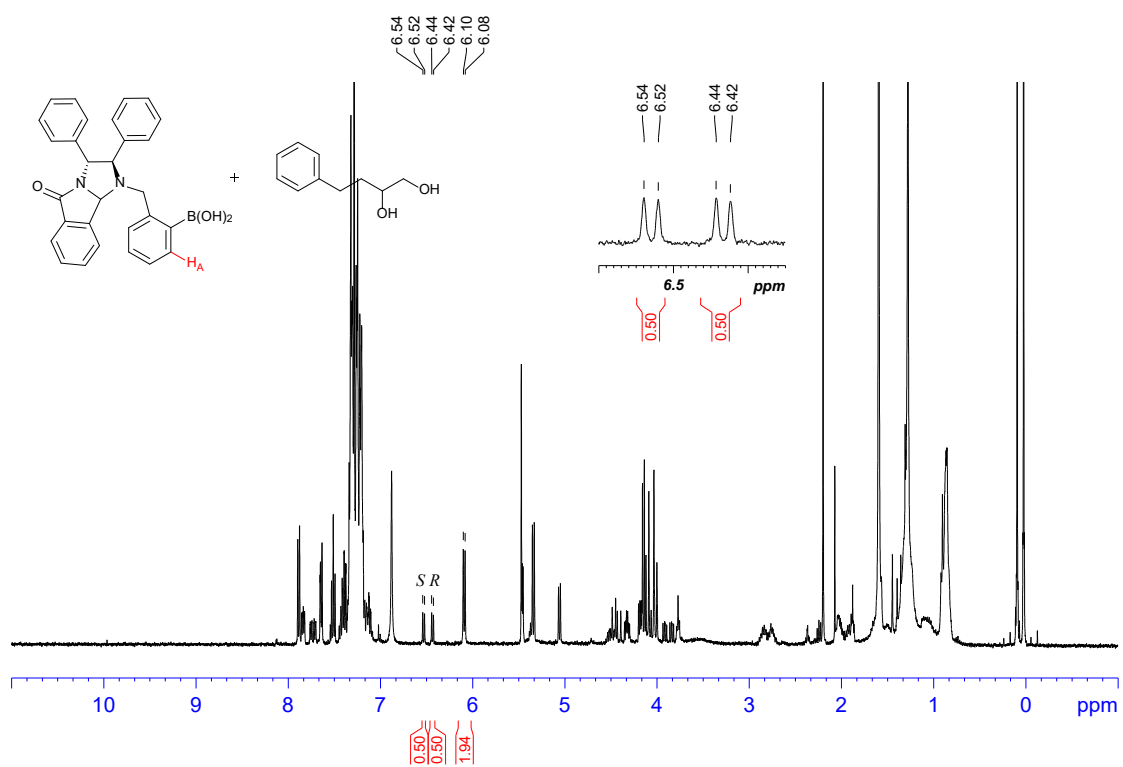


Figure S11. ^1H NMR of boric acid D and *R*-3

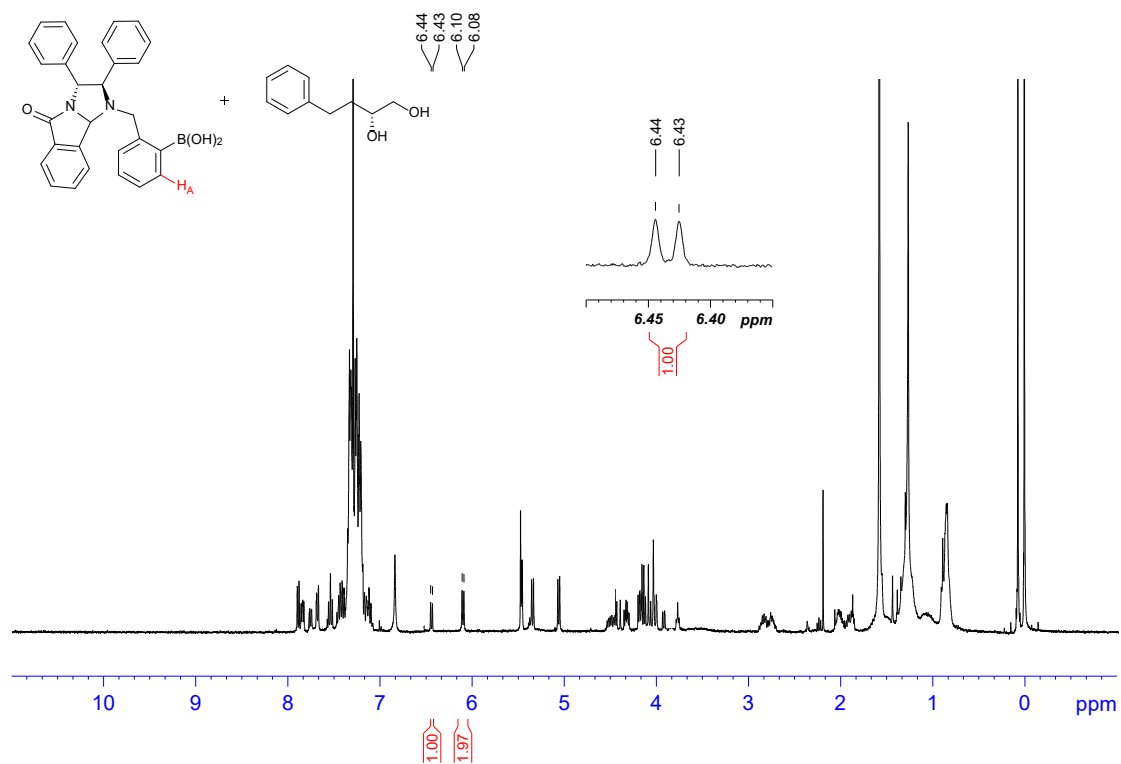


Figure S12. ^1H NMR of boric acid D and racemic 4

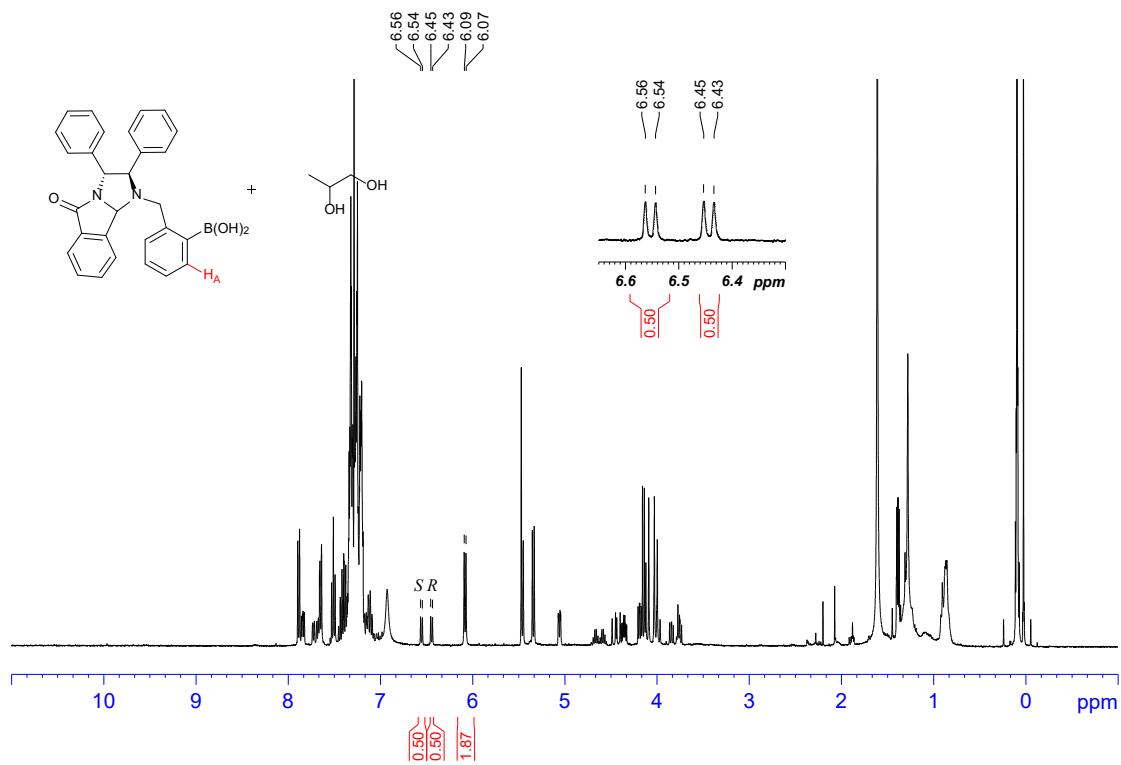


Figure S13. ¹H NMR of boric acid D and S-4

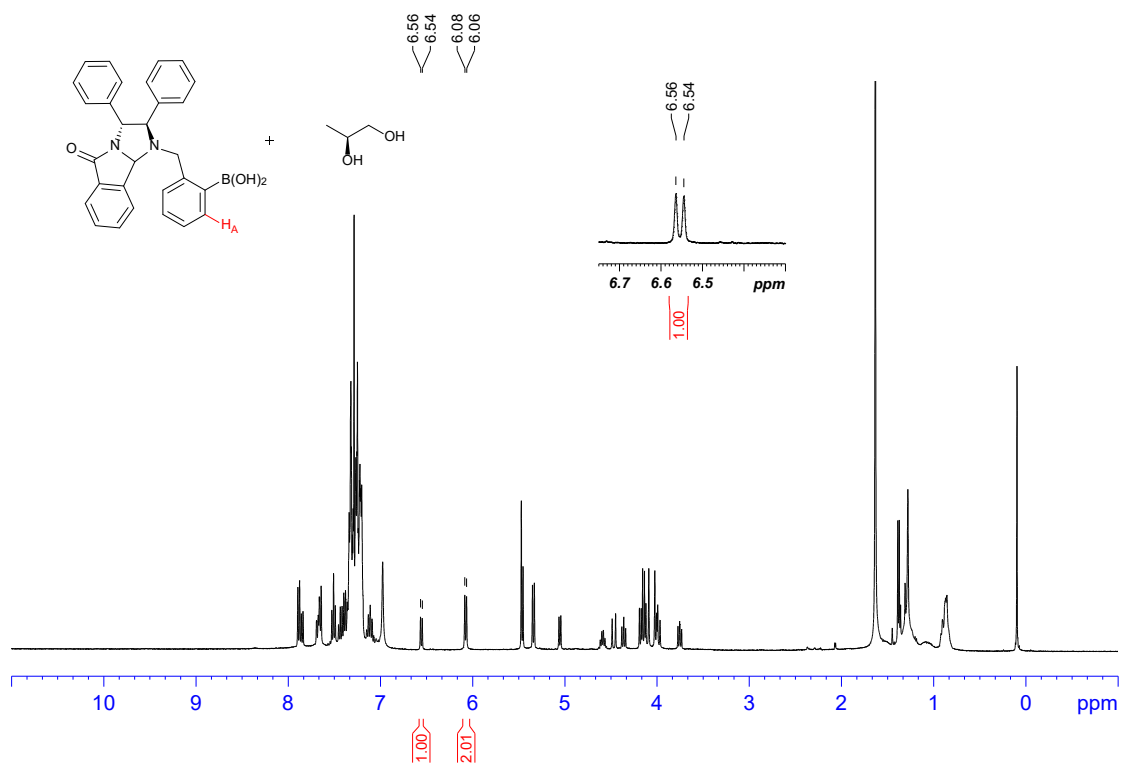


Figure S14. ¹H NMR of boric acid D and racemic 5

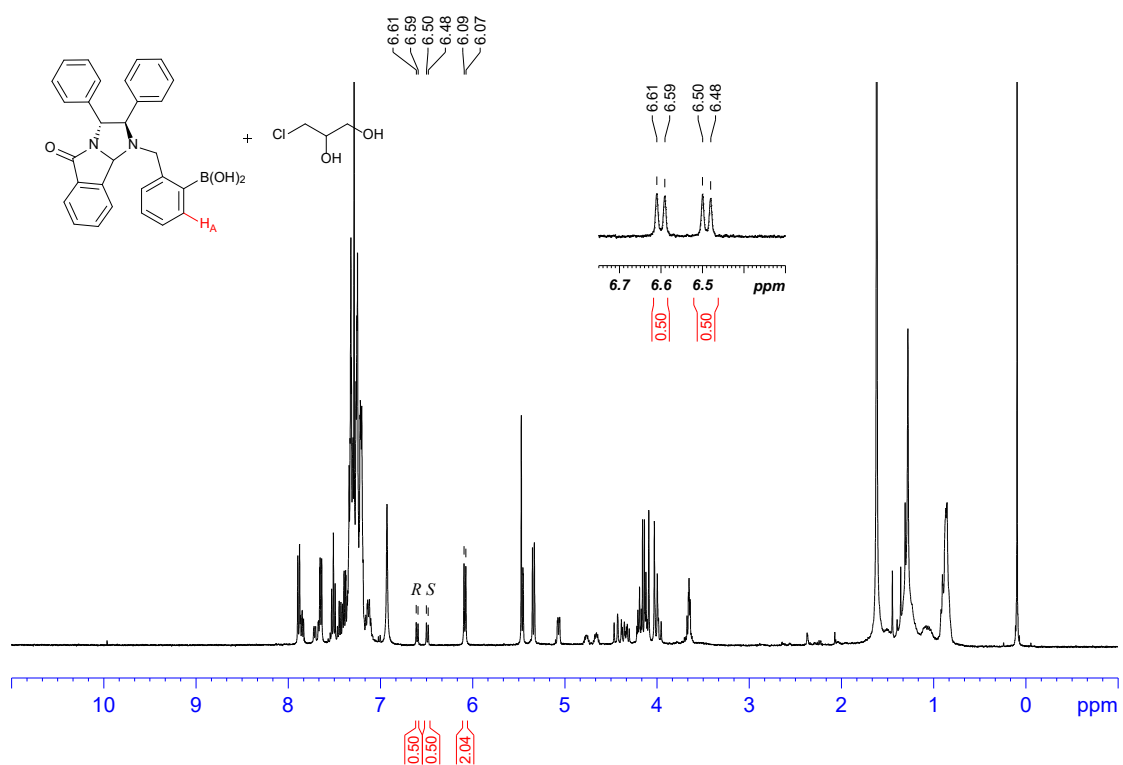


Figure S15. ¹H NMR of boric acid D and S-5

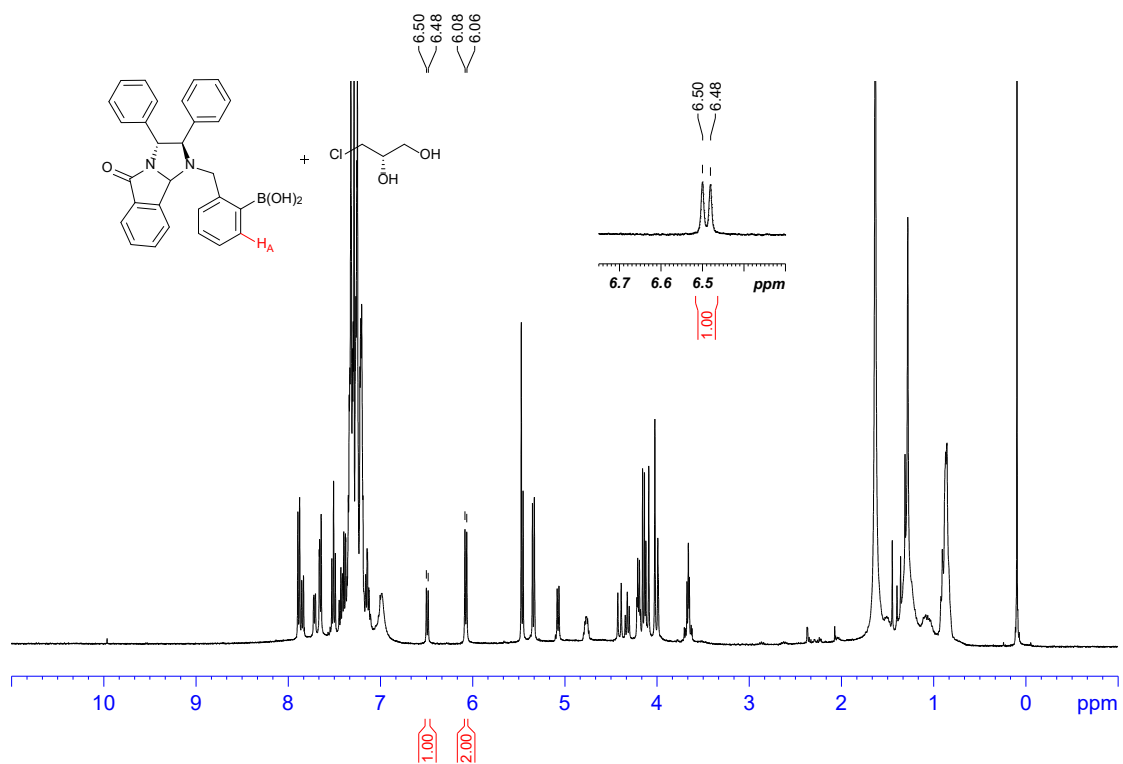


Figure S16. ¹H NMR of boric acid D and racemic 6

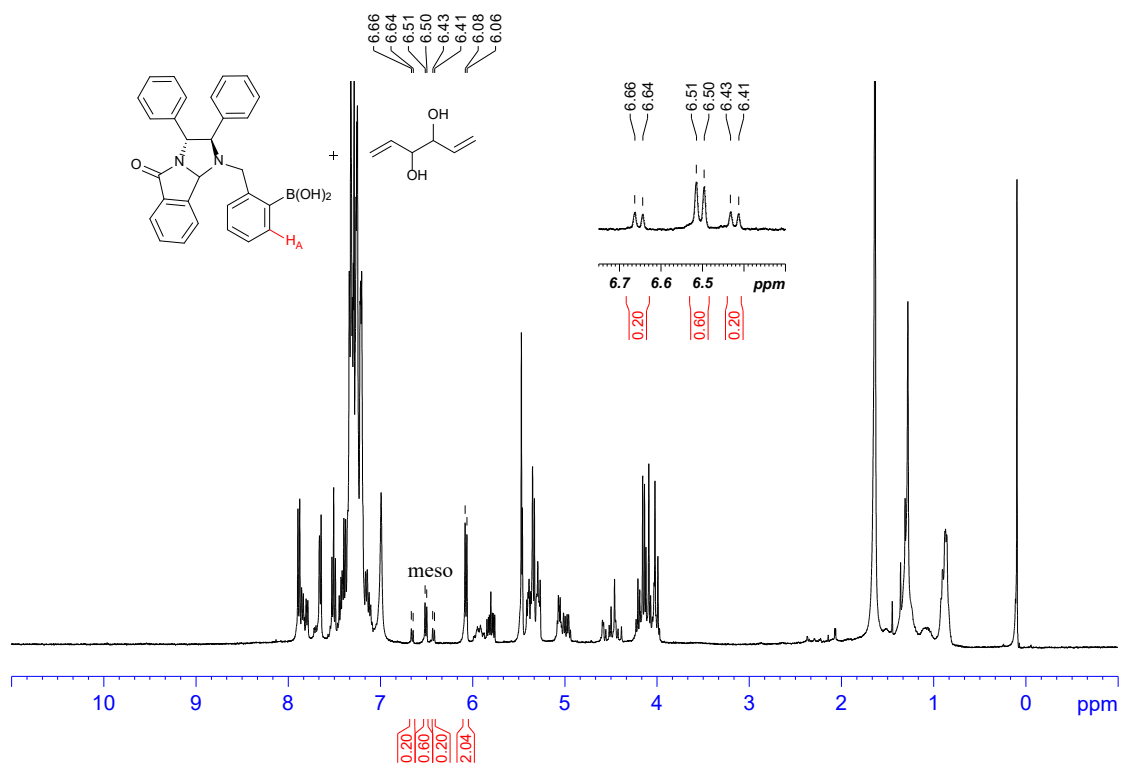


Figure S17. ¹H NMR of boric acid D and racemic 7

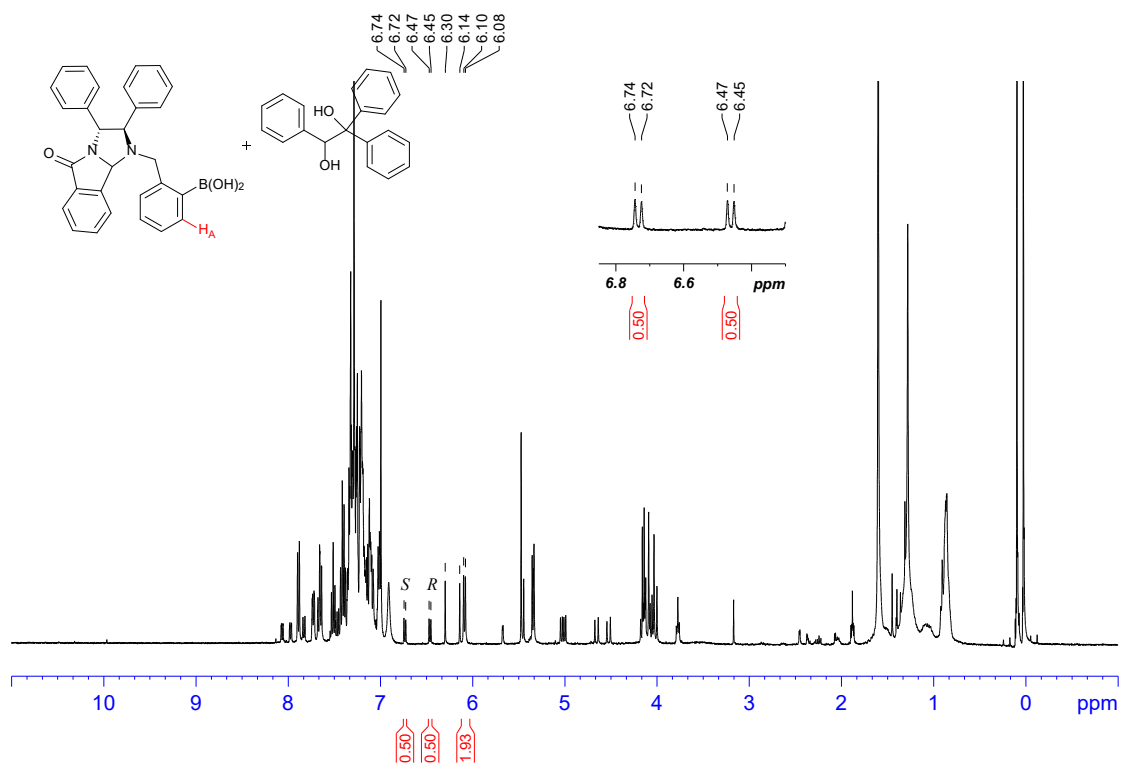


Figure S18. ¹H NMR of boric acid D and R-7

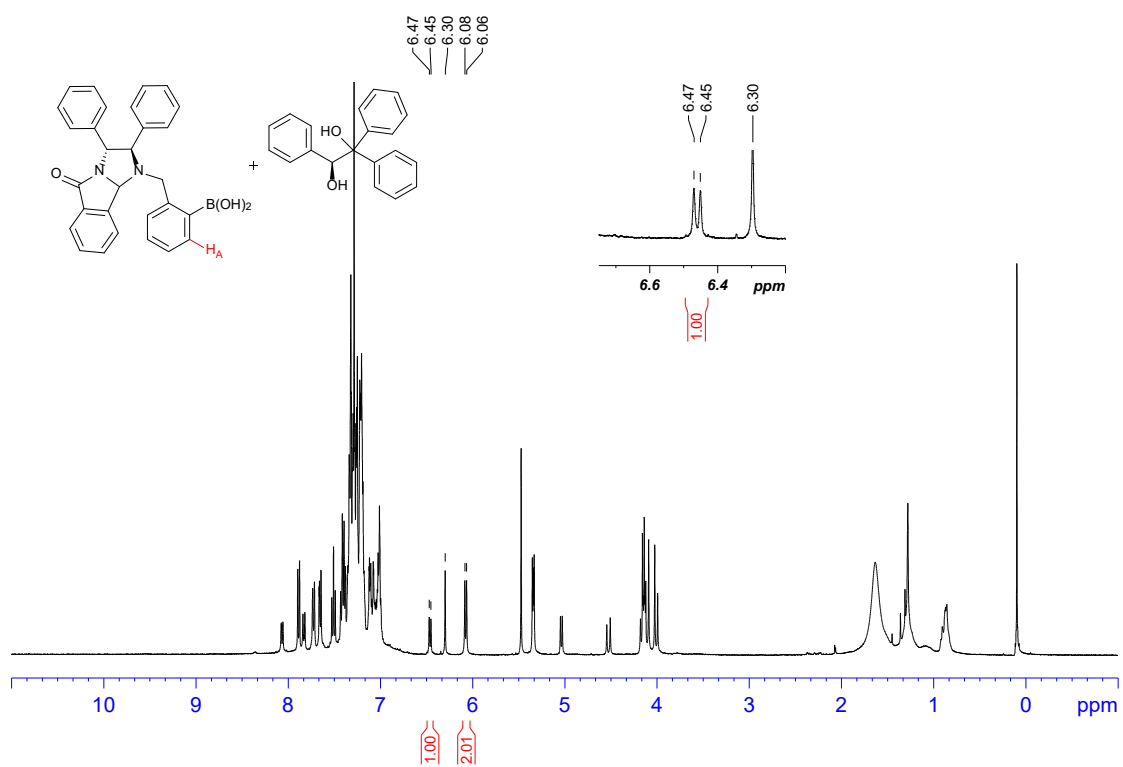


Figure S19. ^1H NMR of boric acid D and racemic **8**

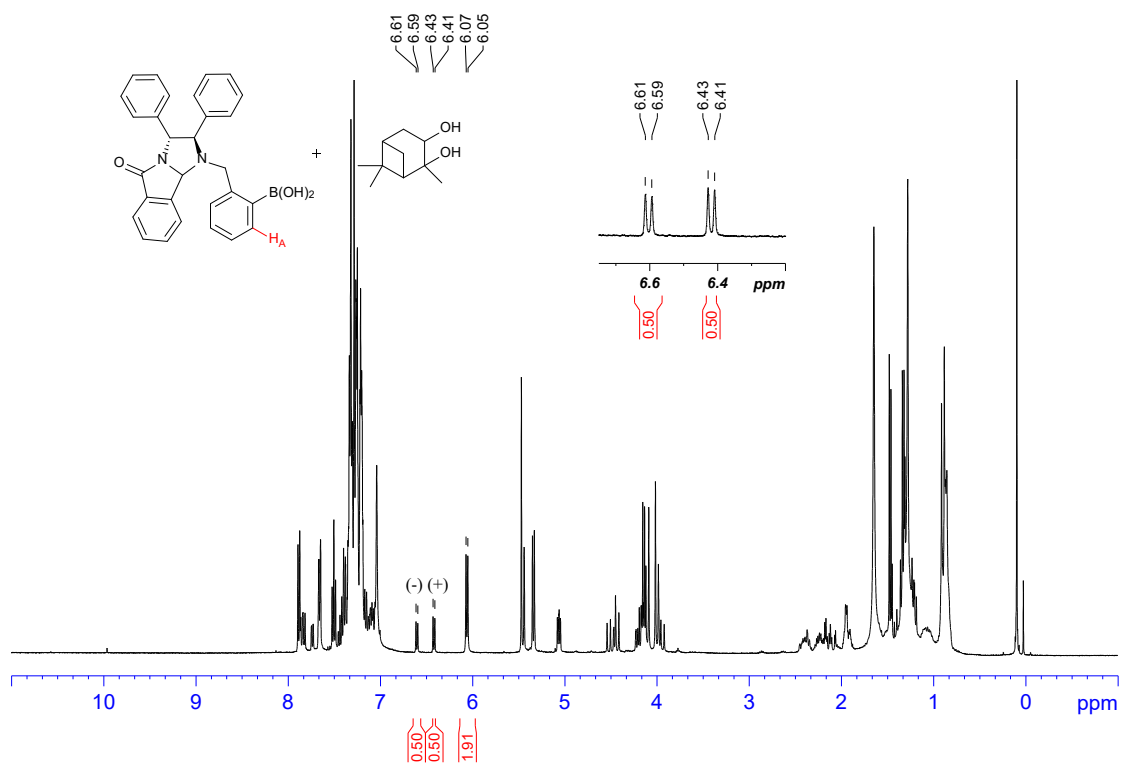


Figure S20. ^1H NMR of boric acid D and (*1R,2R,3S,5R*)-(-)-**8**

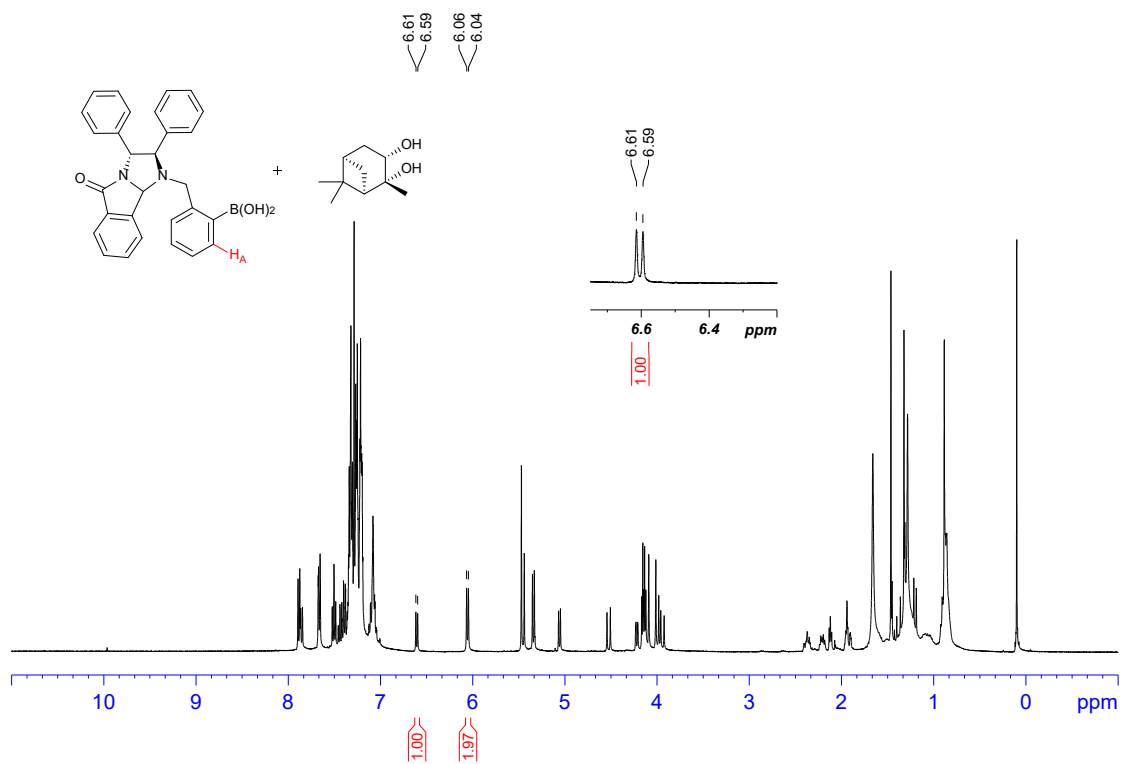


Figure S21. ¹H NMR of boric acid D and racemic **9**

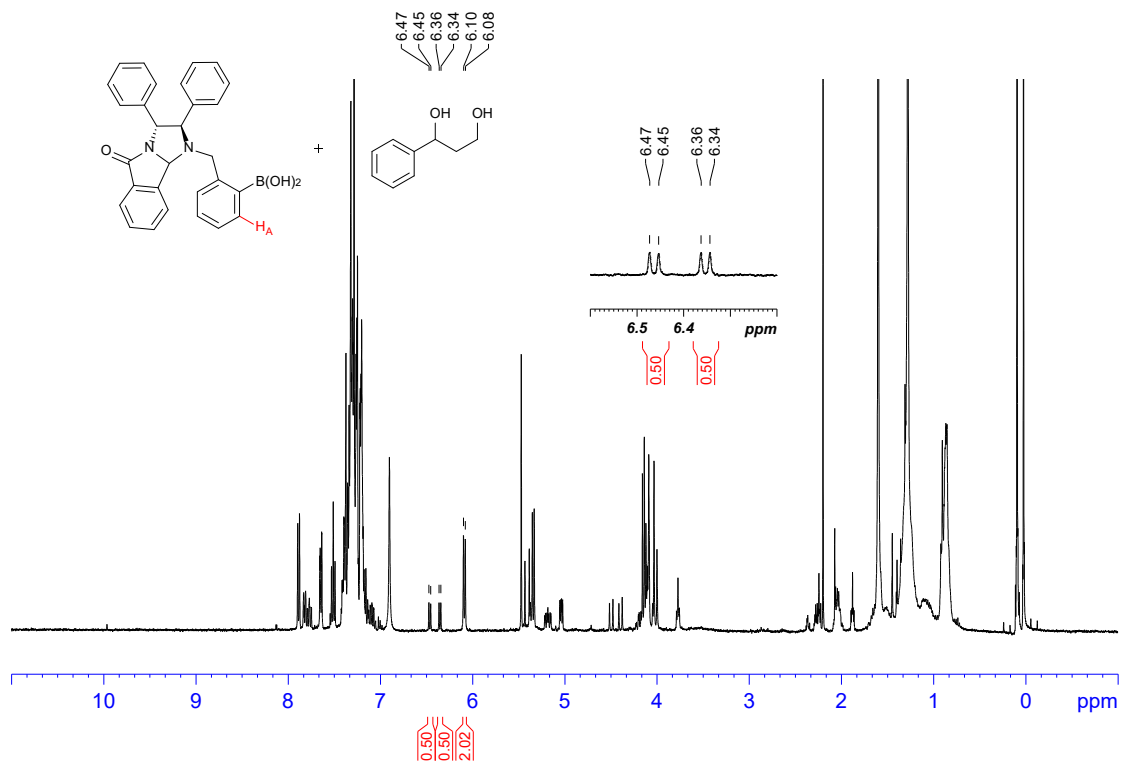


Figure S22. ¹H NMR of boric acid D and racemic **10**

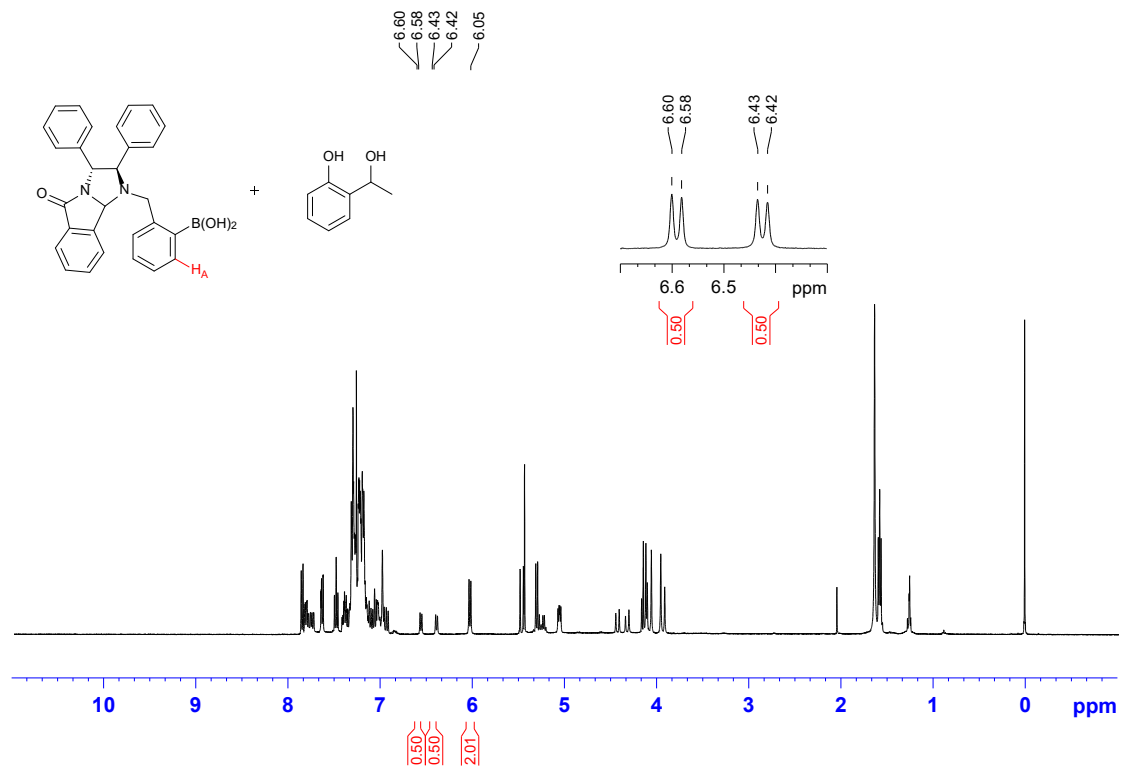


Figure S23. ^1H NMR of boric acid D and racemic **11**

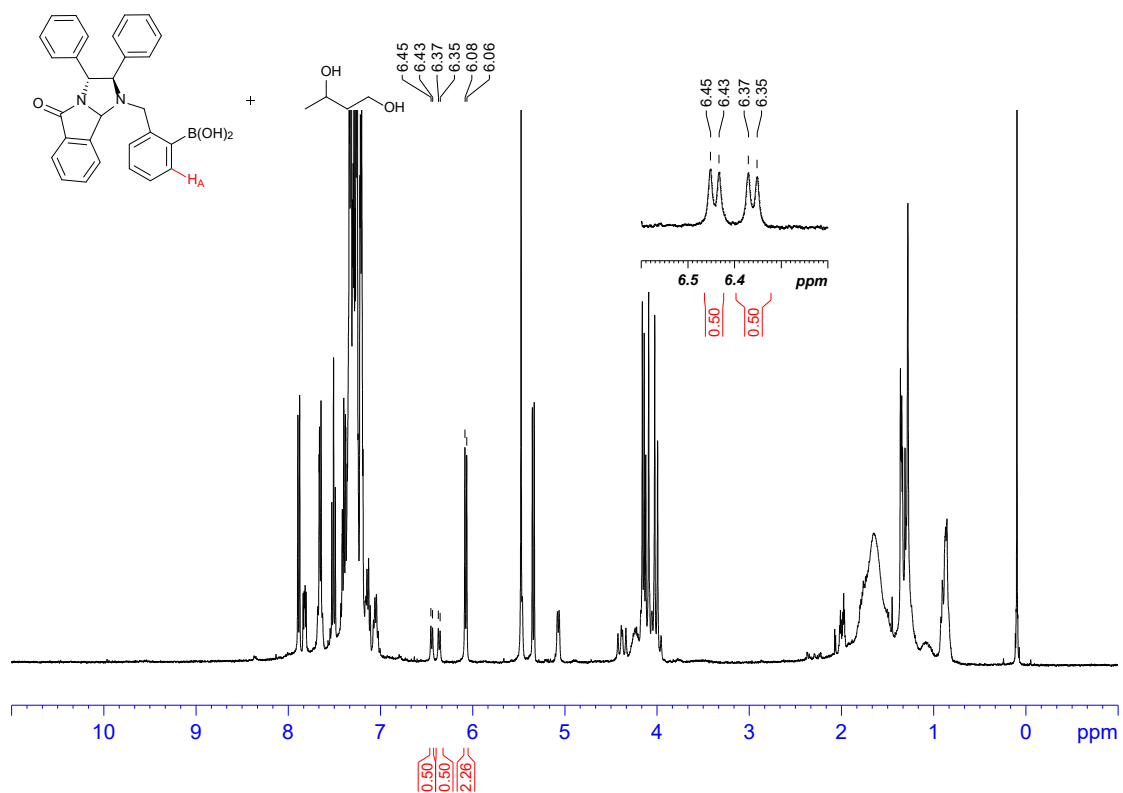


Figure S24. ^1H NMR of boric acid D and racemic **12**

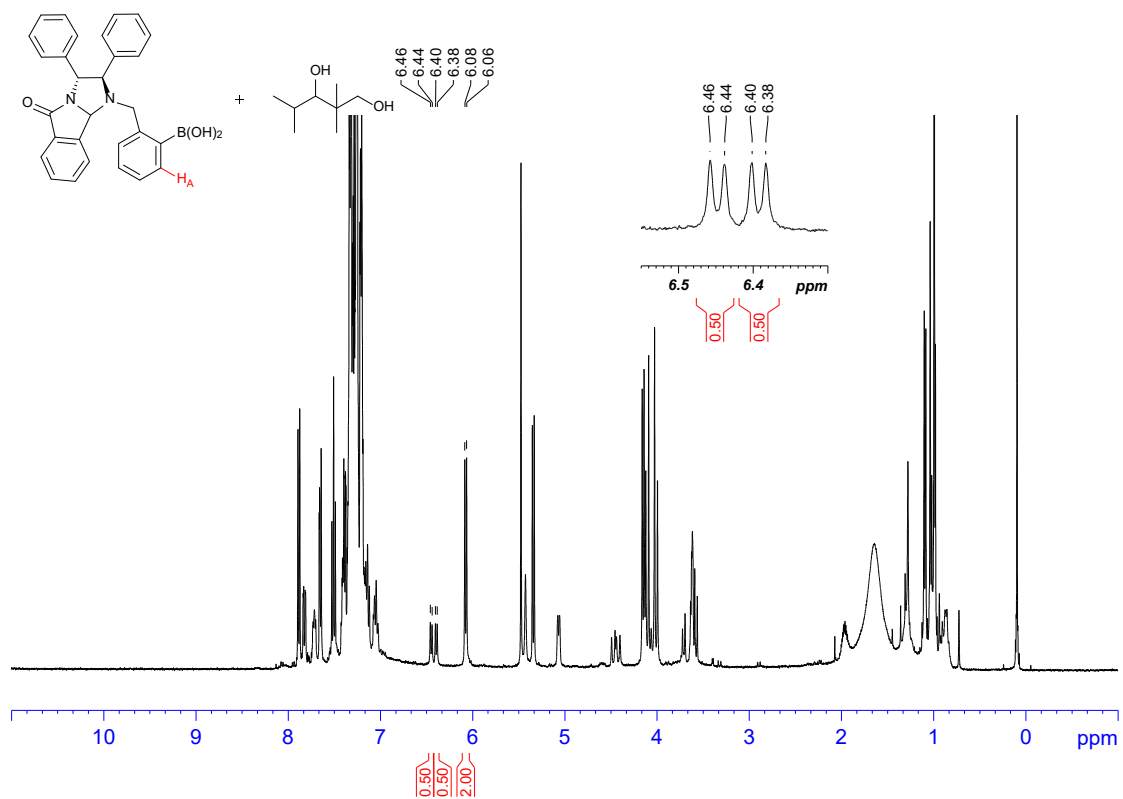
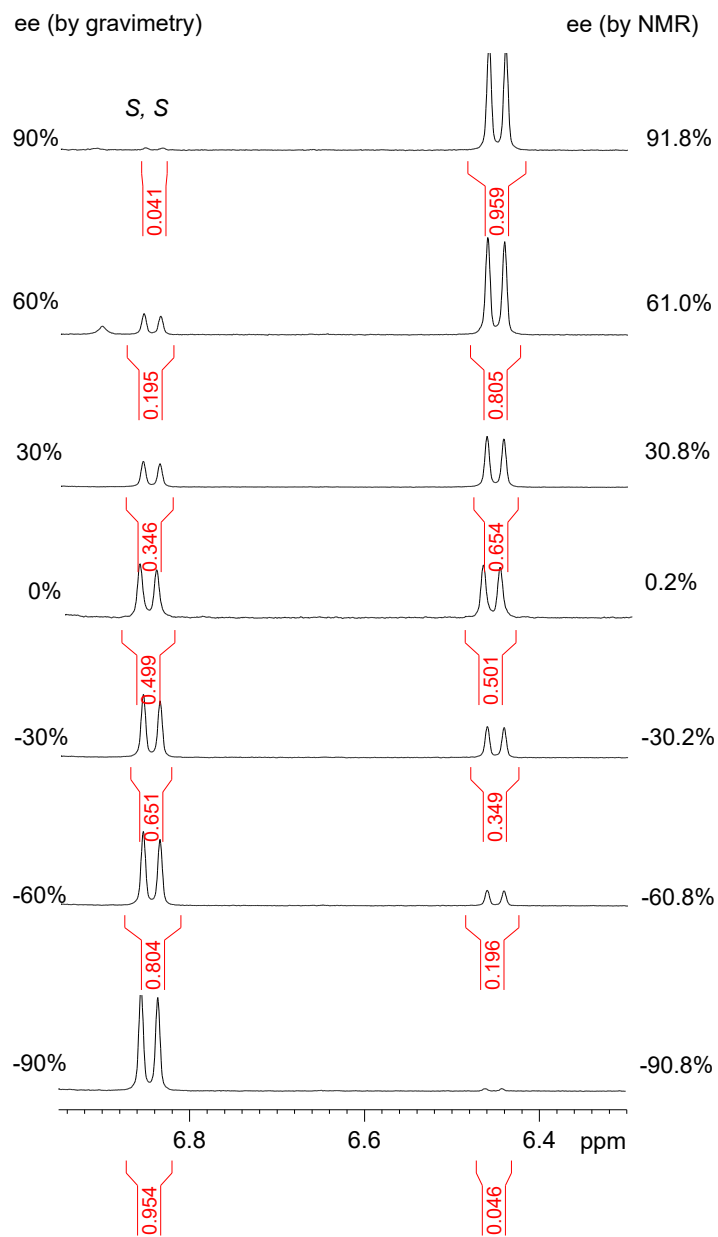


Figure S25. Ee values of nonracemic **1** determined by gravimetry and ^1H NMR spectra with boric acid D.



Ee values were defined in terms of (*R,R*)-**1**.